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A Comparative Evaluation of Different Sediment Quality Guidelines for Metal and Metalloid Pollution in the Xiangjiang River, Hunan, China

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Abstract To evaluate intensively the quality of the sediments contaminated by heavy metals and metalloids in the Xiangjiang River, 52 surface sediment samples were collected at 13 sites and different combinations of empirical and theoretical sediment quality indexes, the consensus-based sediment quality guidelines, sediment toxicity degree, and equilibrium partitioning method were applied. The average contents of Cd, Pb, Cu, Zn, Hg, Cr, and As in the sampled surface sediments were significantly higher than the background values of trace elements in soils of Hunan Province, China. Moreover, speciation fraction analyses revealed that Cd, Hg, and Pb in the sediments were dominated by the more bioavailable organic or exchangeable fractions, whereas the major species of As and Cr were the less bioavailable residual fractions after strong acid treatment. In addition, all indexes showed that these metals posed a median-high degree of toxic risk to benthic organisms in sediments from nearly all of the sampling sites along the Xiangjiang River. Cd, followed by Cu and Pb, erected the most severe ecological risk. Pearson correlation and linear regression analyses between the mean PEC quotients, sediment toxicity degree, interstitial water criteria toxic units, and sediment pollution index showed that these indexes were relatively consistent to assess the quality of sediments contaminated by heavy metals and metalloids in the Xiangjiang River. Our results will facilitate the proposal of proper sediment quality guidelines for the Xiangjiang River.

Sediment is a sink for contaminants, such as heavy metals and metalloids, in aquatic ecosystems (Rezaei et al. 2009; Wu et al. 2015). High concentrations of metals in sediments are detrimental to aquatic ecosystems due to their biological virulence and nondegradation in the natural environment. Sediment represents another implicit contaminant source through interactions between bottom sediment and overlaying water, resulting in severe ecological toxic effects on public health (Chai et al. 2015; Liu et al. 2014; Nielsen et al. 2010; Wang et al. 2011). Therefore, it is critical to establish SQGs for assessing aquatic ecosystems to protect human health.

Numerous empirical and theoretical SQGs for freshwater and marine ecosystems have successfully been proposed to assess toxic risks of pollutants in different sediments (Bednarova et al. 2013; De Deckere et al. 2011). Empirical methods, such as the water quality approach (WQA), the background approach (BA), the consensus-based sediment quality guidelines (CBSQGs), and the biological effect database approach (BEDA), have been applied to assessing the correlation between sediment pollution and toxic reactions (Huo et al. 2013; MacDonald et al. 2000). In empirical SQGs, CBSQGs with threshold/probable effect levels (TELs/PELs) and effects range low/median (ERLs/ERMs) are popular criteria to assess the sediment toxicity (Liu and Shen 2014). Mean probable effect concentration quotients (mPECQs) based on CBSQGs guidelines are used to evaluate the combined effects of multiple contaminants in sediments (Farkas et al. 2007; MacDonald

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et al. 2000). Modified experiential criteria, such as the sediment toxicity degree (STd) and contamination severity index (CSI), are recently generated from statistical analyses (Jamshidi-Zanjani and Saeedi 2013; Pejman et al. 2015). The equilibrium partitioning approach (EqPA), a typical theoretical method, is based on the thermodynamic equilibrium of sediment and water, which increases the accuracy of the assessment of the sediment quality (Gao et al. 2015). EqPA also can identify the causal relationship between a pollutant and its biological response in sediment through its concentrations in pore water. Therefore, EqPA is suitable to predict the potential benthic biotic effects of different types of sediments without biological tests (Burgess et al. 2013; Han et al. 2014). United States Environmental Protection Agency (USEPA) reports that equilibrium partitioning sediment benchmarks (ESBs) account for bioavailability in sediments and the potential effects of individual metals and mixtures of metals in the aquatic environment, thus providing an ecologically relevant benchmarks (Hansen et al. 2005). Sediment pollution index (SPI) based on EqPA also was established to evaluate the potential risk of adverse biological effects on benthic organisms of sediment contaminants (Han et al. 2014).

As of now, there are not established SQGs for rivers, although the empirical and theoretical methods are feasible and effective. The main reasons are as follows: (1) The calibration and validation of biological experiments are conducted using different test organisms with variable sensitivity to pollutants, and as a result, these SQGs are dramatically different (Bay et al. 2012). (2) The hydrographic, geographical, and physicochemical complexities of a specific region influence the biological effectiveness and toxicity of contaminants (Vidal and Bay 2005). (3) SQGs are established based on different hypotheses, applying dissimilar restrictions and following separate standards, and as a consequence, it is easily hampered to generate consistent estimates of sediment quality (Jiang and Sun 2014). Hence, to rate the quality of sediments affected by heavy metals and metalloids more accurately, the intensive comparison of applied SQGs is possibly an appropriate option.

The Xiangjiang River, one of the major tributaries of the Yangtze River, has been severely contaminated by heavy metals and metalloids due to the mining and smelting of nonferrous metals (zinc, lead, and copper) in its basin for hundreds of years. It has been a critical environmental and public health concern in Hunan province and has resulted in increased attention to the metal contamination status and ecological risk in the river (Chai et al. 2010, 2016, 2017; Hu et al. 2015; Zhu et al. 2013). However, SQGs for the Xiangjiang River are rare and involve only individual assessment methods (Han et al. 2014). To estimate in detail the quality of the contaminated sediments in the Xiangjiang River, we utilized CBSQGs, STd, and EqPA to evaluate the contamination of metals in the surface sediments and to investigate statistically their correlation. Our results provide support for developing quality standards for the local sediments and facilitate the treatment of heavy metal and metalloid pollution in the Xiangjiang River.

Materials and Methods

Study Area and Sample Collection

The Xiangjiang River is the largest drainage system in Hunan province, China. It meanders 856 km, covering 43.57% of the area of the province. With the mining and smelting of lead, zinc, and copper developing in the basin for hundreds of years, the river system has been severely contaminated by heavy metals and metalloids, as a result of discharging industrial wastewater, leaching form hazardous solid waste and precipitating air-borne pollutants (Zhao et al. 2015; Zhu et al. 2013).

As shown in Fig. 1, we selected 13 sampling sites from 5 regions in the Xiangjiang River: (1) the origin of the Xiangjiang River of Hunan province: Yongzhou (YZ-1); (2) areas with clusters of mining and smelting factories: Hengyang (HY-1, HY-2, HY-3, and HY-4) and Xiangtan (XT-1 and XT-2); (3) urban and densely populated areas with abandoned chemical and metal factories: Changsha (CS-1, CS-2, CS-3, and CS-4); and (4) agricultural areas from the mouth of the Xiangjiang River to Dongting Lake: Yueyang (YY-1 and YY-2). Four samples at 100-m intervals in each sampling site were randomly collected, and their average metal contents were determined for the sampling site. The samples were collected from the top 0–15 cm of the surface sediments using a sediment sampler (ZYC-200B, Hangzhou Yijie Technology Co., Ltd., Hangzhou, China) and stored in a polyethylene bag with frozen ice packs. The samples were delivered to our laboratory and stored at −20 °C before analyses. The sediment samples were desiccated at 60 °C for 14 days, followed by desiccation at 110 °C for 2 days, and then they were homogenized, crushed, and passed through 150-µm nylon sieves (100 meshes) for determination of the metal concentration and speciation analyses. According to the potential contamination profiles derived from the mining and smelting of lead, zinc, and copper, we chose the seven heavy metals and metalloids (Cd, Pb, Cu, Zn, Hg, Cr, and As) that have serious toxicity and cover large emission areas in the Xiangjiang River (Chai et al. 2010; Hu et al. 2015).

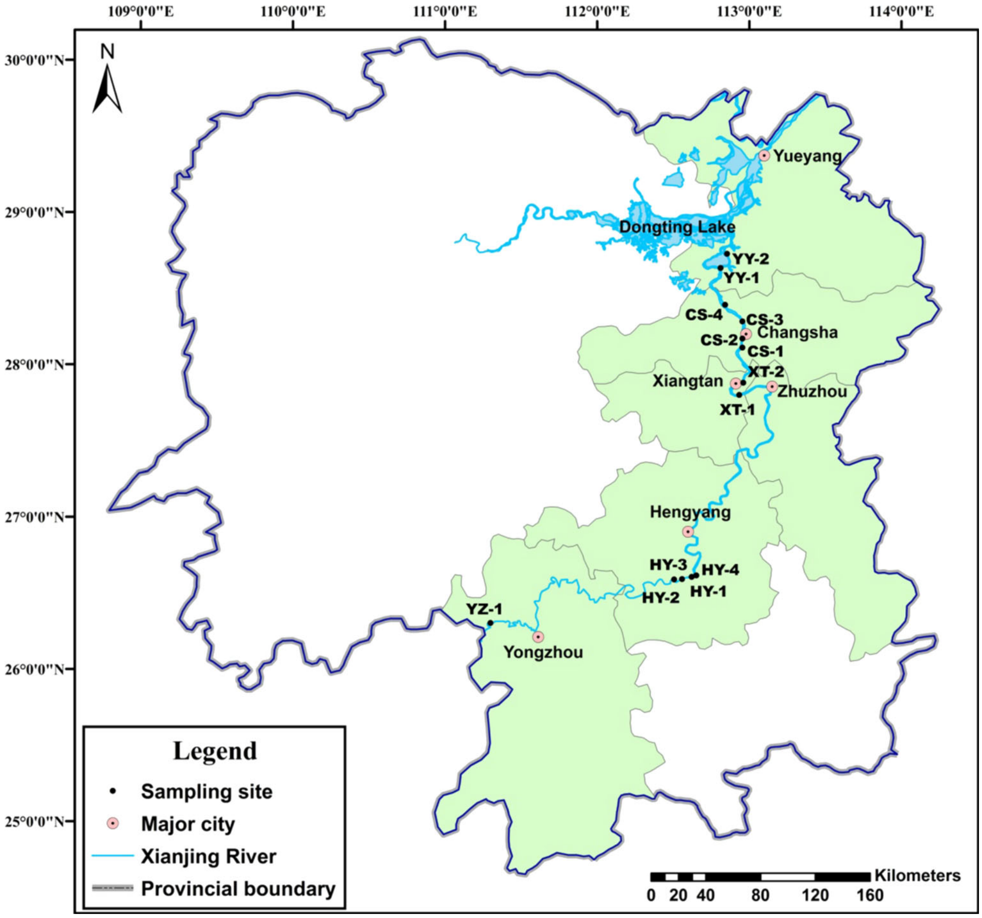


Fig. 1 Sampling sites in the Xiangjiang River

Analyses of Heavy Metals and Metalloids

Interstitial Water

Fresh sediment samples were centrifuged at 6000 rpm for 30 min (H1650, Xiangyi Centrifuge Instrument Co., Ltd., Changsha, China), followed by that the supernatant was filtered by 0.45-μm filter membranes (NSF02, Haining Dacheng Filtration Equipment Co., Ltd., Haining, China) to collect the interstitial water (Han et al. 2014; Louriño-Cabana et al. 2011). The concentrations of Cd, Pb, Cu, Zn, and Cr in the interstitial water were measured using an inductively coupled plasma mass spectrometer (ICP-MS, Agilent 7500 series, Santa Clara, CA). The contents of Hg and As were determined by an atomic fluorescence spectrophotometer (AFS-810, Beijing Titan Instrument Corp., Beijing, China).

Total Metal and Metalloids Concentration

The heavy metals and metalloids in the sediments were determined by following the previously published methods (Chai et al. 2017; Irvine et al. 2009; Wang et al. 2010; Zhu et al. 2013). Briefly, 0.100 g of pretreated sediment was digested on an electric heating plate with a mixture of acids (3 ml HCl, 4.5 ml HNO_3, and 1.5 ml HF). The concentrations of Cd, Pb, Cu, Zn, and Cr in the sediments were determined using an ICP-MS (Agilent 7500 series). In addition, 300.00 mg of the identical sediment sample was digested with 10 ml of aqua regia at 95 °C for 2 h, followed by the addition of 5 ml of HCl, 5 ml of thiocarbamide, and replenishing aqua regia to 50 ml. The concentrations of Hg and As in the sediments were determined using an atomic fluorescence spectrophotometer (AFS-810, Beijing Titan Instrument Corp., Beijing, China).

Speciation Fraction

The speciation fractions of metals in the sediments were obtained using Tessier's Sequential Extraction Procedure (Frankowski et al. 2010; Wang et al. 2010). The five binding forms of metals, exchangeable, carbonate, iron or manganese oxide, organic and sulfide, and residual lattice binding, were extracted with the following reagents: $MgCl_2$, NaAc (pH = 5.0), $NH_2OH{\cdot}HCl$, $NH_4Ac\text{-}H_2O_2$ (pH = 2), and strong acid ($HCl + HNO_3 + HF + HClO_4$), respectively. Concentrations of the speciation fractions of Cd, Pb, Cu, Zn, and Cr were determined using an ICP-MS (Agilent 7500 series). The speciation fractions of Hg and As were examined by an atomic fluorescence spectrophotometer (AFS-810, Beijing Titan Instrument Corp.).

Quality Assurance

To guarantee the quality of the analyses, laboratory quality assurance and control methods were implemented, including standard operating procedures, standard calibration, reagent blank analyses, and repeated analyses. All experiments were repeated, and the results were reported as the average values. Blanks and China Stream Sediment Reference Materials (GBW07309 (GSD-9) and GBW07311 (GSD-11)) were used for quality control, and the recoveries of the standard reference metals were 91–105%.

Sediment Quality Evaluation

Consensus-Based Sediment Quality Guidelines

Consensus-based sediment quality guidelines (CBSQGs), including consensus-based threshold effect concentrations (TECs), consensus-based probable effect concentrations (PECs), and mPECQs, were applied according to the previous studies (Farkas et al. 2007; Ingersoll et al. 2000; MacDonald et al. 2000). The consensus-based TECs and PECs were calculated by determining the geometric mean of the published TEC-type and PEC-type values in the previous study, respectively (MacDonald et al. 2000). Sediment samples were expected to be non-toxic if the measured concentrations of a chemical substance were lower than the corresponding TECs. Conversely, samples were predicted to be toxic if the concentrations of pollutants exceeded parallel PECs.

To evaluate the combined effects of multiple contaminants in the sediments, mPECQs were applied (Farkas et al. 2007). The formula for calculation is as follows:

$$\mathrm{mPECQs} = \frac{\sum_{i=1}^{n} \frac{C_i}{\mathrm{PEC}_i}}{n} \tag{1}$$

C_i is the concentration of pollutant i in the sediment, PEC_i is the corresponding PEC value of pollutant i, and n is the total number of pollutants. When $\mathrm{mPECQ} < 0.1$, the sediment sample is predicted to be nontoxic and the incidence of toxicity is relatively low (<25%). When $1 < \mathrm{mPECQ} < 5$, the incidence of toxicity is 70–75%. When mPECQ >5, the sediment sample is predicted to be toxic, and the incidence of toxicity is more than 75% (Farkas et al. 2007; Ingersoll et al. 2000).

Sediment Toxicity Degree

The STd index was proposed by Jamshidi-Zanjani and Saeedi (2013). This index uses the effects range median (ERM) values (Droge et al. 2008) as the limit of toxic and adverse effects on organisms. The proposed formulae are as follows:

$$\mathrm{STd} = \sum_{i=1}^{n} W_i \left(1 + \frac{C_i}{\mathrm{ERM}_i}\right)^2 \tag{2}$$

$$W_i = \text{relative Eigenvalue}_i \times \text{relative loading value}_i \tag{3}$$

W_i, the site-specific factor, is the weight of metal i calculated by formula (3) in which the relative eigenvalue and relative loading value are calculated by principal component analysis (PCA). C_i is the concentration of metal i. ERM_i is the ERM value of metal i. n is the total number of metals. The toxicity levels of STd are as follows, the value of STd less than 1.0 indicates no toxicity, $1 \leq \mathrm{STd} \leq 1.5$ indicates a low toxic level, $1.5 < \mathrm{STd} \leq 2$ means a low-median toxic level, $2 < \mathrm{STd} \leq 3$ reveals a median toxic level, $3 < \mathrm{STd} \leq 4$ is a median-high toxic level, and $\mathrm{STd} > 4$ indicates high toxicity (Jamshidi-Zanjani and Saeedi 2013).

Equilibrium Partitioning Approach

The USEPA first proposed the EqPA in 1985 (Planas et al. 2006), and the standards for heavy metals and metalloids were established by Holland (Van Der Kooij et al. 1991) and the United Kingdom (Webster and Ridgway 1994). According to the EqPA, SQGs are calculated as follows:

$$\mathrm{SQG} = K_\mathrm{p} \times \mathrm{WQC} \tag{4}$$

WQC is the adopted water quality criteria; the availability of the WQC is a vital step of EqPA (Van Der Kooij et al. 1991; Webster and Ridgway 1994). Because the WQCs for the chronic toxicity of the seven metals in rivers are not available in China, the WQCs of the USEPA were applied in our study (Gooch-Moore et al. 2011). In terms of the fluvial WQCs of the USEPA, the criterion continuous concentration (CCC) indicates the maximum concentration of some pollutant at which organism communities in an aquatic ecosystem can be exposed indefinitely without adverse effects, whereas the criterion maximum concentration (CMC) is the minimum concentration of some pollutant at which aquatic organism communities are impacted in a short time (Balistrieri et al. 2007). The partitioning coefficient (K_p) is the ratio of the bioavailable concentration of a metal in the sediment solid phase (ρ_s) to its concentration in interstitial water (ρ_w):

Table 1 Contents of the seven metals in the sediments of the Xiangjiang River and values of CBSQGs for the metals ($\mu g\ g^{-1}$)

Sites	Cd	Pb	Cu	Zn	Hg	Cr	As
YZ-1	4.62	33.59	56.23	94.28	0.90	61.98	15.75
HY-1	16.56	118.19	150.12	377.65	0.42	89.26	97.58
HY-2	6.73	46.44	165.25	119.76	0.13	93.25	112.36
HY-3	10.59	298.33	169.25	521.57	0.48	79.36	109.84
HY-4	10.90	183.75	200.12	424.40	0.51	80.25	129.98
XT-1	24.83	130.01	78.25	617.22	0.80	109.35	42.37
XT-2	8.32	46.31	85.54	146.95	0.95	95.36	43.21
CS-1	22.05	160.67	69.35	418.83	0.83	104.26	42.36
CS-2	20.73	142.17	95.36	404.09	0.76	118.36	49.32
CS-3	8.37	22.77	101.26	119.33	1.54	97.36	50.24
CS-4	7.43	88.50	89.26	226.02	0.21	92.21	45.26
YY-1	10.37	106.28	59.36	315.38	0.72	94.74	11.88
YY-2	18.39	108.47	63.25	407.90	0.96	97.23	6.82
Average	13.06	114.26	106.35	322.56	0.71	93.31	58.23
Background[a]	0.126	29.7	27.3	94.4	0.116	71.4	15.7
TEC	0.99	35.80	31.60	121.00	0.18	43.40	9.79
PEC	4.98	129.00	149.00	459.00	1.06	111.0	33.00
ERM	9.60	218	270	410	0.71	370	70

TEC threshold effect concentrations, *PEC* probable effect concentration (MacDonald et al. 2000), *ERM* effects range median (Jamshidi-Zanjani and Saeedi 2013)

[a] Background: the soil trace element background for Hunan Province (China National Environmental Monitoring Center 1990)

$$K_{\mathrm{p}} = \frac{\rho_{\mathrm{s}}}{\rho_{\mathrm{w}}} \tag{5}$$

The residual and acid volatile sulfide binding fractions of metals that are not involved in the distribution of the sediment–water phase equilibrium also should be considered (Gao et al. 2015). Therefore, the formula for the SQGs is modified as follows:

$$\mathrm{SQG} = K_{\mathrm{p}} \times \mathrm{WQC} + [\mathrm{M}]_{\mathrm{R}} + [\mathrm{M}]_{\mathrm{AVS}} \tag{6}$$

$[\mathrm{M}]_{\mathrm{R}}$ is the concentration of residual metal fractions and $[\mathrm{M}]_{\mathrm{AVS}}$ is the concentration of metals in acid volatile sulfides. However, the acid volatile sulfide binding contents in most samples were not detectable, and thus, the content of acid volatile sulfides in formula (6) was ignored in our study.

Given that the SQG value based on EqPA did not involve combined contamination effects, interstitial water criteria toxic units (IWCTUs), one of the ESBs was used to assess the toxicity of metal mixture to benthic organisms in sediments (Ankley et al. 1996; Hansen et al. 2005). The IWCTU is calculated as follows:

$$\mathrm{IWCTU}_i = \frac{\rho_{i\mathrm{w}}}{\mathrm{FCV}_i} \tag{7}$$

$\rho_{i\mathrm{w}}$ is concentration of metal i in interstitial water, and FCV is the WQC final chronic value of metal i in interstitial water. When the sum of IWCTUs is not more than 1, it indicates that metal mixtures in sediments should not cause direct toxicity to benthic organisms.

The summed integrated sediment pollution index (SPI) was applied to evaluating the toxic risk of compound pollution in the sampling sites (Han et al. 2014). The integrated SPI is defined as follows:

$$\mathrm{SPI1} = \sum \frac{10}{\mathrm{SQG\text{-}Low}_i} \times \rho_i \quad (\rho_i \le \mathrm{SQG\text{-}Low}_i) \tag{8}$$

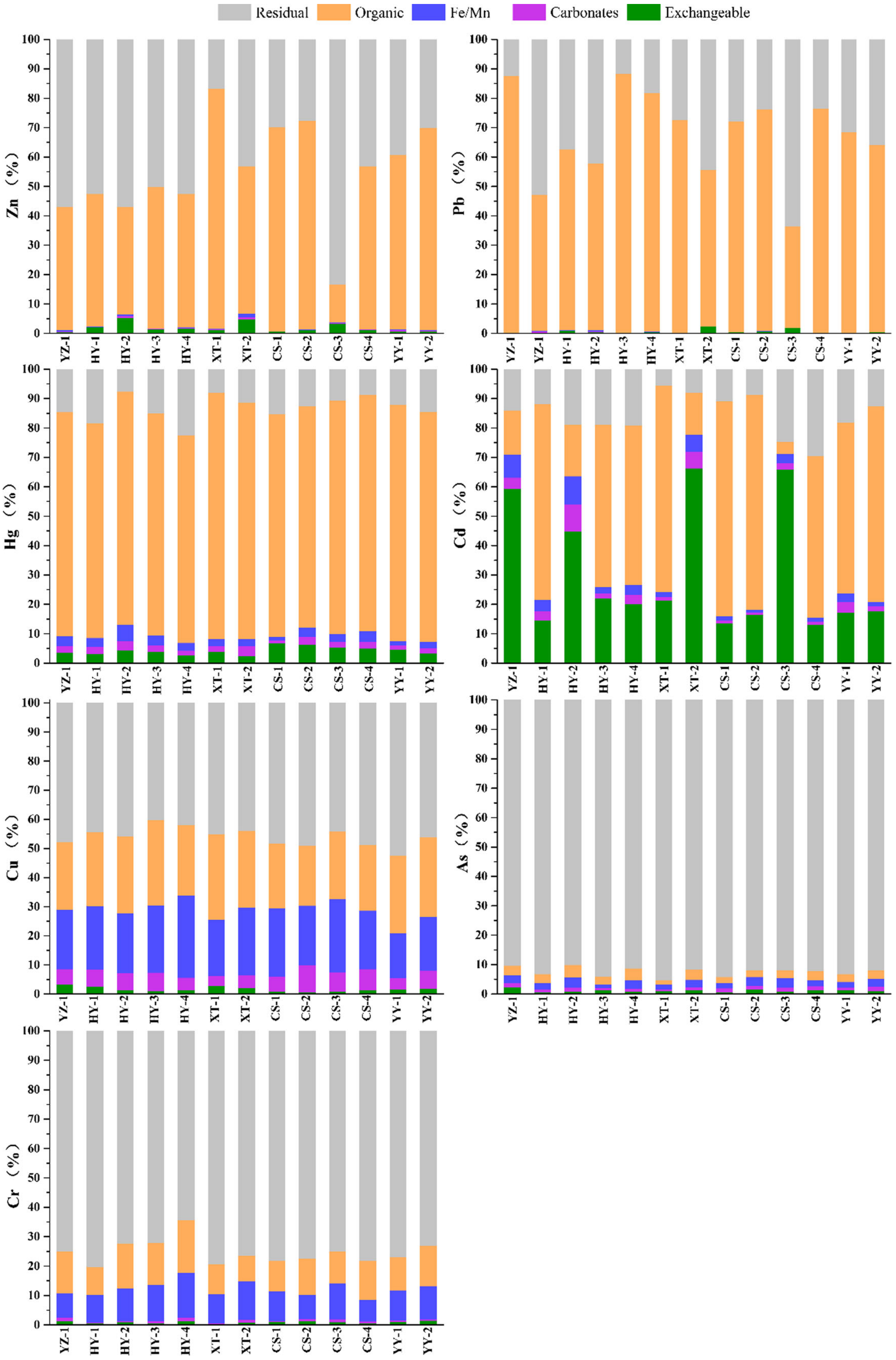
$$\mathrm{SPI2} = \sum \left(10 + \frac{10}{\mathrm{SQG\text{-}Mid}_i - \mathrm{SQG\text{-}Low}_i} \times (\rho_i - \mathrm{SQG\text{-}Low}_i)\right) \quad (\mathrm{SQG\text{-}Low}_i < \rho_i \le \mathrm{SQG\text{-}Mid}_i) \tag{9}$$

$$\mathrm{SPI3} = \sum \left(20 + \frac{10}{\mathrm{SQG\text{-}High}_i - \mathrm{SQG\text{-}Mid}_i} \times (\rho_i - \mathrm{SQG\text{-}Mid}_i)\right) \quad (\mathrm{SQG\text{-}Mid}_i < \rho_i \le \mathrm{SQG\text{-}High}_i) \tag{10}$$

$$\mathrm{SPI4} = \sum \left(30 + \frac{10}{\mathrm{SQG\text{-}High}_i} \times (\rho_i - \mathrm{SQG\text{-}High}_i)\right) \quad (\mathrm{SQG\text{-}High}_i < \rho_i) \tag{11}$$

$$\mathrm{SQG\text{-}Low}_i = K_{\mathrm{p}} \times \mathrm{CCC} + [\mathrm{M}]_{\mathrm{R}} \tag{12}$$

$$\mathrm{SQG\text{-}High}_i = K_{\mathrm{p}} \times \mathrm{CMC} + [\mathrm{M}]_{\mathrm{R}} \tag{13}$$


Residual
Organic
Fe/Mn
Carbonates
Exchangeable
Zn（%）
Pb（%）
Hg（%）
Cd（%）
Cu（%）
As（%）
Cr（%）
0
10
20
30
40
50
60
70
80
90
100
YZ-1
HY-1
HY-2
HY-3
HY-4
XT-1
XT-2
CS-1
CS-2
CS-3
CS-4
YY-1
YY-2

◀**Fig. 2** Fraction speciation of the seven metals in the sampled surface sediments of the Xiangjiang River

$$\text{SQG-Mid}_i = (\text{SQG-Low}_i + \text{SQG-High}_i)/2 \qquad (14)$$

$$\text{SPI} = \text{SPI1} + \text{SPI2} + \text{SPI3} + \text{SPI4} \qquad (15)$$

ρ_i is the concentration of pollutant i; SQG-Low$_i$, SQG-Mid$_i$, and SQG-High$_i$ are the different SQG levels of pollutant i. The SPI standards are as follows, $\text{SPI} \le 10$ indicates low toxic risk, $10<\text{SPI} \le 20$ indicates moderate toxic risk, $20 < \text{SPI} \le 30$ indicates considerable toxic risk, and $\text{SPI} > 30$ indicates high potential toxic risk.

Comparison Between mPECQs, STd, IWCTUs and SPI, and Statistical Analyses

Principal component analysis (PCA) was performed for the data set, following the previous protocols (Chai et al. 2017; Gu et al. 2012). Briefly, Kaiser–Meyer–Olkin (KMO) and Bartlett's sphericity tests were implemented to examine the validity of PCA. Pearson correlation analysis was performed to identify the relationship between mPECQs, STd, IWCTUs, and SPI in our study. The results of the four methods were also compared by linear regression with the goodness of fit test (R^2). All mathematical and statistical calculations were conducted using SPSS 22 for windows (IBM, Inc., Armonk, NY).

Results and Discussion

Contamination Profiles of Heavy Metals and Metalloids

The concentrations of the seven heavy metals and metalloids in the surface sediment samples from the Xiangjiang River and the soil trace element background in Hunan Province, China (China National Environmental Monitoring Center 1990) are shown in Table 1. Generally, the average contents of Cd, Pb, Cu, Zn, Hg, Cr, and As were 13.06, 114.26, 106.35, 322.56, 0.71, 93.31, and 58.23 $\mu g\ g^{-1}$, respectively, which were much higher than their background values. In particular, the average contents of Cd and Hg were 103.65 and 6.21 times the corresponding background values, respectively. Geographically, the highest concentrations of Cd, Pb, Cu, Zn, and As were clustered in the sites of Hengyang (HY-1, HY-2, HY-3, and HY-4) and Xiangtan (XT-1), which is consistent with the locations of major mining and smelting factories of Pb, Cu, and Zn in the two areas (Zhu et al. 2013). Interestingly, Hg and Cr were relatively equally contaminated in all the sampled sediments.

Speciation of Heavy Metals and Metalloids

The relative contents in the speciation fractions of the seven metals in the sampled sediments are shown in Fig. 2. The metals in the sampled sediments were classified into two groups in terms of their biological effectiveness: group I consisted of Cd, Hg, Pb, Zn, and Cu, with the major species being bioavailable, and group II consisted of As and Cr, with reside being dominant. The average relative bioavailable contents (except the residue state) of Cd, Hg, Pb, Zn, and Cu were 83.95, 86.85, 73.69, 60.86, and 52.21%, respectively (Fig. 2). Remarkably, Cd in the sediments was predominately in the organic state (14.02–60.87%; average: 48.14%), followed by the exchangeable state (13.01–47.92%; average: 30.27%). The organic states of metals (the association of a metal with the organic matter in the sediment) are potentially hazardous, secondary ecological pollutants (Yang et al. 2009, 2016). Exchangeable metals are adsorbed on clay, humus and other components also sensitive to environmental changes and are easily transported and transformed (Fig. 2) (Chai et al. 2015). Moreover, exchangeable heavy metals and metalloids reflect impact of the recent anthropogenic pollution and have severe biological toxicity due to their bioavailability (Jiang and Sun 2014). Similarly, the major speciation fractions of Hg and Pb were in the organic state, accounting for 79.49 and 73.11% of the total on average, respectively (Fig. 2). In contrast, the residue states of As and Cr were dominant in the sampled sediments, and the average ratios were 94.06 and 79.79%, respectively. The residue state is generally found in silicate and primary and secondary minerals with long-term stability in sediments. Therefore, Cd possibly poses the highest ecological risk in the surface sediments of the Xiangjiang River, followed by Hg, Pb, Zn, and Cu, whereas As and Cr have less potential ecological risk. However, the metals with high ecological risk fractions do not show equivalently high concentrations in the samples. The possible reason is that the speciation fractions of the metals are shaped by their hydrographic and physicochemical characteristics, as well as indigenous microbial communities in the sediments, and not only by the contamination resources (Jiang and Sun 2014; Yang et al. 2017).

Consensus-Based Sediment Quality Guidelines

Consensus-based sediment quality guidelines have, so far, been proposed for 28 metals and organic chemicals of concern in freshwater sediments. The values of the consensus-based TECs and PECs are shown in Table 1 (MacDonald et al. 2000). By comparing the concentrations of the seven metals with those of the standards in Table 1, the contents of Cd and As dramatically exceed the values of the consensus-based TECs, and respectively ranged from 0.93 to 4.99 (average: 2.62) and from 0.36 to 3.94 (average: 1.76) times of the corresponding consensus-based PECs, which indicates that the two pollutants have severe potential toxicity to the sampled sediments. The concentrations of Pb, Zn, Cu, Hg, and Cr were slightly lower than the consensus-based PECs but much higher than the consensus-based TECs, suggesting that these metals possibly pose adverse risk on the aquatic ecosystems in the Xiangjiang River.

The mPECQs of heavy metals and metalloids can indicate the potential toxicity of all contaminants in one sampling site (MacDonald et al. 2000). As shown in Fig. 3a, the mPECQs of most of the sampling sites exceeded 1 but were lower than 5, implying that the incidence of biological toxicity ranged from 70 to 75%. Therefore, all sampled sections of the Xiangjiang River were seriously polluted by the seven metals, which pose high toxicity risk, especially at the sampling sites YZ-1, XT-2, CS-3, CS-4, and YY-1.

Sediment Toxicity Degree

The seven metals in all the sampling sites were analyzed by principal component analysis (PCA) to calculate the relative eigenvalue and relative loading values. The KMO and Bartlett's sphericity results were 0.712 and 191.08 ($df = 21$, $p < 0.01$), respectively, which indicated that PCA was practicable (Gu et al. 2014a, b). The weights of the selected variables based on eigenvalues using PCA are shown in Table 2, respectively. The variables were split into two clusters by PCA: (1) Cu, Pb, and As; and (2) Cd, Zn, Hg, and Cr (Table 2). It is possible that Cu, Pb, and As originated from anthropogenic actives (the mining and smelting of Cu and Pb), whereas Cd, Zn, Hg, and Cr were potentially derived from another anthropogenic source (discharge of metallurgical waste and smelting of Zn). It also is possible that similar chemical behaviors of metals account for such cluster. For example, the Cu and Pb have a strong association of binding sites on organic matter (Chakraborty and Chakrabarti 2008; Impellitteri et al. 2002).

The W_i values of the seven metals measured by the proportion of eigenvalues and their loading values from PCA ranged from 0.041 (Hg) to 0.185 (As) (Table 2). The STd of the sampled sediments showed the degree of sediment toxicity at all studied areas along the Xiangjiang River. As shown in Fig. 3b, only the YZ-1 sediment sample was categorized at the low-median toxic level, whereas HY-2, XT-2, CS-3, CS-4, and YY-1 were categorized at the median toxic level. HY-1, CS-1, CS-2, and YY-2 were considered as median-high toxic level, and

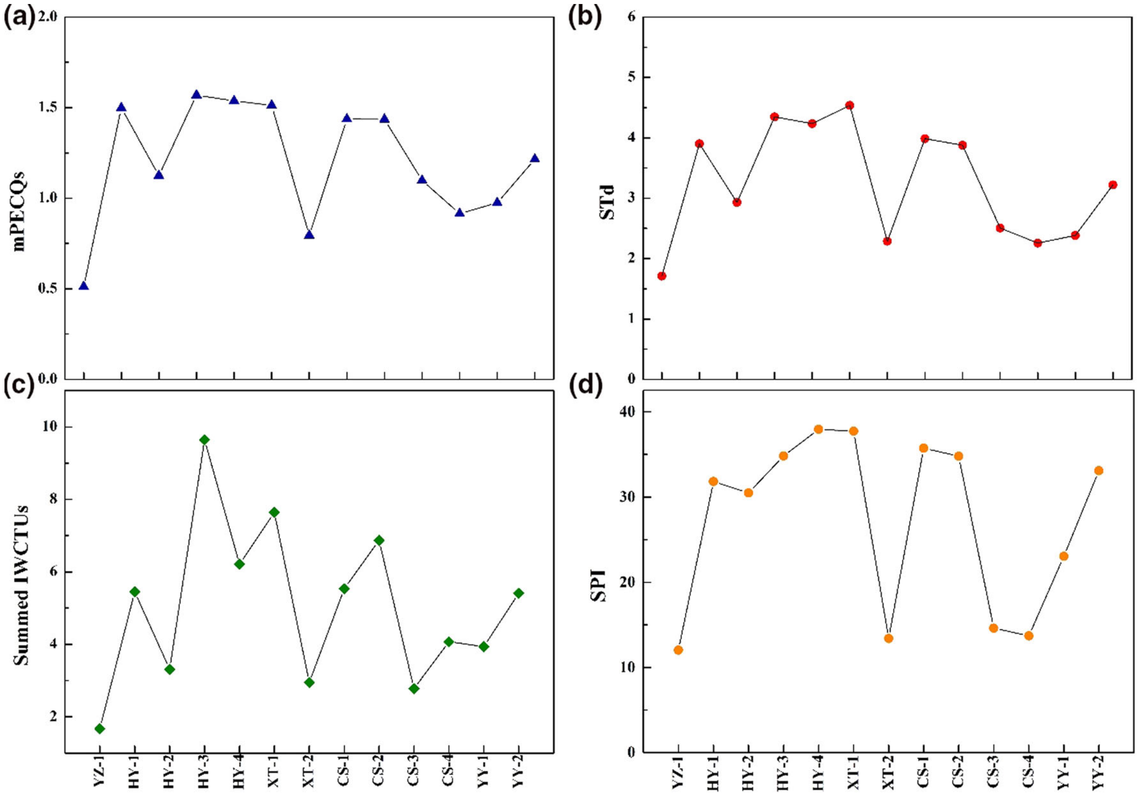


Fig. 3 mPECQs (**a**), STd (**b**), Summed IWCTUs (**c**), and SPI (**d**) of the sampled surface sediments in the Xiangjiang River

Table 2 Weight of selected variables based on eigenvalues using PCA

PC	Eigenvalues	Relative eigenvalues	Variables	Loading values	Relative loading values on same cluster	W*i*
1	2.882	0.534	Pb	0.775	0.309	0.165
			Cu	0.863	0.344	0.184
			As	0.872	0.347	0.185
			Total		1	
2	2.518	0.466	Cd	0.948	0.349	0.163
			Zn	0.777	0.286	0.133
			Hg	0.243	0.089	0.041
			Cr	0.750	0.276	0.129
			Total		1	
Total	5.400	1				1

HY-3, HY-4, and XT-1 were categorized as high toxicity. Generally, the mean value of the STd was 3.24, which suggested that sediments in the Xiangjiang River had a median-high potential of toxic effects on the benthos. In particular, the sediments in Hengyang and Xiangtan had more serious toxic risks.

Equilibrium Partitioning Method

The EqPA accounts for the variable biological availability of chemicals and allows for the incorporation of the appropriate biotic effects of concentrations in different types of sediments (Wepener et al. 2000). The bioavailable concentrations (ρ_s), concentrations in the interstitial water (ρ_w), and sediment–water phase equilibrium distribution coefficients (K_p) of the seven metals from all of the sampling sites along the Xiangjiang River are shown in Table 3. The K_p values in our study were generally consistent with previous studies on sediments of the Xiangjiang River, but our values were significantly different from the K_p values of other watersheds due to variants in the distribution of sediment particle size and organic matter percentage between different fluvial systems (Chen et al. 2007; Han et al. 2014; Lin et al. 2007).

To calculate the CCCs and CMCs of metals, it was necessary to quantify the water hardness. The average water hardness of the Xiangjiang River is 52.65 mg L^{-1}. The calculated results of the sediment quality criteria of the seven metals in the Xiangjiang River are shown in Table 4. SQG-low based on CCC refers to the threshold concentration of the pollutant, whereas at lower concentrations chronic biological effects on benthic organisms in the sediment would be rare. The concentration of pollutant exceeding SQG-high based on CMC would easily cause acute biological effects on benthic organisms.

Interstitial water criteria toxic units is one of interstitial water benchmark approaches for the derivation of the ESBs for individual metals and mixtures of metals (Ankley et al. 1996; Hansen et al. 2005). For freshwater sediments, the FCVs are hardness dependent for all of the metals under consideration, which are used as the effect concentrations (Table 4). The summed IWCTUs of all sampling sites exceed 1 (Fig. 3c; Table 5), which indicate that the metal mixtures in sediments cause direct toxicity to benthic organisms. The effects observed in toxicity tests or in faunal analyses with the sampled sediment should principally be a result of Cd, Pb, and Cu. It results from that the concentrations of Cd, Pb, and Cu in the most sampled sediments exceed the corresponding WQC FCV (Table 5) (Hansen et al. 2005; Suter and Tsao 1996).

The results of SPI revealed that all of the seven metals posed high potential toxic risk in the sampling sites of the Xiangjiang River, except for the YZ-1, XT-2, CS-3 and CS-4 sites, which showed moderate toxic risk, and YY-1, which had notable toxic risk (Fig. 3d). The results showed that the pollution in the middle reaches was more serious, especially at the Hengyang and Changsha sites, which suggest that such pollution may be derived from local chemical, mining, and smelting industrial emissions (Sun et al. 2011).

Correlation of mPECQs, STd, IWCTUs, and SPI

We used four indexes, the mPECQs (a representative empirical method), STd (a combination of empirical data and statistics), IWCTUs (an integrated interstitial water benchmark), and SPI (a classical theoretical method), to assess the sediment quality of the Xiangjiang River. Pearson correlation and linear regression analyses were implemented between the four methods. The result showed

Table 3 Bioavailable concentration, metal concentration in the interstitial water, and partition coefficients of the seven metals in the Xiangjiang River

Sites	Bioavailable concentration of metal (ρ_s) (μg g^{-1})							Metal concentration in the interstitial water (ρ_w) (μg L^{-1})							Partition coefficients (K_p) (L g^{-1})						
	Cd	Pb	Cu	Zn	Hg	Cr	As	Cd	Pb	Cu	Zn	Hg	Cr	As	Cd	Pb	Cu	Zn	Hg	Cr	As
YZ-1	3.97	15.86	25.72	40.53	0.37	12.83	0.91	0.23	0.42	3.85	1.49	0.08	2.64	0.21	17.27	37.76	6.68	27.2	4.93	4.86	4.32
HY-1	14.60	73.89	83.50	179.38	0.26	17.62	6.58	1.42	1.27	10.49	8.32	0.04	3.21	1.54	10.28	58.18	7.96	21.56	7.37	5.49	4.27
HY-2	5.46	26.86	91.89	51.44	0.76	18.42	7.57	0.47	0.56	10.66	2.03	0.16	2.86	1.35	11.62	47.96	8.62	25.34	4.83	6.44	5.61
HY-3	8.59	263.54	94.12	259.62	0.26	15.65	7.40	0.71	6.89	12.02	9.68	0.03	3.24	1.27	12.1	38.25	7.83	26.82	9.13	4.83	5.83
HY-4	8.82	150.22	111.24	201.68	0.24	15.83	8.75	0.93	2.51	12.89	9.35	0.03	3.51	2.03	9.48	59.85	8.63	21.57	7.37	4.51	4.31
XT-1	23.45	94.42	42.86	513.63	0.33	22.56	1.96	2.6	2.17	4.87	20.57	0.07	4.98	0.45	9.02	43.51	8.8	24.97	4.6	4.53	4.36
XT-2	7.67	25.79	46.85	83.57	0.26	19.65	2.00	0.74	0.58	5.24	3.05	0.07	3.68	0.29	10.36	44.47	8.94	27.4	3.79	5.34	6.91
CS-1	19.66	115.74	35.85	294.04	0.35	22.66	2.46	1.35	2.35	4.59	11.54	0.07	3.69	0.35	14.56	49.25	7.81	25.48	5.16	6.14	7.02
CS-2	18.91	108.46	49.36	292.31	0.35	25.69	2.85	1.64	3.05	5.33	12.54	0.07	4.95	0.47	11.53	35.56	9.26	23.31	4.84	5.19	6.07
CS-3	6.30	8.29	52.35	20.50	0.49	21.15	2.87	0.59	0.19	7.23	0.79	0.13	4.68	0.54	10.67	43.61	7.24	25.95	3.77	4.52	5.31
CS-4	5.24	67.66	46.16	128.44	0.06	20.00	2.62	0.68	1.66	6.23	5.96	0.01	4.62	0.32	7.7	40.76	7.41	21.55	6.44	4.33	8.18
YY-1	8.49	72.70	28.26	191.67	0.26	21.90	0.80	0.75	1.64	4.25	8.17	0.06	4.65	0.13	11.32	44.33	6.65	23.46	4.02	4.71	6.16
YY-2	16.08	69.55	30.12	284.99	0.31	22.47	0.46	1.35	2.31	3.26	11.84	0.08	4.72	0.08	11.91	30.11	9.24	24.07	4.1	4.76	5.75

Table 4 Sediment quality criteria for the seven metals in the Xiangjiang River

	Cd	Pb	Cu	Zn	Hg	Cr	As
K_p (L g^{-1})	11.23	43.98	8.28	24.56	5.66	4.92	5.72
WQC (CCC) (μg L^{-1})[a]	0.15	1.25	5.37	55.67	0.77	11	150
WQC (CMC) (μg L^{-1})[a]	1.08	34.59	7.68	74.84	1.4	16	340
FCVs[b]	0.64	1.11	6.56	60.68	1.3	10	190
$[M]_R$ (μg g^{-1})	1.88	29.83	47.82	127.84	0.12	71.26	51.52
SQG-low (μg g^{-1})	3.56	102.79	92.28	1495.09	4.47	125.38	909.52
SQG-high (μg g^{-1})	14.00	1551.09	111.41	1965.90	8.04	149.98	1996.32
SQG-Mid (μg g^{-1})	8.78	826.94	101.84	1730.49	6.25	137.68	1452.92

[a] *WQC* water quality criteria, *CCC* criterion continuous concentration, *CMC* criterion maximum concentration; *Source*: (Gooch-Moore et al. 2011)

[b] *FCVs* final chronic values; *Source*: (Suter and Tsao 1996)

Table 5 Interstitial water criteria toxic units of the seven metals in the Xiangjiang River

Sites	Cd	Pb	Cu	Zn	Hg	Cr	As	$\sum$ IWCTUs
YZ-1	0.359	0.378	0.587	0.025	0.062	0.264	0.001	1.676
HY-1	2.219	1.144	1.599	0.137	0.031	0.321	0.008	5.459
HY-2	0.734	0.505	1.625	0.033	0.123	0.286	0.007	3.314
HY-3	1.109	6.207	1.832	0.160	0.023	0.324	0.007	9.662
HY-4	1.453	2.261	1.965	0.154	0.023	0.351	0.011	6.218
XT-1	4.063	1.955	0.742	0.339	0.054	0.498	0.002	7.653
XT-2	1.156	0.523	0.799	0.050	0.054	0.368	0.002	2.951
CS-1	2.109	2.117	0.700	0.190	0.054	0.369	0.002	5.541
CS-2	2.563	2.748	0.813	0.207	0.054	0.495	0.002	6.881
CS-3	0.922	0.171	1.102	0.013	0.100	0.468	0.003	2.779
CS-4	1.063	1.495	0.950	0.098	0.008	0.462	0.002	4.077
YY-1	1.172	1.477	0.648	0.135	0.046	0.465	0.001	3.944
YY-2	2.109	2.081	0.497	0.195	0.062	0.472	0.000	5.416

that the summed IWCTUs and SPI had a moderately high correlation and the correlation coefficient (r) was 0.797 ($P < 0.01$), which was consistent with the linear regression analysis of the pair (Fig. 4a; $R^2 = 0.601$), although the two indexes derive from EqPA. The reason is possible that IWCTUs are calculated the concentrations of metals in interstitial water, while SPI is based on the contents of metals both in interstitial water and sediments (Ankley et al. 1996; Han et al. 2014; Hansen et al. 2005). The summed IWCTUs also had significantly positive correlations with STd ($r = 0.893$, $P < 0.01$) and mPECQs ($r = 0.859$, $P < 0.01$), respectively, which also were supported by the results of the linear regression analyses (Fig. 4b, c). Moreover, SPI, another derivation of EqPA, had higher positive correlations with STd ($r = 0.925$, $P < 0.01$) and mPECQs ($r = 0.904$, $P < 0.01$) with increased fitting degrees, respectively ($R^2 = 0.856$ and 0.817; Fig. 4d, e). The STd-mPECQs pair had the highest positive correlations ($r = 0.991$, $P < 0.01$) and fitting degree (Fig. 4f; $R^2 = 0.983$), which possibly result from that they rely on empirical methods and were derived from the same data set (Farkas et al. 2007; Jamshidi-Zanjani and Saeedi 2013). In all, the results of the four different SQG methods were mutually verified.

However, the total dry weight of metal is used in most of the SQGs, which is different from the real status of the metals in the sediments and possibly cause a deviation in the SQGs. The reasons that affect the bioavailability and toxicity of heavy metals and metalloids in the sediments also are not fully considered, such as the combination of metals in sediments (acid volatile sulfur compounds, organic carbon, iron, and manganese oxides, etc.) and physicochemical conditions of the water and interstitial water (pH, redox potential, hardness, basicity, ligand in the metal complex, etc.) (Bay et al. 2012; Vidal and Bay 2005). Due to the different pollution characteristics of different sampling regions, mPECQs cannot be equally applied (Farkas et al. 2007; MacDonald et al. 2000). SPI considers the fraction speciation of the pollutants, but there are differences between the water

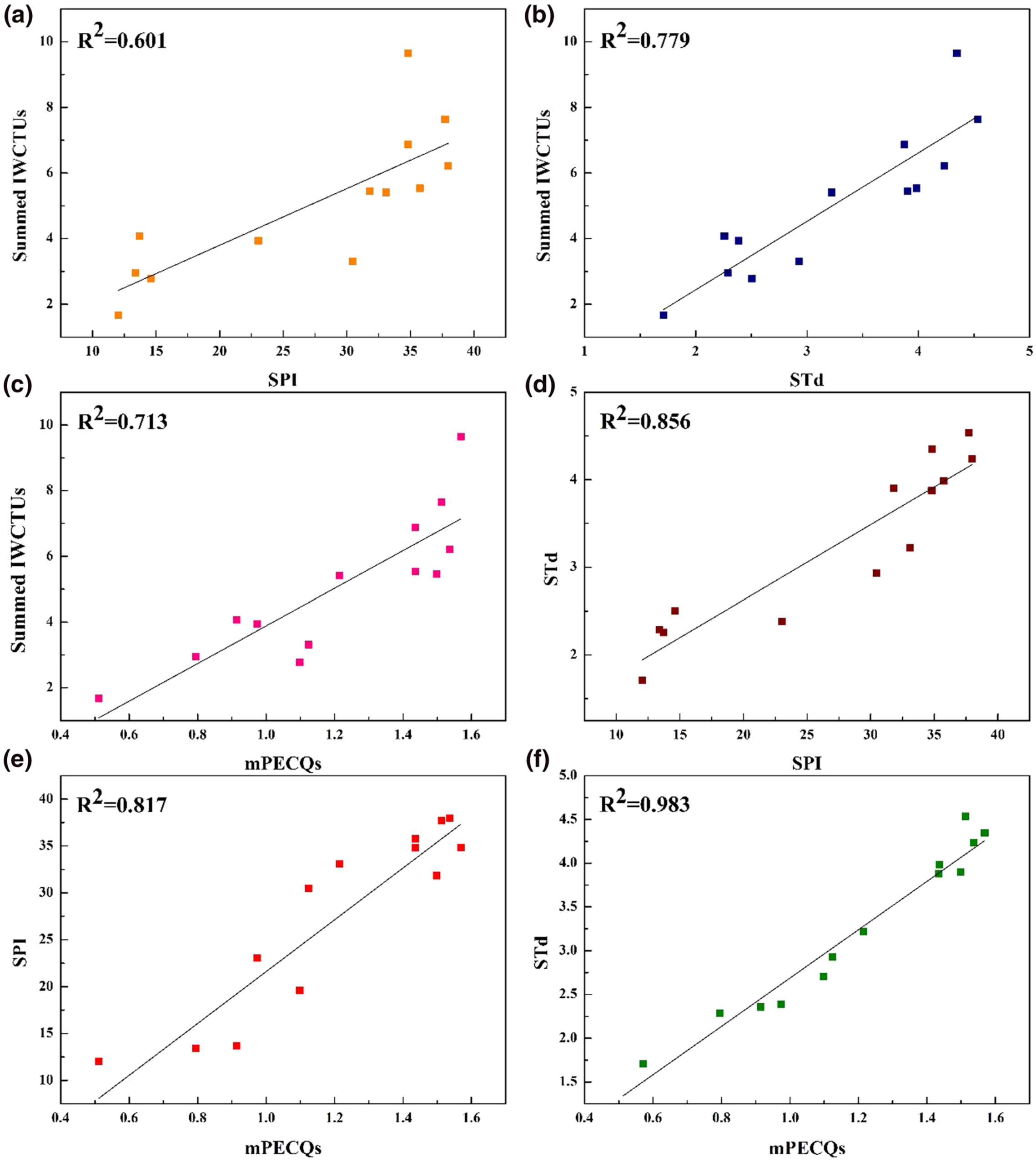


Fig. 4 Scatter plot of the linear regression analyses between mPECQs, SPI, summed IWCTUs and STd for the sampled surface sediments in the Xiangjiang River

quality standards and the actual standards of the interstitial water in sediments (Han et al. 2014). The application of the ESBs, such as IWCTUs to multiple metals is complicated, not only by the chemical interactions of the metals in the sediment-interstitial water system, but also due to their possible toxic interactions (Hansen et al. 2005; Suter and Tsao 1996). Therefore, it is highly recommended that a combination of empirical and theoretical methods is applied to intensively assessing the sediment quality of the Xiangjiang River from different angles.

Conclusions

In our study, we revealed that the average contents of Cd, Pb, Cu, Zn, Hg, Cr, and As in surface sediments of the Xiangjiang River were much higher than the soil trace element background for Hunan Province, especially the contents of Cd (103.65 times the background value) and Hg (6.21 times). Speciation fraction analyses indicated that Cd in the sediments was dominated by organic (average: 48.14%) and exchangeable (average: 30.27%) species, and the major speciation fraction of Hg (average: 79.49%) and

Pb (average: 73.11%) was the organic state. In contrast, an average of 94.06% of As and 79.79% of Cr in the sampled sediments were in the residue state. The results suggest that Cd possibly poses the highest ecological risk to the surface sediments of the Xiangjiang River, whereas As and Cr have less potential ecological risk.

The empirical and theoretical sediment quality indexes, CBSQGs, STd, and EqPA, were applied in our study to assessing the sediment quality of the Xiangjiang River. The results of CBSQGs showed that the contents of Cd and As dramatically exceeded the consensus-based PECs. The concentrations of Pb, Zn, Cu, Hg, and Cr were slightly lower than the consensus-based PECs but much more than consensus-based TECs. The mPECQs from most of the sampling sites ranged from 1 to 5, suggesting that the incidence of biological toxicity of the seven metals in the Xiangjiang River ranged from 70 to 75%. The results of STd indicated that the sampled sediments were considered to have a medium–high degree of toxicity, except at YZ-1. IWCTUs pointed out that the integrated biotoxicity in the Xiangjiang River was mainly derived from Cd, Pb, and Cu. The SPI found that all seven metals posed considerable toxic risk at the sampling sites of the Xiangjiang River, except YZ-1, XT-2, CS-3, and CS-4, which had moderate toxic risk, and YY-1, which had notable toxic risk. Pearson correlation and linear regression analyses between mPECQs, STd, IWCTUs, and SPI showed that these indexes were relatively consistent in assessing the sediments contaminated by heavy metals and metalloids in the Xiangjiang River. To supervise and remediate the metal contamination in the Xiangjiang River, further chemical transformation studies and toxicological assessments for benthic organisms in the sediments of the river are necessary.

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