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REVIEW PAPER

Distribution, fractionation and risk assessment of mercury in surficial sediments of Nansi Lake, China

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Abstract Nansi Lake is composed of four sub-lakes from north to south: Nanyang Lake, Dushan Lake, Zhaoyang Lake and Weishan Lake. An environmental pollution investigation was carried out to determine the fractionation, and pollution assessments of mercury (Hg) in surficial sediments from Nansi Lake. Results showed that the mean concentration of Hg was 3.1 times higher than its background value (0.015 mg kg^{-1}), and the high concentration of Hg which even reached up to five times than the background value in the part of Dushan Lake and Weishan Lake, which indicated that there are obvious spatial differences. The content of Hg was positively correlated with that of total organic carbon, and negatively correlated with that of pH and SiO$_2$ in surface sediments. An improved Tessier sequential extraction procedure was used to study the fractions of Hg in sediments. The results indicated that Hg existed primarily in the fraction of residual, which accounts for 58.4% of total mercury (THg), and the percentage of extractable Hg was only 1.93% of total mercury.

High concentrations of mercury of non-residual phase were found in part lake area from the Nanyang Lake and the Weishan Lake, which indicating a higher potential ecological risk relative to the other lake areas. Based on the values of enrichment factor and geo-accumulation index, most part of Dushan Lake and Nanyang Lake and Weishan Lake were in a moderate pollution. And based on the fractionation of mercury, risk assessment code of Hg exhibited low risks to the environment in Nansi Lake.

Keywords Mercury · Surficial sediments · Fractionation · Contamination assessment · Nansi Lake

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Introduction

Mercury is an important trace metal, which has received serious attention all over the world due to its high toxicity, persistence, bio-accumulation and mobility, and it may produce adverse effects on aquatic organism, water quality and human health (Feng and Bigham 2011). Mercury in the lake system can be scavenged from the overlying water to sediments through a variety of geochemical processes (Sanei et al. 2010). Lake sediments as important "source" and "sink" of lake pollutants played a major role in water ecosystems and are widely used to document the historical and spatial distribution of Hg

contamination (Drevnick et al. 2012). Sediment quality is an evidence to evaluate the water environment, it is necessary to understand the possible environment pollution by anthropogenic activities (Shen et al. 2008). Generally speaking, the mobility, the bioavailability and the toxicity of mercury in sediments depend on its specific geochemical fractions to a great extent (Diop et al. 2014). It has been proved that total concentration of mercury is not enough to evaluate the contaminated sediments, and the pollution assessment critically depends on the geochemical fractionation (Sanjay et al. 2011). The optimized Tessier sequential extraction procedure was applied to determine the mercury fractionation in sediments.

Nansi Lake is an important water supply for the agricultural irrigation and residents life. In the past few decades, with the rapid development of economy and urbanization, Nansi Lake has been influenced by heavy metals, especially mercury (Yang 2004). Nansi Lake is a significant regulating reservoir in the East-Line Project of South-North Water Transfer and a typical shallow lake in China. It is important to understand the distribution and fractionation of mercury in Nansi Lake sediments. The main goals of this study were: (1) to explore the spatial distribution of total mercury (THg) and the relationships between THg and environmental factors such as pH, TOC and SiO_2 in surface sediments of Nansi Lake; (2) to analyze the chemical fractionation of mercury in the sediment; (3) to estimate the pollution level of mercury in sediments of Nansi Lake.

Materials and methods

Study area

Nansi Lake (34°27′–35°20′N, 116°34′–117°°21′E) is in the southwest of Shandong Province, China, and it consists of four sub-lakes from north to south (as is shown in Fig. 1A–D): Nanyang Lake, Dushan Lake, Zhaoyang Lake and Weishan Lake. The catchment area of Nansi Lake is 30,453 km², and the Lake area is 1266 km². It is a typical shallow Lake with an average water depth of 1.46 m. After construction of the secondary dam in Zhaoyang Lake in 1960, Nansi Lake was divided into the upper lake (north part) and the lower lake (south part) (Shen et al. 2008), as showed in Fig. 1. The upper lake including Nanyang Lake,

Dushan Lake and the north part of Zhaoyang Lake, which catchment area is 26,934 km² and accounts for 88.4% of the total drainage basin. There are 29 rivers around the upper lake. The lower lake should refer to the south part of Zhaoyang Lake and the Weishan Lake, which catchment area accounts for 11.6% of the total drainage basin.

Sample collection

In July 2012, all total 205 surface (0–5 cm) sediment samples were collected from Nansi Lake (Fig. 1) by using a gravity corer, and kept in sealed plastic bags with marked labels, and then taken to the laboratory. These samples were air-dried at room temperature, removed the stones and debris. After sufficient grinding, they are sieved through a 100 mesh sieve and used for the measurement of the pH, TOC, SiO_2 and the THg concentrations and fractions.

Analytical methods

About 0.25 g samples were digested with the 4:1 ratio of concentrated HNO_3 and $HClO_4$ mixture solution in Teflon vessels by the microwave heating. The residue was leached with 5 mol L^{-1} HCl, adjust to 25 mL with double-deionized water. The total concentrations of Hg were analyzed by hydride generation-atomic fluorescence spectroscopy (HG-AFS, XGY-1011A, China), Si content was determined by graphite furnace atomic absorption spectrometry (GF-AAS, M6, Thermo Elemental, Franklin, MA, USA), and the major element Si was expressed as percent (%) of element oxides (SiO_2). The pH values of sediments were determined by Leici pH meter PHS-3C in a 1:2.5 (w/v) sediment-deionized water suspension after 1 h agitation.

Measurement of Hg fraction was in accordance with the relevant standard, Technical Requirements for Sample Analysis of Eco-geochemical Evaluation of DD 2005-03 (for Trial Implementation), which established by Geological Survey of China, and adopted the improved Tessier sequential extraction procedure to extract the different fractions of Hg in sediments. Seven operationally defined fractions were determined for Hg: water soluble fraction (F1), ion-exchangeable fraction (F2), carbonate fraction (F3), humic acid fraction (F4), ferric-manganese oxides fraction (F5), strongly organic fraction (F6) and residual fraction (F7). And the seven fractions was extracted by distilled water, magnesium

Fig. 1 Location of sampling sites in surface sediments of Nansi Lake
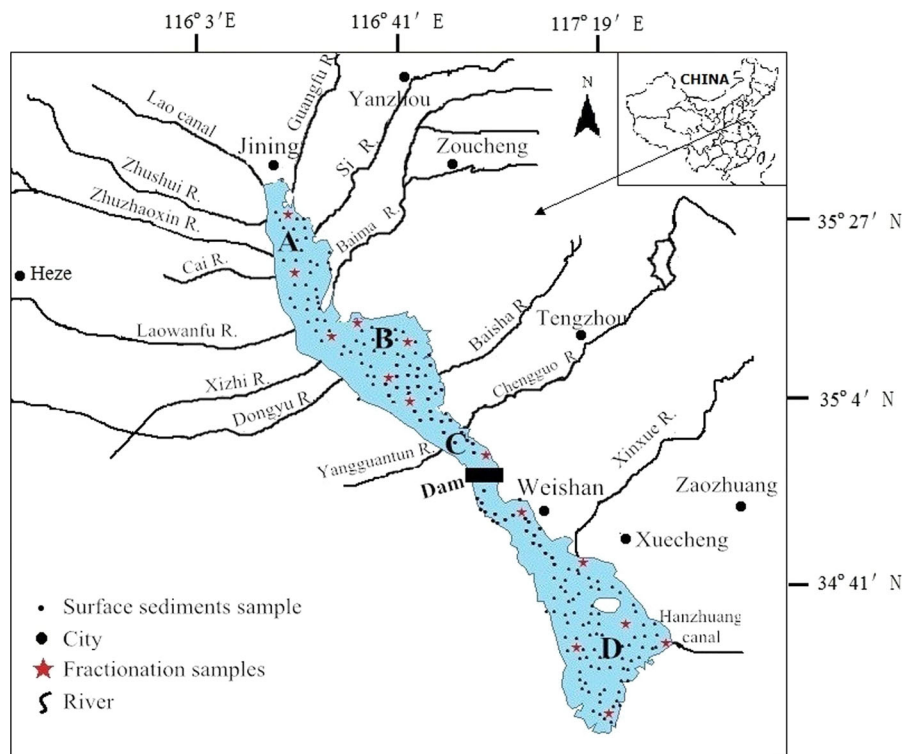
chloride (MgCl$_2$), sodium acetate (NaAc), sodium pyrophosphate (Na$_4$P$_2$O$_7$), hydroxylamine hydrochloride (NH$_2$OH·HCl), hydrogen peroxide (H$_2$O$_2$), hydrofluoric acid (HF), respectively.

Quality control

Quality assurance and quality control in the process of sample analysis were performed using system blank, certified reference materials (GBW07442 and GBW07443) and parallel samples in order to check the possible pollution and to control the precision of experimental data. The detection limit for Hg was 0.4×10^{-3} mg kg^{-1}, and the recoveries of Hg fraction were in the range of 82.3–97.1%, comparing to the total Hg concentration.

Assessment methods

The background value plays an important role in the pollution assessment. Nansi Lake was chocked with mud and sand from the Yellow River, and formed a dumbbell lake (Shen et al. 2008). For this reason, the average contents of heavy metals in sediments of

Yellow River were regarded as the background value of heavy metals in sediments of Nansi Lake. The background values of Hg and Al were 0.015 and 4.87×10^4 mg kg^{-1}, respectively (Yang 2004).

Enrichment factor (EF)

In order to identify the source of nature and anthropogenic for heavy metals, evaluation methods are necessary. It was an evidence that the grain size and mineral materials could lead to variability of heavy metals which came from anthropogenic sources. To compensate the potential influence of grain size and mineral variations on metal concentration, Al, served as conservation element, was been adopted to evaluate anthropogenic sources of trace elements (Muñoz-Barbosa and Huerta-Diaz 2013). The EF was used to indicate the natural or anthropogenic origins of Hg. The EF values were calculated according to the following equation (Kartal et al. 2006):

$$EF = \frac{(C_x/C_{Al}) \text{ sample}}{(C_x/C_{Al}) \text{ background}} \qquad (1)$$

where C_x and C_{Al} represent the concentration of Hg and Al, respectively. The six categories of EF values are recognized: $EF \leq 1$, no enrichment; $1 < EF \leq 2$, minimal enrichment; $2 < EF \leq 5$, moderate enrichment; $5 < EF \leq 20$, significant enrichment; $20 < EF \leq 40$, very high enrichment; and $EF > 40$, extreme enrichment (Wang et al. 2015).

Geo-accumulation index (I_{geo})

Geo-accumulation index was aim to quantify the degree of the environmental pollution in aquatic sediments. The I_{geo} values for Hg as introduced by Muller (1969):

$$I_{geo} = Log_2(C_n/1.5B_n) \qquad (2)$$

where C_n is measured element concentration in lake sediments, and B_n is the geochemical background value of the elements. 1.5 is the background matrix correction due to lithosphere effects. I_{geo} value mainly includes seven levels: $I_{geo} \leq 0$, background concentration (Class 0); $0 < I_{geo} < 1$, unpolluted to moderately polluted (Class 1); $1 \leq I_{geo} < 2$, moderately polluted (Class 2); $2 \leq I_{geo} < 3$, moderate to high pollution (Class 3); $3 \leq I_{geo} < 4$, heavily polluted (Class 4); $4 \leq I_{geo} < 5$, heavily to extremely polluted (Class 5); $5 \leq I_{geo} < 10$, extremely polluted (Class 6) (Soto-Jimenez and Paez-Osuna 2001).

Risk assessment code (RAC)

The RAC is defined as the concentration of trace element exchangeable and carbonates fractions compare to concentration of the total trace element (Singh and Lee 2015). The RAC is an indicator that can quantify the lowest release of mercury to the environment. The mercury in the sediment can be classified using RAC as: no risk, low risk, medium risk, high risk and very high risk, with RAC value ranges of <1, 1–10, 11–30, 31–50 and >50%, respectively.

Results and discussion

The chemical and physical properties of surface sediments in Nansi Lake

Properties of the surface sediments in Nansi Lake are shown in Table 1. The pH values ranged from 7.48 to 8.75, which showed an alkaline character in Nansi lake

sediments. TOC values were in the range 0.16–9.06%, with an average of 2.78%. And the mean TOC content of Nanyang Lake, Dushan Lake, Zhaoyang Lake, Weishan Lake was 1.92, 4.78, 2.94 and 2.60%, respectively. The higher TOC content in Dushan Lake might be due to abundant aquatic plant residue deposited in surface sediment. Organic matter can be used as mercury reservoirs because the presence of large number of humic substances in sediments, which were easy to scavenge Hg from overlying water (Parthasarathi and Raghunadh 2015). SiO_2 was an important part of the coarse particles in lake

Table 1 Statistics of Hg concentration (mg kg^{-1}), TOC and SiO_2 content (%) and pH in surface sediment from Nansi Lake

	Hg	pH	TOC	SiO$_2$
Nanyang lake (N = 37)				
Minimum	0.019	7.48	0.23	40.16
Maximum	0.097	8.75	6.11	64.41
Mean	0.045	8.03	2.47	50.52
SD	0.019	0.30	1.44	5.07
CV	42%	3.8%	58%	10%
Dushan lake (N = 20)				
Minimum	0.031	7.60	0.9	25.76
Maximum	0.072	8.11	6.91	60.16
Mean	0.058	7.71	4.78	37.65
SD	0.013	0.12	1.78	10.93
CV	22%	1.6%	37%	29%
Zhaoyang lake (N = 58)				
Minimum	0.017	7.5	0.16	25.85
Maximum	0.077	8.57	9.06	65.77
Mean	0.042	7.99	2.94	48.96
SD	0.016	0.32	2.65	7.78
CV	38%	4.0%	90%	16%
Weishan lake (N = 90)				
Minimum	0.023	7.53	0.48	28.1
Maximum	0.071	8.63	6.75	57.58
Mean	0.046	7.90	2.60	45.46
SD	0.011	0.24	1.47	5.08
CV	24%	3.1%	57%	11%
Nansi lake (N = 205)				
Minimum	0.017	7.48	0.16	25.76
Maximum	0.097	8.75	9.06	65.77
Mean	0.045	7.93	2.78	46.6
SD	0.015	0.28	2.03	7.53
CV[a]	33%	3.5%	73%	16%

[a] Coefficient of variation

sediments. The amount of SiO_2 ranged from 25.76 to 65.77% in the sediments of Nansi Lake, and the mean content of SiO_2 from the Nanyang Lake was higher than that of the others, and that from Dushan Lake was lower in all the sediments.

The distribution and influence factors of mercury in surface sediments

The content and distribution of mercury

Statistical analysis result of THg content in surface sediments of Nansi Lake is displayed in Fig. 2, which presented a normal distribution. And the THg content ranged from 0.017 to 0.100 mg kg^{-1}. The statistical results of the THg content and environmental impact factors in sediments of Nansi Lake are listed in Table 1. The mean concentration of THg in surface sediments was 0.046 mg kg^{-1}, which was above 3.1 times than the background value (0.015 mg kg^{-1}). And the high concentration of Hg which even reached up to five times than the background values in the part of Dushan Lake and Weishan Lake, which indicated that there is an obvious spatial differences.

Comparatively, the THg content increased in the following order: Zhaoyang Lake < Nanyang Lake < Weishan Lake < Dushan Lake. According to Table 1, the coefficient of variation values higher than 20% in

Fig. 2 Histogram of frequency of total Hg concentration in surface sediments of Nansi Lake

the four lakes and may be affected by anthropogenic sources and the TOC content.

Factors influencing Hg distribution

According to Table 1, the coefficient of variation values of Nanyang Lake was 42%, higher than the other lake area. The coefficient of variation values of Zhaoyang Lake was 38% was taken second place. The coefficient of variation values of Dushan Lake and Weishan Lake all were exceed 22%. The great changes of the coefficient of variation values in different lake area are mainly caused by the human activities in catchment of Nansi Lake, such as metal mining and smelting, coal mining and burning, sewage discharging from the small chemical plant and pesticide spraying (Wang et al. 2014). The large scale of wastewater without effective treatment from the Jining and Heze city was discharged into Nanyang Lake and inflow rivers. According to Fig. 3, apart from Hanzhuang River, other main inflow rivers were all obviously higher Hg contents than the background value of Nansi Lake. The average Hg content of Liangji River, Guangfu River, Dongyu River and Baima River was 0.05, 0.111, 0.041, 0.043 mg kg^{-1}, respectively. Thus, it was an important factor of Hg accumulation in the sediments of Nansi Lake, especially in the upper lake and lake outlet.

The catchment of Nansi Lake have a rich coal resources. There are some large coal-fired power plants such as Jining, Zoucheng, Jiaxiang and Liyan power plants nearby the Nansi Lake. Mercury released from coal combustion passed through atmospheric deposition (dry and wet) into the environment (Fu et al. 2012). The mercury concentration in coal produced by Yanzhou county was 0.188 mg kg^{-1} (Ren et al. 2002), which was much higher than the background value of sediment in Nansi Lake. The local coal combustion was maybe Hg pollution source in Nansi Lake catchment.

Besides anthropogenic impact, sediments were a potential reservoir for Hg, the kind of spatial heterogeneity is affected by environmental factors, such as pH, TOC, particle size distribution. (Soto-Jimenez and Paez-Osuna 2001). It is reported that total organic carbon in sediment plays an important role on controlling mercury deposition process (Parthasarathi and Raghunadh 2015). There is a positive correlation between the TOC and THg ($r = 0.736$, $p = 0.01$) in
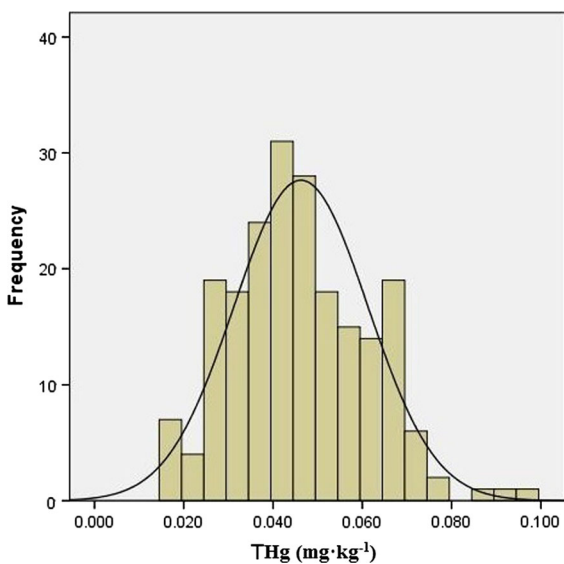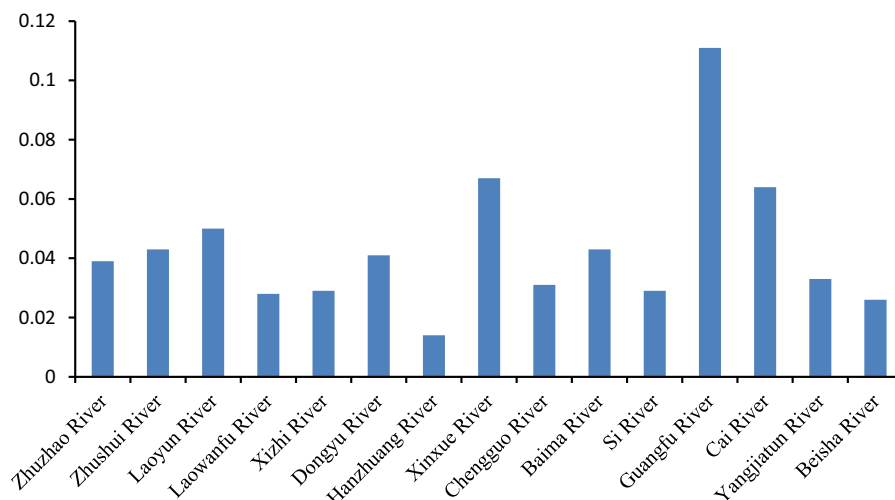
Fig. 3 Content of mercury in the sediment of main inflow rivers

surface sediment of Nansi Lake (Fig. 4). According to Table 1, the mean TOC content in surface sediment of Dushan Lake is 4.78%, which was the higher than that of the other lake area, and the mean SiO_2 content in surface sediment of Dushan Lake is 37.65%, which was the lower than that of the other lake area. It indicated that higher TOC content and lower SiO_2 content was one reason resulting in higher total mercury contents in surface sediment of Dushan Lake than that of the other lake areas.

Similarly, the particle size was also a significant role in the mercury transport, heavy metals are preferentially absorbed by fine particles than coarse ones (Wang and Cheng 2008). Coarse grains do not absorb Hg tightly; conversely, tiny grains could tightly absorb Hg due to their greater surface area (Wang and Cheng 2008). Particle size could reflect the hydrodynamic environment; therefore, the relationship between particle sizes and THg levels is key to understand Hg distribution in sediments. SiO_2 was a major component of coarse particles, the sediments of high SiO_2 content cannot tightly absorb Hg. Therefore, the relationship between SiO_2 and Hg was in negative correlation ($r = -0.677$, $p = 0.01$) (Fig. 4).

The acidity and alkaline not only affect solubility, deposition, adsorption of Hg in sediments, but it also affects the fractionation of Hg by influencing the microbial activity in the sediment (Ding et al. 2009). According to Fig. 4, a negative correlation existed between THg and pH. It was reported that pH values were in the range of 3–5, which the mercury hydroxide

($Hg(OH)_2$) was easier to be adsorbed than the $HgCl_2$, and pH values were more than 5 and continued to rise, which might cause Hg(OH)Cl higher active than $Hg(OH)_2$ (Kinniburgh et al. 1976). According to Table 1, the pH value in surface sediment was most in the range of 7.48–8.75, indicating that sediments in Nansi Lake presented weak alkalinity.

Fractionation of mercury in surface sediments

The total heavy metal concentration is not sufficient to assess the environmental impact of heavy metal contaminant (Parthasarathi and Raghunadh 2015). Chemical fractionation or the sequential extraction of mercury in the sediments of Nansi Lake is a useful technique for predictions of the mercury mobility and bioavailability. Due to be absorbed by organisms directly, the water soluble fraction (F1), ion-exchangeable fraction (F2) and carbonate fraction (F3) of mercury were the most toxic, and they all were called extractable fraction. The humic acid fraction (F4), ferric-manganese oxides (F5) and strongly organic fraction (F6) of mercury were more mobile in a specific sediment environment, which have a potential ecological risk. While the residual fraction (F7) of mercury combined strongly with sediments, which could not be biological assimilated (Singh and Lee 2015). Therefore, the mobility, bioavailability and ecotoxicity of mercury depend on its fractionation rather than its total content.

**Fig. 4** Correlations between THg and pH, SiO₂, TOC in surface sediments of the Nansi Lake ($p < 0.01$)
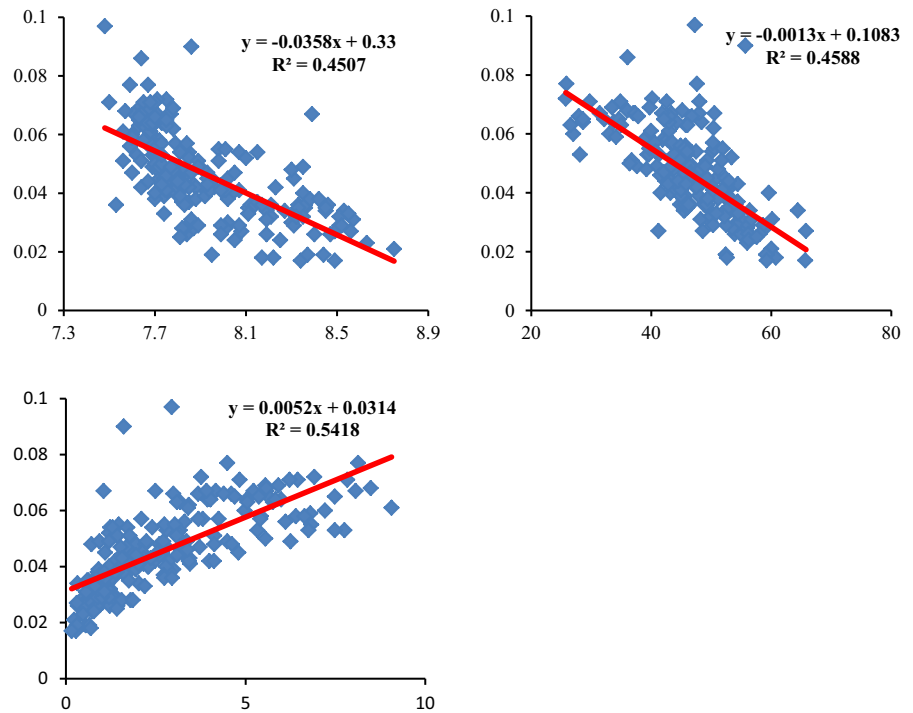
The fractionation results showed that mercury is mainly composed of residue fraction (Fig. 5). The characteristics of the residue fraction of Hg in the sediment were very stable, which exiting in the primary minerals and secondary silicate mineral grid and barely participated in the equilibrium partitioning of sediment and water system, so the residue fractions of mercury on the environment and biology are safe (Singh and Lee 2015).

The second largest proportion of Hg fractions was including the humic acid fraction and ferric-manganese oxides and strongly organic fraction of mercury, which also collectively referred to as the organic fraction, accounts for 31.92% of the total mercury in surface sediments. Bioavailability was defined as the extent of living receptors were exposed to pollutants in soil or sediment (Feng et al. 2015). Although the content of bioavailable mercury only account for small part of total mercury, the concentration of extractable fraction of Hg could still pose a significant risk to surrounding environment.

Except the residue fractions of mercury, the other fractions of mercury are also known as the non-residual fraction, which is an important evidence for

Fig. 5 Percentage distribution of Hg fractionation after the seven-step sequential extraction procedure (*NRF* non-residue fractionation)
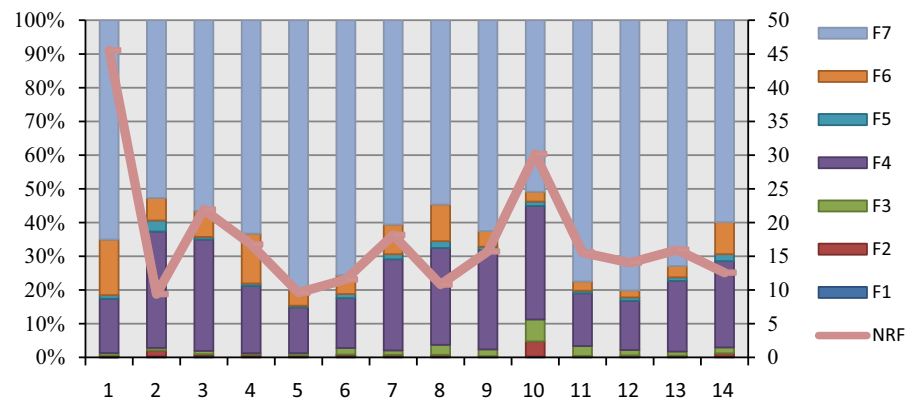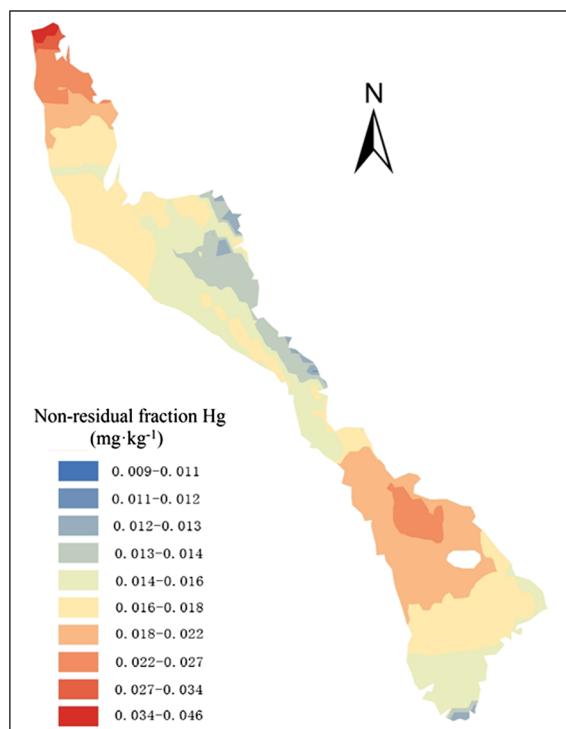
Fig. 6 Non-residual fraction for mercury in surface sediments from Nansi Lake

the direct risks and bioavailability of Hg. The non-residual fraction can be used to indicate the potential ecological risk of Hg in sediments, because that it can be released from the sediment when the factors of water environment changed. Concentration of non-residual fraction mercury is shown in Fig. 6, the high contents of Non-residual fraction were found in the estuary of Nanyang Lake and the north of Weishan Lake, while the other two lakes at a relatively low levels. In other words, Nanyang Lake and Weishan Lake may cause potential pollution to the ecological environment.

Pollution and risk assessment of mercury in sediments of Nansi lake

Enrichment factor (EF) and geo-accumulation index (I_{geo})

The magnitude of value of enrichment factor was used to define the enrichment of single element caused by anthropogenic origin in sediment, and the I_{geo} was applied to quantify the degree of anthropogenic

contamination in sediments (Sekabira et al. 2010). As a result, the space distribution of enrichment factor and geo-accumulation index of Hg in surface sediments are described in Fig. 7, the EF distribution presented high–low–high characteristics from north to south of Nansi Lake. The average EF of Hg was 2.2, 3.1, 1.8, 2.1 in Nanyang, Dushan, Zhaoyang, Weishan Lake, respectively. The minor enrichment ($1 < EF \leq 2$) was recorded in Zhaoyang Lake, the other three lakes were in a condition of moderate enrichment, indicating a certain degree of anthropogenic contribution. The monitoring data indicated that the untreated domestic and industrial wastewater directly discharged into Nansi Lake, especially into the Nanyang Lake and the Dushan Lake, was the mainly source of mercury and led to significantly Hg enrichment (Yang 2004).

The values of I_{geo} in Nanyang Lake, Dushan Lake, Zhaoyang Lake and Weishan Lake were 0.97, 1.31, 0.88, 0.90, respectively. Obviously, Dushan Lake has reached the moderate pollution, and the other lake area was close to moderate pollution.

In a few words, compare to result of the previous study (Yang 2004), Nansi Lake was still in moderately pollution, which indicated the Hg pollution in Nansi Lake did not get to deteriorate. This could be due to the implementation of control measures against pollution in Nansi Lake catchment. It has reported that the average content of mercury in seven types fish is much less than the content of the World Health Organization standards (Shi et al. 2006), which indicated the mercury is not dangerous to aquatic organisms in Nansi Lake.

Risk assessment code (RAC)

By the means of RAC, the mercury exhibited low risks ($1\% < RAC < 10\%$) in most lake area of Nansi Lake, accounted for 94% of the total area, except the part of area in Weishan Lake (Fig. 8). According to the RAC guidelines, it is considered safe for the environment if the exchangeable and carbonate fractions of the mercury in sediment are less than 10% of the total metal concentration (Sanjay et al. 2011). Moreover, there was no significant difference in the risk levels of Hg between four lakes area, suggesting that Hg enrichment is widespread in the lake region.

There are certain differences in the evaluate results between the Potential Ecological Risk Index and the
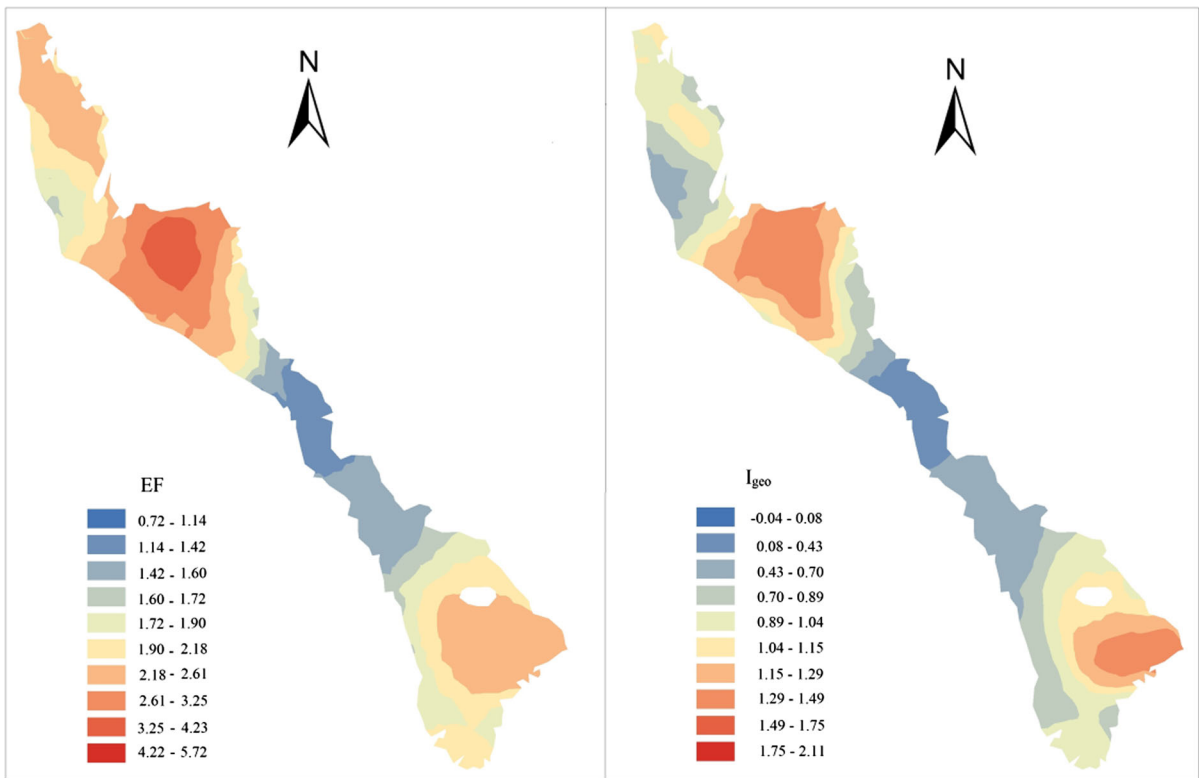
Fig. 7 Spatial distribution of enrichment factor (EF) and geo-accumulation index (I_{geo}) for mercury in surface sediments from Nansi Lake
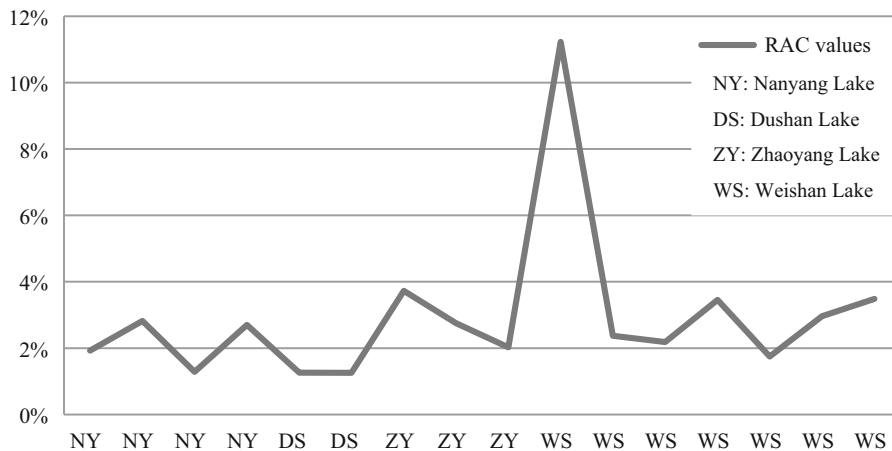

Fig. 8 The RAC values of mercury in surface sediments from Nansi Lake

Risk Assessment Code. Obviously, the Hg concentrations in Nansi Lake have reached or been close to the moderate pollution by EF method, but that exhibited low risks (1% < RAC < 10%) by RAC method. This

was because the former assessment method not only was taking into account the total amount of Hg, but also considering the toxicity of mercury, which results had human subjective weighting. The latter

assessment method was mainly to evaluate the bioavailability of mercury by means of its speciation, and the absolute content of Hg was not considered.

It is difficult to pose a hazard to the environment that the total amount of mercury is relatively high, but its risk level is lower, which showed that the evaluation result is different by the different criteria. In brief, considering the two aspects of THg and fractions of Hg, the contents of mercury in sediments from the Nansi Lake were in low risk, but the special attention should be paid to the management of Hg in Nansi Lake Basin.

Conclusion

Nansi Lake has been seriously influenced by long-term intensive industrial and human activities. Hg concentration ranged from 0.017 to 0.097 mg kg^{-1}, the average value of Hg was 3.1 times higher than its background value. The THg concentration was positively correlated obviously to the content of TOC ($r = 0.736$, $p = 0.01$), but negatively correlated with pH ($r = -0.671$, $p = 0.01$) and SiO$_2$ ($r = -0.677$, $p = 0.01$) in sediments. In addition, the predominant chemical fraction of Hg in sediments was the residual fraction. The proportion of bioavailable Hg was relatively weak, while it also can pose direct risks to surrounding environment. Based on the resulted of the enrichment (EF) and geo-accumulation index (I_{geo}), the Hg concentrations in Nansi Lake have reached or been close to the moderate pollution by EF method, but by the Risk assessment code (RAC), Hg in sediment of Nansi Lake may have low eco-risk.

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