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Elemental sulfur in drain sediments associated with acid sulfate soils

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Abstract

This paper reports the abundance of elemental S in drain sediments associated with acid sulfate soils. The sediments exhibited near-neutral pH (5.97–7.27), high concentrations of pore-water Fe^{2+} (1.37–15.9 mM) and abundant oxalate-extractable Fe (up to 4300 $\mu mol\ g^{-1}$). Maximum acid-volatile sulfide (AVS) concentrations in each sediment profile were high (118–1019 $\mu mol\ g^{-1}$), with AVS often exceeding pyrite-S. Elemental S occurred at concentrations of 13–396 $\mu mol\ g^{-1}$, with the higher concentrations exceeding previous concentrations reported for other sedimentary systems. Up to 62% of reduced inorganic S near the sediment/water interface was present as elemental S, due to reaction between AVS and oxidants such as O_2 and Fe(III). Significant correlation ($r = 0.74$; $P < 0.05$) between elemental S and oxalate-extractable Fe(III) is indicative of elemental S formation by in situ oxidation of AVS. The results indicate that AVS oxidation in near-surface sediments is dynamic in acidified coastal floodplain drains, causing elemental S to be a quantitatively important intermediate S fraction. Transformations of elemental S may therefore strongly influence water quality in ASS landscapes.
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1. Introduction

Sulfidic soils, also termed acid sulfate soils (ASS), underlie large areas of coastal floodplains and are often highly acidic as a result of pyrite oxidation (Dent, 2000). In eastern Australia, ASS landscapes have been intensively modified through the construction of drain networks. The benthic sediments in these drains are known to contain reactive sulfides (measured as acid-volatile sulfide; AVS) at levels that greatly exceed those found in other systems (Bush et al., 2004).
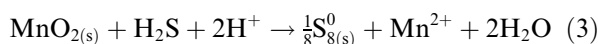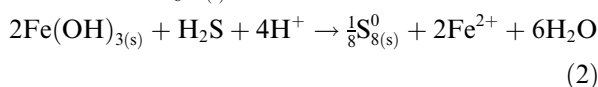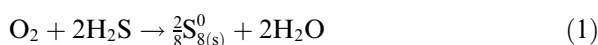
Water in ASS drains is typically acidic, with elevated Fe, SO_4^{2-} and trace metal concentrations (Keene et al., 2003; Johnston et al., 2004). Sulfate reduction and formation of sulfide minerals in drain sediments may improve water quality by sequestering Fe and other metals, and by increasing alkalinity (Evangelou, 1995; Burton et al., 2005). Accumulation of SO_4^{2-} reduction products, such as AVS and $FeS_{2(s)}$, may also represent an environmental hazard, capable of deoxygenating and acidifying overlying

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water during sediment re-suspension events (Sullivan et al., 2002; Bush et al., 2004).

Previous studies examining drain sediments from ASS landscapes have focused on the AVS and $FeS_{2(s)}$-S pools (Bush et al., 2004; Macdonald et al., 2004; Smith and Melville, 2004). Quantification of S species in these previous studies have not included the measurement of elemental S $\left(S_{8(s)}^0\right)$. However, King (1988) and Thode-Andersen and Jørgensen (1989) report that $S_{8(s)}^0$ may be the most abundant short-term SO_4^{2-} reduction product in near-surface sediments. This is a result of incomplete oxidation of pore-water sulfide (produced via SO_4^{2-} reduction) by O_2, Fe^{3+} and Mn^{4+} species (Morse et al., 1987; Aller and Rude, 1988):

$$O_2 + 2H_2S \rightarrow \tfrac{2}{8}S_{8(s)}^0 + 2H_2O \quad (1)$$

$$2Fe(OH)_{3(s)} + H_2S + 4H^+ \rightarrow \tfrac{1}{8}S_{8(s)}^0 + 2Fe^{2+} + 6H_2O \quad (2)$$

$$MnO_{2(s)} + H_2S + 2H^+ \rightarrow \tfrac{1}{8}S_{8(s)}^0 + Mn^{2+} + 2H_2O \quad (3)$$

Elemental S is of particular significance to understanding sedimentary reduced inorganic S (RIS) cycling, as it is known to be important in $FeS_{2(s)}$ formation reactions involving FeS precursors (Schoonen and Barnes, 1991). Quantification of $S_{8(s)}^0$ in drain sediments is also important to managing water quality through the accurate definition of sedimentary $FeS_{2(s)}$ and the calculation of potential acid production due to oxidation of RIS. Despite this importance, the behavior of sedimentary $S_{8(s)}^0$ associated with ASS has not been previously documented.

This study provides the first detailed quantification of $S_{8(s)}^0$ in sediment profiles from coastal floodplain ASS landscapes.

2. Methods

2.1. Sediment collection and handling

Five drain sites (termed T1, T2, B1, B2, and SC), characterized by Fe- and SO_4^{2-}-rich acidic water, were identified from previous research (Keene et al., 2003; Bush et al., 2004; Johnston et al., 2004). The study sites were on coastal floodplains of three major rivers in eastern Australia (Fig. 1). Sites T1 and T2 (28°57′S, 153°23′E) were located on the Richmond River floodplain, sites B1 and B2 (28°19′S, 153°26′E) on the Tweed River floodplain, and site SC (29°30′S, 153°15′E) on the Clarence River floodplain.

Sediment cores (internal diameter of 10 cm; 22 cm length) were retrieved using a push-tube coring device and sectioned (within 4 h of collection) into 2 cm or 4 cm segments using a stainless steel spatula. The depth segments were sectioned rapidly (<2 min) and immediately placed into 50 mL polypropylene vials. The vials were completely filled with sediment and sealed with gas-tight screw-caps. Possible oxidation of reduced species was minimized by rapid sediment sectioning, avoidance of unnecessary sediment disturbance, and by transporting the sediment-filled vials on ice under an inert (N_2) atmosphere.

2.2. General sediment characterization

Sediment preparation for pore-water analysis and RIS speciation was performed under N_2 in order to minimize sediment oxidation. Pore-water was extracted, within 24 h of sediment collection, by centrifugation ($2000g$, 20 min). The extracted pore-water was passed through a 0.45 μm syringe-driven filter. Five milliliter of filtrate was added directly to 1 mL of 20% ZnOAc followed by addition of four drops of 6 M NaOH to preserve pore-water sulfide species (which include H_2S, HS^-, S^{2-}, and aqueous sulfide complexes; collectively denoted as $\sum S_{(aq)}^{2-}$). Pore-water $\sum S_{(aq)}^{2-}$ was subsequently determined by the methylene blue method (Cline, 1969). Aliquots of 0.100–2.00 mL of filtrate were added directly to 1,10-phenanthroline solutions for Fe^{2+} analysis (APHA, 1998). Pore-water SO_4^{2-} concentrations in the remaining filtrate were calculated from S analysis by inductively coupled plasma – atomic emission spectrometry (Perkin-Elmer DV4300), taking into account the concentration of pore-water $\sum S_{(aq)}^{2-}$. Pore-water retrieval and filtration was performed in less than 30 s for each sample to avoid oxidation of reduced pore-water species due to atmospheric exposure.

Sediment moisture content was determined by weight loss due to drying at 105 °C. Sediment pH and Eh (reported versus the standard hydrogen electrode; SHE) were determined, within 4 h of sample collection, by direct insertion of calibrated electrodes into the sediment sample. Total C was determined on oven-dry sediment samples using a LECO-CNS 2000 induction furnace analyzer. Reactive solid-phase Fe(II) and Fe(III) species were extracted (in duplicate) using an anoxic oxalate-extraction procedure (Phillips and Lovley, 1987). This procedure extracts Fe(II) from FeS, $FeCO_3$ and crystalline oxides, and Fe(III) from poorly
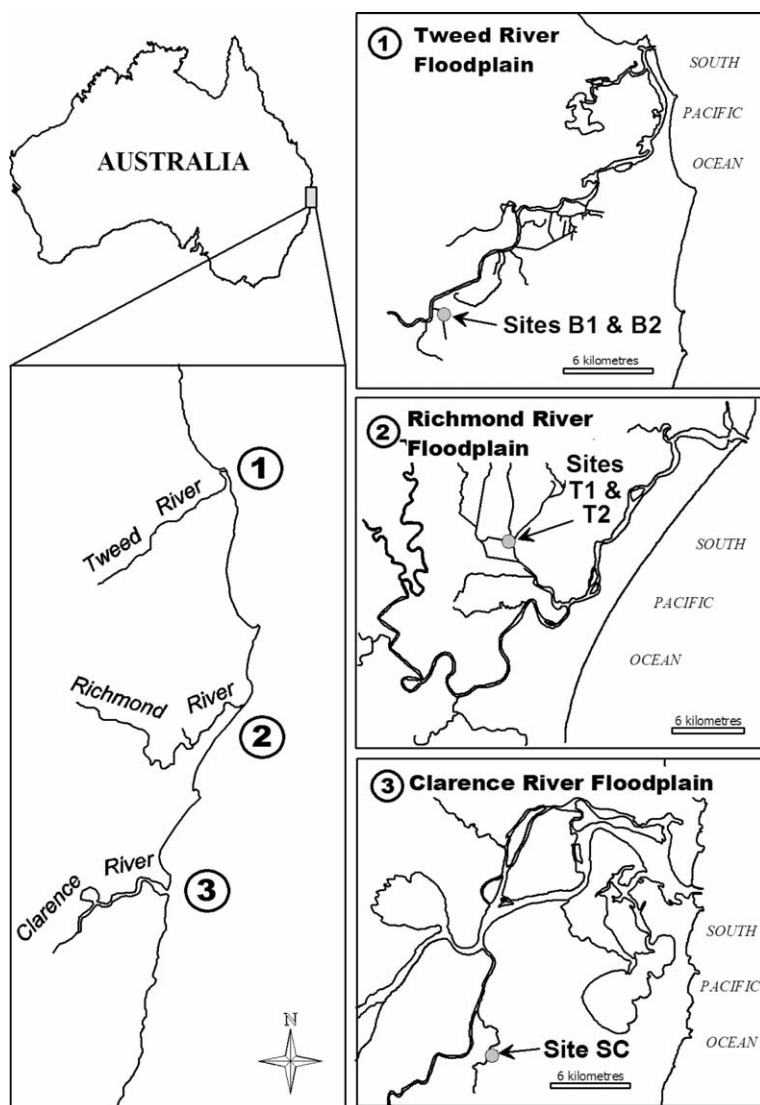
Fig. 1. Location of sites T1, T2, SC, B1, and B2.

crystalline oxides without changing the Fe oxidation state (Phillips and Lovley, 1987; Kostka and Luther, 1994; Thamdrup et al., 1994). Concentrations of oxalate-extractable Fe species were determined using the 1,10-phenanthroline method (APHA, 1998).

Results for all solid-phase properties are presented on a dry weight basis.

2.3. Reduced inorganic S speciation

Acid-volatile sulfide (AVS), $S_{8(s)}^0$ and FeS_2-S were extracted (in duplicate) using a 3-step sequential extraction procedure (Neretin et al., 2004; Burton et al., 2006, in press). This procedure was performed with sediment samples that had been stored frozen, under N_2, for no longer than 4 weeks.

Acid-volatile sulfide was extracted by the cold diffusion procedure, described by Hsieh et al. (2002). These researchers demonstrated that the use of ascorbic acid prevents oxidation of liberated H_2S, to $S_{8(s)}^0$ by Fe^{3+} species during AVS extraction, whilst also preventing extraction of S from $FeS_{2(s)}$. To check that $S_{8(s)}^0$ was not formed during the AVS extraction, the $S_{8(s)}^0$ concentration was determined (as described below) prior to, and following AVS extraction for samples from 0 to 2 cm and 2 to 4 cm depth intervals. The $S_{8(s)}^0$ concentrations
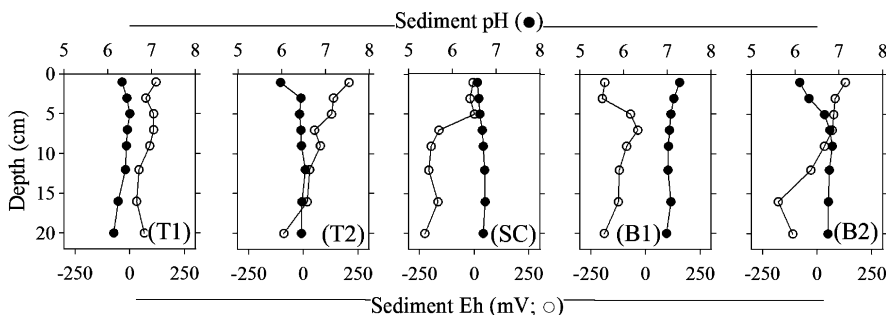
Fig. 2. Sediment pH and Eh (mV, SHE) at sites T1, T2, SC, B1, and B2.

did not change significantly (paired *t*-test; $P > 0.1$) as a result of AVS extraction.

The sediment slurry remaining after AVS extraction was quantitatively transferred to an acetone-washed 50 mL vial, centrifuged (3000g, 10 min) and the supernatant discarded. Elemental S was extracted by shaking the residual sediment with 20 mL of acetone for 24 h (Wieder et al., 1985), followed by a further 10 mL acetone rinse. The acetone-sediment slurry was separated by centrifugation (3000g, 10 min) and the supernatant was transferred to a 250 mL conical flask. The acetone extract was allowed to vaporize to near-dryness at room temperature. Reduced inorganic S was then extracted from the acetone residue by hot, acidified, Cr-reduction (Sullivan et al., 2000). Elemental S is operationally-defined as acetone-extractable, reduced inorganic S (Wieder et al., 1985; White et al., 1989; Neretin et al., 2004).

The acetone-extracted sediment was quantitatively transferred (rinsing twice with 10 mL of 96% ethanol) to a 250 mL conical flask. Chromium-reducible S (CRS) was extracted following Sullivan et al. (2000). This procedure selectively extracts inorganic S with oxidation states $<+6$. When performed after AVS and $S_{8(s)}^0$ extraction, the CRS procedure selectively extracts S from $FeS_{2(s)}$.

The quantity of S, corresponding to each of the three RIS species, was determined via iodometric titration (APHA, 1998).

3. Results and discussion

3.1. General sediment properties

Sediment profiles from sites B2, T2, and SC were overlain by approximately 20–40 cm of acidic (pH 3.5–5) water. Sites T1 and B1 were collected from drain edges and were water saturated (containing 62.1–84.7% water for B1, and 69.8–76.9% for T1), but not submerged. Sediment pH ranged from 5.97 to 7.27 (Fig. 2), and varied little at each site. Sediment Eh generally decreased with increasing depth (Fig. 2), indicating a transition to more reducing, anoxic conditions at greater depth. The development of strongly reducing conditions can be attributed to bacterial degradation of abundant sedimentary organic material (reflected in high mean total C contents of 7.3% at B1, 5.9% at B2, 13.4% at T1, 11.7% at T2, and 7.2% at SC).

The pore-water SO_4^{2-} concentrations were large (1.37–15.9 mM) near the sediment surface, decreasing markedly with depth (Fig. 3). Relatively low SO_4^{2-} concentrations (<0.2 mM) in the lower depth
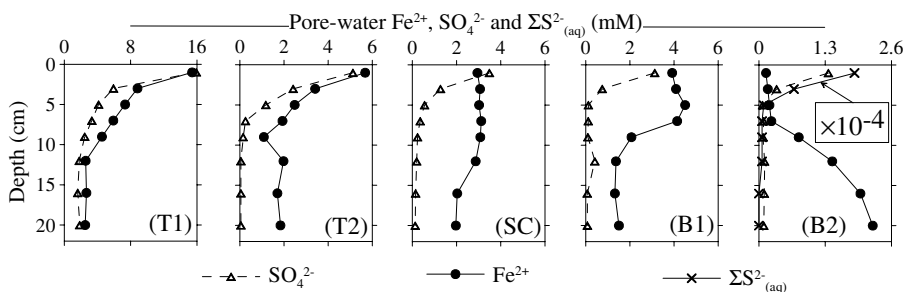

Fig. 3. Pore-water Fe^{2+}, SO_4^{2-} and $\sum S_{(aq)}^{2-}$ at sites T1, T2, SC, B1, and B2. Pore-water $\sum S_{(aq)}^{2-}$ was present at <0.5 μM at all sites, except site B2.

intervals are indicative of SO_4^{2-} consumption by microbially-mediated SO_4^{2-} reduction. Although $\sum S_{(aq)}^{2-}$ is a primary product of SO_4^{2-} reduction, pore-water $\sum S_{(aq)}^{2-}$ was undetectable (i.e., $<0.5\,\mu M$) at all but one location. Measurable pore-water $\sum S_{(aq)}^{2-}$ concentrations occurred in the upper-most sediment layers at Site B2, with $18.8\,\mu M$ near the sediment-water interface decreasing to $<0.5\,\mu M$ at 8 cm (Fig. 3).

The generally low pore-water $\sum S_{(aq)}^{2-}$ concentrations may be due to high Fe^{2+} concentrations (i.e., >2 mM, except at site B2; Fig. 3) and subsequent precipitation of FeS. The dependence of $\sum S_{(aq)}^{2-}$ concentrations on pore-water Fe^{2+} (i.e., $[HS^-] = [H^+]K_{sp}/[Fe^{2+}]$, where $K_{sp} = 10^{-2.95}$ for amorphous FeS; Morse et al., 1987) suggests that

pore-water $\sum S_{(aq)}^{2-}$ is sequestered as FeS in drain sediments from ASS landscapes.

Oxalate-extractable Fe(III) at sites B1, T1, T2, and SC ranged from approximately 2000–4000 $\mu mol\,g^{-1}$ near the sediment surface, with lesser concentrations occurring at site B2 (Fig. 4). The contribution of Fe(II) to oxalate-extractable Fe generally increased with depth, which probably reflects reductive dissolution of Fe(III) oxides under reducing conditions.

3.2. Reduced inorganic S speciation

Maximum AVS concentrations at each site ranged from 118 to 1019 $\mu mol\,g^{-1}$ (Fig. 5). These AVS concentrations are comparable with Bush
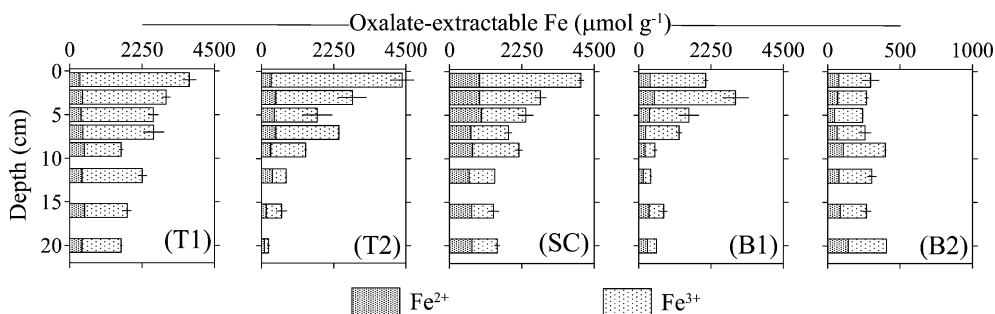

Fig. 4. Oxalate-extractable Fe(II) and Fe(III) in sediments at sites T1, T2, SC, B1, and B2. The data are means of duplicate analyses, with errors bars showing ±1 standard deviation.
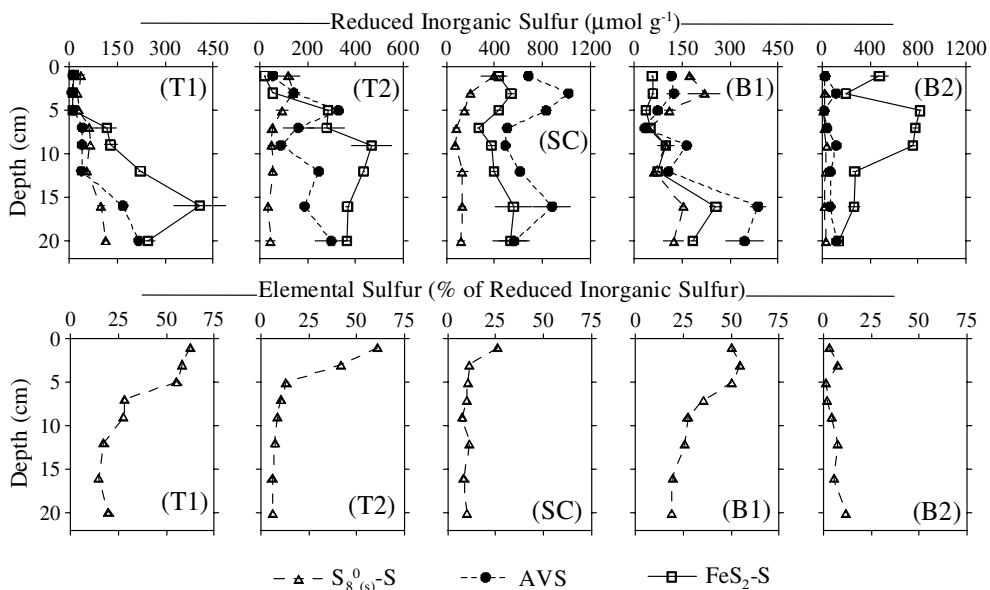

Fig. 5. Vertical distribution of reduced inorganic S species ($S_{8(s)}^0$, AVS and FeS$_{2(s)}$) in sediments at sites T1, T2, SC, B1, and B2. Data points are the means ± standard deviation of duplicate analyses.

et al. (2004), who found approximately 150–2000 $\mu mol\ g^{-1}$ in sediments near sites T1 and T2. In comparison, AVS is typically present in natural estuarine and marine sediments at <90 $\mu mol\ g^{-1}$ (Morse and Cornwell, 1987; Burton et al., 2005), due to efficient conversion of AVS to FeS_2-S (Morse and Rickard, 2004).

The $FeS_{2(s)}$-S concentrations in the sediments examined here ranged from 10 to 815 $\mu mol\ g^{-1}$ (Fig. 5). The ratios of $FeS_{2(s)}$-S to AVS were low (mostly ~0.5–2.0) for sites B1, T1, T2, and SC. In contrast, these ratios were higher at site B2, ranging from 1.2 to 50. Gagnon et al. (1995) suggested that $FeS_{2(s)}$-S to AVS ratios <3 indicate inefficient conversion of AVS to $FeS_{2(s)}$-S, and hypothesized that this may be related to high levels of reactive Fe. The high $FeS_{2(s)}$-S to AVS ratios at site B2 may therefore be attributed to much lower levels of oxalate-extractable Fe at this site compared to the high levels at sites T1, T2, B1, and SC (Fig. 4).

Elemental S in the five sediment profiles described here ranged from 13 to 396 $\mu mol\ g^{-1}$ (Fig. 5). These concentrations are approximately equivalent to 4.0–44.4 $\mu mol\ cm^{-3}$ on a fresh sediment volume basis (calculated from the measured sediment-water content and assuming a dry solids density of 2.5 $g\ cm^{-3}$). These $S^0_{8(s)}$-S concentrations are high in comparison to previous reports of ~2 $\mu mol\ cm^{-3}$ in salt marsh sediments (King et al., 1985), ~8 $\mu mol\ cm^{-3}$ in marine sediments from Denmark (Thode-Andersen and Jørgensen, 1989) and ~10 $\mu mol\ g^{-1}$ in mine pit lake sediments (Meier et al., 2004). It should be noted that these previously reported $S^0_{8(s)}$-S concentrations were in sediments containing substantially lower AVS contents than ASS-associated sediments. In Black Sea bottom-sediments, containing AVS levels (i.e., up to 211 $\mu mol\ AVS\ g^{-1}$) closer to those reported here, Neretin et al. (2004) found $S^0_{8(s)}$-S ranged to ~60 $\mu mol\ g^{-1}$. Similarly, Troelsen and Jørgensen (1982) found approx. 600 $\mu mol\ g^{-1}\ S^0_{8(s)}$-S in surface sediment in a sulfuretum with intensive bacterial sulfide production.

Elemental S comprised substantial proportions (up to 62%) of RIS in near-surface sediments at sites T1, T2, B1, and SC (Fig. 5). This is consistent with King (1988), who found that $S^0_{8(s)}$-S comprised >90% of short-term SO_4^{2-} reduction end-products in the 0–4 cm depth interval of South Carolina salt marsh sediments. King (1988) also found that this proportion decreased to <20% at depths greater than 8 cm below the sediment surface. Similarly,

Thode-Andersen and Jørgensen (1989) found that $S^0_{8(s)}$-S exceeded the AVS concentration and was also the most abundant short-term end-product of SO_4^{2-} reduction in the 0–1 cm depth interval of coastal marine sediments from Denmark.

The proportion of RIS as $S^0_{8(s)}$-S was strongly correlated ($r = 0.74$; $P < 0.05$) with oxalate-extractable Fe(III) (Fig. 6). This correlation is consistent with $S^0_{8(s)}$ formation due to AVS oxidation by Fe(III) (Eq. (2)). However, simultaneous oxidation of both $\sum S^{2-}$ and Fe(II) by O_2 or Mn^{4+} species may also lead to the relationship presented in Fig. 6. Future research is needed to assess the relative importance of O_2, Fe^{3+}, and Mn^{4+} as electron acceptors during oxidation of AVS to $S^0_{8(s)}$.

3.3. General discussion

In most sedimentary systems, RIS is principally composed of $FeS_{2(s)}$-S, with AVS and $S^0_{8(s)}$ comprising only a few percent of RIS (Giblin and Wieder, 1992). This study and previous research (Bush et al., 2004) has demonstrated that drain sediments from coastal, ASS landscapes contain anomalously high AVS contents. Studies in other environments have shown that a substantial proportion of AVS is partially oxidized to $S^0_{8(s)}$ in near-surface sediments (Troelsen and Jørgensen, 1982; King, 1988; Thode-Andersen and Jørgensen, 1989). Therefore, the high $S^0_{8(s)}$ concentrations reported here are probably due to the very high AVS content in ASS-associated drain sediments.

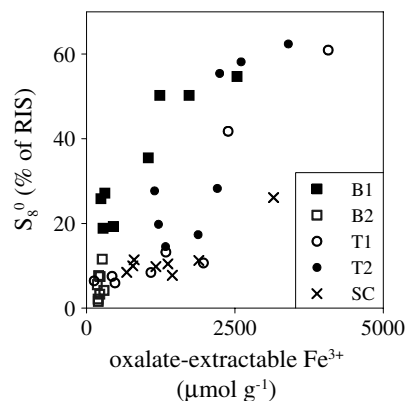Sedimentary $S^0_{8(s)}$ has been described as a very dynamic phase (Howarth and Jørgensen, 1984). It

Fig. 6. Relationship between the percentage of reduced inorganic S (RIS) as $S^0_{8(s)}$ and oxalate-extractable Fe^{3+} in sediments at sites T1, T2, SC, B1, and B2.

may be oxidized to SO_3^{2-} and SO_4^{2-}, reduced to $\sum S^{2-}$, or may be subject to disproportionation into both SO_4^{2-} and $\sum S^{2-}$ (Postgate, 1979; Canfield and Thamdrup, 1996; Bottcher et al., 2001). Elemental S may also react with pore-water $\sum S^{2-}_{(aq)}$ to produce polysulfide ions $\left(S_n^{2-}\right)$ according to (Morse et al., 1987):

$$HS^-_{(aq)} + \frac{x}{8}S^0_{8(s)} \rightarrow S^{2-}_{(n)(aq)} + H^+ \qquad (4)$$
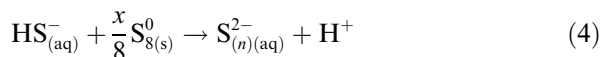
where $x = n - 1$. The S_n^{2-} species produced by this reaction are important to $FeS_{2(s)}$ formation (Luther, 1991; Schoonen and Barnes, 1991):
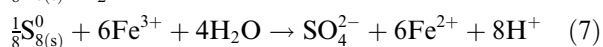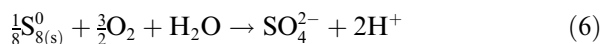
$$FeS_{(aq)} + S^{2-}_{n(aq)} \rightarrow FeS^+_{2(s)}S^{2-}_{(n-1)(aq)} \qquad (5)$$
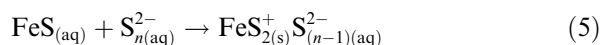
The rates of $S^0_{8(s)}$ formation and loss via these oxidation and reduction pathways have received little attention compared to other aspects of sedimentary S cycling. Troelsen and Jørgensen (1982) speculate that the turn-over time for newly-formed $S^0_{8(s)}$ is in the order of days. Therefore, the high $S^0_{8(s)}$ concentrations reported here probably reflect dynamic formation and oxidation of AVS in near-surface sediments. In this case, $S^0_{8(s)}$ would be continuously produced by AVS oxidation and consumed via oxidation to SO_3^{2-} and SO_4^{2-}, or disproportionation into SO_4^{2-} and $\sum S^{2-}$. The generally low $FeS_{2(s)}$-S to AVS ratios reported here suggest that a small proportion of $S^0_{8(s)}$ would also be lost via Eq. (4) prior to $FeS_{2(s)}$ formation. The rates and relative importance of these individual processes in ASS-associated sediments is worthy of further research.

4. Environmental implications

This study clearly demonstrates that $S^0_{8(s)}$ is often abundant in drain sediments from ASS landscapes. Elemental S is most abundant near the sediment surface, where reactive $\sum S^{2-}$ and oxidants such as Fe^{3+} are in close proximity. This is attributable to formation of $S^0_{8(s)}$ as an intermediate product of AVS oxidation. This reflects the dynamic nature of S cycling in drain sediments from ASS landscapes. As such, water quality improvements associated with SO_4^{2-} reduction and AVS formation may be reversible during early diagenesis.

Elemental S is a potential acidity source in dredged sediments or within ASS-associated waterways during sediment re-suspension events. Oxidation of $S^0_{8(s)}$ may proceed with O_2 or Fe^{3+} as electron acceptors (Burton et al., 2006):

$$\frac{1}{8}S^0_{8(s)} + \frac{3}{2}O_2 + H_2O \rightarrow SO_4^{2-} + 2H^+ \qquad (6)$$

$$\frac{1}{8}S^0_{8(s)} + 6Fe^{3+} + 4H_2O \rightarrow SO_4^{2-} + 6Fe^{2+} + 8H^+ \qquad (7)$$

The great abundance of $S^0_{8(s)}$ in ASS-associated sediments indicates that acidity budgets for ASS waterways should consider Eqs. (6) and (7). However, future research is required to verify the relative importance of acidity released via these competing $S^0_{8(s)}$ oxidation pathways. Overall, the quantitative importance of $S^0_{8(s)}$ in ASS-associated sediments indicates that this intermediate S species may influence water quality in ASS landscapes.

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