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Assessment of heavy metals contamination in soil profiles of roadside *Suaeda salsa* wetlands in a Chinese delta

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ABSTRACT

Five sampling sites (Sites A, B, C, D and E) were selected along a 250 m sampling zone covered by *Suaeda salsa*, which is perpendicular to a road, in the Yellow River Delta of China. Soil samples were collected to a depth of 40cm in these five sampling sites to investigate the profile distributions and toxic risks of heavy metals. Concentrations of heavy metals (As, Cd, Cr, Cu, Ni, Pb and Zn) were determined using inductively coupled plasma atomic absorption spectrometry (ICP-AAS). The results showed that in each sampling site, Cd, Cu, Pb and Zn have approximately constant concentrations along soil profiles and did not show high contamination compared with the values of probable effect levels (PELs). All soils exhibited As and Ni contamination at all sampling sites compared with other heavy metals. The index of geo-accumulation (I_{geo}) values for As in the 20–30 cm soil layer at Site B was grouped into Class IV($2 < I_{geo} \leq 3$), indicating that the soil was moderately to strongly contaminated. Forty percent of I_{geo} values of Cd for all soil samples were grouped into Class IV($2 < I_{geo} \leq 3$) and 75% samples of Site C showed moderately to strongly contaminated level. The Enrichment factor (EF) values of As at Sites B, C, D and E reached significant enrichment level and EF values of Cd at five sampling sites all reached significant enrichment level. The sum of toxic units ($\sum$TUs) values for surface soils of Sites B and C beyond 4 indicated that Sites B and C have severer toxicity compared with other three sampling sites. As and Ni should be paid more attention to avoid potential ecotoxicity due to their high contribution ratios to the $\sum$TUs in *Suaeda salsa* wetlands. Correlation analysis (CA) and principal components analysis (PCA) revealed that Cr, Cu, Ni, Pb and Zn might derive from the common sources, Cd might originate from another, while As might have more complex sources in this study area.

1. Introduction

Located in zones between land and ocean, coastal wetlands are a multifunctional and complex ecosystem with special ecological values and potential resources. However, the rapid development of industry and agriculture around coastal area make the ecosystems more easily destructed. As a result of the physico-chemical processes such as adsorption, ligand exchange and sedimentation, wetland soils act as a source, sink, and transfer for heavy metals (Lau and Chu, 2000; Reddy and DeLaune, 2008). Heavy metals pollution is one of the most typical contaminations caused by anthropogenic activities, because heavy metals can be easily bioaccumulated through food chains and cause heath risks and ecotoxicity even at low levels in the environment (Pejman et al., 2015).

The Yellow River Delta (YRD) is one of the most active regions of land-ocean interactions (Lu et al., 2014). It is estimated that approximately 1.05×10^7 t of sand and soil per year are carried and deposited in the YRD (Xu et al., 2002). The variation in hydrological conditions was observed in the YRD due to the implementation of flow-sediment regulation (Bai et al., 2012). The active hydrological fluctuations in the YRD make the physico-chemical processes of coastal region more complex (Xu et al., 2002; Lu et al., 2014; Zhou et al., 2015). Additionally, the YRD is under great pressure of environmental degradation caused by aquaculture, petrol oil industries, agriculture, road and harbor construction (Bai et al., 2011; 2012). There was an increasing tendency of heavy metals contamination in the YRD during the last decade caused by sustained and intensive human activities (Nie et al., 2010; Bai et al., 2012). Some

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studies on heavy metals contamination have been performed in this region, but little information is available about the profile distributions of heavy metals in *Suaeda salsa* wetland soils affected by road transportation in the YRD.

Therefore, the primary objectives of this study were: (1) to investigate the profiles distribution of heavy metals (e.g., Cd, Cr, Cu, Ni, Pb and Zn) in *Suaeda salsa* wetland soils perpendicular to a road in the YRD, China; (2) to evaluate the contamination levels based on the index of geo-accumulation (I_{geo}) and the enrichment factor (EF) as well as toxic risks using toxic units (TUs); and (3) to identify the possible sources of heavy metals using correlation analysis (CA) and principal components analysis (PCA).

2. Materials and methods

2.1. Study area

This study was conducted in the Yellow River Delta (37°10′ to 38°40′N and 118°30′ to120°10′E), Shandong province, China. It has a warm-temperate and continental monsoon climate, with annual mean precipitation of 640 mm and annual mean evaporation of 1962 mm. The annual mean air temperature is 11.9 °C, with 196 frostless days (Bai et al., 2015). Intrazonal tidal soil and salt soil are the dominant soil types in the study area (Zhang et al., 2013). The dominant vegetation comprises *Phragmites australis*, *Suaeda salsa*, *Tamarix chinensis*, *Imperata cylindrica* and *Salix metsudana* (Zhang et al., 2016).

2.2. Sample collection and analysis

Five sampling sites (Sites A, B, C, D and E) were selected along a 250 m sampling zone covered by *Suaeda salsa*, which was perpendicular to a road (The distances from Sites A, B, C, D and E to the road are 250 m, 200 m, 150 m, 100 m and 50 m, respectively) in the YRD. The top 40 cm soils (sectioned into 0–10 cm, 10–20 cm, 20–30 cm and 30–40 cm) were collected with three replicates, and three replicates of each soil layer were mixed uniformly into one composite sample in the same soil profile and then placed in polyethylene bags and brought to the laboratory. All soil samples were air-dried at room temperature for 3 weeks and sieved through a 2-mm nylon sieve to remove coarse debris and then ground using a pestle and mortar until all particles could pass through a 0.149-mmnylon sieve for the determination of soil chemical properties.

The soil samples were digested with an $HClO_4$-HNO_3-HF mixture in Teflon tubes to determine the concentations of total sulfur (TS), total phosphorous (TP), Al, As, Cd, Cr, Cu, Ni, Pb and Zn. The digested sample solutions were analyzed by inductively coupled plasma atomic emission spectrometry (ICP-AES). Quality assurance and quality control were assessed with duplicates, method blanks and standard reference materials (GBW07401) from the Chinese Academy of Measurement Sciences with each batch of samples (1 blank and 1 standard for each 10 samples). The recoveries of samples spiked with standards ranged from 95% to 105%. Soil organic matter (SOM) was measured using dichromate oxidation (Page et al., 1982). Soil pH was measured in 1:5 soil/water (m/v) suspensions with a Hach pH meter (Hach company, Loveland, CO, USA).

2.3. Contamination and ecotoxic risk assessment

In this study, the index of geoaccumulation (I_{geo}) (Müller, 1979) was used to measure the contamination levels of heavy metals in wetland soils:

$$I_{geo} = \log_2 \frac{C_n}{1.5\,B_n}$$

where C_n is the measured concentration of the element and B_n is the geochemical background concentration of this element. The geoaccumulation values were classified as uncontaminated ($I_{geo} \leq 0$), uncontaminated to moderately contaminated ($0 < I_{geo} \leq 1$), moderately contaminated ($1 < I_{geo} \leq 2$), moderately to strongly contaminated ($2 < I_{geo} \leq 3$), strongly contaminated ($3 < I_{geo} \leq 4$), strongly to extremely contaminated ($4 < I_{geo} \leq 5$), and extremely contaminated ($I_{geo} > 5$). In this study, the reference background values were obtained from the environmental background concentrations of heavy metals in the Yellow River Delta (CNEMC, 1990, Table 1).

Enrichment factor (EF) was used to evaluate the probable exogenous input to selected sample metals. EF was defined as $EF = (M/Al)_{sample}/(M/Al)_{background}$, where M and $M_{background}$ are the determined values of target elements in the sampled soils and the background levels of them, respectively. Al is the normalizing element due to its conservation here (Xiao et al., 2012; Bai et al., 2014). The EF were classified into five classes as follows: (1) EF < 2, minimal enrichment; (2) EF = 2–5, moderate enrichment; (3) EF = 5–20, significant enrichment; (4) EF = 20–40, very high enrichment; and (5) EF > 40, extremely high enrichment (Brady, 1984). Background values of heavy metals were from the China National Environmental Monitoring Center (CNEMC, 1990, Table 1).

To assess the ecotoxicity of heavy metals, the toxic unit (TU) was used to compare the relative impact of various metals in different sites, which is defined as the ratio of the measured concentration to probable effect level (PEL) (Pedersen et al., 1998). The PEL represents the concentration above which adverse effects are expected to occur, and the PEL values for estuarine ecosystem was used in this study (MacDonald et al., 2000, Table 1).

2.4. Statistical analysis

Pearson correlation analysis and principle component analysis were carried out using SPSS 17.0 software package to identify the relationships among different metals. The relationships between heavy metals and selected soil properties were also examined by correlation analysis. Differences were considered to be significantly if $P < 0.05$.

3. Results and discussion

3.1. Heavy metals distribution in soil profiles

The vertical distributions of heavy metals along soil profiles at

Table 1
Reference concentrations of heavy metals.

	Al (mg/kg)	As (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Zn (mg/kg)
TEL[a]	–	7.2	0.68	52.3	18.7	15.9	30.2	124.0
PEL[a]	–	42.6	4.2	160.4	108.2	42.8	112.2	271.0
Background value[b]	62700	10.7	0.095	59	21.1	27.6	21.6	64.5

[a] TEL: threshold effect level; PEL: probable effect level; (Long and Mac Donald, 1998).

[b] Background values in the Yellow River Delta (CNEMC, 1990).

five sampling sites are shown in Fig. 1. At five sampling sites, Cd, Cu, Pb and Zn approximately kept constant concentrations at four soil layers and did not show severe contamination compared with the PEL values. The concentrations of Cr along soil profiles at Sites A and C showed a fluctuation change while the peak values were much lower than the PEL value for Cr. However, As and Ni contamination were observed in the study area and it could be observed that As showed severer contamination compared with Ni. The As concentrations at Sites B, C and D showed an irregular variation with peak values beyond PEL value at Sites B and D while the lowest concentration was observed at Site C at the 20–30 cm soil layer. The possible explaination was that the road transportation might be the improtant pollution sources of As and could greatly affect the roadside wetland soils (Bai et al., 2010). And the highest As levels were observed at the 30–40 cm soil layers of Sites A and E. The Ni concentrations generally kept constant along the soil profiles at Site D. The surface soils exhibited Ni contaminations at Sites B, C and D due to the relatively high concentations in the top 10 cm soils of those sites.

3.2. Contamination assessment of heavy metals

3.2.1. Contamination assessment of heavy metals using I_{geo}

As shown in Fig. 2, with the exception of 20–30 cm soil layer at Site B, the I_{geo} values for As in other soil samples were grouped into Class III ($1 < I_{geo} \leq 2$). The I_{geo} value of the 20–30 cm soil layer at Site B was grouped into Class IV($2 < I_{geo} \leq 3$), indicating that the soil was moderately to strongly contaminated. No contamination for other heavy metals was observed at five sampling sites except for As and Cd. This was in agreement with the results by Bai et al. (2012, 2015). As showed high contamination level in deeper soil layers (10–40 cm) of five sampling sites, which is consistent with the profile distribution of As (Fig. 1). Forty percent of the I_{geo} values of Cd for all samples were grouped into Class IV($2 < I_{geo} \leq 3$). The contamination status seemed severer at Site C for 75% of soil samples showing moderate to strong contamination levels. Additionally, the surface soils of Site C showed higher I_{geo} values than other sampling sites. This might be caused by the traffic emission from the adjacent road. Site C at the distance of 150 m away from the road could be the reference value of the critical influencing distance with higher ecological risks caused by road transportation in the study area.

3.2.2. Contamination assessment of heavy metals using EF

Fig. 3 showed the proportions of soil samples with different enrichment levels in all samples from Sites A, B, C, D and E. Except for Site A where As showed moderate enrichment level, the EF values of As at Sites B, C, D and E reached significant enrichment levels with nearly 50% of soil samples at Site B and approximately 25% of samples at Sites C, D and E, respectively. Nearly 25% of samples showed moderate enrichment level of Zn at Sites A, C and E. Pb showed moderate enrichment level at Site E. The EF values for Cd at five sampling sites reached the significant enrichment level. This is consistent with the assessment result according to I_{geo} values that Cd contributed mostly to the heavy metals contamination in the study area. Additionally, As and Cd might be the primary toxic metals in the study area due to high EF values for the two anthropogenic elements. Similar results were also presented by Bai et al. (2015).

3.2.3. Ecotoxic assessment of heavy metals using TU

Toxic units (TUs) are defined as the ratios of the determined concentrations to the values of PEL, which were used to normalize the toxicities caused by various heavy metals and hence allowed the camparison of their relative effects (Pedersen et al., 1998). The sum of toxic units ($\sum$TUs) was used to estimate the potential acute toxicity of heavy metals in the study area. Fig. 4 illustrated the TU, $\sum$TUs and the contribution ratio of each heavy metal at each soil layer of five sampling sites. The $\sum$TUs values for all soil samples ranged from 2.19 to 4.67, and 10% of the samples showed the $\sum$TUs values exceeding 4, which implied that there were moderate

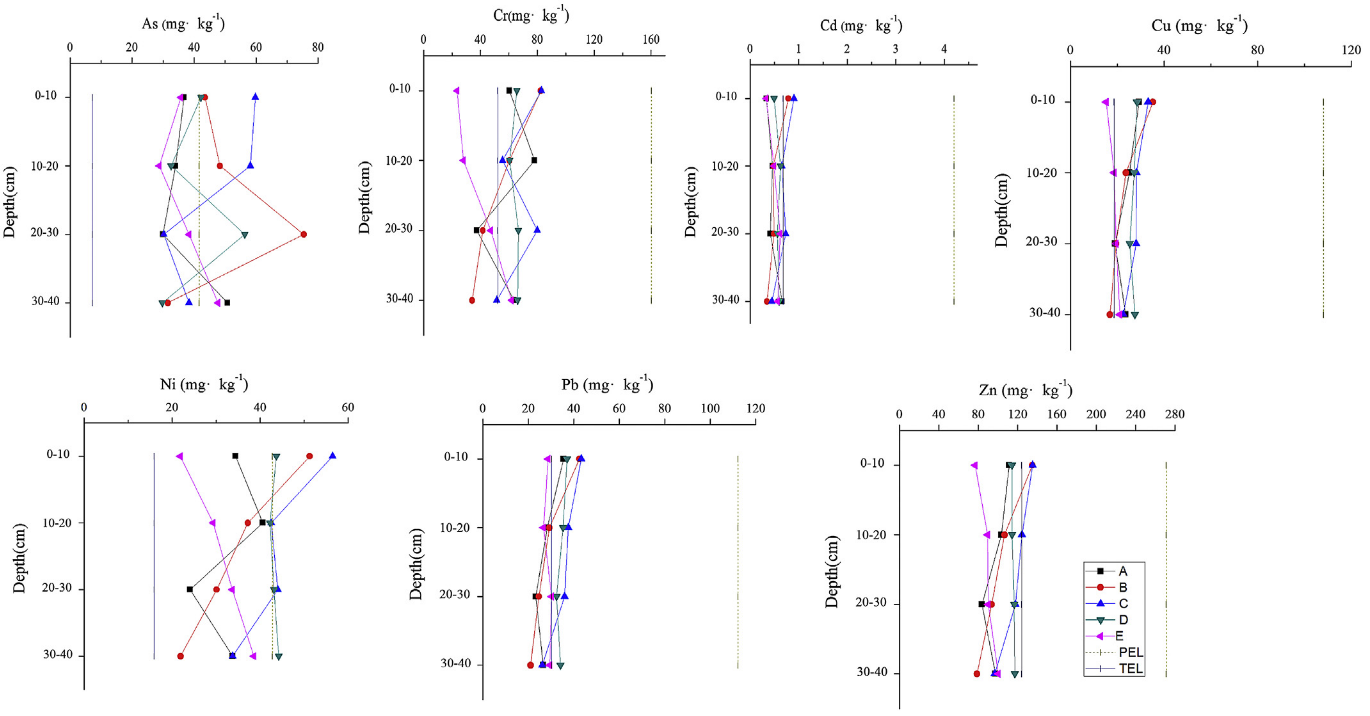

Fig. 1. Vertical distribution of heavy metals in soil profiles at five sampling sites (Sites A, B, C, D and E).

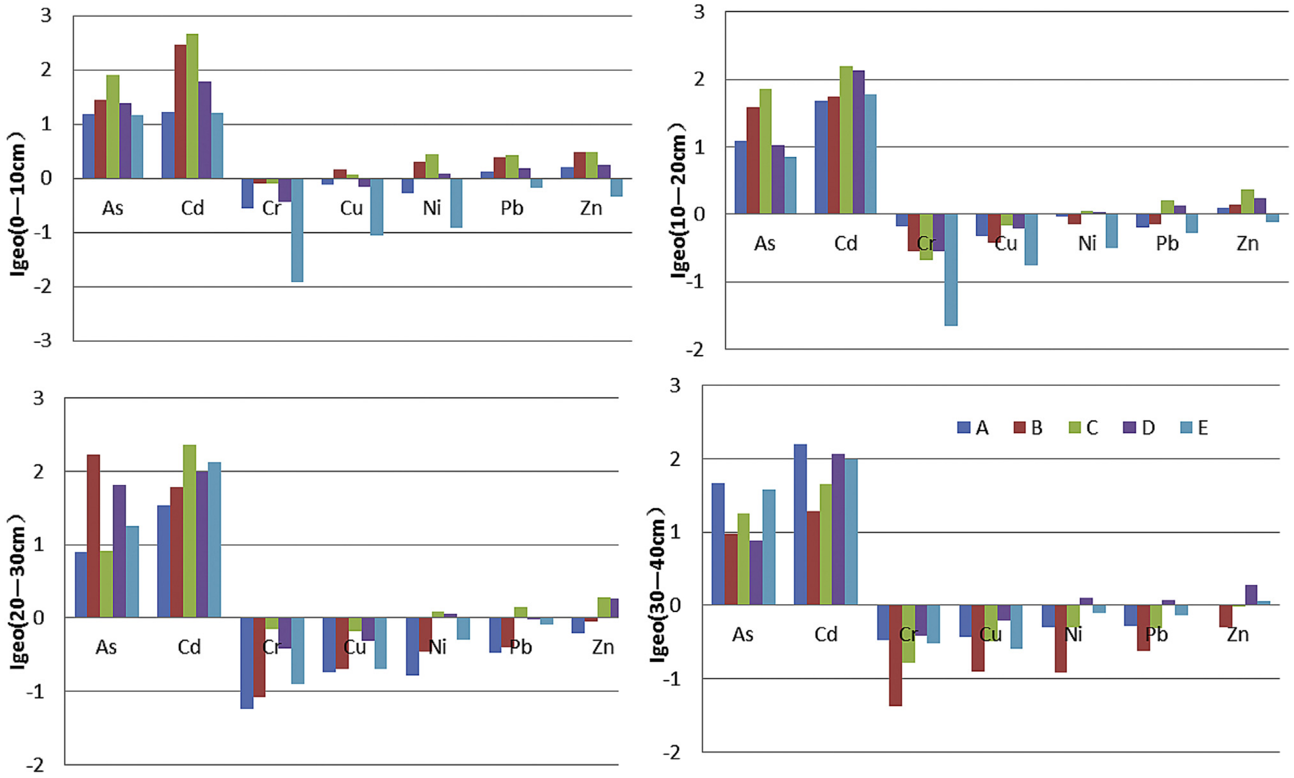

Fig. 2. Geoaccumulation indices (I_{geo}) of As, Cd, Cr, Cu, Ni, Pb, and Zn at five sampling sites with four soil layers.

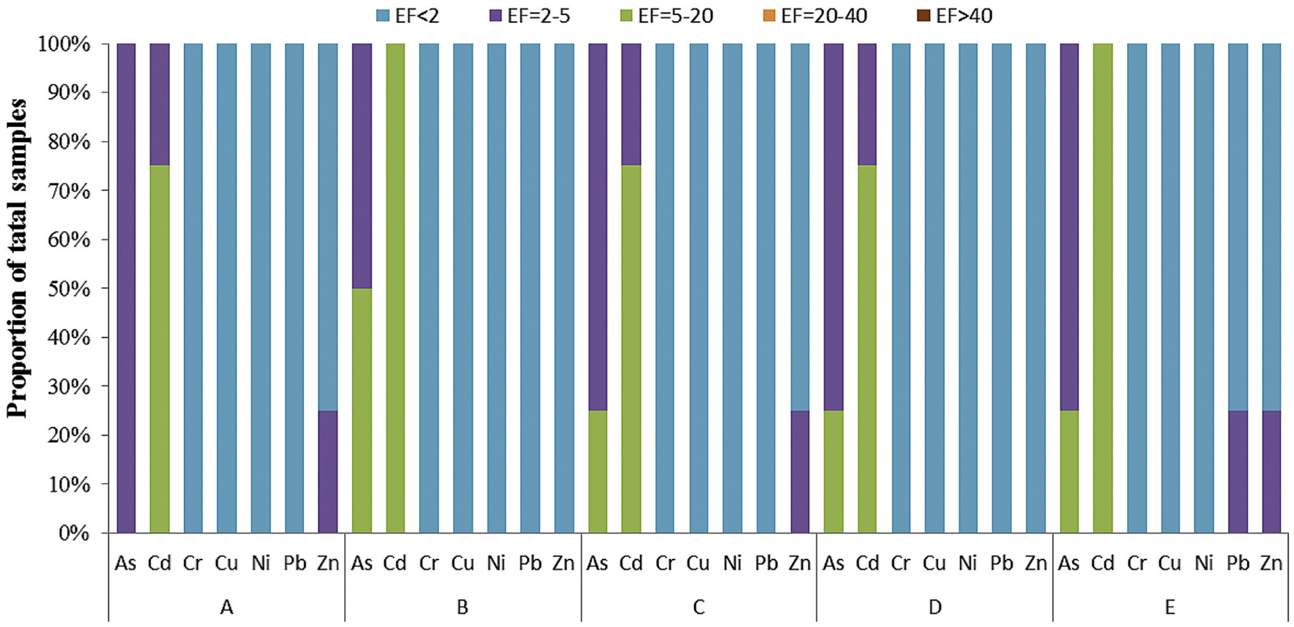

Fig. 3. Proportions of contamination levels of heavy metals in all soil samples of each sampling site.

toxicity to wetland ecosystem in this study area (Pedersen et al., 1998). The ∑TUs values for surface soils of Sites B and C beyond 4 indicated that the surface soils of both sites (200 m and 150 m away from the road, respectively) had severer toxicity compared with another three sampling sites. The mean values for TU of soil samples decreased in the following order: As (1.25 ± 0.56) >Ni (0.92 ± 0.40) >Zn (0.39 ± 0.11) >Cr (0.33 ± 0.18) >Pb (0.29 ± 0.10) >Cu (0.23 ± 0.09) >Cd (0.15 ± 0.07). As and Ni contributed more than other heavy metals to the potential toxicity of soils in the *Suaeda salsa* wetlands and the ratios of As and Ni to ∑TUs in all samples were 35.44 ± 14.37% and 25.36 ± 6.02%, respectively, while the results of the assessment using I_{geo} and EF indicated the highest contamination level and significant enrichment of Cd (Figs. 2 and 3). This might be caused by the significantly higher reference values of PEL than those of background values in the YRD for Cd (Table 1) (Xiao et al., 2012; Lu et al., 2014).

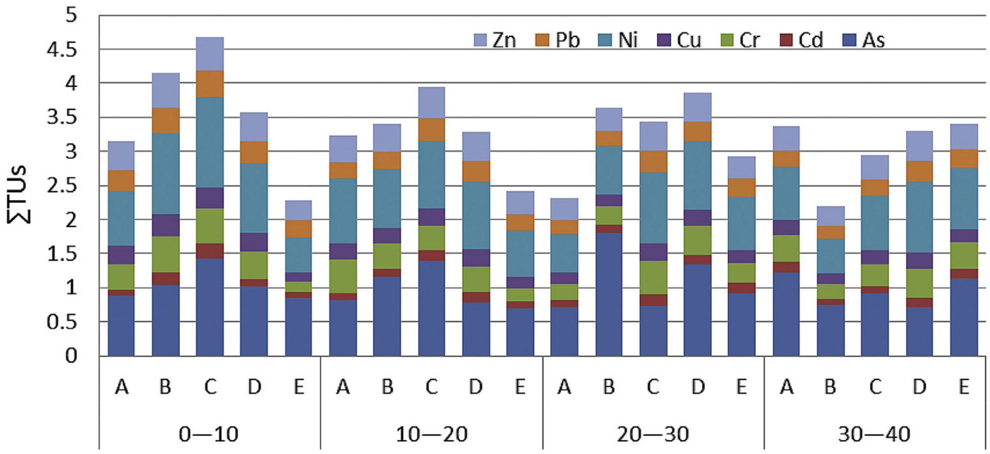

Fig. 4. The toxic unit (TU) of each heavy metal and the sum of toxic units (∑TUs) of seven heavy metals in soil profiles of five sampling sites.

Table 2
Results of principle component analysis of heavy metals (two principle components were selected).

Elements	Component matrixes	
	PC1	PC2
As	0.438	−0.417
Cd	0.203	0.846
Cr	0.845	0.294
Cu	0.773	0.203
Ni	0.930	−0.039
Pb	0.927	−0.174
Zn	0.912	−0.216
Initial Eigenvalues	4.100	1.095
Variance%	58.579	15.649
Cumulative%	58.579	74.228

3.3. *Identification of possible sources*

Principle component analysis (PCA) and Pearson correlation analysis (CA) have been applied widely to identify the sources of heavy metals in many studies (Mico et al., 2006; Karim et al., 2014; Lu et al., 2014). The results of PCA (Table 2) showed that two principle components (PC1, PC2) were extracted with eigenvalues > 1, accounting for 74.22% of the total variance. The PC1 explaining 58.57% of the total variance showed positive relation to Cr, Cu, Ni, Pb and Zn. Meanwhile, the significant correlations between them were also observed in Table 3. The PC2,which was only highly related to Cd, explained 15.64% of the total variance. The result of PCA indicated that Cr, Cu, Ni, Pb and Zn might derive from a common source and Cd might originate from another source. And As might have more complex sources due to weak loading factors in both PCs. Soil organic matter often acts as carriers of heavy metals due to its strong capacity for complexing metallic contaminants (Bai et al., 2012), but SOM was not significantly correlated with heavy metals in our study (Table 3). This might be associated with the lower SOM levels in the YRD.

4. Conclusions

The present study illustrated that As and Cd exhibited higher contamination levels compared with other heavy metals. The surface soils showed severe contamination which might be related to the road transportation. Generally, As, Cd and Ni should be paid more attention to avoid potential ecotoxicity in the study area. Higher As contamination levels in surface soils at the distance of 150 m away from the road, showing the critical influcing distance with higher ecological risks of As caused by road transportation in the study area. Therefore, proactive measures should be taken to prevent heavy metals contamination caused by the intensive anthropogenic activities. Moreover, the fractions of heavy metals and their toxic risks need to be further studied to better understand the contamination dynamics and ecotoxicity of heavy metals in roadside wetland soils in coastal region.

Table 3
A correlation matrix between heavy metals and selected soil properties in the study area.

	As	Cd	Cr	Cu	Ni	Pb	Zn	TS	TP	PH	SOM
As	1										
Cd	0.051	1									
Cr	0.235	0.316^{a}	1								
Cu	0.088	0.131	0.726^{b}	1							
Ni	0.395^{b}	0.212	0.710^{b}	0.578^{b}	1						
Pb	0.331^{a}	−0.004	0.656^{b}	0.747^{b}	0.869^{b}	1					
Zn	0.446^{b}	0.049	0.682^{b}	0.534^{b}	0.895^{b}	0.865^{b}	1				
TS	0.128	−0.186	0.461^{b}	0.734^{b}	0.394^{b}	0.700^{b}	0.528^{b}	1			
TP	0.347^{a}	−0.027	0.627^{b}	0.505^{b}	0.607^{b}	0.698^{b}	0.766^{b}	0.681^{b}	1		
PH	0.223	−0.13	−0.182	-0.654^{b}	−0.07	−0.219	0.156	-0.376^{a}	0.115	1	
SOM	-0.423^{b}	0.04	−0.13	0.117	−0.152	−0.135	-0.323^{a}	−0.082	−0.211	-0.526^{b}	1

[a] Represents significant correlation at the level of $p < 0.05$.
[b] Represents significant correlation at the level of $p < 0.01$.

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