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Original Research

Distribution and Ecological Risk Assessment of Heavy Metals in Sediments in Chinese Collapsed Lakes

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Abstract

Surficial sediments and water (27 samples each) were collected from three representative lakes collapsed by mining activities in Huaibei, China. Contents of heavy metals (Sb, As, Cd, Pb, Zn) in both water and sediments were detected via atomic absorption spectrophotometry and atomic fluorescence spectrometry, respectively. The variation coefficient (*C.V.*) of each heavy metal as well as the partition coefficient (*Kd*) between surficial sediments and water was calculated. The ecological risks of heavy metals in collapsed lakes were assessed via geoaccumulation index (I_{geo}), and we estimated the Pearson correlation coefficients between heavy metals. Together with the real situations around collapsed lakes, we resolved the sources of heavy metals. We found that the contents of heavy metals in water were basically below Surface Water Quality Standard I. The contents of heavy metals in the majority of sediment samples exceeded the background levels in the Yangtze River and Huai River areas, but were below the threshold effect concentration. The spatial distributions of heavy metals in surficial sediments were not uniform, as the *C.V.s* indicate medium variations. Analysis of Partition coefficient showed that Lieshan collapsed lake (LSH) was under severe risk. An analysis of I_{geo} revealed gentle-medium ecological risks from Sb, As, and Cd. The classification and frequency distributions of I_{geo} of five heavy metals indicate that the ecological risk of Cd was the largest. The ecological risk was lowest in Yangzhuang collapsed lake (YZH). These results were confirmed by analysis of *C.V.* and *Kd*. Analysis of heavy metal sources showed that the inputs of Sb and As into the three collapsed lakes were human-driven and very complex, probably due to point-source pollution, including leaching from gangue piles and industrial discharge. Cd mainly originated from non-point-source agricultural pollution, while Pb and Zn might mainly originate from natural sources.

Keywords: heavy metal, sediment, collapsed lake, ecological risk, geoaccumulation index (i_{geo})

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Introduction

The extensive use of downhole coal mining in China has resulted in soil destruction, the formation of large-area mining-caused subsidence zones [1], and ponding and lake development in subsidence zones. These consequences have altered the ground ecoenvironment. The exploitation and use of coals is accompanied by the formation of abundant coal gangue and burnt waste, which are usually piled near or on the banks of subsidence lakes in mining areas. However, these wastes may damage the surrounding environment [2]. Six of the trace elements found in coals (Hg, As, Se, Pb, Cr, Cd) are considered to be the most noteworthy environmentally sensitive elements [3]. Se, Pb, Zn, Cd, and Sb are very toxic and readily available in the environment. Since heavy metals are chronically circulating between water and particles, heavy metal pollution is more severe than organic matter or microorganism pollution [4]. The existing relevant research is focused on heavy metals in surface water and shallow groundwater [5], but is rarely targeted at the sediments of subsidence lakes. According to the priority pollutant lists (heavy metals) in and outside China as well as the situations in Huaibei mining areas, we selected five heavy metals (Sb, As, Pb, Zn, and Cd) that might be environmentally risky.

Heavy metal pollution is characterized by latency, wide source, non-degradability, and environmental accumulation [6], and heavy metals entering lakes are eventually deposited in lake sediments [7]. Once the water environment is changed, the heavy metals in the sediments would, through a series of physical, chemical, and biological processes, enter waters again [8]. Besides the heavy metal "source" and "sink" functions [9], the quality of lake sediment environment is closely associated with the well-being and sustainability of the whole aquatic ecosystem. Since the chemical behaviors and ecological effects of heavy metals in aquatic ecosystems are very complex, research about heavy metals in sediments has become a hotspot [10].

Existing research on the storage, conversion, and levels of heavy metals in sediments is focused on natural lakes, but rarely on mining-induced collapsed lakes. Especially in the arid and semi-arid areas of northern China, these artificial lakes would largely affect the local hydrology, climate, and living. Coal-producing areas were found with serious environmental degradation and pervasive gangue piling. Also, coal transportation and combustion causes modest pollution of water sediments. Thus, studying the distributions and ecological risks of heavy metals in collapsed lakes is very meaningful for reasonable water use in water-deficient areas.

The objective of the present study is to thoroughly uncover the distribution law and ecological risk assessment of the selected heavy metals (Sb, As, Cd, Pb, Zn) in different collapsed lakes of Huaibei, and thereby to provide scientific exact evidences of controlling heavy metal contamination in mine sediments. We will investigate:

1. The variation coefficients of heavy metal concentrations in sediments of different collapsed lakes, and how different these are among the selected heavy metals.
2. What are the contents of sediment-borne heavy metals among three collapsed lakes, and what factors may impact the space distribution characteristics of the selected heavy metals in sediment?

Materials and Methods

Study Area

Huaibei coalfield, located in northern Anhui Province, is a major coal industrial base in eastern China. This area is rich in coal reserves under good layer deposit conditions. Over 60 years of cumulative exploitation, coal mining caused large areas of land subsidence. So far, there are some 38 collapsed lakes in Huaibei mining area, with a subsidence water area of ~32.9 km^2. These lakes pond throughout the year and are 2-3 m deep on average (greatest depth 10 m). The large-area collapsed lakes have changed the morphology in Huaibei. These artificial lakes are very different from some natural lakes, thus analyzing and monitoring the contents/distributions of sediment-borne heavy metals in these lakes would facilitate the reasonable utilization of collapsed lakes and provide scientific reference for treatment and research of heavy metal pollution in coal mining areas.

Sample Collection and Processing

We selected three representative collapsed lakes in Huaibei mining area: Xiangcheng (XCH), Yangzhuang (YZH), and Lieshan (LSH). From each lake we set three sampling sites – to the eastern, middle, and western parts. In total, we distributed nine sampling points in each lake (Fig. 1) and samples were collected in August 2015. All sampling points were positioned using GPS. In total, 27 samples below a water depth of 50 cm were collected and put into 5-L polyethylene bottles until used. In addition, 27 samples (~1 kg each) of surficial sediments were acquired and stored in a refrigerator. After the water samples were transported to the laboratory, 250 ml of water were separated from each sample immediately and HNO_3 was added until pH was < 2. The samples were filtered through 0.45-μm membranes and stored at 4ºC. In the laboratory, the sediment samples were put in a freeze-drying box, impurities were removed, they were ground with a mortar, passed through a 100-mesh sieve, stored in polyethylene bottles, sealed with polyethylene bags, and stored in a freezer until used.

Element Analysis

The samples were digested and instrumentally analyzed as per U.S. Environmental Protection Agency (EPA) methods. Specifically, 0.2500 g from each sediment

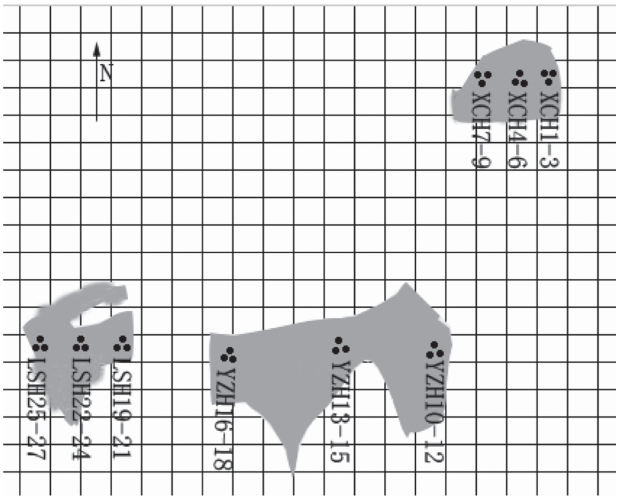

Fig. 1. Sampling sites in each collapsed lake.

sample was accurately weighed and put into a microwave digestion tank to which were added 4 mL of HNO_3, 5 mL of HF, and 2 mL of perchloric acid, successively. Then the tank was put into a microwave digestion instrument, and after 30 min of pre-digestion at 50ºC the substances were digested following a preset program. After that, when the temperature dropped to 40ºC, the tank was taken out and put on an electric heating plate, followed by ~1 h of acid removal at 180ºC. After that, the digestion tank was cleaned with ultra-pure water several times each with little water. Then the sample was completely removed to a sample bottle, which was diluted with deionized water to 20 mL. Pb, CD, and Zn were measured using an AAS-210 atomic absorption spectrophotometer (Beijing Beifenruili Instrument Co., Ltd, China), and Sb and As were detected via an AFS-2002 hydride generation-atomic fluorescence spectrometer (HGAFS, Beijing Jinsuokun Technology Developing Co., Ltd, China). The spiked recovery rate was 94.1-105.2%.

Heavy Metal Pollution Assessment

So far, the single-factor index and Nemerow index are commonly used to assess heavy-metal pollution in soil or sediments. These two indices are capable of comprehensive assessment, but unable to determine the artificial pollution of heavy metals during supergene. This problem can be resolved using the geoaccumulation index (I_{geo}), which makes up for the shortcomings of other indices. Since the 1960s, I_{geo} (also called Müller index) has been the most widely used index to quantify heavy metal pollution in sediments and other substances. It is expressed as follows:

$$I_{geo} = Log_2 \frac{C_n}{1.5 \times B_n} \quad (1)$$

…where C_n is the content of an element in sediments, B_n is the chemical background value of this element in sediments, and 1.5 is the coefficient considering the variation of background values due to regional differences in rocks and is used to characterize the geological and sedimentary effects.

Results and Discussion

Distribution of Heavy Metals in Overlying Water and Sediments

Table 1 lists the content ranges of five heavy metals in the overlying water and sediments of the three collapsedlakes, as well as a comparison with surface water quality standards and background levels. Clearly, the contents of all five heavy metals in overlying water of all three collapsed lakes are below Water Quality Standard I, except that the maximum Sb content is 4.26 μg/L and should be paid great attention. Moreover, the contents of all heavy metals (except Zn) in surficial sediments at all sampling points are above the background levels in the Yangtze and Huai river areas to different degrees (Table 1). The background values of As, Pb, Cd, and Zn are based on projects that are a geochemical survey of the Yangtze-Huaihe River basin of Anhui province, including 663 sediment samples from nine lakes. But these monitoring data were not published. The background values of Sb in soil samples from Huaibei coal mines was 4 mg•kg^{-1}[12]. In particular, average Sb content is 6.67 mg/kg, and average As, Pb, and Cd contents are 25.71, 0.26, and 74.83 mg/kg, respectively, which are 2.92-, 1.08-, and 1.6-fold above the standards, respectively. Compared with a threshold effect concentration (TEC) [13-14], only As content is above the standard and 2.6-fold of TEC. A comparison shows that Sb, As, Pb, and Cd in all three collapsed lakes will cause potential ecological hazards.

Coefficient of variation (*C.V.*) reflects the dispersity of data among different sampling points. Generally, *C.V.*<10%, 10-100%, and >100% are considered weak, medium, and severe variability, respectively [15]. A larger environmental *CV* of an element indicates more interference from artificial factors [16]. As for heavy metals in overlying water, the *C.V.s* of all elements are very large as "medium variation." The *C.V.s* of four heavy metals exceed 0.50 and *C.V.* of Sb is up to 0.86. The minimum *C.V.* is 0.47 from As. The *C.V.s* in the overlying water change as Sb > Pb > Cd > Zn > As.

The *C.V.s* of all heavy metals in sediments belong to medium variation. The *C.V.s* of Sb and As are larger (0.49 and 0.63, respectively). The *C.V.s* of five heavy metals change in order to As>Sb>Cd>Zn>Pb. Generally, the *C.V.s* of heavy metals are smaller in sediments versus overlying water, indicating that the overlying water is more vulnerable to the interference of human activities compared with sediments. The Sb, As, and Cd in sediments are subjected to more human interference. The *C.V.* of As is significantly larger in overlying water versus sediments, indicating that 1) As in overlying water is under very severe human interference, and 2) the migration and conversion

Table 1. Contents of heavy metals in water and sediments from three mining-related collapsed lakes in Huaibei.

Element	Sb	As	Pb	Cd	Zn
	Overlying water (μg/L)				
Minimum	1.53	1.75	0.68	N.D.	3.6
Maximum	4.26	15.8	4.7	0.14	36.5
Mean	3.25	9.6	1.67	0.07	12.6
S.D.	0.27	4.7	0.86	0.04	7.6
C.V.	86.20%	47.30%	74.60%	68.50%	53.80%
EQS-SW	5	50	10	1	50
	Sediments (mg/kg)				
Minimum	1.79	3.8	21.78	0.08	48.43
Maximum	11.64	37.43	48.65	3.47	127.59
Mean	6.67	25.71	31.26	0.26	74.83
S.D.	1.32	6.27	7.54	0.07	43.25
C.V.	49.75%	63.37%	24.81%	48.66%	36.27%
TEC	-	9.79	35.8	0.99	121
Background level	-	8.8	29	0.162	83
Fold above background level	-	**2.92**	1.08	**1.60**	0.90

Note: EQS-SW: Environmental Quality Standard for Surface Water

of As between overlying water and sediments are very weak. Results of *C.V.s* indicate that human activities are dominant factors affecting the variation of heavy metals in the collapsed lakes of Huaibei mining areas.

As reported, the *C.V.*s of Zn, Cd, and Pb in sediments from Dongdagou are very large, indicating the possible existence of point source pollution [17]. Sediments as an important indicator for water environment quality assessment have long been a focus [18-19]. Pollutants from multiple sources – including industry, agriculture, and transport – accumulate in sediments, then the continuous accumulation of toxic substances in sediments is extended through a series of exchange actions to the biological habitat areas, which via food chains will severely endanger the ecoenvironment and human health. Heavy metals, because of extensive sources, environmental accumulation, and persistent risks, become a major type of pollutant that largely affects sediment quality.

With chronic stability, heavy metals in sediments directly endanger the bottom-living organisms, and may be released to the overlying water where they would threaten the whole aquatic ecosystem [20]. The spatial distributions of five heavy metals in sediments of the three lakes are showed in Fig. 2. The distributive characteristics in the sediments of collapsed lakes are similar between Sb and As: LSH>XCH>YZH. The distributive characteristics of Pb are YZH>XCH>LSH. The distributive characteristics are similar between Cd and Zn: XCH>LSH>YZH.

The contents of sediment-borne heavy metals are different among the three collapsed lakes, which is due to the age of the lake formation and distance from coal piles. XSH and LSH are older and have stopped collapsing and become stable, while YZH is still young and unstable.

A coal gangue hill is located to the northeast of LSH. After soaking and leaching by rain, the heavy metals contained in the coal gangue will be gradually released to the environment, converge into lakes, and finally precipitate in sediments. Heavy metals in collapsed lakes may also originate from coal mining, washing, combustion and transport, which should be studied further.

YZH is a reserve water source for Huaibei City and basically is not threatened by any point source pollution. Thus, the heavy metal pollution in YZH is less severe than other two lakes. In comparison, XCH and LSH are near industrial areas, and received abundant industrial wastewater, which aggravated the heavy metal pollution in sediments.

Partition Coefficient of Heavy Metals between Overlying Water and Sediments

Partition coefficient is an important physiochemical parameter that portrays the behaviors of heavy metals in a water environment and reflects the migrating ability and potential ecohazard of pollutants between water-phase and solid-phase. Partition coefficient is the concentration ratio of a heavy metal in sediments relative to overlying water when the water-sediment two-phase system is balanced. Partition coefficient is expressed as:

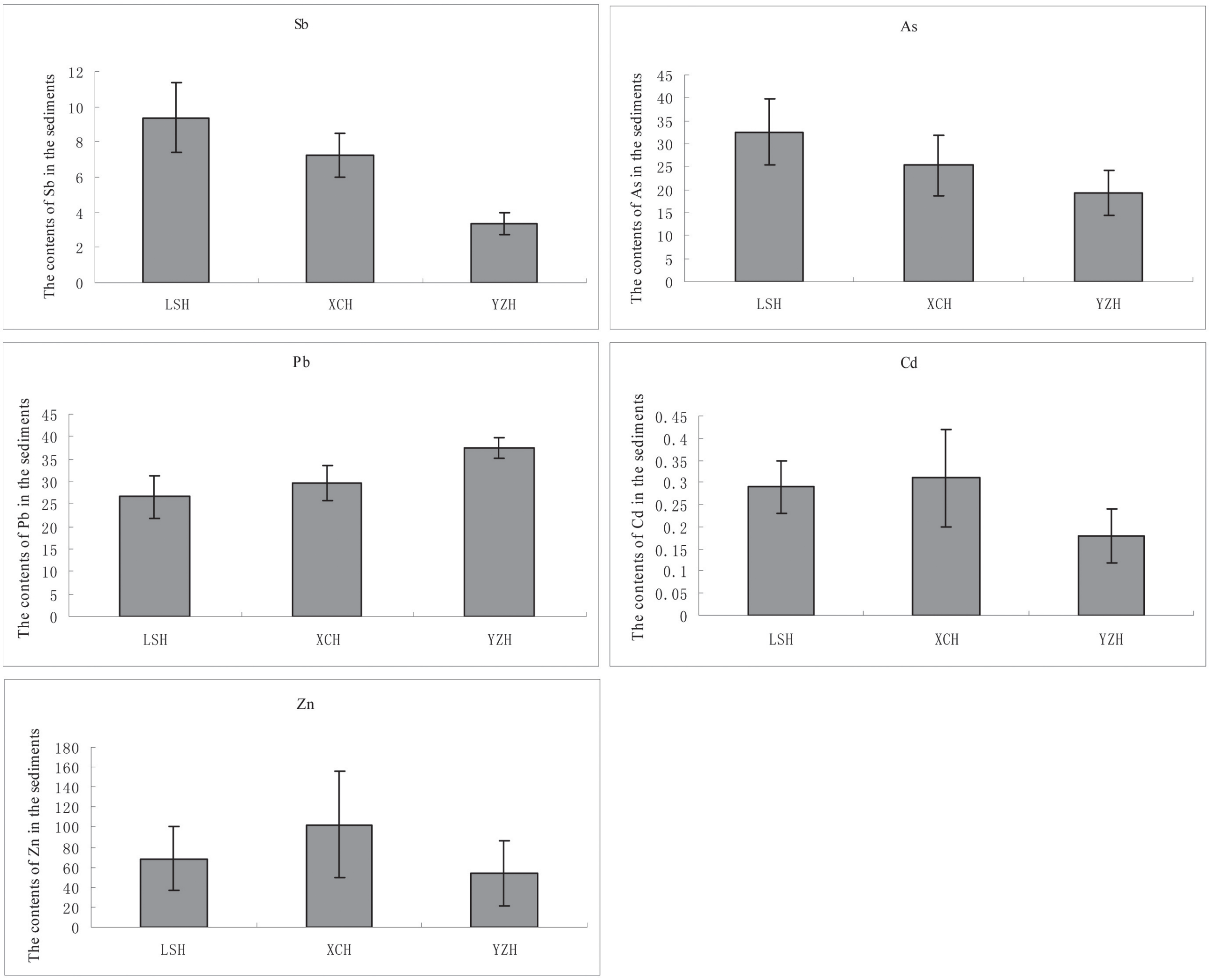

Fig. 2. Distributions of different heavy metals among three collapsed lakes.

$$K_d = Cs/Cw \tag{2}$$

…where C_s/(mg·kg^{-1}), C_w/(mg·L^{-1}) are the contents of a heavy metal in sediments and overlying water, respectively, under a balanced system.

The means, ranges of partition coefficient, and number of samples listed in Table 2 were cited from the U.S. EPA [21] (except for Sb data). Fig. 3 is a box plot of K_d of heavy metals in sediments of three collapsed lakes. The upper, middle, and lower lines on the box represent the upper four percentile, median, and lower four percentile, respectively; the extended upper and lower edges represent 5th/95th percentiles, respectively. Table 2 and Fig. 3 show the K_d underlying heavy metals between

Table 2. Partition coefficient (logK_d) (L·kg^{-1}) of heavy metals in sediments and water.

	logK_d of heavy metals in sediments and water (L·kg^{-1})				
	Sb	As	Pb	Cd	Zn
Mean	-	2.5	5.1	3.6	3.7-
Range	-	1.6-4.3	2.0-7.0	0.5-7.3	1.5-6.2
Number of samples	-	18	24	21	18
Study area (N = 27)	3.5-4.3	3.4-4.5	4.1-4.7	2.3-2.9	3.4-4.6
LSH (N = 9)	3.7	3.79	4.22	2.42	3.63
XCH (N = 9)	3.73	3.8	4.25	2.62	3.86
YZH (N = 9)	3.98	3.9	4.33	2.64	3.79

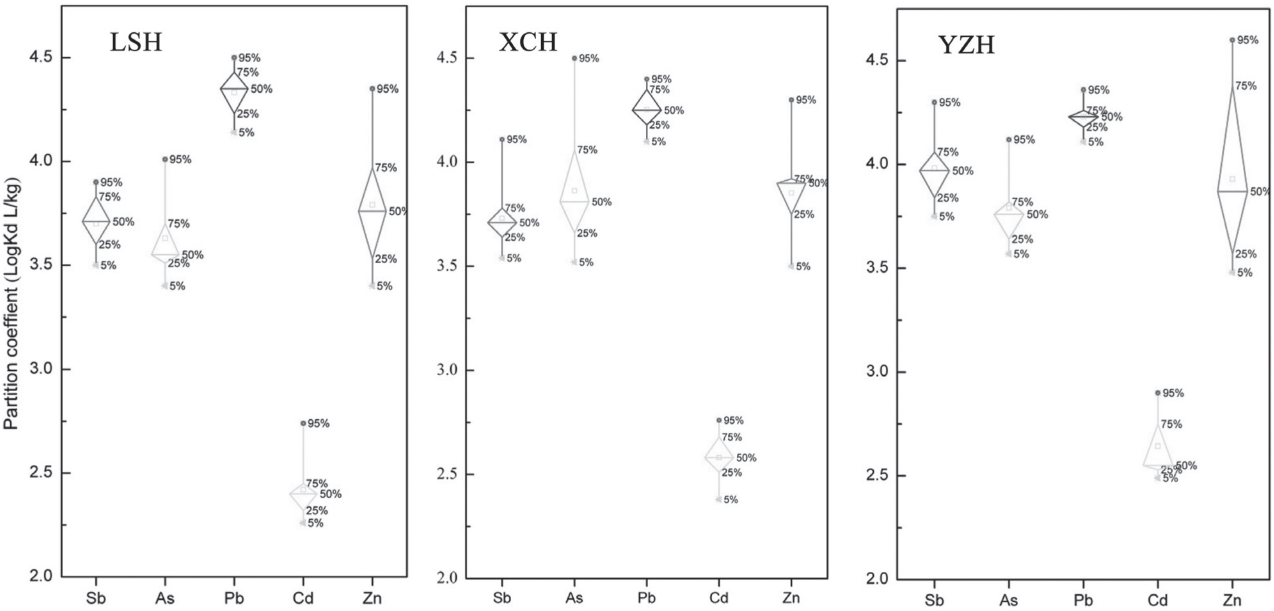

Fig. 3. Partition coefficient of heavy metals elements from collapsed lakes.

solid-water phases in collapsed lakes of Huaibei. Clearly, the K_ds in the water-sediment system are different among all elements, probably because of differences in chemical properties and adsorption characteristics among elements, which lead to the difference in two-phase partition among pollutants.

The logK_d of each heavy metal in LSH is significantly lower compared with other lakes (except Pb), probably because the competitive adsorption drove the dissolution of heavy metals from sediments to the overlying water, thus raising the heavy metal contents in the overlying water, indicating that the ecological risk of LSH is relatively higher. K_d of Cd is relatively lower among all heavy metals in three lakes, indicating that the ecological risk of Cd is very high.

As reported, metal concentrations are positively correlated with toxicity unit, and the strong acidic pH (pH<3.5) enhanced the metal toxicity by increasing metal activity and bioavailability [22]. Adsorption is a major route in which heavy metals are transferred from liquid phase to solid phase. The adsorption of heavy metals in sediments and suspended matter is driven by ion exchange, exclusive adsorption, nonexclusive adsorption, and complex adsorption. As reported, the chemical behaviors of heavy metals (As, Cd, Cr, Cu, Ni, Pb, and Zn) in the sediment-water interface are affected by environmental dredging, especially Zn and Cd (increasing rates 482.98% and 261.07%, respectively), probably due to some characteristics of suspended particulate matter (SPM). [23].

Geoaccumulation Index (I_{geo}) of Heavy Metals in Collapsed Lakes

As shown in Table 3, average I_{geo} of Cd is 0.115, while 2.3%, 4.7%, 12.3%, and 32.6% of sampling points belong to severe, medium-severe, medium, and gentle-medium pollution, respectively; average I_{geo} of As is 0.453, while 43.7% and 18.6% of sampling points belong to above-gentle and medium pollution, respectively; average I_{geo}

Table 3. Classification and frequency-based distribution of heavy metal I_{geo} in three collapsed lakes.

Class	I_{geo}	Pollution degree	Pollution frequency (%)				
			Sb	As	Pb	Cd	Zn
0	≤0	No pollution	63.7	56.3	91.6	48.1	84.7
1	(0, 1)	Weak-medium	36.3	25.1	8.4	32.6	15.3
2	(1, 2)	Medium	0	18.6	0	12.3	0
3	(2, 3)	Medium-severe	0	0	0	4.7	0
4	(3, 4)	Severe	0	0	0	2.3	0
5	(4, 5)	Severe-very severe	0	0	0	0	0
6	(5, 10)	Very severe	0	0	0	0	0

of Sb is 0.316, while 36.3% of sampling points belong to gentle-medium pollution; average I_{geo} of Zn is -0.734, while 15.3% of sampling points belong to gentle-medium pollution; and average I_{geo} of Pb is -0.477, while 8.4% of sampling points belong to gentle-medium pollution.

According to I_{geo}, the pollution degrees change in the order Cd>As>Sb>Zn>Pb. Nearly half of sampling points were polluted by Cd and As, showing very high ecological risks. About 36.3% of sampling points belong to gentle Sb pollution and above. These phenomena should hardly be ignored.

The average I_{geo} changes among heavy metals as As>Sb>Cd>Pb>Zn, indicating that As and Sb pollution is very severe. Together with Fig. 2, it it clear that As and Sb levels in sediments of LSH are very high and indicate very severe ecological risks.

Analysis of Heavy Metal Pollution Sources

Heavy metals mainly originate from natural sources (weathering erosion of soil, rocks) and anthropogenic sources (sewage discharge from human production and life, e.g., industrial water, agricultural water, and living water are discharged directly) [24]. The sources of heavy metals in sediments are correlated with the sedimentary parent materials of rocks and minerals, and also with the emissions from human production and life [25]. In a certain area, the heavy metals in sediments are relatively stable. Thus, studying the correlations among heavy metals in sediments helps to reveal whether the sources of heavy metals in sediments are the same. A higher correlation indicates more similarity.

The Pearson correlations among five heavy metals in each sampling point were computed on SPSS 17.0. Results show that in LSH, Sb, As, and Cd are significantly correlated pairwise (all P<0. 01), but Zn is not significantly correlated with other heavy metals. In XCH, Sb, As, and Cd are significantly correlated pairwise (all P<0. 05).

In aquatic environments, Sb and other compounds mainly originate from the wastewater discharge from diverse manufacturing industries [26], Cd generally is a mark of insecticide and fertilizer use from agricultural activities [27], while Pb indicates motor vehicle pollution [28]. Combining the *C.V.*s of heavy metals in sediments as well as the actual situations around collapsed lakes, we think that in LSH and XCH, Sb, As, and Cd come mainly from anthropogenic sources; Sb and As mainly originate from point sources including leaching from gangue piles and industrial discharge, while Cd mainly comes from non-point-source agricultural discharge. Correlation analysis can reveal the sources of the different heavy metals in sediments, and correlation coefficients are usually performed by SPSS statistical software. If there is a relationship between heavy metals, they may have similar sources and vice versa. In YZH, Pb and Zn are not significantly correlated with other heavy metals, so Pb and Zn may come mainly from natural sources.

Conclusions

By analyzing the heavy metal contents in three collapsed lakes, we find the three collapsed lakes already suffered gentle heavy metal pollution, especially in LSH and XCH. One possible reason is the relatively older ages of these two lakes. Analysis of partition coefficient shows that LSH is under a very severe ecohazard.

The spatial distributions of heavy metals in surficial sediments are not uniform, as the *C.V.s* indicate medium variations. Average *C.V.s* of Sb, As, Pb, and Cd are all above the background levels, but below TEC (except As).

I_{geo} is used to assess ecological risks of heavy metals in sediments. Results show that Sb, As, and Cd impose gentle-medium ecological risks. The classification and frequency distributions of I_{geo} indicate that the largest ecological risk comes from Cd. The I_{geo}s and classification of five heavy metals in the three collapsed lakes show that ecological risk is lowest in YZH. These results were confirmed by *C.V.*s and K_ds.

Analysis of heavy metal sources shows that the input of Sb and As into the three collapsed lakes was human-driven and very complex, probably due to point-source pollution, including leaching from gangue piles and industrial discharge. Cd mainly originated from non-point-source agricultural pollution, while Pb and Zn may mainly originate from natural sources.

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