Subject Area 10: State-of-Art in China – Research Progress (Section Editor: Yong-Guan Zhu)

Vertical Distribution of Acid-volatile Sulfide and Simultaneously Extracted Metals in Mangrove Sediments from the Jiulong River Estuary, Fujian, China

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Abstract

Background. Acid-volatile sulfide (AVS) is operationally defined as sulfides in sediment, which are soluble in cold acid, and is reported as the most active part of the total sulfur in aquatic sediments. It is a key partitioning phase controlling the activities of divalent cationic heavy metals in sediment.

Methods. In order to examine this in mangrove environments, six sites were selected along the Jiulong River Estuary in Fujian, China, which had previously been reported to be polluted by heavy metals. Sediments were sampled from 0–60 cm depth at each site, and the spatial distribution of AVS and SEM (simultaneously extracted metals: copper, cadmium, zinc, and lead) were determined.

Results and Discussion. The results indicate that the AVS concentrations had a spatial variation, ranging from 0.24 to 16.10 µmol g^{-1} sediment dry weight. The AVS concentration in the surface layer is lower than that of the deeper sediment, with peak values in the 15–30 cm horizon. There was no correlation between the AVS value and organic matter content or total dissolved salts, but a significant positive correlation of AVS with surface sediment (0–5 cm) moisture content was found. This indicates that water logged sediments tend to have a high AVS value. The amount of SEM was within the range of 0.33–2.80 µmol g^{-1} sediment dry weight and decreased with sediment depth.

Conclusions. There was a marked variation in AVS and SEM among different sites studied. AVS concentrations were generally lower in the surface sediments, while SEM concentrations slightly decreased with the depth. Higher concentrations of SEM found in the upper layers of the sediments confirm the earlier suggestions that this study area may suffer from increasing heavy metal pollution.

Recommendations and Perspectives. When monitoring environmental impacts by using AVS, the micro and large-scale spatial variation as well as vertical distribution need to be estimated to avoid misleading results. Both AVS and SEM concentrations in different sediment layers should be taken into account in assessing the potential impact of heavy metals on the biotic environment.

Keywords: Acid-volatile sulfide (AVS); China; heavy metals; Jiulong River Estuary; mangroves; sediment; simultaneously extracted metals (SEM)

Introduction

In recent years, much attention, with respect to predicting the potential bioavailability of divalent toxic heavy metals in anoxic sediments, has been given to the importance of Acid Volatile Sulfide (AVS) (Ankley et al. 1991, Di Toro et al. 1992, Hansen et al. 1996). Acid-volatile sulfide, which is operationally defined as sediment sulfides that are soluble in hydrochloric acid (Howard and Evans 1993), has been proposed as a possible indicator of metal toxicity since it can effectively react with metals to make them unavailable to biota (Liber et al. 1996). Many studies which were based on field collection or laboratory-spiked sediments have revealed that the molar ratio of AVS and SEM (simultaneously extracted metals: which usually contain cadmium, copper, lead, zinc and nickel) is able to predict the availability of various heavy metals for different organisms (Ankley et al. 1991, Di Toro et al. 1992, Hansen et al. 1996, Liber et al. 1996). However, some other studies have shown that AVS concentration has seasonal and spatial variation (Besser et al. 1996, Howard and Evans 1993, Mackey and Mackay 1996, Oehm et al. 1997), and this variability has been recognized as a problem in applying AVS normalization to metal bioavailability in aqueous sediments (Besser et al. 1996). This spatial variation may be expected to occur in inter-tidal situations such as salt marshes and mangroves where seasonal changes and sediment spatial heterogeneity are also commonplace (Mackey and Mackay 1996).

Mangroves are an important inter-tidal habitat usually found in tropical and subtropical coasts, and are recognized as playing an important role in protecting embankments against tides, removing pollutants. However, mangroves are often at risk from heavy metal pollution due to a variety of anthropogenic, metal-producing activities, ranging from shipping, port development and manufacturing to sewage and storm water discharge (Mackey and Mackay 1996). Mangrove sediments act as sinks and sources of heavy metals because of their high sulfide content. The present study therefore aimed at investigating AVS and SEM values in a subtropical mangrove forest in the Jiulong River Estuary, China and their distribution through the sediment profiles. We used these to assess the potential bioavailability of sediment heavy metals to mangrove fauna and flora. At the same time, we assessed which stratum of sediment should be selected to measure the AVS concentration and should be used to predict the heavy metal pollution of the mangrove sediment.

1 Material and Methods

1.1 Study area

The mangrove forest studied is located in the estuary of Jiulong River (24º24′N, 117º55′E), Fujian province, Southeastern China. The area studied is about 233.3 ha, mainly consisting of a regrowth and mature *Kandelia candel* (L.) Druce community, and mixed with some other mangrove species (*Aepiceras corniculatum, Avicennia marina*). It is one of the best *K. candel* mangrove forests protected in China. The previous studies in this area have indicated that there is heavy metal pollution (Zheng et al. 1996).

1.2 Sampling and handling of sediment samples

To avoid temporal variability, samples were collected during a single low tide period on August 3, 2003. A total of six sample sites along the beach were selected and located with GPS. Four sites (site1, site 3, site 4 and site 5) were located in the mangrove forest, with another two sites (site 2 and site 6) being located in the barren beach without mangrove plants. The vegetation and location of sample sites are described in **Table 1**.

At each sample site, three replicate sediment cores (within a 5 m×5 m area) from 0–60 cm were taken by using PVC pipes (pre-washed with 14% nitric acid; diameter: 63 mm, length: 70 cm), and immediately capped and sealed with tape to prevent oxygen diffusion and then transferred to the laboratory where they were stored at –20°C. The sediment in the cores was drawn out and cut with a plastic knife into 0–2 cm, 2–5 cm, 5–10 cm, 10–15 cm, 15–20 cm, 20–25 cm, 25–30 cm, 30–35 cm, 35–40 cm, 40–50 cm, 50–60 cm segments under nitrogen gas (N_2). Note that, in some cores, the bottom end of the core dropped out of the pipe during extraction, and so only 0–40 cm or 0–50 cm cores could be utilized.

1.3 Sediment analysis

Analyses of AVS and SEM concentration in sediments followed the cold-acid purge-and-trap method of Allen et al. (1993) and were conducted within two weeks of sampling. Percent recovery was estimated from the analyses performed on a standard prepared from appropriate quantities of standardized Na_2S solution. The mean percent recovery (±1S.D.) was 87.27±6.7% (n=5). The amount of AVS was determined spectrophotometrically at 670 nm, and the solubilized metals (SEM) were determined by atomic absorption spectrophotometer (AAS, Model AA-6800, Shimadzu, Kyoto). The moisture content of the sediment samples, necessary to calculate AVS and SEM levels on the basis of dry weight, was

determined by measuring the weight loss after drying at 105°C for 24 h at the same time.

After the sediments had been air dried, their physical-chemical parameters were analyzed based on standard methods (Lu 1999). The pH (pH-H_2O) values were measured in a 1:5 solid:liquid suspension. Cation exchange capability (CEC) was determined by using sodium acetate and ammonium acetate solutions, and the organic matter content was determined by oxidation with potassium dichromate. The particle size distribution was measured with a hydrometer, and was fractionated into 3 size classes: sand (2 mm–50 μm), silt (50–1 μm) and clay (<1 μm). Total dissolved salt content was measured by the mass method (water: sediment=5:1). All the values are the mean of three replicates from the same site. All reagents were Merck analytical grade or Suprapur quality, and all materials (bottles, filters, etc.) were acid-cleaned prior to use.

1.4 Statistical analysis

All results presented and discussed are based on mean values and standard deviation (S.D.) of three replications. All statistical analysis was carried out by using the SPSS statistical software, and statistically significant differences between groups were tested using DMRT and LSD.

2 Results and Discussion

2.1 Physical-chemical parameters of sediments

A total of 177 sediment samples were collected and analyzed. The means of some general physical-chemical characteristics of the sediments at different sites and depths were as follows: Site 1 (0–40 cm sediment profile) – mean moisture 45.34% (range: 44.03–48.40%), pH 6.75 (5.70–7.61), clay 19.38% (16.14–23.74%), organic matter 2.86% (2.12–3.50%), CEC 16.81 cmol kg^{-1} (15.51–18.20 cmol kg^{-1}), and total dissolved salt 1.96% (1.59–2.15%); Site 2 (0–40 cm sediment profile) – moisture 41.51% (40.41–43.28%), pH 7.22 (7.03–7.45), clay 16.90% (15.97–17.63%), organic matter 2.12% (1.60–2.33%), CEC 16.53 cmol kg^{-1} (14.88–18.01 cmol kg^{-1}), and total dissolved salt 1.17% (0.94–1.71%); Site 3 (0–60 cm sediment profile) – moisture 42.77% (40.01–46.28%), pH 6.65 (6.00–7.13), clay 25.09% (22.14–26.65%), organic matter 2.47% (1.61–3.02%), CEC 16.40 cmol kg^{-1} (15.01–17.85 cmol kg^{-1}), and total dissolved salt 1.61% (1.36–1.85%); Site 4 (0–50 cm sediment profile) – moisture 44.08% (41.34–49.10%), pH 6.42 (5.36–6.97), clay 24.28% (22.33–26.37%), organic matter 2.30% (1.71–2.71%), CEC 14.81 cmol kg^{-1} (13.60–18.68 cmol kg^{-1}), and total dissolved salt 1.65% (1.43–1.82%); Site 5 (0–40 cm

Table 1: The description of sample sites

Location	Site	Elevation (m)	Vegetation description
Tiantou	1: 24º23.690′N, 117º55.469′E	1.27	Pure *Kandelia candel* forest, heights: 2–3 m, base diameter: 5–6 cm
Chennei	2: 24º23.657′N, 117º55.185′E	1.89	Barren sediment, located at the *Kandelia candel* forest edge (5 m), heights: 1–1.5 m, base diameter: 3–4 cm
	3: 24º23.694′N, 117º54.791′E	1.45	Pure *Kandelia candel* forest, heights: 4–5 m, base diameter: 7–8 cm
	4: 24º23.665′N, 117º54.551′E	1.13	Mainly *Kandelia candel* plants; some *Bruguiera gymnorrhiza* plants mixed, heights: 2–3 m, base diameter: 5–6 cm
Chaoputou	5: 24º23.621′N, 117º54.290′E	1.32	Man-made *Kandelia candel* forest, heights 1.5–2 m, base diameter 2–3 cm
	6: 24º23.345′N, 117º54.107′E	0.86	Barren sediment. Low tide zone

sediment profile) – moisture 44.01% (43.45–44.87%), pH 6.73 (6.13–7.05), clay 20.08% (17.78–22.54%), organic matter 2.11% (1.27–2.34%), CEC 15.77 cmol kg^{-1} (15.00–16.65 cmol kg^{-1}), and total dissolved salt 1.16% (0.83–1.44%); and Site 6 (0–60 cm sediment profile) – moisture 48.37% (43.51–53.16%), pH 6.61 (5.93–7.23), clay 14.86% (13.12–16.54%), organic matter 2.30% (1.84–2.75%), CEC 15.25 cmol kg^{-1} (13.00–17.19 cmol kg^{-1}), and total dissolved salt 0.78% (0.46–1.45%).

At each sample site, the moisture content showed a trend of decreasing with increase in sediment depth. This was consistent with the findings of Martynova (2003), and may be related to the variation of bulk density and porosity of sediment with depth. The pH values of most layers of the sediment were acidic, with only some pH values of surface samples being higher than 7.0. Mangrove sediment had a relatively lower pH value than that of the barren sediment. In vertical distribution, pH values showed a decreasing tendency with increase in sediment depth. This may be related to the active microbial decomposition of mangrove litter and hydrolysis of tannin in mangrove plants which releases various kinds of organic acids, and oxidation of FeS_2 and FeS to H_2SO_4 (Tam and Wong 2000). Organic matter (O.M.) content had a higher variability among the replicates (3.73–33.83%), and this may due to micro-scale heterogeneity in the sediment. There was no relationship between O.M. content and sample site location or sediment depth. However, the site closer to the water usually had relatively higher salt contents. At the same time, the surface sediments had higher salt concentrations than the deeper sections. Sediments at all six sites were composed mostly of silt and clay with little sand.

2.2 The vertical distribution of AVS and SEM

The spatial variations of AVS and SEM in the sediments of the six sample sites are shown in **Fig. 1**. The results indi-cated that AVS concentrations showed spatial variation and ranged from 0.24 to 16.10 µmol g^{-1} sediment dry weight. The AVS concentration in the surface layer was lower than that of deeper layers, and had its peak value 15 to 30 cm below the surface. These results were consistent with the findings of some other reports of variation in AVS concentration with depth (Howard and Evans 1993, Oehm et al. 1997). Considering the strongly reducing nature and high sulfide content of mangrove sediments (Silva et al. 1990), as well as the sample season (August) and related research results (0.33–22.61 µmol g^{-1} in surficial sediments, Mackey and Mackay 1996), higher AVS values in the study area sediments were expected. However, the AVS values in the present study were very low compared with those in literature. The concentration of AVS in sediments is determined both by the rate at which AVS is produced and by the rate at which it is lost by oxidation or diffusion. The supply of organic matter, the rate of SO_4^{2-} reduction, and the redox status of the sediments affect AVS concentrations. In the present study the redox status may be most important, and this will be determined by a number of factors. Firstly, surface sediments are subjected to daily flooding by well buffered seawater (Clark et al. 1998), and in order to sample different areas within the tide zone, sampling was conducted at low water, the presence of oxygen will result in the oxidation of sulfide (Zehl and Einax 2005). Secondly, mangrove sediment is an important feeding site for many aquatic organisms and bioturbation by benthic organisms may change the redox condition, and reduce the AVS concentration (Peterson et al. 1996, Williamson et al. 1999). Finally, sediment aeration by oxygen released from mangrove roots may increase the Eh value in the deeper layers (Clark et al. 1998), and reduce AVS (Almeida et al. 2005).

Analysis of variance of the AVS concentration among different sample sites and different sediment layers indicated that there was a significant difference between sites at the same
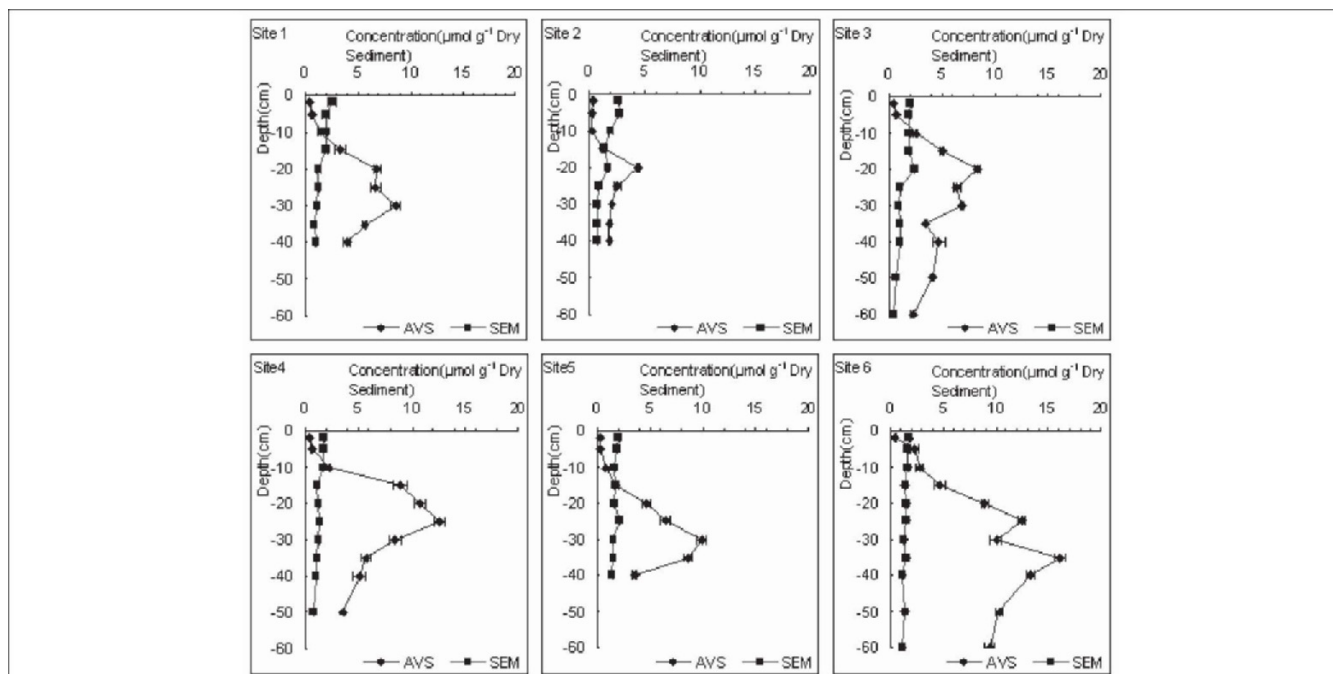

Fig. 1: Sediment profiles of AVS and SEM at six sample sites from the Jiulong River Estuary of Fujian, China

Table 2: Correlation coefficient of AVS with sediment moisture content, organic mater content and total dissolved salt content, Jiulong River Estuary, China (n=6)

Depth (cm)	Moisture content	Organic matter	Total dissolved salt
0–2	0.89*	0.75	0.06
2–5	0.82*	0.14	−0.416
5–10	0.71	0.09	0.14
10–15	0.22	0.52	0.42
15–20	0.01	0.44	0.21
20–25	0.77	−0.15	−0.08
25–30	0.7	0.1	0.04
30–35	0.86 *	−0.03	−0.61
35–40	0.3	−0.04	−0.6

* The mean difference is significant at the 0.05 level

layer. Multiple comparisons (DMRT and LSD) showed that site 2 had a relatively low AVS concentration, and usually had a significant difference with the other sites at the same depth. Site 6 usually had the highest AVS concentration except at 10–15 cm and 15–20 cm depths. Both site 2 and site 6 were located at the edge of the mangrove forest. However, site 6 was situated at the low tide mark and had the highest moisture content, while site 2 was at a higher elevation and had the lowest moisture content of the six sample sites. Correlation coefficients revealed, however, no significant relationships found between AVS value and organic matter content or salt content. A significantly positive relationship between AVS values and surface sediment moisture content across different sites (**Table 2**) indicated that water logged sediment tended to have a high AVS value (Mackey and Mackay 1996).

SEM values slightly decreased with sediment depth (see Fig. 1). This result was consistent with previous reports (Yu et al. 2001), and may be attributed to the higher total heavy metal content (data not shown), higher organic matter content and less anoxic conditions in surficial sediment. Hansen et al. (1996) believed that the difference between the molar concentrations of SEM and AVS ([SEM]–[AVS]) could provide important insights into the extent of additional available binding capacity. The differences between the molar concentrations of SEM and AVS in this study were shown in **Fig. 2**. In surface sediments, the differences were positive, and then decreased with depth. Between 20 and 35 cm below the surface, it reached its peak negative value, and then increased with depth. In the surface layer of the sediments, the amount of AVS was too low to bind all reactive metals as sulfides. In the deeper layers

of the sediment, owing to the low Eh values and long deposition time, sulfide may be transformed into sulfate, which is too stable to bind reactive metals.

3 Conclusions

There was a marked variation in AVS and SEM among different sites studied. AVS concentrations were generally lower in the surface sediments, while SEM concentrations slightly decreased with the depth. The AVS value of surface sediment was related to its moisture content, and water logged sediments tended to have a high AVS value.

Higher concentrations of SEM were found in the upper layers of the sediments. The differences between the molar concentrations of SEM and AVS ([SEM]–[AVS]) were positive in surface sediments, these confirmed the earlier suggestions that this study area may suffer from increasing heavy metal pollution.

The differences of both AVS and SEM concentrations among different sites or sediment layers indicated that when monitoring environmental impacts by using AVS, the micro and large-scale spatial variation as well as vertical distribution need to be estimated to avoid misleading results.

4 Recommendations and Perspectives

When monitoring environmental impacts by using AVS, the micro and large-scale spatial variation as well as vertical distribution need to be estimated to avoid misleading results. Both AVS and SEM concentrations in different sediment layers should be taken into account in assessing the potential impact of heavy metals on the biotic environment.
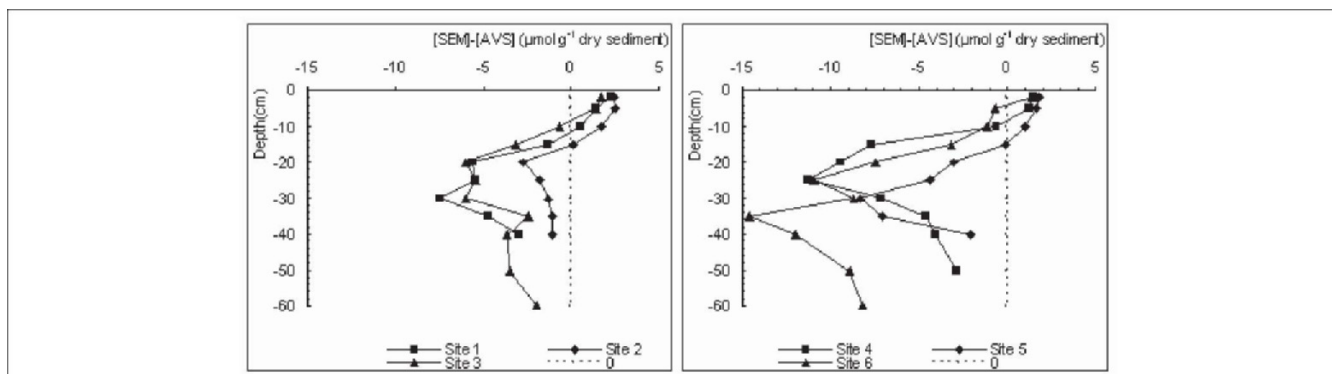

Fig. 2: Variation of the difference between the molar concentrations of SEM and AVS ([SEM]–[AVS]) with the sediment depth in mangrove sediments from the Jiulong River Estuary, China

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The Role of a Salt Marsh Plant on Trace Metal Bioavailability in Sediments
Estimation By Different Chemical Approaches *

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Abstract

Goal, Scope and Background. The presence or absence of vegetation can condition sediment characteristics. The main aim of this work was to investigate the influence of the sea rush *Juncus maritimus* on metal (Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn) availability to organisms living on or in estuarine sediments, from Douro River (NW Portugal), by comparing the characteristics and chemical behaviour of rhizosediments (collected within the plant assemblage) and those of sediment (collected around the plant). In order to evaluate whether and how sediment characteristics condition the role of plants on metal availability, sandy and muddy sediments colonised by *J. maritimus* were studied in parallel.

Methods. Metal availability was estimated by enzymatic digestion with pepsin (ED), which may provide an estimate of metal availability to organisms living at estuarine sediments. Nevertheless, since no consensus exists yet on the most suitable methodologies to estimate metal bioavailability in sediments, two more conventional approaches, BCR sequential extraction (SE) and AVS/SEM model, were also used, in parallel, and the information these approaches provided was compared with that provided by ED. Total-recoverable metal contents were determined by atomic absorption spectrophotometry after sediment digestion using a high-pressure microwave system.

Results and Discussion. Plants could concentrate metals around its roots and rhizomes. In addition, they were capable of oxidizing (release of oxygen by the roots) the anaerobic medium surrounding their roots in muddy sediment (reducing AVS). As sulphide oxidation renders metals (Cd, Cu, Ni, Pb and Zn) into more soluble forms, according to the AVS/SEM model, metals from muddy sites would be more available in rhizosediment than in sediment. The SE approach led to a similar conclusion. Nevertheless, the results provided by ED pointed at opposite conclusion, particularly for Cd and Zn, indicating less availability at rhizosediments than in the surrounding sediment. ED results were interpreted as a consequence of an enrichment of the rhizosediment in organic ligands exuded by the roots or liberated by dead plants. The effect of complexation of metals by organic compounds, which ED could not decompose/dissolve, seemed to overcome that caused by sediment oxidation. In general, a comparison of the information about metal availability by ED, SE, AVS/SEM, showed that it did not always match and in few cases it was even contradictory.

Conclusion and Outlook. Therefore, a thorough evaluation of the metal availability in sediments requires a combination of different chemical approaches, so as to take into consideration differences in ways of organism exposure (interstitial water and/or ingestion of sediment particles).

Keywords: Enzymatic digestion; estuarine sediments; *Juncus maritimus*; metal availability; salt marsh plants; sequential extraction