

Original Research

Pollution Evaluation and Sources Identification of Heavy Metals in Surface Sediments from Upstream of Yellow River

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Abstract

Recently, the water quality of the second longest River in China (the Yellow river) is deteriorating severely. To investigate the heavy metals contaminated degrees, potential risks and pollution sources of heavy metals in the river, we measured the concentrations of 8 heavy metals (Fe, Mn, Cu, Ni, Zn, Cr, Pb and Cd) from 122 surface sediment samples collected along the upstream Yellow River. According to the results of enrichment factor (EF) analysis and the geoaccumulation index (I_{geo}) evaluation, we found Cd accumulated significantly for moderately contaminated level and the other heavy metals existed at uncontaminated levels. We conducted risk assessment by using calculation of the sediment quality guidelines (TEC-PEC SQGs) and potential ecological risk index (RI), and the results implied that the concentrations of Ni, Cr and Cd had potential harm for aquatic organisms and the upstream Yellow River had integral no toxic. Moreover, the results of multivariate analysis (principle component analysis, hierarchical cluster analysis and correlation analysis) indicated that Fe, Mn, Cu, Zn, Cr and Cd in the river primarily originated from natural sources, while Ni and Pb mainly derived from different human activities. In addition, partial Cu and Cd come from anthropogenic sources of Ni discharge.

Keywords: heavy metals, pollution evaluation, surface sediment, Yellow River

Introduction

The Yellow River, the second longest river in China, plays the crucial roles in providing the drinking water for millions inhabitants and using water for industrial and agricultural activities in the arid region of north China. However, recent studies have reported that

the water quality of the Yellow River is deteriorating severely because of the enormous pollutants discharged in the river through human activities and tributaries [1-2]. Currently, the Yellow River annually receives about 3.367 billion cubic meters of sewage, but only 48.6% of wastewater and tributaries satisfy quality criteria [3]. Thus, it is properly imperative to research the pollutions of the Yellow River.

Heavy metals are concerned and considered significant pollutants of aquatic systems [4] because

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of their toxic, stable or persistent characters [5-7]. Some heavy metals can store in the tissue of biota and may finally hazard human health through food chains [8-12]. In the aquatic systems, the heavy metals can originate from both natural sources (e.g. rock weathering, soil erosion, volcanic eruption and forest fires) and human sources (e.g. metallurgical industries, mining and smelting of metals, wastewater discharge) [13-14].

Sediments are crucial and inherent parts of aquatic systems. Various kinds of harmful and toxic pollutants were accumulated in sediments of aquatic systems [15-16]. These pollutants in the sediments can be released and possibly give rise secondary pollution of overlying water when electrical conductivity, pH, chemical oxygen demand and oxidation reduction potential of sediments altered [17-19]. Approximately, 85% of heavy metals in the aquatic systems are deposited in the sediments [19-21]. Therefore, sediments can act as the reservoirs and indicators for heavy metals.

At present, there have been some reports on heavy metal pollution in sediment in Yellow River basin. However, these studies selected small areas or special environmental areas in the Yellow River basin, such as the middle Yellow River reaches between Hekou Town in Inner Mongolia and Zhengzhou, Henan and Baotou section [22-23], estuary [24], a heavy industry area [25-26] and the wetland [27-28]. Their samples were taken from different parts of the Yellow River basin, such as main streams or tributary streams, floodplains or alluvial areas [25]. These previous research were limited in small and specific areas. This study area covers the whole of upper Yellow River and all samples were collected in riverbed of mainstream of the upper

Yellow River. In our study, the concentrations of 8 heavy metals were measured from 122 surface sediment samples collected along the upstream Yellow River. The objectives of the study were: (1) to assess the toxicity of the sediment; (2) to investigate the spatial distribution maps of heavy metals contamination; (3) to explore dominating sources of heavy metals in sediments of the upstream Yellow River. The results may provide valuable scientific basis for the government to make reasonable policies of managing the regional water environment.

Material and Methods

Study Area

The Yellow River is the largest water resources of northwest regions of China, providing agricultural irrigation water for 15% and domestic water for 12%. Approximately 40% of its runoff comes from the upstream Yellow River [29-30]. The upstream Yellow River starts from the Bayan Har Mountains of Qinghai province to Hekou Town (Togtoh County) of Inner Mongolia autonomous regions through four provinces for Qinghai, Gansu, Ningxia and Inner Mongolia and three metropolitan area for Xining, Lanzhou and Yinchuan. The length and basin area of the upstream Yellow River is 3,472 km and 386,000 km², accounting for 63.5% of the total length and 51.4% of the total area respectively. In decades, the amount of domestic and industrial sewage discharged into the upstream Yellow River shows an increasing trend and water quality is deteriorating gradually [31]. In addition, for the sake

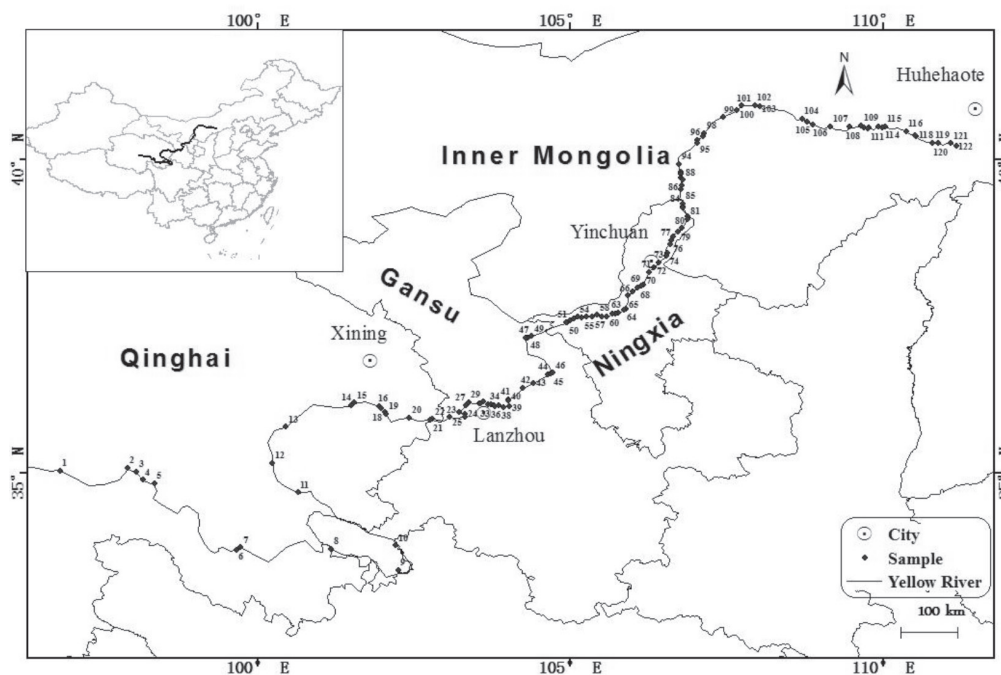


Fig. 1. Map of the study area and locality of sampling sites.

of hydroelectric generation, flood control and water supply a series of dams are built which have altered the quantity and quality of river sediment.

Sample Collection

122 surface sediment samples (0-5cm) were collected along the upstream Yellow River using a grab sampler (Fig. 1). Locations of all sampling sites were determined by a global positioning system and their surrounding environments were written down in the notebook. These samples were sealed in clean polyethylene bags and transported to the soil laboratory of Lanzhou Jiaotong University. These samples need to be air-dried, ground, digested and frozen specifically before determination.

Chemical Analyses

After air dried, all samples passed a 2 mm nylon sieve to remove the organic debris and a 0.063 mm mesh sieves through grinding. At last they were frozen for further analysis [32]. For the determination of heavy metal concentrations, 0.5g sediment sample was accurately weighed, completely digested by successively adding concentrated HCL-HNO₃-HClO₄-HF and heated at 180°C in a Teflon container. When the liquid containing sediment sample turned into a small golden droplet, it was diluted with 100 ml deionized water, filtered with a membrane and measured the concentrations of heavy metals with inductively coupled plasma mass spectrometer (ICP-MS).

In this study, glassware was washed used de-ionized water after soaking them in 15% HNO₃ for the further experimentation. The treatment of every sample was triplicate and the analytical datum were average values of the triplicates in this study.

Multivariate Statistical Analyses

Principle component analysis (PCA) can transform the original heavy metal variables into several principal components (PC_s) which reveal the similarity between heavy metals and confirm the potential pollution sources of heavy metals in sediments of aquatic systems [33-34]. Kaiser-Meyer-Olkin (KMO) and Bartlett's sphericity tests were conducted to verdict whether the data can be used for PCA.

Hierarchical cluster analysis (HCA), a useful data classification technique, was conducted to sort all heavy metals on the basis of their similarities [6, 35]. The similarity degrees about metals were measured by using Ward's method and squared Euclidean distances. The results of similarity can be shown in a visual dendrogram. Correlation analysis (CA) was also implemented to determine the relationship among metals and further verify the results obtained by multivariate analysis. Analyses were conducted using SPSS software 22.0 for Windows.

Estimation of Sediment Contamination

Numerous sediment quality guidelines (SQGs) are widely used to evaluate contaminant toxicity or risks to aquatic ecosystems by comparing sediment contaminant concentration with the corresponding quality guidelines [9,36]. Consensus-based SQGs which were determined by calculating the geometric mean of the published SQGs are likely to be directly relevant for assessing freshwater sediments that are influenced by multiple sources of contaminants [37]. These synthesized guidelines included a threshold effect concentration (TEC) and a probable effect concentration (PEC). TECs were interpreted to represent chemical concentrations below which adverse effects rarely occur. In contrast to TECs, PECs were intended to represent chemical concentrations above which adverse effects were likely to be frequently observed.

Mean PEC quotient (M-PEC-Q) was also useful ecological risk assessment to recognize and prioritize regions of potential hazards with respect to quality of sediments [36-38]. M-PEC-Q was calculated using the following equation [37-39]:

$$M - PEC - Q = \frac{\sum_{i=1}^n \frac{C_i}{PEC_i}}{n}$$

...where C_i was the concentration of metal in sediments, PEC_i was the corresponding guideline values for the element i and n was the number of metals. In the evaluation, sediment samples were predicted to be not toxic if M-PEC-Qs were less than 0.5. In contrast, sediment samples were predicted to be toxic when M-PEC-Qs exceeded 1.5.

Enrichment factor (EF) was widely used as an appropriate approach to determine the pollution degree in sediments of aquatic ecosystems. To identify anomalous metal concentrations, a normalization metal was employed. In this study, Mn element was used as reference material [20, 40]. EF was calculated as:

$$EF = \frac{(\frac{M}{Mn})_{\text{sample}}}{(\frac{M}{Mn})_{\text{background}}}$$

...where $(\frac{M}{Mn})_{\text{sample}}$ was the ratio of metal (M) and Mn concentrations of the sample, and $(\frac{M}{Mn})_{\text{background}}$ was the ratio of metal (M) and Mn concentrations of the background. The background concentrations of Fe, Cu, Ni, Zn, Cr, Pb, and Cd were 47200, 45, 68, 95, 90, 20, and 0.3 mg/kg, respectively [41]. The EF levels were interpreted as reported in Table 1.

Müller (1969) proposed the geoaccumulation index (I_{geo}) to evaluate the heavy metal contamination in sediments, which was applied using the following equation:

Table 1. Classification standard of enrichment factor (EF) and geoaccumulation index(I_{geo}).

EF classes	Enrichment level	I _{geo} value	I _{geo} Class	Contamination level
EF<1	No enrichment	I _{geo} ≤0	0	Uncontaminated
EF = 1-3	Minor enrichment	I _{geo} =0-1	1	Uncontaminated/moderately contaminated
EF = 3-5	Moderate enrichment	I _{geo} =1-2	2	Moderately contaminated
EF = 5-10	Moderately severe enrichment	I _{geo} =2-3	3	Moderately/strongly contaminated
EF = 10-25	Severe enrichment	I _{geo} =3-4	4	Strongly contaminated
EF = 25-50	Very severe enrichment	I _{geo} =4-5	5	Strongly /extremely contaminated
EF>50	Extremely severe enrichment	I _{geo} >5	6	Extremely contaminated

$$I_{geo} = \log_2\left(\frac{C_n}{1.5B_n}\right)$$

...where C_n was the measured concentration of trace metal in sediment, B_n was the geochemical background value of the corresponding metal [41]. The factor of 1.5 was used to minimize the effects of possible variations in the background values due to lithogenic effects. A seven-level classification of I_{geo} was defined in Table 1.

The potential ecological risk index (RI) introduced by Hakanson (1980) was used to assess the degree of heavy metal pollution in sediment according to the toxicity of heavy metal and the response of the environment [42]. RI was calculated using the following formulas:

$$RI = \sum_i^m E_r^i = \sum_i^m T_r^i C_f^i = \sum_i^m T_r^i C_n^i / C_r^i$$

...where RI was the sum of all risk factors for heavy metals in sediment, E_rⁱ was the monomial potential ecological risk factor of each heavy metal, T_rⁱ was the toxic-response factor for heavy metal i. According to Hakanson (1980), T_r for Cu, Ni, Zn, Cr, Pb, Cd are 5, 5, 1, 2, 5, and 30 respectively. C_fⁱ was the contamination factor of heavy metal i, C_nⁱ was the measured concentration of heavy metal i, C_rⁱ was the background of heavy metal i. Standards of ecological risk levels were shown in Table 2 [42-43].

Results and Discussion

Evaluation of Heavy Metal Pollution

To estimate the degree of metals contamination in the surface sediments of the upstream Yellow River, EF and the I_{geo} for each element were calculated. The median EF values for Fe, Mn, Cu, Ni and Pb were <1, indicating no enrichment of these metals in the sediment. The median EF values for Zn and Cr showed minor enrichment while Cd indicated moderately severe enrichment due to anthropogenic inputs (Fig. 2a). The median I_{geo} values except for Cd were less than 1, placing these metals into the uncontaminated, while the I_{geo} value of Cd was mostly higher than 1 in upstream Yellow River for moderately contaminated level (Fig. 2b). The median of I_{geo} values for these metals decreased in the order of Cd>Cr>Zn>Mn/Fe/Ni>Cu/Pb, which is similar with that observed from EF values. Overall, the mean EF was 1.47 and ranged from no enrichment to minor enrichment level, and the mean I_{geo} was -0.88 and belonged to uncontaminated level in the upstream Yellow River (Fig. 3).

To evaluate the toxicity and potential risk of heavy metals in sediments of the upstream Yellow River, SQGs and RI were applied. A comparison of the metals concentrations with the TEC and PEC values showed that 0% of the samples exceeded the PEC for all metals except Ni and Cr. The concentrations of Ni and Cr in 25.4% and 36.9% sediment samples were higher than their corresponding PEC, respectively, implying potential harm for aquatic organisms. Approximately

Table 2. Standards of potential ecological risk index of E_rⁱ and RI suggested by Hakanson (1980).

E _r ⁱ	Level	RI	Level
E _r ⁱ <40	Low risk	RI<95	Low risk
40≤E _r ⁱ <80	Moderate risk	95≤RI<190	Moderate risk
80≤E _r ⁱ <160	Considerable risk	190≤RI<380	Considerable risk
160≤E _r ⁱ <320	High risk	RI≥380	Very high risk
E _r ⁱ ≥320	Very high risk		

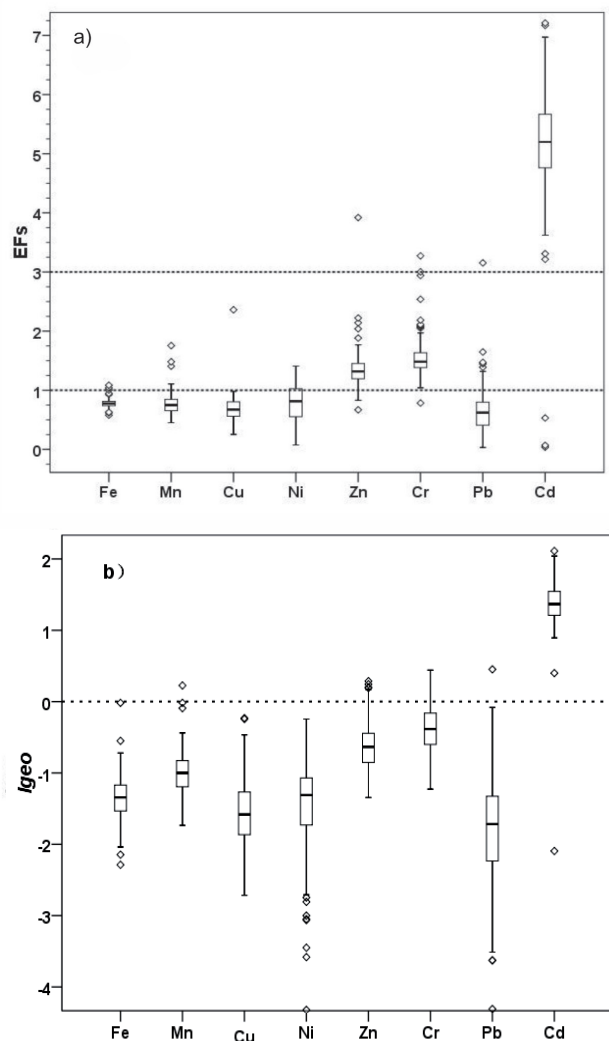


Fig. 2. Boxplots of a) enrichment factors (EF) and b) geoaccumulation indexes (I_{geo}) of heavy metals in the surface sediments of the upstream Yellow river.

84.4% for Cd was between the TEC and PEC, also suggesting potential adverse effects. In contrast, 89.3% for Cu, 87.7% for Zn and 99.2% for Pb were below

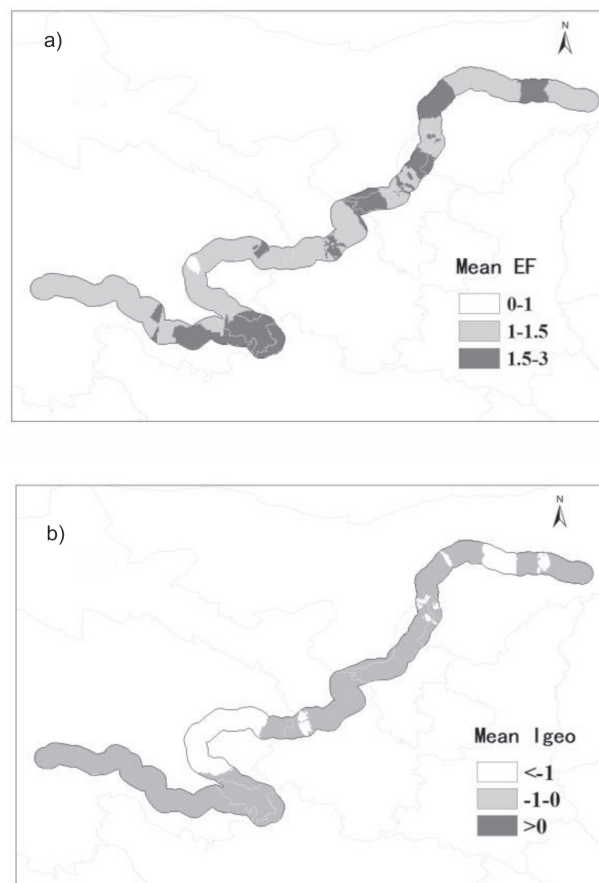


Fig. 3. The spatial distribution of a) enrichment factors (EF) and b) geoaccumulation indexes (I_{geo}) of heavy metals in the surface sediments of the upstream Yellow river.

their TEC, indicating a low probability of adverse effects (Table 3). To further evaluate their toxic risk, the PEC quotients (PEC-Q) were calculated according to the definition reported by MacDonald. (2000) [37]. The mean PEC-Q varied from 0.08 to 0.96 mg/kg (with a mean value of 0.41mg/kg), indicating integrally no toxic in the upstream Yellow River (Table 3, Fig. 4a). The maximal PEC-Qs for all metals except for Ni

Table 3. Percentages of sediments samples above PEC, between TEC and PEC, and below TEC, and summary description of PEC quotients for heavy metals in the upstream of Yellow River.

SQGs	Cu	Ni	Zn	Cr	Pb	Cd
TEC	31.6	22.7	121	43.40	35.8	0.99
PEC	149	48.6	459	111.00	128	4.98
>PEC (%)	0	25.4	0	36.9	0	0
Between TEC and PEC (%)	10.3	58.2	12.3	63.1	0.8	84.4
<TEC (%)	89.3	16.4	87.7	0	99.2	15.6
Mean PEC-Q	0.16	0.81	0.21	0.96	0.08	0.24
Minimal PEC-Q	0.07	0.11	0.12	0.52	0.004	0.002
Maximal PEC-Q	0.39	1.77	0.38	1.65	0.32	0.39

Table 5. Correlation coefficients between different heavy metal elements (n=122).

	Fe	Mn	Cu	Ni	Zn	Cr	Pb	Cd
Fe	1							
Mn	0.907**	1						
Cu	0.40**	0.493**	1					
Ni	0.045	0.066	0.266**	1				
Zn	0.497**	0.561**	0.180**	0.232*	1			
Cr	0.521**	0.559**	0.253**	0.114	0.523**	1		
Pb	0.113	0.128	0.174	0.047	0.201*	0.217*	1	
Cd	0.529**	0.493**	0.465**	0.330**	0.360**	0.515**	0.136	1

Levels of significance: *P<0.05; **P<0.01.

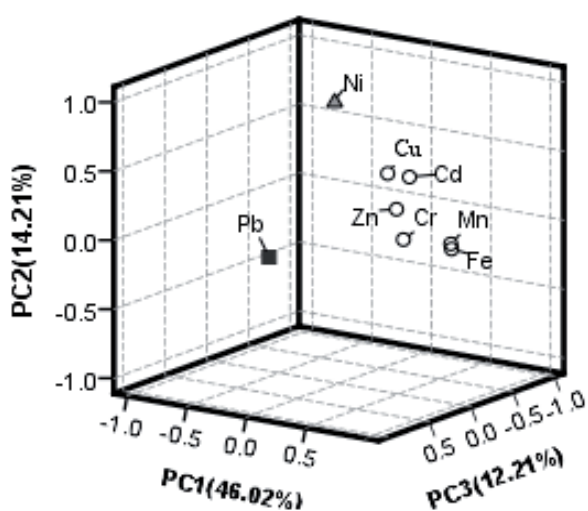


Fig. 5. Principal component loading plot for metal variables.

for Cu, suggesting these metals might come from the erosion of the parent rock and its weathering crusts and they might be mainly control sources of pollution. PC2 (18.09% of the total variance) has strong positive loading on Ni, moderate positive loading for Cu and Cd, weak positive loading for Mn and negative positive loading for Fe. The results showed Ni, Cu and Cd likely originate from similar anthropogenic pollution sources. However, Cu and Cd also show moderate to strong positive loading on PC1, suggesting that the sources of Cu and Cd could be both natural and anthropogenic. PC3 (13.4% of the total variance) demonstrates strong positive loading for Pb and negative positive loading for Fe, Mn and Ni, suggesting Pb has another different anthropogenic source.

To confirm the association obtained from CA and PCA, HCA was preformed to find out the relationship between heavy metals. The dendrogram with single linkage Euclidean distance was showed in Fig. 6. The

Table 6. Total variance explained and rotated component matrix of principal components analysis.

	PC1	PC2	PC3
Fe	0.92	-0.004	-0.04
Mn	0.94	0.04	-0.01
Cu	0.51	0.50	0.12
Ni	-0.04	0.92	-0.002
Zn	0.65	0.30	0.23
Cr	0.71	0.09	0.24
Pb	0.09	0.03	0.97
Cd	0.61	0.49	0.03
Eigenvalue	3.28	1.45	1.07
% Total variance	40.95	18.09	13.40
% Cumulative variance	40.95	59.04	72.44

Extraction method: principal component analysis. Rotation method: Varimax with Kaiser normalization.

Rescaled Distance Cluster Combine

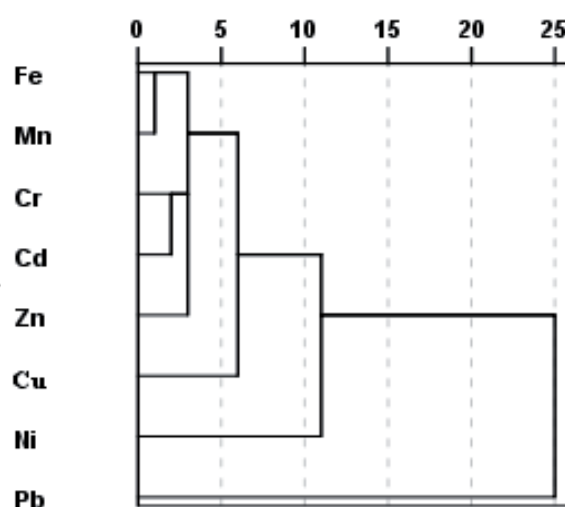


Fig. 6. Dendrogram of cluster analysis for parameters in the sediments of study area.

distance axis represented the degrees of association between groups of variables, the lower the value on the axis, the more significant the association. In this dendrogram, all 8 parameters were grouped into three statistically meaningful clusters. Cluster 1 contained Fe, Mn, Cu, Zn, Cr and Cd which could mostly be originated from natural sources. Cluster 2 and Cluster 3 contained Ni and Pb, respectively. Ni and Pb derived from different anthropogenic sources. Overall, the results of CA, PCA and HCA were consistent.

Conclusions

Pollution evaluation and sources identification of heavy metals (Fe, Mn, Cu, Ni, Zn, Cr, Pb and Cd) were analyzed in surface sediments from upstream of Yellow River. Based on the background values presented by Turekian in 1961, all of the heavy metals had different accumulated levels for the order: Cd>Cr>Zn>Mn/Fe/Ni>Cu/Pb, which was similar to that by Reference [30]. Among these metals, Cd enrichment was significant, while the upstream Yellow River was basically uncontaminated, which was attested by the values of EF and I_{geo} . According to TEC-PEC SQGs and RI, only Ni and Cr in part of the sites had potentially harmful effects and the other metals had no toxic in the upstream Yellow River. In light of multivariate analyses (CA, PCA and HCA), Fe, Mn, Zn Cr, Cu and Cd were mostly originated from natural sources, while Ni and Pb mainly derived from different anthropogenic sources and a small number of Cu and Cd come from anthropogenic sources of Ni discharge. Therefore, it is necessary for the government to control quality of sewage discharge containing Ni.

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Conflict of Interest

The authors declare no conflict of interest.

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