Seasonal Dynamics of Trace Elements in Tidal Salt Marsh Soils as Affected by the Flow-Sediment Regulation Regime

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Abstract

Soil profiles were collected in three salt marshes with different plant species (i.e. Phragmites australis, Tamarix chinensis and Suaeda salsa) in the Yellow River Delta (YRD) of China during three seasons (summer and fall of 2007 and the following spring of 2008) after the flow-sediment regulation regime. Total elemental contents of As, Cd, Cu, Pb and Zn were determined using inductively coupled plasma atomic absorption spectrometry to investigate temporal variations in trace elements in soil profiles of the three salt marshes, assess the enrichment levels and ecological risks of these trace elements in three sampling seasons and identify their influencing factors. Trace elements did not change significantly along soil profiles at each site in each sampling season. The highest value for each sampling site was observed in summer and the lowest one in fall. Soils in both P. australis and S. salsa wetlands tended to have higher trace element levels than those in T. chinensis wetland. Compared to other elements, both Cd and As had higher enrichment factors exceeding moderate enrichment levels. However, the toxic unit (TU) values of these trace elements did not exceed probable effect levels. Correlation analysis showed that these trace elements were closely linked to soil properties such as moisture, sulfur, salinity, soil organic matter, soil texture and pH values. Principal component analysis showed that the sampling season affected by the flow-sediment regulation regime was the dominant factor influencing the distribution patterns of these trace elements in soils, and plant community type was another important factor. The findings of this study could contribute to wetland conservation and management in coastal regions affected by the hydrological engineering.

Introduction

Sediment contamination in rivers, lakes, reservoirs, and wetlands has been widely reported in the developing countries as a result of the intense land use in agricultural and urban environment. Contamination of sediments with trace elements is a major concern [1,2]. Moreover, these trace elements can be transferred and carried downstream into wetland ecosystems, and accumulate in wetland soils [3,4]. Wetland soils serve as “source” and “sink” of these chemical pollutants [5]. For example, salt marshes and estuarine sediments can retain these metals as metal sulfides [5,6]. However, seasonal hydrological changes and water level fluctuation can affect Eh and pH of wetlands, thus resulting in mobilizing trace elements [5,7].

Several studies on large river delta have focused on source identification of trace elements and organic pollutants in surface sediments [6,8,9,10]. Some studies have shown that heavy metals can be accumulated and retained in wetland soils for a long time period [11], and they would not vary significantly over seasons in salt marshes under natural conditions [12]. The global regulation of rivers and steams by building reservoirs and dams has brought great effects on downstream ecosystems [13]. Recently, Bai et al. [4] have reported that the flow-sediment regulation contributed to trace elements (i.e. Arsenic, Cadmium, and others) accumulation in surface wetland soils of the Yellow River Delta as bed sands can carry and settle these trace elements to the downstream due to strong hydraulic flushing [12,14]. However, less information is available on the dynamics and fate of these trace elements in estuarine wetlands after the flow-sediment regulation.

The Yellow River Delta (YRD) is one of the most active regions of land-ocean interaction, and a national nature reserve was established to better protect this newly-formed wetland ecosystem and maintain biological diversity [15]. However, with the rapid development of agriculture, fisheries, and the extensive exploitation of the Shengli oilfield (It is the second largest oilfield in China and is mainly located in both sides of the Yellow River Estuary, which was originally built in 1962 and the working area has covered approximately 4.4×104 km²), the Yellow River Delta was greatly impacted by the intense human activities, leading to serious wetland degradation [16]. Moreover, the flow-sediment regulation regime has shown a significant influence on trace element accumulation [4] and wetland plant distribution [16] in the Yellow River Delta.
The primary objectives of this study were: (1) to determine the dynamic changes of selected trace elements including As, Cd, Cu, Pb, and Zn in wetland soils covered by different dominant plant species (i.e., Phragmites australis, Saeda salsa and Tamarix chinensis) of the Yellow River Delta; (2) to assess enrichment levels of these trace elements and determine their ecological risks using enrichment and toxic indicators; and (3) to identify the relationships among trace elements and other selected soil properties.

Materials and Methods

Ethics statement

Our study area is located in the Yellow River Delta wetland nature reserve, which is owned by the Chinese government. We obtained a specific permit from the administration bureau of the Yellow River Delta National Nature Reserve to conduct this study in the reserve. Moreover, our sampling sites were not located in any strictly protected areas and the field studies did not involve endangered or protected species.

Study area

The study area is located in the Yellow River Delta of China (37°37’48” to 37°48’36”N and 119°01’ to 119°21’E) (Figure 1). It has a temperate, continental monsoon climate with distinct seasons. The average annual air temperature is 12°C, with 196 frost-free days, and the annual mean precipitation is 552 mm [17]. Soils are characterized as loamy fine sand or coarse sand in all soil layers with clay content of 5-7% and can be classified as aquents, aquolls and fluvaquents [18]. The dominant vegetation in tidal salt marshes includes herbaceous plants such as Phragmites australis and Saeda salsa and woody plant namely Tamarix chinensis [17]. The vegetation succession in this delta follows the order Saeda salsa → Tamarix chinensis → Phragmites australis, and plant distributions are dominantly controlled by soil salinity. The study area was consistently influenced by the flow-sediment regulation of the upstream Xiaolangdi Reservoir from June and July since 2002 [4]. Xiaolangdi Reservoir began storing water in 1999, and considerable siltation occurred in the reservoir after commissioning, with a total sediment trapping of 32.47 × 10⁸ t from 1997 to 2007 [19]. The flow-sediment regulation of the upstream Xiaolangdi Reservoir has been enforced from June to July since 2002 [17]. For this study, three sampling sites with different dominant plant species (Table 1) were selected. The three sites are approximately 100 m away from each other and they were consistently inundated by tidal seawater (twice every day).

Sample collection and analysis

Soil samples were collected using a soil auger (4.8-cm diam.) from three sampling sites in summer (August, 2007), fall (November, 2007), and spring (April, 2008). Well-developed root systems were observed to depths of 50 cm at P. australis wetland and T. chinensis wetland, whereas the root zone was confined to depths of 15-20 cm at S. salsa wetland. Therefore, in each sampling season, the top 50 cm soils (sectioned into 0-10, 10-20, 20-30, and 30-50 cm) were collected with three or five replicates in P. australis wetland and T. chinensis wetland, and the top 20 cm soils (sectioned into 0-10 and 10-20 cm) were sampled with four replicates in S. salsa wetland. A total 120 soil samples were collected, including 36 samples in P. australis wetland, 60 samples in T. chinensis wetland, and 24 samples in S. salsa wetland. All soil samples were placed in polyethylene bags and brought to the laboratory, where they were air dried at room temperature for three weeks. Some air-dried soils of selected two profiles at each site in each sampling season were used for soil particle size analysis. All the other air-dried soil samples were sieved through a 2-mm nylon sieve to remove coarse debris and then ground with a pestle and mortar until all particles passed a 0.149-mm nylon sieve for the determination of soil chemical properties. Soil bulk density cores were also correspondingly collected using a 104 cm³ cylinder from each soil layer of each soil profile, oven dried at 105°C for 24 h, and weighed for the determination of bulk density (BD) and soil moisture.

Plant samples for the aboveground parts (including stems and leaves) were also collected at each soil sampling site in summer. They were placed in paper bags after clean washing and transported to the laboratory. All plant samples were oven dried at 65°C for 48 h, and ground into fine powder for the determination of trace metals. Soil and plant samples were digested with an HClO₄-HNO₃-HF mixture in Teflon tubes to analyze total contents of Al, As, Cd, Cu, Pb, and Zn. The solutions of the digested samples were analyzed by inductively coupled plasma atomic absorption spectrometry (ICP-AAS). Quality assurance and quality control were assessed using duplicates, method blanks and standard reference soil materials (GBW07401) and standard reference plant materials (GBW07602) from the Chinese Academy of Measurement Sciences with each batch of samples (1 blank and 1 standard for each 10 samples). The recovery rates of samples spiked with standards ranged from 99 to 106% for soils, and from 92–119% for plants. Soil organic matter (SOM) was measured using dichromate oxidation [20]. Soil pH and salinity were determined in the supernatants of 1:5 soil and water mixtures using a pH meter or salinity meter. Soil particle size was analyzed using a Laser Particle Size Analyzer (Microtrac Inc., USA).

Enrichment factor (EF)

Enrichment factor (EF) was selected to evaluate possible anthropogenic inputs of observed sample metals[21]. EF was defined as $EF = (M_{sample} / M_{background}) / (M_{reference} / M_{background})$, where $M_{sample}$, $M_{reference}$ and $M_{background}$ are the determined contents of targeted elements (e.g., As, Cd, Cu, Pb, and Zn) in soil samples and their background contents, respectively; $M_{reference}$ is used the reference element because it is a conservative lithogenic element presumed not to be enriched due to local contamination [22]. Contamination categories were classified based on EF values: EF<2, deficiency to minimal enrichment; EF = 2–5, moderate enrichment; EF = 5–20, significant enrichment; EF = 20–40, very high enrichment; EF>40, extremely high enrichment [23]. Background values for $M_{background}$ and $M_{reference}$ in this study were referenced to background contents in the loess materials of the Yellow River [24]. Background contents of 62700 μg g⁻¹ for Al, 10.7 μg g⁻¹ for As, 0.095 μg g⁻¹ for Cd, 21.1 μg g⁻¹ for Cu, 21.6 μg g⁻¹ for Pb and 64.5 μg g⁻¹ for Zn were used.

Toxic units (TUs)

Toxic units (TUs) are used to normalize the toxicities of various trace elements to allow for the comparison of the relative effects. TUs were defined as the ratios of the observed contents to the probable effect level (PEL) values [25]. PEL values represent the thresholds of chemical contents above which adverse effects are likely to occur. The PEL values for marine and estuarine ecosystem was used in this study (Table 2)[26].
Biological Concentration Factors (BCFs)

Biological concentration factor is the ratio of metal content in the aboveground parts of plants to metal content in surrounding soils, which can more accurately describe the plant’s uptake potential than plant metal content [27].

Statistical analysis

Pearson correlation analysis was performed to determine the relationships between trace elements and selected soil properties and the relationships between Al, trace elements, and sand and clay contents in selected soil profiles. Principal component analysis (PCA) was used to discriminate soil samples with similar or different contamination patterns and identify their influencing factors. Analysis of variance (ANOVA) analysis was implemented to analyze the differences in trace elements between sampling sites and seasons. Differences were considered to be significant at $p \leq 0.05$. Statistical analysis was performed using the SPSS 16.0 and Canoco 4.5 software packages for Windows.

Results and Discussion

Soil characteristics in three salt marshes

Selected physical-chemical properties in the top 20 cm soils of three salt marshes of the YRD are summarized in Table 2. No significant differences in soil moisture among three seasons in each sampling site due to consistent tidal flooding condition. However, Soils in $P. australis$ wetland contained higher moisture contents in spring and summer compared to $T. chinensis$ wetland due to less sand contents in $P. australis$ wetland ($p<0.05$; Table 2). Bulk density at each sampling site of the three was lower in spring than in summer and fall ($p<0.05$), but there were no significant differences among three sites in each season. With the exception of higher SOM level in $P. australis$ wetland than those in $T. chinensis$ and S. salsa wetlands in fall ($p<0.05$), no significant differences were observed among three sites in summer and spring. Compared to other seasons, higher SOM content appeared in $P. australis$ wetland in fall, while it was higher in $T. chinensis$ wetland in spring ($p<0.05$), which was likely caused by plant litter inputs and decomposition [5]. All soil samples of three sites exhibited higher salinities in spring than in summer or fall ($p<0.05$), which

Table 1. Ecological characteristics of three sampling sites in different sampling seasons.

| Site          | Location                                      | Replicates | Sampling time | Plant height (m) | Plant density (m$^{-2}$) |
|---------------|-----------------------------------------------|------------|---------------|------------------|--------------------------|
| $P. australis$ | 37°43'35.1" to 37°43'59"N 119°12'42" to       | 3          | Spring        | 0.1–0.3          | 35–50                    |
|               | 119°13′21″E                                    |            | Summer        | 1.8–2.2          | 230–280                  |
|               |                                                |            | Fall          | 1.8–2.2          | 190–230                  |
| $T. chinensis$| 37°42'47" to 37°42'52"N 119°13'49" to          | 5          | Spring        | 1–1.6            | 15–30                    |
|               | 119°14′15″E                                    |            | Summer        | 1–2              | 15–30                    |
|               |                                                |            | Fall          | 1–2              | 15–30                    |
| $S. salsa$    | 37°43'16" to 37°43'34"N 119°13'36" to          | 4          | Spring        | 0.05–0.15        | 100–220                  |
|               | 119°13′53″E                                    |            | Summer        | 0.20–0.40        | 280–580                  |
|               |                                                |            | Fall          | 0.30–0.45        | 136–520                  |

Note: Spring = April; Summer = August; Fall = November.

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Table 2. Summary of physical-chemical properties in the top 20 cm soils at three sampling sites during three sampling seasons.

| Site          | Moisture (%) | BD (g cm⁻³) | pH | SOM (g kg⁻¹) | Clay (%) | Sand (%) |
|---------------|--------------|-------------|----|--------------|----------|----------|
| P. australis  |              |             |    |              |          |          |
| Spring        | 268 ± 2.9    | 1.4 ± 0.1   | 8.4 ± 0.1 | 6.9 ± 0.9   | 15 ± 0.3 | 52 ± 1.3 |
| Summer        | 280 ± 4.4    | 1.8 ± 0.1   | 8.2 ± 0.6 | 6.7 ± 1.0   | 28 ± 0.5 | 23 ± 1.6 |
| Fall          | 270 ± 3.1    | 1.8 ± 0.1   | 7.0 ± 3.0 | 6.2 ± 0.8   | 34 ± 0.0 | 61 ± 4.3 |
| Fall          | 250 ± 2.7    | 1.9 ± 0.1   | 5.9 ± 2.2 | 6.8 ± 1.4   | 17 ± 0.4 | 84 ± 1.0 |
| S. salsa      |              |             |    |              |          |          |
| Spring        | 238 ± 1.6    | 1.5 ± 0.1   | 5.9 ± 2.2 | 6.8 ± 1.4   | 17 ± 0.4 | 84 ± 1.0 |
| Summer        | 250 ± 1.6    | 1.8 ± 0.1   | 6.5 ± 0.9 | 6.5 ± 1.0   | 20 ± 0.2 | 60 ± 3.1 |
| Fall          | 238 ± 1.6    | 1.8 ± 0.1   | 6.5 ± 0.9 | 6.5 ± 1.0   | 20 ± 0.2 | 60 ± 3.1 |
| P. australis  |              |             |    |              |          |          |
| Spring        | 260 ± 2.3    | 1.8 ± 0.1   | 8.2 ± 0.6 | 6.7 ± 1.0   | 28 ± 0.5 | 23 ± 1.6 |
| Summer        | 270 ± 4.4    | 1.8 ± 0.1   | 8.2 ± 0.6 | 6.7 ± 1.0   | 28 ± 0.5 | 23 ± 1.6 |
| Fall          | 250 ± 2.7    | 1.9 ± 0.1   | 5.9 ± 2.2 | 6.8 ± 1.4   | 17 ± 0.4 | 84 ± 1.0 |
| P. australis  |              |             |    |              |          |          |
| Spring        | 268 ± 2.9    | 1.4 ± 0.1   | 8.4 ± 0.1 | 6.9 ± 0.9   | 15 ± 0.3 | 52 ± 1.3 |
| Summer        | 280 ± 4.4    | 1.8 ± 0.1   | 8.2 ± 0.6 | 6.7 ± 1.0   | 28 ± 0.5 | 23 ± 1.6 |
| Fall          | 270 ± 3.1    | 1.8 ± 0.1   | 7.0 ± 3.0 | 6.2 ± 0.8   | 34 ± 0.0 | 61 ± 4.3 |

was associated with the freshwater inputs from the flow-sediment regulation [4]. Moreover, T. chinensis wetland showed higher soil salinity ($p<0.05$) than P. australis wetland or S. salsa wetland in three seasons as T. chinensis wetland was further away from the river channel (Figure 1). However, no significant differences in pH values were observed among three wetlands in spring and fall except that T. chinensis wetland had lower pH values ($p<0.05$) than S. salsa wetland in summer. Both T. chinensis and S. salsa wetlands showed higher pH values ($p<0.05$) in fall compared to spring and summer, which might be associated with weaker soil respiration in fall as the dissolved CO$_2$ in overlying water could decrease pH values [28]. All sampling sites showed higher clay contents in summer than in spring and fall and sand contents were also lower in both P. australis and S. salsa wetlands in summer ($p<0.05$; Table 2). This indicated that the flow and sediment regulation in summer brought more clay and silt contents to the tidal salint marshes [4]. However, no significant differences in sand or clay contents were observed between spring and fall, which was associated with tidal seawater erosion [4]. Moreover, soils in P. australis and S. salsa wetlands contained more clay contents and less sand contents in summer compared to T. chinensis wetland ($p<0.05$). This might be caused by the fact that T. chinensis wetland is located at the farther distances from the Yellow River Channel (Figure 1). In both spring and fall, P. australis wetland had higher clay contents than T. chinensis and S. salsa wetlands.

Spatial and temporal variations in trace elements in marsh soils

Profile distributions of these trace elements in three seasons are shown in Figure 2. Generally, trace element contents exhibited a decreasing tendency with depth in each season except for As, Cd and Zn in P. australis wetland and Cd, Cu and Zn in S. salsa wetland in summer. Prusty et al. [29] also reported that trace elements such as As, Cu, Pb, and Zn decreased with increasing depths in wetland ecosystems. This was greatly associated with plant cycling which lead to trace elements upwards movement through plant litters and return to surface soils [29,30]. However, clear seasonal changes in the contents of these trace elements were observed along soil profiles (Figure 2). Among three seasons, most soil profiles of three sampling sites exhibited the highest levels of As, Cd and Zn in spring, followed by summer, whereas the lowest levels in fall. As for Cu and Pb, lower levels were also observed in fall compared to summer and spring. The average contents of As, Cd and Zn in the top 20 cm soils in P. australis and T. chinensis wetlands were significantly higher in summer than in fall ($p<0.05$; Table 3). Similarly, much higher average contents of these trace elements in the top 20 cm soils were also observed in S. salsa wetland in summer compared to fall ($p<0.05$; Table 3). This is inconsistent with the results by Roychoudhury [12], who presented that trace metal contents in surface soils were relatively lower in summer than in winter due to higher wetland plant uptake and standing stocks of metals in summer. Additionally, Eihklen and Kirchner [31] reported that higher water contents, lower salinities and bulk densities increased the potential mobility of metals in soil solutions. However, all trace elements generally reached their higher levels in summer in this study, although all soils contained lower salinities and higher water contents in three sampling sites after the flow-sediment regulation, and then fell to the lowest levels in fall. Therefore, the higher trace element contents observed in summer might be attributed to exogenous inputs of trace metals (e.g., water and sediment inputs from the flow-sediment regulation) [4,32]. The increased plant uptake of trace elements in the late fall could explain the lower trace element contents in these marsh soils in this season, as plants possess efficient root-to-shoot
translocation systems that are activated at the end of the growing season [33].

Both Cu and Pb contents in soil profiles were generally lower in T. chinensis wetland than those in P. australis and S. salsa wetlands in spring and summer, whereas higher levels of As and Cd in soil profiles were observed in T. chinensis wetland in summer (Figure 2). Table 3 also showed that the average contents of trace elements in the top 20 cm soils were significantly higher in P. australis and S. salsa wetlands in spring compared to T. chinensis wetland ($p < 0.05$). Meanwhile, higher levels of Cu and Pb in P. australis wetland and higher Cd contents in S. salsa wetland were observed in fall compared to other sites ($p < 0.05$). In summer, P. australis and S. salsa wetlands exhibited greatly higher Cu and Pb contents than T. chinensis wetland ($p < 0.05$, Table 3). The differences in Cu and Pb between sites or sampling seasons were most likely related to SOM, sand and clay contents (Tables 4 and 5). Additionally, lower Cu and Pb levels in the top 20 cm soils in T. chinensis wetland in summer compared to S. salsa wetland might be caused by higher plant uptake as the aboveground parts of T. chinensis had higher BCFs values for both Cu and Pb compared to S. salsa ($p < 0.05$; Figure 3). However, it is difficult to explain much higher BCFs values of T. chinensis for Cd with higher soil Cd levels ($p < 0.05$; Table 2) than P. australis. Therefore, the variations in trace element levels among the three sampling sites might be influenced by the multiple environmental factors including plant communities, soil properties, and water levels etc. [34,35,36]. As shown in Figure 4, all trace elements generally showed similar hand-type distribution patterns, indicating higher levels of these trace elements in P. australis and S. salsa wetlands higher compared to T. chinensis wetland except for some soil samples with higher levels of As and Cd. These trace elements increased linearly with increasing Al content at each sampling site, which implies that these trace elements and Al might have common origin.

Assessment of trace element pollution by sediment quality guidelines (SQGs)

The extent of trace element pollution was assessed by comparing element contents in marsh soils to the sediment quality criteria developed by the Chinese Marine Sediment Quality Criteria (GB 18668-2002) [37] and the sediment quality guidelines (SQGs) [23] (Table 3). The Chinese Marine Sediment Quality Criteria indicated that approximately 85–95% of all collected soil samples in three sampling sites during three seasons exceeded the Class I criteria (suitable for fisheries and natural waters) but fell within the scope of Class II criteria (used for industrial and tourism sites) for As and Cd, and all samples were below the Class I criteria for Cu, Pb and Zn (Table 3). This supplies that most soil samples in this region have been moderately polluted by As and Cd. As shown in Figure 2, the SQGs also indicated that almost all soil samples along soil profiles at three sites fell within the range between the threshold effect levels (TELs) and probable effect levels (PELs) for As and Cu in each of the three seasons, even some samples of three sites exceed PELs in summer. Moreover, all soil profile samples at each site exceeded TELs in summer, whereas they were below TELs in both spring and fall. However, approximately 85% of soil profile samples in this region didn’t exceed TELs for Pb in three seasons, and almost all soil profile samples at each site exhibited much lower Zn contents than TELs in each season. This implies higher potential risks of As, Cu and Cd in soil profiles of this region. Therefore, it is still required to control heavy metal pollution (i.e., As, Cu and Cd) in this region, as higher trace elements may make it more difficult to restore the degraded living habitat [36].

Figure 2. Profile distributions of As and heavy metals in marsh soils with P. australis (a), T. chinensis (b) and S. salsa (c) in three sampling seasons.
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Although the contents of trace elements were lower in the YRD than in most large rivers and estuaries [4], trace element pollution in the YRD is increasingly serious due to continuous disturbances caused by human activities and sediment movements [36,38]. Trace element contents in the upstream loess were used as indicators to determine the enrichment levels for trace elements in the soils. Figure 5 shows the proportions of enrichment levels in marsh soils at three sampling sites in each season. Cd levels in all soil samples were at the significant enrichment level in three seasons. As levels in almost 95% of soil samples were at a moderate enrichment level. Comparatively, more than 90% of soil samples showed minimal enrichment levels of Cu, Pb and Zn in three sampling sites in each season. Both P. australis and T. chinensis wetlands showed higher enrichment levels of Cd and lower enrichment of Cu, Pb, and Zn in summer than in both spring and fall, which was associated with higher BCFs of Cu, Pb and Zn and lower BCFs of As and Cd at both sites (Figure 3). Moreover, almost all EF values of As and Cd and more than 50% of EF values of Cu, Pb and Zn for all soils in three seasons exceeded 1.5, indicating that a significant portion of metal is delivered from non-crustal materials, or non-natural weathering processes, so anthropogenic sources might be an important contributor [39]. Bai et al. [4] also presented that the flow-sediment regulation could elevate the levels of trace metals.

**Ecotoxicity assessments of trace elements**

The potential acute toxicities of contaminants in soil samples can be estimated using the toxic unit (TU) index, which is the ratio of the determined content to the probable effect level (PEL) [26]. The TUs, the sum of toxic units ($g_TU$) and relative contributions of trace elements at each soil layer in the YRD are illustrated in Figure 6. The mean TUs value of each element at three sites decreased in the order of As$>Zn> Cu > Pb > Cd$ in each of

| Table 3. Mean contents of As and heavy metals in the top 20 cm soils of three sampling sites during the whole sampling period (mg kg$^{-1}$ dry weight). |
|---|---|---|---|---|---|
| $P. australis$ sites | | | | | |
| Summer | 35.8±2.4$^{a1}$ | 0.9±0.1$^{a1}$ | 30.7±5.5$^{a1}$ | 28.56±5.3$^{a01}$ | 95.1±11.0$^{a1}$ |
| Fall | 23.0±4.8$^{b1}$ | 0.5±0.1$^{b12}$ | 29.2±1.5$^{b1}$ | 25.27±1.3$^{b1}$ | 74.7±5.7$^{b1}$ |
| Spring | 29.8±3.5$^{c1}$ | 0.6±0.1$^{c1}$ | 33.5±4.3$^{c1}$ | 32.9±2.4$^{c1}$ | 91.2±10.4$^{c1}$ |
| $T. chinensis$ sites | | | | | |
| Summer | 41.9±3.2$^{a2}$ | 1.2±0.2$^{a2}$ | 23.0±2.3$^{a2}$ | 22.4±2.8$^{a2}$ | 93.9±13.1$^{a2}$ |
| Fall | 24.7±3.4$^{b2}$ | 0.5±0.0$^{b2}$ | 22.1±1.4$^{b2}$ | 19.4±2.8$^{b2}$ | 72.0±10.7$^{b2}$ |
| Spring | 24.1±4.5$^{c2}$ | 0.4±0.0$^{c2}$ | 21.4±1.6$^{c2}$ | 20.1±1.8$^{c2}$ | 62.5±4.5$^{c2}$ |
| $S. salsa$ sites | | | | | |
| Summer | 40.5±6.4$^{a2}$ | 0.9±0.1$^{a2}$ | 37.7±1.7$^{a2}$ | 29.9±1.9$^{a2}$ | 106.3±3.8$^{a2}$ |
| Fall | 25.2±1.8$^{b2}$ | 0.6±0.0$^{b2}$ | 22.0±2.1$^{b2}$ | 19.9±2.4$^{b2}$ | 66.9±6.2$^{b2}$ |
| Spring | 34.5±1.4$^{c1}$ | 0.7±0.0$^{c1}$ | 34.5±3.7$^{c1}$ | 33.7±4.5$^{c1}$ | 92.1±9.5$^{c1}$ |

Sediment Quality Criteria of China*

| Class | As | Cd | Cu | Pb | Zn |
|---|---|---|---|---|---|
| Class I | 20 | 0.5 | 35 | 60 | 150 |
| Class II | 65 | 1.5 | 100 | 130 | 350 |
| Class III | 93 | 5 | 200 | 250 | 600 |

**SQGs**: Sediment Quality Guidelines for marine ecosystem; TEL: threshold effect level; PEL: probable effect level [23].

Enrichment factors of trace elements in marsh soils

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Ecotoxicity assessments of trace elements

The potential acute toxicities of contaminants in soil samples can be estimated using the toxic unit (TU) index, which is the ratio of the determined content to the probable effect level (PEL) [26]. The TUs, the sum of toxic units ($\Sigma TU$) and relative contributions of trace elements at each soil layer in the YRD are illustrated in Figure 6. The mean TUs value of each element at three sites decreased in the order of As$>Zn> Cu > Pb > Cd$ in each of

| Table 4. Relationships between soil texture and Al and heavy metals in typical profiles from three sampling sites in three sampling seasons. |
|---|---|---|---|---|---|---|
| Al | As | Cd | Cu | Pb | Zn |
| Sand | 0.743** | 0.306* | 0.259* | 0.805** | 0.753** | 0.121 |
| Clay | 0.758** | 0.305* | 0.263* | 0.850** | 0.756** | 0.276** |

* represents significant correlation at the level of $p < 0.05$; ** represents significant correlation at the level of $p < 0.01$. n = 60.

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three seasons. As and Cd in all soil samples had much higher TUs in summer compared to spring and fall. Generally, the TUs decreased with depth along soil profiles. The TUs for surface soils (0–10 cm) in S. salsa wetland in spring or in T. chinensis wetland in summer were much higher than those for all the other soil samples of the three sites, but neither exceeded the moderate toxicity level (ΣTUs = 4) [25]. The order of the TUs in the top 20 cm soils of three sampling sites in spring was S. salsa wetland > P. australis wetland > T. chinensis wetland. Additionally, the TUs for all soil layers at the three sites reached higher levels in summer than in fall and spring. The TUs for each layer were higher in P. australis and S. salsa wetlands than T. chinensis wetland in spring, whereas they exhibited similar TUs levels at three sites in summer and fall.

The values of the ΣTUs increased from spring to summer and then decreased to their lowest levels in fall. This corresponded to the temporal variations in trace element contaminations during the three sampling seasons which was closely related to the input flow containing trace elements from upstream during the flood-sediment regulation period from June to July. Tixier et al. [40] presented that the storm water exhibited the seasonally varying toxicity of trace elements with high toxicity in spring and recovery in fall, which was caused by the Terraview–Willow field storm water management facility. The TUs for all soil layers at the three sites reached higher levels in summer than in fall and spring. The TUs for each layer were higher in P. australis and S. salsa wetlands than T. chinensis wetland in spring, whereas they exhibited similar ΣTUs levels at three sites in summer and fall.

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Table 5. Correlation matrix among trace elements and selected soil properties for three sampling sites.

| Moisture | Bd | SOM | Salinity | pH | S | Al | As | Cd | Cu | Pb | Zn |
|----------|----|-----|----------|----|---|----|----|----|----|----|----|
| Moisture | 1.000 | -0.210* | 0.143 | -0.236* | -0.242* | 0.478* | 0.113 | 0.347* | 0.507** | 0.295** | 0.137 |
| Bd      | -0.210* | 1.000 | -0.198* | -0.173 | -0.097 | -0.017 | -0.026 | 0.048 | -0.037 | 0.009 | 0.297** |
| SOM     | 0.143 | -0.198* | 1.000 | -0.262** | 0.077 | 0.144 | 0.047 | 0.212* | 0.097 | 0.344 | 0.173 |
| Salinity | -0.236* | -0.173 | -0.262** | 1.000 | 0.109 | 0.193 | 0.037 | 0.017 | 0.009 | 0.037 | 0.212* |
| pH      | -0.242* | -0.097 | 0.077 | 0.109 | 1.000 | 0.139 | 0.037 | 0.017 | 0.009 | 0.037 | 0.212* |
| S       | 0.478* | 0.017 | 0.144 | 0.193 | 0.139 | 1.000 | 0.237** | 0.111 | 0.371** | 0.507** | 0.144 |
| Al      | 0.113 | 0.047 | 0.047 | 0.037 | 0.037 | 0.237** | 1.000 | 0.141 | 0.507** | 0.295** | 0.137 |
| As      | 0.347* | 0.017 | 0.193 | 0.037 | 0.037 | 0.347** | 0.507** | 1.000 | 0.371** | 0.507** | 0.137 |
| Cd      | 0.507** | 0.009 | 0.212* | 0.139 | 0.037 | 0.507** | 0.507** | 0.137 | 1.000 | 0.507** | 0.507** |
| Cu      | 0.295** | 0.137 | 0.097 | 0.344 | 0.173 | 0.344 | 0.507** | 0.371** | 0.507** | 1.000 | 0.371** |
| Pb      | 0.137 | 0.173 | 0.344 | 0.173 | 0.173 | 0.173 | 0.507** | 0.371** | 0.507** | 0.507** | 1.000 |
| Zn      | 0.137 | 0.173 | 0.173 | 0.173 | 0.173 | 0.173 | 0.173 | 0.173 | 0.173 | 0.173 | 1.000 |

* represents significant correlation at the level of \( p < 0.05 \).
** represents significant correlation at the level of \( p < 0.01 \).

Figure 3. Biological concentration factors of the aboveground parts of plants in each site. abc Different letters on the error bars represent significant differences \( (p < 0.05) \). doi:10.1371/journal.pone.0107738.g003

Relationships between trace elements and soil physicochemical properties

The correlation analysis between trace elements and soil physicochemical properties was performed to assess possible co-contamination from similar sources. As shown in Table 4, significantly positive correlations among all trace elements (i.e., As, Cd, Pb, Cu, and Zn) were observed, suggesting that these trace elements are closely related and may originate from the same sources.
elements in marsh soils might originate from common source. Figure 4 also exhibited that the linear relationships between Al and these trace elements at each sampling site, which implies that these trace elements and Al were greatly associated with the upstream loess materials with higher Al contents of the Yellow River (62700 mg kg$^{-1}$) [24]. Moreover, Al and these trace elements were significantly correlated with sand and clay contents (Table 4), indicating that higher clay contents in August (Table 2) would contribute to these trace element accumulation. Therefore, the upstream flow-sediment regulation in June could take main responsibility for the accumulation of these trace elements in the delta in the studied period due to no other pollution source inputs in the study area. This is also supported by the result of Bai et al. [4], who presented that the upstream flow-sediment regulation contributed to elevating trace element contents in surface soils in this delta.

Table 5 showed the negatively linear correlations between salinity and Al, Cu, Pb and Zn in the study area ($p<0.01$). This is in agreement with the results presented by Du Laing et al. [45], indicating increasing salinity might enhance the mobility of these metals in soils, as the chloro complex formation and the substitution of Ca$^{2+}$ and Mg$^{2+}$ in the exchange positions could promote the mobility of heavy metals [44]. Additionally, Manousaki et al. [45] found that salinity could favor metal accumulation in shoots of salt cedars, thus decreasing soil metal contents. Bulk densities exhibited negative correlations with Cu and Pb in all soils, implying that lower bulk densities in saturated substances with higher SOM favored the accumulation of both metals. Numerous studies reported that SOM and soil pH played important roles in determining the fates of heavy metals in soils [11,46]. SOM can act as a major sink for heavy metals due to its strong complexing capacity for metallic contaminants [47]. In this
study, SOM showed a positive linear relationship with Cu, Pb and Zn ($p<0.01$; Table 5). Most researchers presented that subalkaline environments could decrease the mobility of metals and increase the ability of soil to stabilize metals [11,36,48]. However, in this study, soil pH values were negatively correlated with As and Cd ($p<0.01$; Table 5). This was most likely due to plant uptake or leaching as alkalinization will increase metal leaching at higher pH ranges (8–10) [46,49]. Sulfur exhibited significantly positive correlations with Cu, Pb and Zn ($p<0.01$) due to the deposition of metal sulphates (i.e., CuS, PbS and ZnS) under anaerobic conditions [4]. Moreover, Kumpiene et al. [46] reported that the presence of one contaminant (e.g., Cu or Pb) can decrease the stabilization efficiency of another contaminant (e.g., Zn) due to competition for sorption sites. Therefore, heavy metals levels in the marsh soils could be impacted by such factors as moisture, salinity, SOM, pH values, sulfur and the interactions between trace elements.

To assess the relationships between spatial distribution patterns of trace elements and several environmental factors (including 5 soil parameters), PCA was implemented to characterize the variations in trace element contents among sampling sites. The factor loading scores in the biplot (Figure 7) exhibited a clear separation amongst *P. australis*, *T. chinensis* and *S. salsa* wetlands and amongst spring, summer, and autumn along the first principal component (PC) accounting for 75.1% and the second PC accounting for 16.0% of the variance. As shown in Figure 7, three main groups can be clearly identified. The red plate contained the soils collected in spring, the yellow plate contained the soils collected in summer, and the blue plate included the soils collected in fall. It appears that the sampling seasons play an important role in shaping the spatial distribution patterns of As and heavy metals in marsh soils. However, the soils in *T. chinensis* wetland in spring did not fall into the red plate but entered or attached to the blue plate, suggesting that the distribution patterns
of trace elements in those soils are more similar to those in \textit{T. chinensis} and \textit{S. salsa} wetlands in fall than those in \textit{P. australis} and \textit{S. salsa} wetlands in spring.

PCA exhibited that the strongest determinants of the distribution patterns of trace elements in spring were soil moisture, S, SOM and pH value, while the strongest influencing factors in fall were soil salinity and soil texture. Obviously, salinity, water content and soil texture controlled the distribution patterns of trace elements in \textit{T. chinensis} and \textit{S. salsa} wetlands, while SOM, S and soil pH values were the dominant factor in \textit{P. australis} wetland. All trace elements with the longest arrows in the summer group testified the highest accumulations of trace elements in summer, especially in \textit{P. australis} and \textit{S. salsa} wetlands with herbaceous plants. The results obtained from the PCA were consistent with the results derived from the correlation analysis.

Although spatial distribution patterns of trace elements were dominantly controlled by sampling seasons due to the flow-sediment regulation, the effects of plant species on their distributions in soils could not be ignored due to different plant accumulation capabilities for different species (Figure 3). As shown in Figure 7, in the same season plate, sampling sites with the same plant community were clustered together. This was because plant roots can significantly affect plants’ abilities to transfer metals [50,51], as they employed a variety of ways to alleviate the stresses from root zone pollution through increasing the rate of root exudation as a result of losses of membrane integrity or of breakdowns in normal cell metabolism [52,53]. The fixation and stabilization of trace elements by plants originating from physiological characteristics (e.g., root exudation in response to underground stress) were different between herbaceous plants (\textit{P. australis} and \textit{S. salsa}) and woody plant (\textit{T. chinensis}). Additionally, ecological characteristics of plants (e.g., plant density) might also affect the responses of plants to trace elements, especially under the interactive impacts of water and salt in the tidal salt marshes. This implies that marsh soils with different plant communities might maintain different capabilities for immobilizing trace elements at the same pollution level (Figure 3). Further studies are needed to investigate the impacts of land cover changes on these trace elements.

### Conclusions

Obvious spatial and seasonal changes in trace elements in wetland soils in the intertidal salt marshes were observed. The higher trace element contents (especially As and Cd) in summer compared to spring and fall suggested the contribution of the flow-sediment regulation (from June to July) to pollutants from upper reaches deposition and accumulation in the delta and higher self-purifying capabilities of these salt marshes. This implies that these tidal salt marshes can alleviate the effects of the upstream flow-sediment regulation. Higher trace element contents in these soils were closely correlated to soil properties such as soil moisture, salinity, sulfur, SOM, soil texture and pH value. It is noted that sampling seasons had dominant impacts on the spatial distribution patterns and ecotoxicity of trace elements in soils in this study. Therefore, seasonal changes in trace element contamination should be taken into account in tidal wetlands especially in tidal wetlands with great hydrological fluctuations. We also observed that marsh soils with herbaceous plants (\textit{P. australis} and \textit{S. salsa}) had higher trace element accumulation than woody plant (\textit{T. chinensis}). Although all soil samples showed lower ecotoxicity, higher ecotoxicity of As in each of the three seasons should attract our concerns in this region. Moreover, it is important to continuously monitor these trace elements in coastal wetland soils to identify the ecological and environmental effects of hydrological engineering. The findings of this study could contribute to wetland conservation and management in coastal regions affected by the hydrological engineering. Further studies of the final fate and transport of these trace elements and plant uptake mechanism under water and salt stress in coastal wetlands will greatly contribute to trace element pollution control in coastal region.

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### Author Contributions

Conceived and designed the experiments: JB RX. Performed the experiments: QZ QL JW. Analyzed the data: JB KR. Contributed reagents/materials/analysis tools: JB KR. Wrote the paper: JB RX KR.

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