Spatial and seasonal distributions of soil sulfur in two marsh wetlands with different flooding frequencies of the Yellow River Delta, China

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Abstract
Soil samples from 0 to 100 cm depth were collected in eight sampling sites (Sites A1, B1, C1 and D1; Sites A2, B2, C2 and D2) respectively along two 250-m length of sampling zones (Zone N and Zone S) from the Yellow River channel to a tidal creek in the short-term flooding and seasonal flooding wetland of the Yellow River Delta of China in fall of 2007 and spring of 2008 to investigate spatial and seasonal distribution patterns of total sulfur (TS) and their influencing factors. Our results showed that TS contents in both seasons in surface soil in Zone N increased with increasing distances away from the Yellow River channel except for Site D1 in fall. However, TS contents in both seasons in surface soil in Zone S showed a tendency of increasing and then decreasing with increasing distances away from the Yellow River channel. Relative to spring, the mean TS contents in surface soils were slightly higher in fall in Zone N, whereas an opposite tendency was observed in Zone S. The mean TS stocks were obviously higher in fall with 934.36 g/m² and 877.77 g/m² compared to those in spring with 729.85 g/m² and 696.78 g/m² in Zone N and Zone S, respectively. TS contents and stock generally declined with depth along soil profiles with one accumulation peaks at a soil increment of 40–60 cm. Topsoil concentration factors also indicated that TS had shallower distribution in soil profiles. Soil total carbon (TC), total nitrogen (TN), soil organic matter (SOM), salinity and silt exerted positive loadings on TS, while soil pH and depth exhibited a negative correlation with TS content.

1. Introduction
Coastal wetland is one type of wetlands, which is frequently considered as the first recipients of high nutrient runoff via overland or groundwater flow (Caffrey et al., 2007). Wetland soils are the primary component of the global biogeochemical S cycle, acting as a source and sink for various S species and mediating changes of oxidation states (Wang et al., 2006). Sulfur, along with nitrogen (N), phosphorus (P) and potassium (K), plays an important role in many biogeochemical processes, such as participating in the composition of protein, aminophenol and chlorophyll, controlling the metabolism of carbohydrates in the photosynthesis process, and influencing the respiration and the stress-resistance of plants (Sun et al., 2013). It is not only an only growth-limiting plant nutrients but also indirectly affects the use efficiency of other plant nutrients, such as N (Bona and Monteiro, 2010).

Sulfur cycle, especially the process of transformation of organic S to inorganic sulfate (mineralization) and the reverse process (incorporation of sulfate into soil organic compounds or immobilization) in wetland soils is greatly influenced by soil moisture (Scherer, 2009), Freney et al. (1975) and Williams (1967) presented that the drying and re-wetting events in wetlands could improve the amount of sulfur mineralization. With the exception of hydrology, sulfur fractions in wetland soils were also significantly influenced by other soil properties, including soil organic matter (SOM) (Li et al., 2009), soil pH (Tanikawa et al., 2014) and particle size (Hao and Wang, 2004). Moreover, sulfur and sulfur cycle also impacted by soil microorganism, which was sensitive to the local environmental conditions and anthropogenic activity (Wu et al., 2013, 2015; Zeng et al., 2015). Previous studies have examined the dynamics of soil sulfur fractions under forest (Vannier and Guillet, 1994), cropland (Yang et al., 2007), grassland (Nguyen and Goh, 1992) and tropical soils (Schmidt et al., 2012). However, little information is available on the spatial and temporal distribution of soil S in different flooding frequency wetland in the coastal regions (Krairapanond et al., 1991; Li et al., 2009). A better understanding
of spatial and seasonal distribution of soil S in coastal wetlands could provide theoretical basis for wetland sulfur cycle, wetland conservation and management.

The primary objectives of this study were (1) to investigate horizontal distributions of total sulfur (TS) contents in surface soils (0–10 cm) in two coastal wetlands with different flooding frequencies along two sampling zones from the Yellow River channel to a tidal creek; (2) to characterize their vertical distributions in soil profile (0–100 cm) from the wetland in different sampling dates; (3) to reveal relationships between soil S and other selected soil properties.

2. Materials and methods

2.1. Study area

The study area is located in the Nature Reserve of the Yellow River Delta (37°35′ N–38°12′ N, 118°33′ E–119°20′ E) (Fig. 1) in Dongying City, Shandong Province, China. It is the most complete and youngest wetland ecosystem which covers approximately 15.3 × 10⁴ ha². It has a semi-humid continental monsoon climate with distinct four seasons. The average annual temperature is 11.7–12.6 °C. The average annual precipitation is 530–630 mm, 70% of them concentrate in summer (May–July) which often leads to the summer flooding or short-term flooding in this region and the average annual evaporation is 1900–2400 mm. Meanwhile, the flow-sediment regulation regime was operated during the period from late June to early July every year since 2002, which can contribute to flooding during this period from July to October. The five dominant species in the Yellow River Delta are reed (Phragmites australis), Suaeda salsa (Suaeda heteroptera), cogon grass (Imperata cylindrica), salt cedar (Tamarix chinensis), and willow (Salix matsudana) (Xie et al., 2011). Generally, soil type in this delta mainly includes coastal saline alluvial soil and marsh soil.

2.2. Sample collection and analysis

Two typical marsh wetlands with different flooding frequencies were selected in Yellow River Delta. Based on the differences in soil moisture and salinity, four sampling sites (A1, B1, C1, D1) were selected along 250-m length of sampling zone between the Yellow River channel and a tidal creek in short-term flooding wetland (Zone N, only flooded by overbank flow for a short time after the water and sediment regulation regime of the upstream Xiaolangdi Reservoir) and the other four sampling sites (Sites A2, B2, C2, D2) were selected along a 250-m length of sampling zone perpendicular to the Yellow River Delta in seasonal flooding wetland (Zone S, submerged by river floods during the period of water and sediment regulation and retained inundation with certain water levels until the end of the rainy season due to higher groundwater level and low terrain in this region) in fall (November of 2007) and spring (April of 2008). Soil profiles with three replicates were collected and stratified at depths of 0–10, 10–20, 20–40, 40–60, 60–80, and 80–100 cm. A total of 288 soil samples were obtained. All soil samples were placed in polyethylene bags and brought to the laboratory at once. All soil samples were air-dried under natural condition for 3 week and sieved using a 100-mesh sieve after removing recognizable plant litters, stones and other impurities, and then stored in a plastic zip-lock bag before analysis. Another soil core (4.8 cm diameter) for each soil layer of each sampling plot in both seasons were collected for the determination of soil water content and bulk density (BD).

TS was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES) after digestion with HNO₃/HClO₄ (volume ratio 10:1). Quality assurance and quality control were assessed using 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 every 10 samples). The recoveries of samples spiked with standards ranged from 99 to 106%. Soil water content (WC) and soil bulk density (BD) were measured by oven-drying at 105 °C for 24 h. Soil particle size was analyzed using a Laser Particle Size Analyzer (Microtrac Inc., USA). SOM was determined using dichromate oxidation (Walkley and Black, 1934). Soil PH and salinity were measured in the suspension of 1:5 soil/water (m/v) using a pH meter and salinity meter, respectively. TC and TN were measured using the Elemental Analyzer (Vario El, Elementar Co., Germany). The physical and chemical properties of the top 10 cm in Zone N and Zone S are listed in Table 1.

Total sulfur stock (TSS) at certain soil layer of each sampling plot in each sampling date was calculated by Eq. (1):

\[
TSS = \frac{BD_i \times TS_i \times h}{100}
\]  

(1)

where TSS (g/m²) is TS stock; BD_i (g/cm³) is soil BD of layer i; h (cm) is soil depth; TS_i (mg/kg) is TS content at soil layer i (i = 1, 2, 3, 4, 5 and 6).

2.3. Statistical analysis and graphing

Principal component analysis (PCA) and person correlation analysis were performed to identify the relationship among soil S and selected environmental factors. One-way ANOVA analysis was conducted to test the difference of soil properties between two
seasons in two different flooding periods wetlands. Differences were considered significant if \( p < 0.05 \). Statistical analysis was conducted using SPSS 16.0 software package, and linear graphs and contour maps were conducted using Origin 8.0 and Surfer 10.0 software packages, respectively. Kriging was used as an unbiased weighted linear interpolation method to conduct counter maps.

### 3. Results

#### 3.1. Horizontal distribution of total sulfur contents in surface soils in both seasons

Contents of TS and selected soil properties in surface soils (0–10 cm) in Zone N and Zone S are summarized in Table 1. Contents of TN and TC in the top 10 cm soils were higher in Zone N than those in Zone S in two sampling dates, while TS was higher in Zone N in November and in Zone S in April. Meanwhile, no significant differences were observed of TN and TC between Zone N and Zone S in two sampling dates. However, there were significant differences of TS between Zone N and Zone S in two sampling dates. Additionally, there were no significant differences of other physicochemical properties between Zone N and Zone S in two sampling dates except for salinity. Generally, obviously lower contents of SOM, BD and moisture were observed in April compared to those in November both in Zone N and Zone S. However, the top soils exhibited much lower soil pH values in November both in Zone N and Zone S.

The horizontal distribution patterns of TS contents in the top 10 cm soils in Zone N and Zone S in both spring and fall were illustrated. As shown in Fig. 2, TS contents in top soils in fall were slightly lower than those in spring (\( p < 0.05 \)) in Zone S. TS contents in top soils followed the order Site B2 (817.5 mg/kg) > Site D2 (639.0 mg/kg) > Site C2 (565.6 mg/kg) > Site A2 (424.2 mg/kg) and Site B2 (1168.7 mg/kg) > Site A2 (591.6 mg/kg) > Site C2 (588.7 mg/kg) > Site D2 (518.3 mg/kg) in fall and spring, respectively. TS levels generally increased and then decreased with increasing distances away from the Yellow River channel. However, TS contents in top soils in spring were obviously lower than those in fall (\( p < 0.05 \)) in Zone N. TS contents in top soils followed the order Site C1 (1196.2 mg/kg) > Site D1 (743.1 mg/kg) > Site B1 (654.4 mg/kg) > Site A1 (555.3 mg/kg) and Site D1 (666.8 mg/kg) > Site C1 (610.2 mg/kg) > Site B1 (337.4 mg/kg) > Site A1 (275.8 mg/kg) in fall and spring, respectively. TS levels generally increased with increasing distances away from the Yellow River channel. Moreover, TS content in Site B2 was higher in Zone S in two sampling dates, while higher TS contents were observed at Sites C1 and D1 in Zone N in two sampling seasons.

#### 3.2. Vertical distributions of total sulfur contents along soil profiles in both seasons

Fig. 3 shows the vertical distributions of TS contents in Zone N and Zone S in both seasons. Generally, TS contents decreased with depth along soil profiles in fall and spring in Zone S, except for the patches at the 60–80 cm soil depth of Site A2 in fall. The patches with lower TS contents appeared at the distance of 50 m and 200 m away from the Yellow River channel in fall and spring in Zone S, respectively. However, TS contents increased with the increasing distances away from the Yellow River channel in Zone N. Moreover, TS was accumulated in bottom soils at the distance from 250 m away from the Yellow River channel in spring in Zone N.

As shown in Fig. 4, the mean TS contents in Zone N and Zone S exhibited similar profile distributions in both seasons. The mean TS content in soil profiles was higher in spring (490.1 mg/kg) than that in fall (361.4 mg/kg) in Zone N, whereas the mean TS content in soil profiles was higher in fall (507.3 mg/kg) than that in spring (488.5 mg/kg) in Zone S. Additionally, there was an accumulative peak in the soil increment of 40–60 cm in Zone N and Zone S in both seasons.

#### 3.3. Profile distribution of soil S stocks in both seasons

Fig. 5 shows TSS at each soil depth of Zone N and Zone S in both seasons. TS stock in the 0–100 cm soil depth ranged from 825.1 to 916.4 g/m² and 590.6 to 864.2 g/m² at four sampling sites in fall and spring in Zone S, respectively. Meanwhile, they didn't show big differences in TS stocks between different soil depth except for higher accumulation in the 60–80 cm soil depth of Site D2 in fall and the top soils (0–10 cm) of Site C2 in spring in Zone S. Similarly, in Zone N, the highest TS stocks in the 0–100 cm soil depth were observed at Site C1 (1172.8 g/m²) in fall and Site D1 (982.3 g/m²) in spring among four sites due to higher contribution of TS stocks at the soil depth of 0–10 cm and 40–60 cm, respectively. Generally,
Fig. 2. Horizontal distributions of total sulfur in surface soils (0–10 cm) in fall (a) and spring (b) in Zone N (n) and Zone S (s).

Fig. 3. Vertical distributions of total sulfur in 100 cm soil depth in fall (a) and spring (b) in Zone N (n) and Zone S (s).
the mean TS stocks showed a tendency of decrease, increase and then slight decrease along soil profiles in Zone N and Zone S in both seasons, whereas a big accumulation peak of TS stocks appeared at the 40–60 cm soil depth in both seasons (Fig. 6). Additionally, the mean TS stocks for each soil layer in Zone N and Zone S were significantly higher in fall (Fig. 6).

3.4. Topsoil concentration factors (TCFs) of total sulfur in both seasons

The topsoil concentration factor represented the relative contribution of certain nutrient content at the 0–10 cm depth to the total content in the top 1 m on a basis of bulk density (Jobbágy and
Jackson, 2001). Fig. 7 showed TCFs for TS in Zone S and Zone N in both fall and spring. TCFs for TS at four sampling sites in Zone S and Zone N in both seasons were generally higher than 0.1, which means that the vertical distribution of sulfur showed shallower distribution in soil profiles. TCFs for TS at four sampling sites were higher in spring in Zone S, while higher TCFs for TS at the other four sampling sites could be found in fall in Zone N. Moreover, TCFs for TS were higher in both seasons at Site B2 and Site C1 in Zone S and Zone N, respectively.

3.5. Relationships between sulfur and other selected soil properties

To identify the relationship among all the types of sulfur and other selected soil properties, the PCA algorithm in the Canoco package was used, as illustrated in Fig. 8. SOM, TC, TN, Salinity and slit had positive loadings on the TS contents. The relationship between the soil pH and sulfur contents exhibited a negative correlation, and the depth generally presented a negative relationship with sulfur contents.

Table 2 also indicates the relationship between TS and selected soil properties in profile soils. TS in profile soils showed significantly positive correlation with TC, TN, SOM, salinity, WC and slit (p < 0.01, Table 2), but it was significantly negative correlation with soil depth, pH and sand (p < 0.01). Moreover, TS had not significantly correlations with BD (p < 0.05). Additionally, TS had significantly positive correlation with clay (p < 0.05), which was consistent with the result of the PCA.
4. Discussion

4.1. Horizontal changes of total sulfur contents of surface soils

It is important to quantify the temporal distribution of TS in the surface soils as they could be more easily leached by run-off than other soil layers (Gudimov et al., 2011). The lowest TS contents were observed in the surface soils of Site A1 and Site A2 in both sampling seasons, which were closely related to the lateral seepage of the Yellow River. Meanwhile, the higher TS contents were observed in the surface soils of Site C1 and Site D1 with S. salsa vegetation in Zone N. This was likely related to the input of the sea water from the tidal creek, since sea water could bring the external sulfur to Site C1 and Site D1. Moreover, Sun et al. (2013) stated that the high percent of soil S in S. salsa vegetation indicated that it was the circulation hing in the process of S cycle, which could prevent the S from being lost easily. However, the higher TS content was observed in the surface soils of Site B2 in Zone S. The reason can be interpreted that anaerobic respiration of the obligate anaerobic bacteria such as desulfovibrio under the condition of high soil moisture content can generates FeS and FeS$_2$ through sulfate reduction reaction which could fixed the sulfate during the water and sediment regulation regime of the Xiaolangdi Reservoir (Luo et al., 2014; Zheng and Wang, 2012). After the wetland plants absorb these sulfates, litter with the higher total sulfur then return to the soil, so that the total sulfur content in the soil in Site B2 can increase greatly (Lin et al., 2009). In addition, hydrological conditions also had an important effect on sulfur contents. Under flooded environment, the decomposition rates of SOM were lower (Bradley et al., 2007). Additionally, wetland soils saturated with enough flow can provide rich nutrients for microorganisms, which could result in the acceleration of litter decomposition (Anderson and Smith, 2002). We can also come to a conclusion that the different distribution patterns of TS in two belts were not completely consistent as the distance away from the Yellow River, it was mainly because the spatial variability of landform, vegetation type and soil hydrology in sampling zones (Hughes, 1990). Beverly et al. (1995) have testified that S accumulation varied with plant growth rhythm and ecological characteristics. As the most typical environmental factors in the Yellow River estuary, salinity gradients might also influence the S accumulation in plants directly (Sun et al., 2013). Braekke (1990) have showed that the changes in water condition (drainage) occurring in Scots pine (Pinus sylvestris) and Norway spruce (Picea abies) wetlands had significant influences on nutrient (N, P, K and S) accumulation.

4.2. Dynamic changes of total sulfur contents in soil profiles

Profile distributions of sulfur showed an obvious decrease with depth (Fig. 4). TS had significantly negative correlations with soil depths (p < 0.01, Table 2), which were mainly dependent on the spatial heterogeneity of SOM, plant root system distribution and soil properties (Wang et al., 2003; Stark, 1994). This is consistent with the result of Hao and Wang (2004). Additionally, higher sulfur contents generally occurred in soils 0–10 cm and 40–60 cm (Figs. 3 and 4). Biological cycling generally moves nutrients upwards because some proportion of the nutrients absorbed by plants are transported aboveground and then recycled to the soil surface by litterfall and throughfall (Stark, 1994). Therefore, plant S cycles could contribute to the shallower distribution of S in soil profiles. Meanwhile, the sulfate leaching could explain S accumulation in deeper soils. Jobbágy and Jackson (2001) have reported that leaching could move nutrients downward and might increase nutrient contents in deeper soils. Korb et al. (2002) also have stated that leaching is one common means of S loss from the surface. Area with the highest sulfate leaching is associated with high precipitation, irrigated conditions, coarse-textured soils, shallow soils, and soils with low anion exchange capacity. Compared to spring, higher TS stock was observed in soils in fall (Fig. 6), which might be related to the high bulk density in fall (Table 1). Topsoil concentration factors ranged from 0 to 1 and approached 0.1 when the vertical distribution was homogeneous throughout the soil profile (Jobbágy and Jackson, 2001). Therefore, the profile distribution of sulfur was heterogeneous since these relative values were almost more than 0.1 (Fig. 7). Moreover, the higher TCFs of TS in Site C1 and Site D1 in Zone N were also associated with these soil properties, such as SOM, soil texture, salinity, etc. in wetland ecosystems.

4.3. Effects of environmental factors on the spatial distribution of soil S

Most researchers have shown that the variation in soil S is related to changes in environmental factors, such as SOM, pH, clay content, salinity and soil moisture in soils and sediments (Zhou et al., 2007; Solomon et al., 2001). A significant positive correlation between TS and SOM was observed in this study (p < 0.01, Table 2; Fig. 8). Ghamakh et al. (2009) also found a positive relationship between C and S mineralization in soil. Additionally, the metabolic activities of microorganisms might also be promoted by SOM and influence sulfur transformation in soil (Tanikawa et al., 2014). Meanwhile, the significant and positive correlation between soil nitrogen, carbon and sulfur indicated that the intrinsic

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Table 2

Correlation coefficient matrix of total sulfur and other selected soil properties in soil profiles.

<table>
<thead>
<tr>
<th></th>
<th>TS</th>
<th>TSS</th>
<th>TC</th>
<th>TN</th>
<th>SOM</th>
<th>pH</th>
<th>Salinity</th>
<th>BD</th>
<th>WC</th>
<th>Sand</th>
<th>Slit</th>
<th>Clay</th>
<th>Depth</th>
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<tr>
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<tr>
<td>TSS</td>
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<tr>
<td>TC</td>
<td>0.527</td>
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<tr>
<td>TN</td>
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<td>0.802*</td>
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<tr>
<td>SOM</td>
<td>0.502*</td>
<td>0.116</td>
<td>0.759*</td>
<td>0.696*</td>
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<tr>
<td>pH</td>
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<tr>
<td>Salinity</td>
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<td>0.257</td>
<td>0.181</td>
<td>0.113</td>
<td>0.196</td>
<td>−0.331*</td>
<td>1.000</td>
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<tr>
<td>BD</td>
<td>0.067</td>
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<td>0.041</td>
<td>−0.270*</td>
<td>0.082</td>
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<tr>
<td>Sand</td>
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<td>−0.288*</td>
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<td>−0.466*</td>
<td>−0.509*</td>
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<td>0.113</td>
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<td>−0.432*</td>
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<td>0.161</td>
<td>−0.076</td>
<td>0.042</td>
<td>1.000</td>
</tr>
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</table>

TS: total sulfur; TSS: total sulfur stock; TC: total carbon; TN: total nitrogen; SOM: soil organic matter; BD: bulk density; WC: water content.

* p < 0.05 level significant correlation.

** p < 0.01 level significant correlation.
relationships between these nutrients as they are integral components of soil organic matter and exist in organic matter in certain proportion (Wang et al., 2006).

Correlation coefficient values (Table 2) indicated significant and positive correlation of TS, clay (p < 0.05) and slit (p < 0.01), but negative correlation with sand (p < 0.01). These results are in conformity with earlier reports (Mishra et al., 1990; Tripathi et al., 1997). Lin et al. (2009) also showed that when the soil contained a greater amount of finer-sized particles, the contents of clay minerals are higher, then the specific surface area is larger and has a higher adsorption capacity of elements.

The negative correlation between the soil pH and the TS content could be explained by the fact that a lower pH will improve the oxidation of FeS2 and FeS, which would contribute to a decreased in the soil S storage (Lin et al., 2009). Hao and Wang (2004) also found that the content of TS decreased with the increase of soil pH value in typical wetland soils in the Sanjiang Plain. Additionally, a significantly positive correlation between BD and TSS was also observed in this study (Table 2; Fig. 8). Sun et al. (2013) also proved that high bulk density is conductive to sulfur storage in wetland soils.

In this study, the WC was positively correlated with TS (Table 2), which is inconsistent with the result of Sun et al. (2013), who presented that the SO42− in topsoil could be easily deoxidized which might induce significant S loss. However, the salinity was significant and positive correlation with TS (p < 0.01), which was in agreement with the conclusion reported by Zeng et al. (2010) and Lin et al. (2009). Therefore, soil properties such as SOM, TC, TN, clay, pH and salinity were the main factors influencing soil TS distribution in the Yellow River Delta.

5. Conclusions

This paper investigated spatial and seasonal distribution patterns of soil S content and stock and their influencing factors along a hydrological gradient in two belts of the Yellow River Delta in two seasons in this study. Results have demonstrated that Site A1 and Site A2 that were located near the Yellow River had lowest soil TS contents. While Site C1 and Site D1 in Zone N had the higher TS contents, which were located near the tidal creek. Additionally, Site B2 in Zone S had the higher TS contents due to its higher water content and plant S cycle. TS stock in this region was significantly higher in fall. Moreover, vertical distributions of sulfur in two belts in two sampling dates showed a decrease with depth or shallower vertical distribution. A correlation analysis indicated that the S distribution was dependent on the properties of the soil. High SOM, TC, TN, salinity and slit were significantly correlated with soil S contents, while pH value was significantly negative with soil S contents. Generally, the changes of TS contents at spatial and temporal scales might be controlled dominantly by plant cycling and sulfur processes, which need to be further studied to testify their influencing mechanisms. Therefore, the findings of this study regarding spatial distribution patterns of soil S are quite valuable for investigating S processes in estuary wetland and discerning the relationships of ecotones with respect to their water and salinity conditions.

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