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Original Research

Geochemical Baseline Establishment, Pollution Assessment, and Source Apportionment of Heavy Metals in Estuary Sediments of Northwestern Taihu Lake, China

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Abstract

The scientific and reasonable evaluation of heavy metals pollution in estuarine sediments is crucial for characterizing the environmental quality of the lake, however, it is still unavailable due to the lack of local geochemical baseline concentrations (GBCs) and thus applying background values of soils. In this study, the statistical method of cumulative frequency was employed to obtain the GBCs, which were applied as reference standards for the pollution assessment of heavy metals and source apportionment in estuary sediments of northwestern Taihu Lake. The results showed that the GBCs of Cd, Cu and Zn were higher than soil background values in Jiangsu Province while Ni and Pb displayed a small gap between GBCs and background values. Cd, Ni and Zn presented a degree of moderate pollution or moderate to strong pollution according to geo-accumulation index (*Igeo*). The pollution load index (*PLI*) showed that 75% of samples ranged from non-pollution to moderate pollution level while 2.94% of samples exhibited a moderate to high pollution level. The possible sources of heavy metals in the estuary sediments were quantitatively identified by positive matrix factorization (PMF), including agricultural source (23%), the combined contribution of natural sources and traffic emissions (36%) and industrial source (41%).

Keywords: geochemical baseline, heavy metals, estuary sediments, pollution assessment, source apportionment

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Introduction

Due to the concealment, persistence, non-degradability, and toxicity, heavy metal contamination poses a serious threat to lake ecosystems through bioaccumulation and biomagnification during the migration processes [1, 2]. The lacustrine estuary, as the interaction area between surface runoff and lake water, is a complex ecosystem affected by multiple biogeochemical factors as well as human activities. The estuary sediment acts as a main sink for allochthonous heavy metals carried by the inflow rivers around lakes, which records abundant information of environmental evolution associated with heavy metal pollution [3, 4]. Due to the weaker hydrodynamic exchange capacity of the estuary water than inflow rivers, most heavy metals from inflow rivers are easily trapped in estuary sediments of lakes through the adsorption, deposition, and desorption of suspended matter [5-7]. Meanwhile, the estuary sediment can serve as a significant releasing source of heavy metals when the physicochemical conditions change at the sediment-water interface of lakes [8]. The sedimentary heavy metals releasing into the overlying water will cause the secondary pollution and directly endanger the ecological environments of lakes [9]. Therefore, the scientific and reasonable evaluation of heavy metal pollution in estuarine sediments is crucial for characterizing the environmental quality of lakes and exploring the impact of human activities on the aquatic environments.

The geochemical baseline and the environmental background values are generally selected as the reference standards in assessment calculations of heavy metals pollution. The geochemical baseline concentrations (GBCs) refer to the natural level of heavy metals in the soils or sediments of a specific small-scale study area when the series of data in the study area were considered as a reference [10, 11]. Geochemical baseline is the upper limit of the normal range of heavy metal concentrations in the environment at present, reflecting whether the current levels of heavy metals is influenced by human beings [12]. Environmental background values usually reflect the concentrations of heavy metals in the undisturbed sediments or soils of large-scale natural environmental areas [13]. The accurate estimation of environmental background values for lake sediments needs to collect and measure lots of sediment samples from various types of lakes (e.g., different nutritional levels or distributed in different altitudes) affected by less anthropogenic activities [14, 15]. This estimation process of background values encounters difficulties in practice, and the background values of soils are often applied in the pollution assessment of heavy metals in lake sediments. Due to the difference of physicochemical properties between soils and sediments, the evaluation results of heavy metal pollution may be somewhat controversial. Therefore, the establishment of geochemical baseline can provide a meaningful reference standard for the pollution assessment of heavy metals in lake sediments when the environmental background values of local lake sediments are lacking.

Taihu Lake connects the plain river network of the most developed economy area in China, and thus it receives a large amount of heavy metal contaminants from anthropogenic sources [16]. At present, heavy metal pollution in sediments is still one of the main problems threatening the ecological environment of the lake [17]. Intensive studies have mainly focused on heavy metals in far shore sediments of the lake, including the pollution assessment based on background values, source identification, and evolution history [18, 19]. However, the establishment of local geochemical baseline for pollution assessment of heavy metals in estuarine sediments is still scarce. Therefore, the objective of this study is to establish the geochemical baseline of heavy metals in sediments, and further to assess the pollution levels and quantitatively identify the sources of heavy metals in estuary sediments of northwestern Taihu Lake. This study can provide effective information for scientific and reasonable evaluation of heavy metals pollution in estuary sediments of lakes highly affected by human activities.

Materials and Methods

Study Area and Sampling

Taihu Lake (30°55’40”-31°32’58”N, 119°52’32”-120°36’10”E), as the third largest freshwater lake in China, is located at the core of the Yangtze Delta (Fig. 1). The northwestern Taihu Lake adjoining Wuxi City and Changzhou City connects most of the inflow rivers, with an average depth of 1.9 m [20]. The industrial sewages, including a large amount of heavy metal pollutions from chemical engineering, metallurgy manufacture, papermaking and textile dyeing in the two industrialized cities, are discharged into inflow rivers, and further accumulate in estuary sediments of the lake. Therefore, the heavy metal pollution in estuary sediments of northwestern Taihu Lake has become a major threat to the ecological environment of Taihu Lake.

According to the distribution characteristics of inflow rivers around Taihu Lake, the surface sediment samples (0-10 cm) were collected from 68 estuary sites in August 2015 using a Peterson sediment sampler (Fig. 1). The sample locations were accurately recorded using the global positioning system (GPS). Triplicate surface sediment samples from each site were well mixed and then transported to the laboratory for further analysis.

Sample Analysis

After removing large particles, the freeze-dried sediment samples were powdered using agate grinding

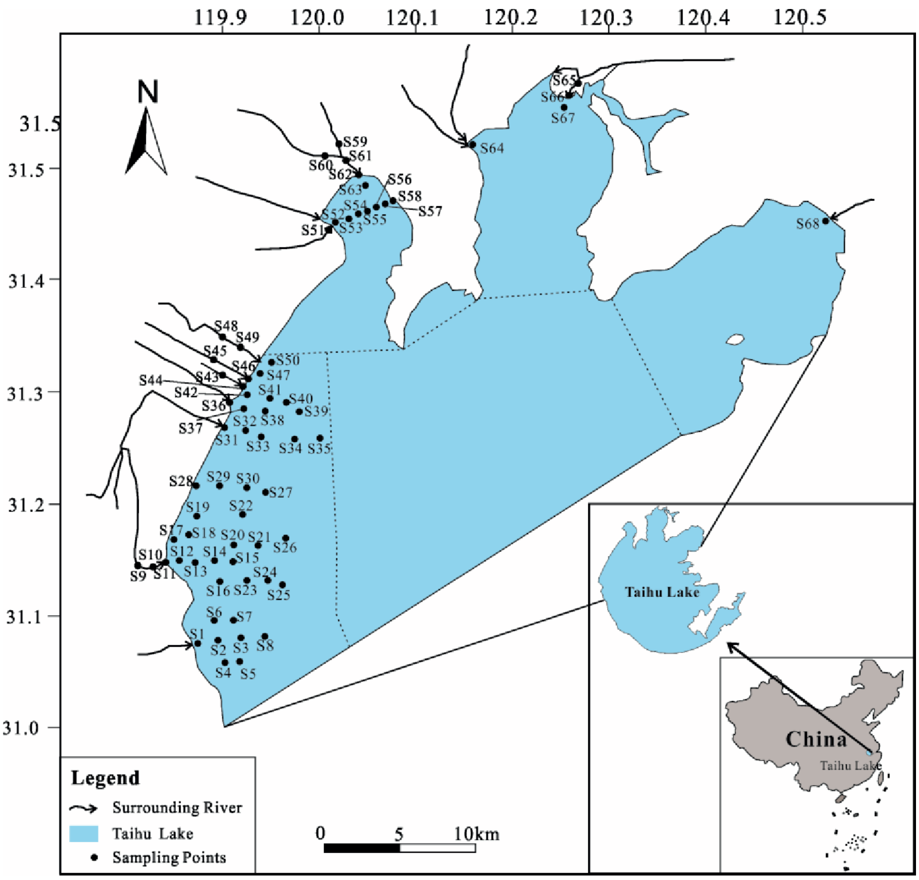

Fig. 1. Sediment sampling sites of northwestern Taihu Lake.

machine, and then passed through the 0.075 mm screen. The fine-grained sediment samples were digested by EPA 3051 method (US EPA, 2004). The contents of the representative heavy metals (Cu, Zn, Ni, Pb and Cd) in sediments were determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Perkin Elmer Inc. NEXION 300X, USA). The glass wares and polyethylene utensils were fully soaked in 2 mol·L^{-1} HNO_3 for more than 24 h before the pretreatment and measurement of samples. During the whole experiment process, the ultrapure water was used for cleaning experimental vessels and preparing solutions. National standard sediment materials (GSD-7, GBW-07307, China), blanks and duplicate samples were measured to perform the quality control of the instrumental analysis procedures and meet the precision and accuracy of corresponding standards (National Environmental Protection Standard of the People's Republic of China, 2017).

Geochemical Baseline Calculation

The relative cumulative frequency curve is usually applied for calculating the GBCs of heavy metals [21, 22], including two basic calculation processes. Firstly, the data on heavy metal concentrations were performed concentrations normal distribution test and box chart statistics to eliminate the data outliers and make the remaining data basically comply with normal distribution. Secondly, a relative cumulative frequency curve was plotted based on decimal coordinates where the heavy metal concentrations in accordance with normal distribution and their corresponding relative cumulative frequencies were set as abscissas and the ordinate, respectively. Generally, there are one or two inflection points on the curve. The lower inflexion point (IP) means the upper limit of natural concentrations, and the average or median value of heavy metal concentrations lower than the upper limit is considered as the GBCs in the study area. The higher IP represents the lower limit of concentrations is affected by human activities. Based on the comparative analysis about IP calculation methods, the optimized method proposed by Fan Kai was applied in this study [23].

Pollution Assessment of Heavy Metals

Geo-accumulation index *(Igeo)* can reflect the pollution level of a single heavy metal element in sediments or soils [24]. *Igeo* value of each heavy metal in estuary sediment samples can be calculated as follows:

$$\mathrm{Igeo} = \log_2[C_s/(1.5C_B)] \quad (1)$$

where C_s and C_B are the measured concentration of individual heavy metal and the calculated geochemical baseline value of this element in study area, respectively. The coefficient 1.5 is an introduced parameter considering the fluctuation of background value caused by diagenesis. The ranges of *Igeo* values and the corresponding pollution degree can be defined as seven levels [24, 25].

Pollution load index *(PLI)* calculated based on pollution indexes (*PI*) of all involved heavy metals is an effective tool to clarify the overall contamination degree of these metal elements in a sampling site or study area [26]. The *PI* and *PLI* can be calculated based on the following equations:

$$\mathrm{PI_i = C_{si}/C_{Bi}} \tag{2}$$

$$\mathrm{PLI} = \sqrt[n]{\mathrm{PI_1 \times PI_2 \times PI_3 \cdots PLI_n}} \tag{3}$$

where C_{si} is the measured concentration of heavy metal i in the sediment sample, C_{Bi} is the geochemical baseline value of element i in study area, and n is the element number of analyzed heavy metals. According to the summary of previous studies [27, 28], *PI* values can be classified into four contamination levels. Moreover, the standards of pollution assessment for *PLI* values can be divided into six categories or four levels ranging from non-pollution to extremely strong pollution [26, 29].

Positive Matrix Factorization (PMF) Model for Identifying Sources of Heavy Metals

PMF is a multivariate receptor model for the quantitative analysis of pollution sources, which was proposed by Paatero and Tapper [30]. The PMF model sets the original sample data as a matrix *X* which is factorized into a factor score matrix (*G*), a factor load matrix (*F*) and a residual matrix (*E*) [31]. This principle can be expressed as follows:

$$\mathrm{X_{ij}} = \sum_{k=1}^{p}(\mathrm{G_{ik} \times F_{ik}}) + \mathrm{E_{ij}} \tag{4}$$

The optimal matrices *G* and *F* are obtained by minimizing the objective function *Q* when the PMF model is applied for the factorization of original matrix *X*. The function *Q* is determined as follows:

$$\mathrm{Q} = \sum_{i=1}^{n}\sum_{j=1}^{m}\left(\frac{\mathrm{E_{ij}}}{\mathrm{U_{ij}}}\right) \tag{5}$$

where U_{ij} means the uncertainty of heavy metal *j* from the *i*-th sample, and it can be calculated as follows:

$$\mathrm{U_{ij}} = \begin{cases} \frac{5}{6}\times\mathrm{MDL}, & \mathrm{X_{ij} \le MDL} \\ \sqrt{\left(\sigma_i \times \mathrm{X_{ij}}\right)^2 + (\mathrm{MDL})^2}, & \mathrm{X_{ij} > MDL} \end{cases} \tag{6}$$

where σ_i expresses the relative standard deviation of the concentration of heavy metal *i*, and *MDL* is the method detection limit for the contents of heavy metals in sediments.

Data Analysis and Presentation

The distribution characteristics of concentrations and pollution levels of heavy metals as well as the relationship curves between relative cumulative frequencies and corresponding heavy metal concentrations were analyzed by Origin 9.0 (OriginLab Corporation, Massachusetts, USA). The normal distribution analysis and statistical boxplot of heavy metal concentrations and inflection point calculations of relative cumulative frequencies were performed using SPSS 19.0 (SPSS, Inc., Chicago, IL, USA). Sampling sites in study area were presented by CorelDRAW Graphics Suite X7 (Corel Corporation, Ottawa, Canada) and ArcGIS 10.2 (Environmental Systems Research Institute, Inc., California, USA).

Results and Discussion

Concentration Distribution of Heavy Metals

Fundamental statistical characteristics of heavy metals (Cd, Cu, Ni, Pb and Zn) in estuary sediments of northwestern Taihu Lake were presented in Table 1. The concentrations of Cd, Cu, Ni, Pb and Zn in estuary sediments of northwestern Taihu Lake were in a range of 0.09-9.94 mg/kg, 11.76-207.52 mg/kg, 15.37-142.89 mg/kg, 18.09-57.50 mg/kg, and 4 4.34-435.82 mg/kg, respectively. The average concentrations of five heavy metals were obviously higher than corresponding soil background values in Jiangsu Province [32]. Concentrations of Cd in 66 samples (97.1%), Cu in 61 samples (89.7%), Ni in 62 samples (91.2%), Pb in 57 samples (83.8%) and Zn in 66 samples (97.1%) exceeded the background values. Therefore, from the perspective of comparison with the background values, the five heavy metals showed a high enrichment level in the study area. The coefficient of variation (*CV*) of heavy metals were found in the order of Cd>Cu>Zn>Ni>Pb. According to the *CV* division standards in previous studies [33], Cd and Cu concentrations showed a high variability with *CV* of 0.976 and 0.760, respectively, suggesting that their sources were more likely to be affected by various anthropogenic activities.

The normal distribution test for the contents of Cd, Cu, Ni, Pb and Zn was performed when the abnormal values of these contents outside the 1.5-fold quartile difference were gradually excluded by box chart statistics (Fig. 2). After eliminating the data outliers, the skewness coefficients and kurtosis coefficients of Cd, Cu, Ni, Pb and Zn were 0.264 and -0.565, 0.846 and 0.562, 0.890 and 0.271, 0.001 and -0.0.534, and 0.107 and 0.057, respectively. These values were all less than 1.0, indicating the remaining data for Cd, Cu, Ni, Pb and

Table 1. Descriptive statistics of Cd, Cu, Ni, Pb, and Zn concentrations (mg/kg) in estuary sediments of northwestern Taihu Lake.

	N	Cd	Cu	Ni	Pb	Zn
Mean	68	1.87	52.43	54.96	34.15	146.39
Maximum	68	9.94	207.52	142.89	57.50	435.82
Minimum	68	0.09	11.76	15.37	18.09	44.34
Standard deviation	68	1.82	39.84	29.28	8.30	88.72
Coefficient of variation	68	0.976	0.760	0.533	0.243	0.606
Background value[a]		0.13	22.30	26.70	26.20	62.60
Geochemical baseline value		0.9	32.3	26.1	27.0	72.8

[a] Soil background values in Jiangsu Province, China. N is the number of samples.

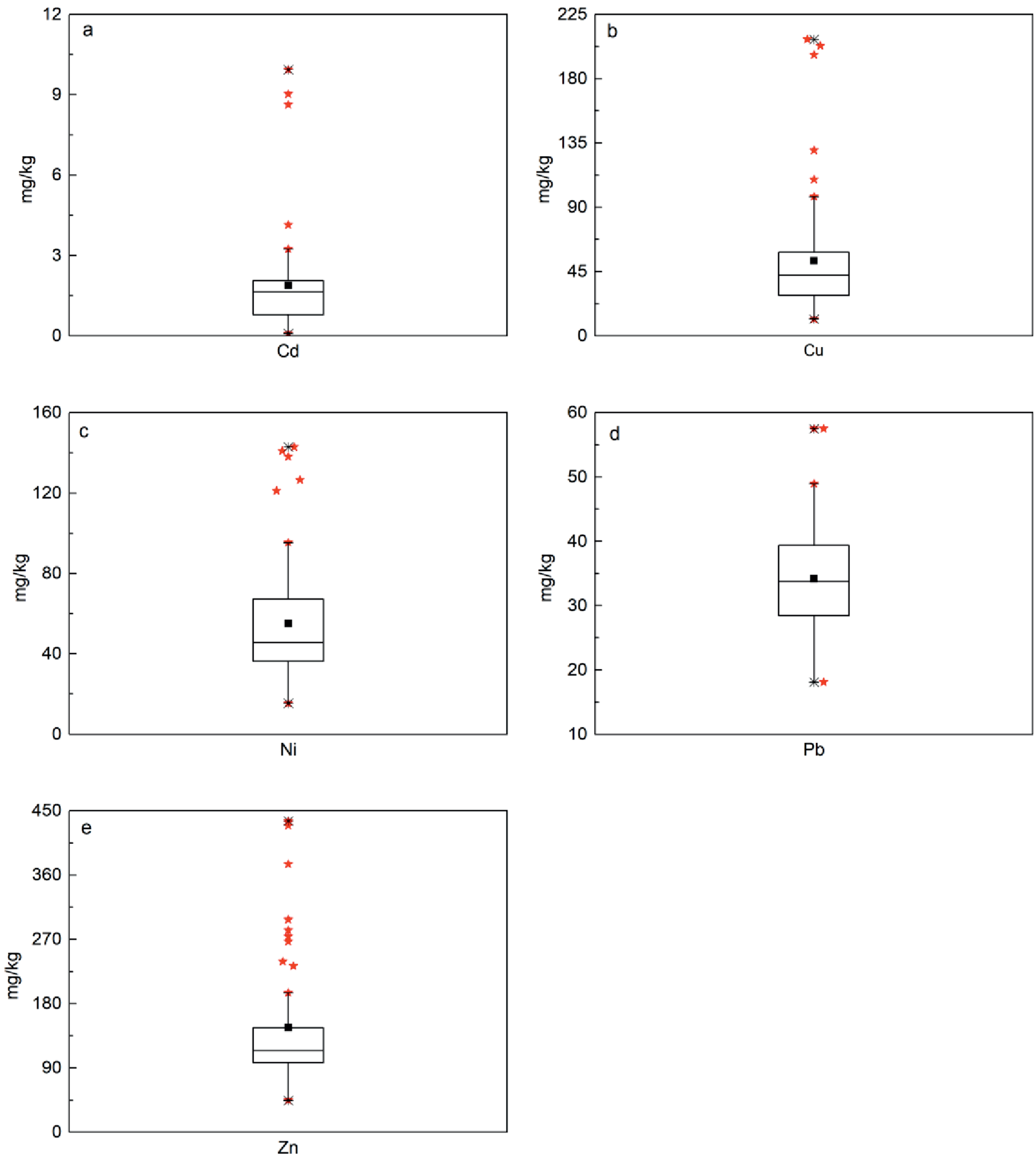

Fig. 2. Statistical box-plots of Cd, Cu, Ni, Pb, and Zn concentrations in estuary sediments of northwestern Taihu Lake.

Zn contents basically could meet the normal distribution [34, 35].

Establishment of Geographical Baseline

After removing the abnormal points of Cd, Cu, Ni, Pb and Zn concentrations, the relative cumulative frequency curve of concentrations for each element was shown in Fig. 3. All the curves existed two inflexion points (IP1 and IP2 in Fig. 3) based on the relationship between heavy metal contents and their relative cumulative frequency. Zn and Ni showed more sample points above IP2 than other three heavy metals, indicating the contaminations of Zn and Ni were greatly affected by human activities in study area [11, 22, 36]. In relative cumulative frequency curves, the average value of sample concentrations below IP1 was used as the geochemical baseline value for each heavy metal. Consequently, the geochemical baseline values of Cd, Cu, Ni, Pb and Zn were 0.9 mg/kg, 32.3 mg/kg, 26.1 mg/kg, 27.0 mg/kg, and 72.8 mg/kg (Table 1). The geochemical baseline values of Cd, Cu and Zn were higher than background values in Jiangsu Province while Ni and Pb presented a small gap between baseline values and background values.

Pollution Assessment of Heavy Metals

Pollution Assessment of Heavy Metals Based on Igeo

The *Igeo* assessment results of heavy metals in estuary sediments of northwestern Taihu Lake were displayed in Fig. 4. The *Igeo* ranges of Cd, Cu, Ni, Pb,

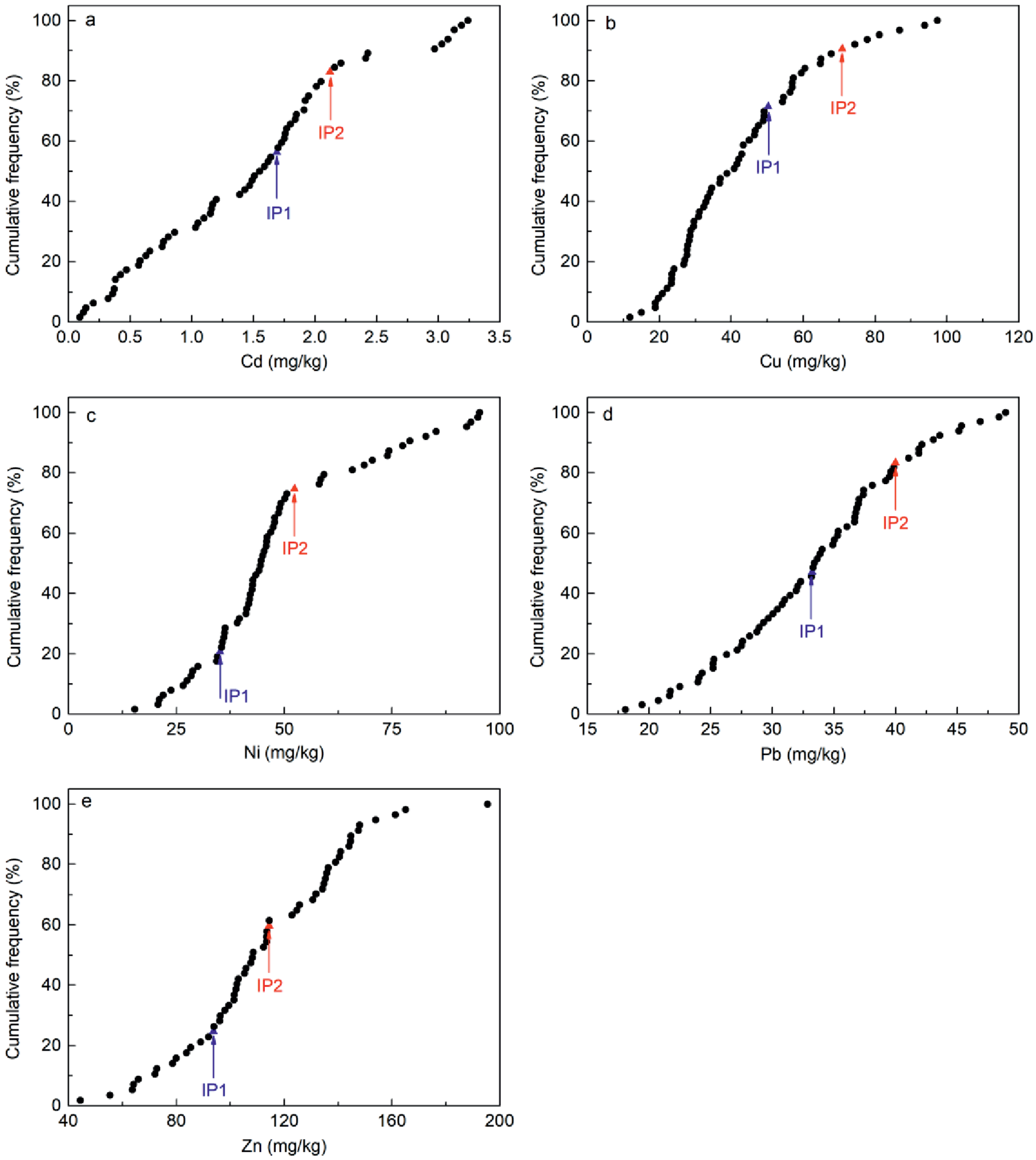

Fig. 3. Relative cumulative frequency curves of Cd, Cu, Ni, Pb, and Zn concentrations in estuary sediments of northwestern Taihu Lake.

and Zn were -3.88-2.91, -2.04-2.10, -1.35-1.87, -1.16-0.51 and -1.30-2.00, respectively. The average *Igeo* values of five heavy metals were ranked as follows: Ni>Zn>Cd>Cu>Pb. Generally, the contamination levels of these heavy metals (*Igeo*<3.0) were less than strong pollution according to the contamination degrees of *Igeo* category. Based on the statistical definitions of the median value, the 25th and the 75th quantiles, more than 50% of Cd, Ni and Zn samples presented the *Igeo* values higher than 0. Meanwhile, some samples of Cd (11 sites), Ni (12 sites) and Zn (11 sites) showed a degree of moderate pollution or moderate to strong pollution, indicating a certain contamination of the three elements in study area. The contamination level of Cu was less than Cd, Ni and Zn, because the *Igeo* values of Cu suggested that over 60% of samples were not polluted by Cu. More than 75% of Pb samples presented a non-pollution state.

The *Igeo* values of five heavy metals in these sampling sites showed the various and differential distribution characteristics. The sampling sites S43 and S44, located in the inflow river into Taihu Lake from Hongxiang Port, presented the *Igeo* values of all five heavy meals were more than 0, indicating this inflow river was conducive to the accumulation of heavy metals in estuary sediments of Taihu Lake [37]. The *Igeo* values of five heavy meals in site S68 near the junction of Taihu Lake and Wangyu River also ranged from 0.42 to 1.45, which clarified Wangyu River had an important influence on the contamination of heavy metals in estuary sediments of Taihu Lake [16]. Four heavy metals in 16 sampling sites and three heavy metals in 14 sampling sites showed a contamination level above slight pollution in accordance with *Igeo* values. Therefore, these regional distribution characteristics of heavy metal pollution indicated the non-ignorable effects of allochthonous inputs through inflow rivers on the heavy metal pollution of Taihu Lake.

Pollution Assessment of Heavy Metals Based on PLI

PI values of five heavy metals in 68 estuary sediment sites of northwestern Taihu Lake were summarized in Fig. 5a). *PI* values of Cd, Cu, Ni, Pb and Zn varied between 0.1 and 11.2, 0.4 and 6.4, 0.6 and 5.5, 0.7 and 2.1, 0.6 and 6.0, respectively. The sample proportions exhibiting a moderate contamination level (1≤*PI*<3) for five heavy metals were much higher than the other three degrees (*PI*<1, low contamination; 3≤*PI*<6, considerable contamination; *PI*>6, very high contamination), indicating the accumulation of Cd, Cu, Ni, Pb and Zn in most sampling sites were affected by anthropogenic factors [27, 28]. The *PI* values of Cd and Cu in 4.41% of sediment samples was higher than 6.0, indicating a high contamination level of the two elements in some sampling sites. The *PI* values of Cd, Cu, Ni and Zn ranging from 3.0 to 6.0 were respectively observed in 11.8%, 4.4%, 17.7% and 16.2% of samples, implying the sampling sites with a considerable contamination level of Ni and Zn were more than the other three heavy metals. Moreover, 27.9%, 35.3%, 7.4%, 19.1% and 10.3% of

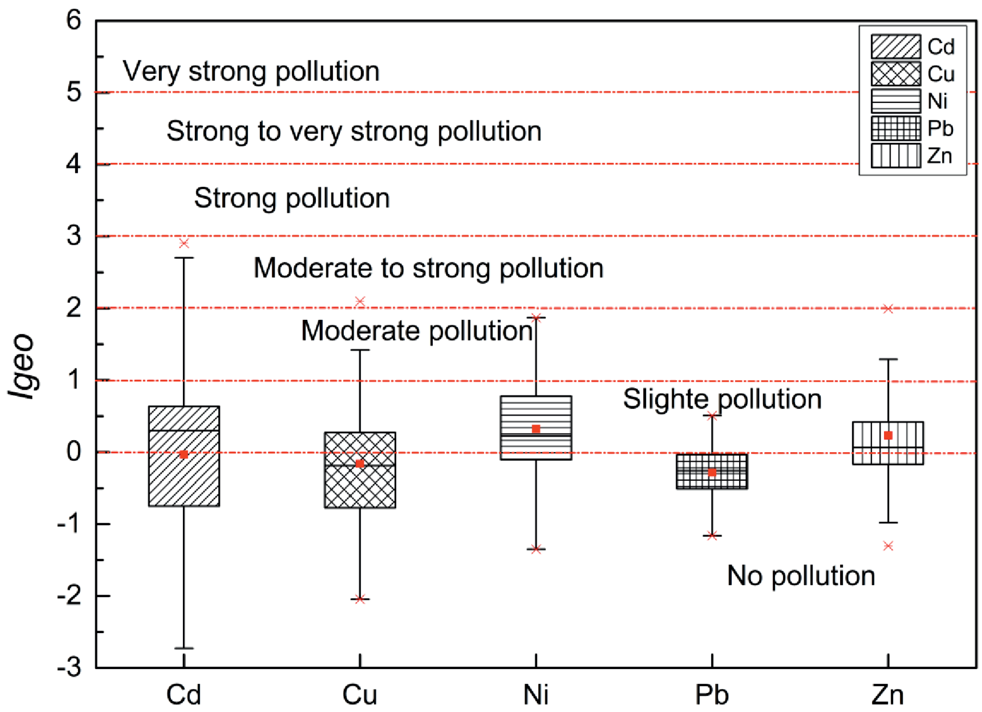

Fig. 4. Boxplots of pollution assessment results of Cd, Cu, Ni, Pb, and Zn in estuary sediments of northwestern Taihu Lake based on *Igeo* values. The black horizontal line and the small red square within the box present the median value and mean value, and the ends of box mean the 25th and 75th quantiles.

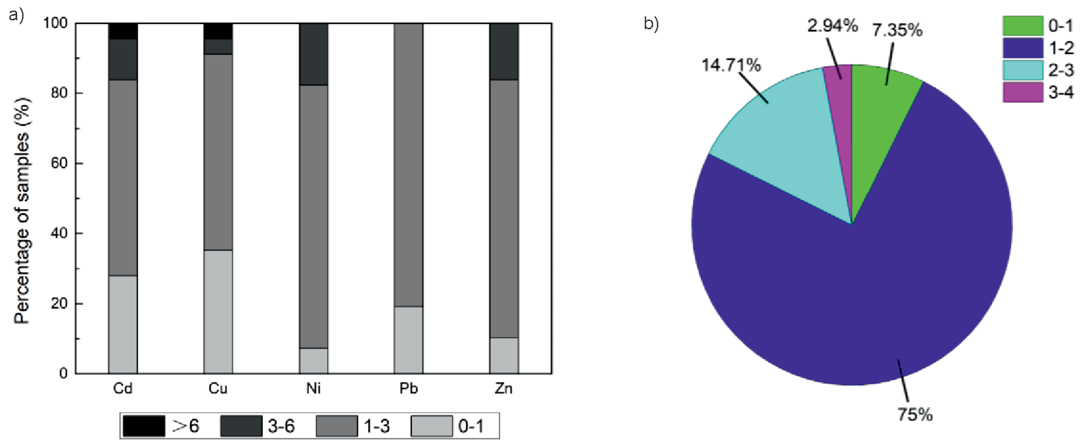

Fig. 5. The statistical characteristics for *PI* a) and *IPL* b) values of Cd, Cu, Ni, Pb, and Zn in estuary sediment samples of northwestern Taihu Lake.

total samples presented Cu, Cd, Ni, Pb and Zn at a low contamination level. Over the entire study area, *PI* values of Cd and Cu covered all the four evaluation levels. This wide coverage of evaluation levels clarified the various sources of two elements and their further accumulation in sediments affecting by complicated biogeochemical factors, corresponding with the results of *CV*.

The *PLI* values were calculated based on *PI* values of Cd, Cu, Ni, Pb and Zn to assess the comprehensive pollution level of five heavy metals in study area (Fig. 5b). In this study, the *PLI* values involved with four pollution grades [26, 29], i.e., non-pollution ($0<PLI\leq1$), non-pollution to moderate pollution ($1<PLI\leq2$), moderate pollution ($2<PLI\leq3$), and moderate to high pollution ($3<PLI\leq4$). Only 7.35% of samples showed that the *PLI* values of five heavy metals were less than 1, indicating most of the study area was comprehensively polluted by heavy metals. Moreover, the *PLI* values in 75% of the samples ranged from 1 to 2, which revealed the main pollution level of heavy metals in study area was non-pollution to moderate pollution, corresponding with the assessment results of *Igeo*. It should be noted that the *PLI* values ranging from 3 to 4 in 2.94% of the samples implied a moderate to high pollution level. These samples were located near the inflow mouth of Caoqiao River in Taihu Lake, and thus the allochthonous sources

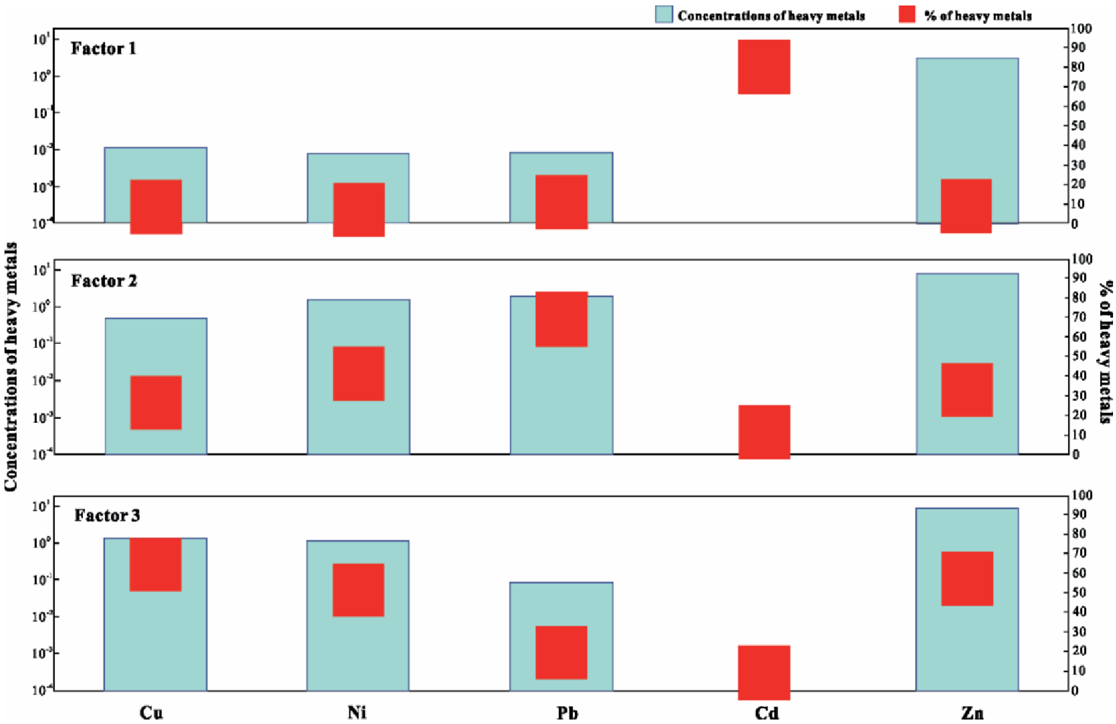

Fig. 6. Contributions of each factor (source) to each kind of heavy metal based on PMF model.

carried by this inflow river should be considered as an important contributor to heavy metal contamination in estuary sediments of northwestern Taihu Lake.

Source Apportionment by PMF

The number of factors was set to 3, 4 and 5 when the EPA PMF 5.0 was running for source apportionment of heavy metals in study area. The number of optimal factors was determined by comparing the difference between Q_{robust} (the optimal solution of the objective function Q in robust mode during the PMF running) and Q_{true} (the true value of the objective function Q) under factor numbers. Q_{robust} and Q_{true} were the closest when the number of factors was 3. The determination coefficients of all heavy metals were higher than 0.6, indicating that the model data presented a good fitting effect. Therefore, this model can realize the identification of heavy metal sources in the original data of this study based on the determined number of factors.

The results of the PMF source apportionment for all the heavy metals considered in the study were presented in Fig. 6. Factor 1 primarily constituted of Cd (80.1%) which accounted for less contribution rate to other factors. Meanwhile, Cd presented a high contamination level according to *Igeo* values. In study area, the enrichment of Cd mainly was related with the long-term use of pesticides and chemical fertilizers in traditional agricultural production [38, 39]. Many farmlands were distributed around northwestern Taihu Lake, which was an important part of production base cultivating rice mulberry and tea in China. Therefore, factor 1 was considered as the agricultural source, accounting for 23% of the source contribution.

Factor 2 primarily comprised of Pb (69.5%), Ni (41.4%), Zn (33.0%) and Cu (26.3%). Pb presented the much lower *CV* values than other heavy metals (Table 1), and *Igeo* values of Pb in most sites indicated a non-pollution state (Fig. 4). This indicated that Pb partly derived from soil parent materials, deposited in the sediment of northwestern Taihu Lake through various natural conditions. Moreover, Pb was an important indicator of traffic emissions. Many sample points above IP2 and *Igeo* values for Zn and Ni clarified the enrichment of Zn and Ni were greatly affected by human activities in study area (Fig. 2). Previous Reports have been documented that traffic emissions were the primary responsible for sediment contamination pertaining Cu, Ni and Zn besides Pb [40, 41]. Massive traffic emissions were produced by the combustion of gasoline and diesel, the leakage of fuel oil and lubricating oil and engine mechanical wea from motor ships on the lake. Therefore, factor 2 can be considered as natural sources and traffic emissions, explaining 36% of the source contribution.

Factor 3 mainly contributed to the concentrations of Cu, Ni and Zn, with its proportion of 65.1%, 51.6% and 57.8%, respectively. Meanwhile, Cu, Ni and Zn presented the high pollution levels based on *Igeo* values, which were mainly associated with electroplating, machinery manufacturing, metal smelting, leather production and other in Wuxi City and Changzhou City. Therefore, factor 3 was defined as the industrial source, taking over 41% of the total contribution.

Conclusions

This study established the local GBCs of heavy metals in estuary sediments of Taihu Lake by relative cumulative frequency. The pollution of heavy metals in surface sediments was assessed based on the established baseline values. The results showed that the geochemical baseline values of Cd, Cu, Ni, Pb and Zn were 0.9 mg/kg, 32.3 mg/kg, 26.1 mg/kg, 27.0 mg/kg, and 72.8 mg/kg, respectively. Cd, Ni and Zn presented a degree of moderate pollution or moderate to strong pollution according to *Igeo* values. The *PLI* values showed that 75% of the samples ranged from non-pollution to moderate pollution level while 2.94% of the samples showed a moderate to high pollution level. The allochthonous inputs through inflow rivers had the non-ignorable effects on the heavy metal pollution of Taihu Lake. The possible sources of heavy metals were quantitatively identified by PMF, including agricultural source (23%), the combined contribution of natural sources and traffic emissions (36%) and industrial source (41%). The established geochemical baseline well solved the lack of reference standards for the pollution assessment of heavy metals in the sediments of freshwater lakes, which is also helpful to identify the pollution sources of heavy metals.

Acknowledgments

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Conflict of Interest

The authors declare no conflict of interest.

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