

ACIDIFICATION OF FRESHWATER WETLANDS: COMBINED EFFECTS OF NON-AIRBORNE SULFUR POLLUTION AND DESICCATION

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Abstract. In recent decades, SO_4^{2-} concentrations have increased in groundwater and surface water of freshwater wetlands. For many minerotrophic peatlands, S originating from SO_4^{2-} -polluted groundwater and surface water is a more significant source of SO_4^{2-} than the actual atmospheric deposition of S compounds. Lowered groundwater tables in wetlands, as a result of either natural or anthropogenic desiccation, may cause acidification because of concomitant geochemical oxidation processes. The impact of the enhanced availability of reduced S compounds, due to preceding SO_4^{2-} pollution, on these processes was tested in a mesocosm experiment, using soil cores including vegetation from a mesotrophic wet meadow. The soils had been maintained in waterlogged condition for seven months, using two environmentally relevant SO_4^{2-} concentrations (2 and 4 mmol L^{-1}). The groundwater table was reduced in two successive steps: 10 cm below soil surface, and complete desiccation. Control pretreated soils did not show a decrease in soil pH during desiccation, due to adequate buffering by bicarbonate. However, both SO_4^{2-} -pretreated groups showed a significant drop in pH (from 6.5 to 4.5) caused by additional sulfide oxidation, leading to high SO_4^{2-} concentrations (10 and 16 mmol L^{-1} , respectively). Cation exchange and acidification-related solubilization processes induced the mobilization of base cations and potentially phytotoxic metals like Al. Nutrient concentrations in soil moisture were influenced strongly by SO_4^{2-} pretreatment, showing distinct patterns for P, N and K. Therefore, S polluted groundwater and surface water may severely increase the sensitivity of wetlands to desiccation. The results are discussed in relation to wetland management.

Key words: sulfur, sulfate, desiccation, acidification, nutrients, water quality, pollution, wetlands

1. Introduction

The effects of increased sulfur input from atmospheric deposition on the biogeochemistry and ecological functioning of wetlands have received much attention (e.g. Gorham, 1958, 1976; Johnson, 1979; Schindler *et al.*, 1980; Cook *et al.*, 1986; Reuss *et al.*, 1987). However, in many minerotrophic freshwater wetlands, SO_4^{2-} concentrations in surface and sediment water have increased recently from less than 0.2 mmol L^{-1} to concentrations of 0.5 mmol L^{-1} and higher (over 3 mmol L^{-1} ; Lamers *et al.*, in press). It is unlikely that these values can be attributed solely to enhanced atmospheric S input. The affected wetlands receive large amounts of S from groundwater and surface water, sources contaminated by anthropogenic SO_4^{2-} dumping and weathering of marine geological deposits. Allochthonous river water, containing much higher SO_4^{2-} concentrations than the autochthonous water, is used to compensate for desiccation (Roelofs, 1991; Smolders and Roelofs, 1993; Koerselman *et al.*, 1993). Furthermore, the influx of groundwater from surrounding agricultural land supplies high amounts of SO_4^{2-} (Kölle and Schreck, 1982; Stuyfzand, 1993; Jansen and Roelofs, 1996).

In earlier studies, the eutrophication observed in peatlands receiving only low amounts of nutrients could be attributed to increased SO_4^{2-} loading from river water (Roelofs, 1991; Smolders and Roelofs, 1993). In anoxic soils and sediments, increased SO_4^{2-} reduction

rates are known to cause eutrophication via sulfide induced phosphate mobilization and/or enhancement of the mineralization rate (Ohle, 1954; Boström *et al.* 1982; Caraco *et al.*, 1989; Roelofs, 1991; Smolders and Roelofs, 1993; Lamers *et al.*, in press). A considerable amount of the resultant sulfide precipitates with metals (in particular with iron), thereby increasing the pool of reduced S in soils and sediments.

Desiccation of wet soils and sediments may be accompanied by acidification because of proton production by chemical processes, such as chemical oxidation of reduced Fe and sulfide, and by oxygen-dependent microbiological processes, like enhanced nitrification rates and oxidation of Fe and sulfide. Acidification due to oxygen availability during drought is well known from research on acid SO_4^{2-} soils (van Breemen, 1975) and acid mine drainage (Barton, 1978; Harries and Ritchie, 1983; Wieder, 1989), in areas containing high amounts of iron monosulfide (FeS) and/or pyrite (FeS_2). Acidification due to desiccation also has been shown for poorly buffered pools (Vangenechten *et al.*, 1981; Schuurkes *et al.*, 1988), in which the acid neutralizing capacity (ANC) of the sediment was insufficient to prevent the drop in pH caused by oxidation of accumulated airborne S.

In recent decades, groundwater levels in many wetlands worldwide have dropped by a few decimeters up to several meters. This is a result of extensive hydraulic operations for agricultural purposes and of increased water extraction. Consequently, the extent, duration and frequency of desiccation have increased in many wetlands and in pastures with vegetation types dependent upon a high ground water table. Furthermore, global climate change is thought to have a serious negative impact on the amount of precipitation during summer (IPCC, 1995). Desiccation therefore has become one of the principal problems for wetland conservation. The increased availability of iron sulfides, as indicated above, may severely enhance the sensitivity to dehydration, concomitantly affecting the ecological functioning of these wetlands during drought.

To evaluate the combined effects of preceding high S loads and desiccation, both on soil acidification and on general biogeochemistry, a mesocosm experiment was set up, using soil cores with vegetation from a microtrophic wet meadow. The cores had been maintained in waterlogged condition for seven months with three different environmentally relevant SO_4^{2-} concentrations (Lamers *et al.*, in press) before they were subjected to desiccation. It was hypothesized that the increased S loads severely influence soil biogeochemistry during desiccation.

2. Materials and Methods

Soil cores were collected from a mesotrophic wetland meadow in the nature reserve 'De Bruuk' near Nijmegen, The Netherlands ($51^\circ 45'N$; $5^\circ 58'E$). The upper 10 cm, used for the experiment, included moderately decomposed peat containing loam and living roots, classified as Rhizic Hydromoder (Green *et al.*, 1993). The vegetation was dominated by *Carex nigra* and could be considered *Caricion nigrae* (Ellenberg, 1988). In total, 18 sods (diameter 18 cm, depth 12 cm), including vegetation, were cut to fit tightly into plastic containers. Perforations in the bottom of the containers were covered from inside with plastic gauze to prevent loss of soil, and each container was suspended in another larger plastic container (12 L; Figure 1). The containers were placed in a climate control chamber, with a light level of $110 \mu\text{E m}^{-2} \text{ s}^{-1}$, a daily photoperiod of 12 hr, an ambient air temperature

of 20 °C, and an air humidity of 50-60 % saturation. Each experimental flow-through unit received water from its own stock container through black silicon tubes, at a flow speed of 10 L wk⁻¹ maintained by peristaltic pumps. The water level in the outer container could be manipulated by changing the overflow level (Figure 1). During both the preceding waterlogging period of seven months and the subsequent phases presented here, three different SO₄²⁻ concentrations, added as Na₂SO₄ (0, 2 and 4 mmol L⁻¹, indicated as [0], [S2], [S4]), were used in addition to the basic composition (Table I). Each experimental group consisted of six replicates, with random assignment to the three SO₄²⁻ concentration treatments. During pretreatment, the inflow tubes were placed in the centre of the top of the inner container, enabling a vertical water flow through the soil (Figure 1).

TABLE I
Chemical composition of the basic medium used. For the SO₄²⁻ treatments, either 2,000 or 4,000 μmol L⁻¹ Na₂SO₄ was added.

Constituent	Concentration (μmol L ⁻¹)	Constituent	Concentration (μmol L ⁻¹)
Ca	2,000	K	5.0
Mg	400	Mn	0.9
Na	2,000	Zn	0.7
HCO ₃ ⁻	2,000	Cu	0.2
Cl	4,885	B	0.8

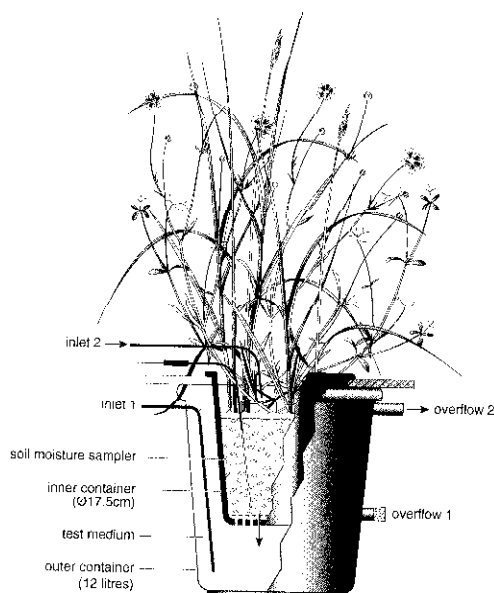


Fig 1. Experimental set-up for one unit. The water level in the soil could be manipulated by using different overflow levels.

Three soil moisture samplers (Rhizon SMS-10 cm; Eijkelkamp Agrisearch Equipment, The Netherlands) were placed in each container, and the collected samples were pooled in order to collect a representative pore water sample. Variation among the subsamples was moderate (standard deviation, at most, 10-15 % of the mean for all parameters measured). The removal of the sods did not result in any experimental artifacts; further details provided by Lamers *et al.* (in press). After the groundwater table had been reduced to 10 cm below soil surface (-10 cm) for nine weeks, to simulate moderate desiccation, the soils were further dehydrated for six weeks by removing the medium from the outer container. After this period, the water table was restored to -10 cm for three weeks and subsequently to the original waterlogged (pretreatment) situation.

Soil redox potential measurements were carried out in triplicate in each pot at 5 cm depth, using a mV-meter with a platinum wire electrode and a Ag/AgCl (3 M KCl) reference electrode. Values were converted to the potential relative to the normal hydrogen reference electrode (E_h). The pH was determined with a standard Ag/AgCl electrode and alkalinity was estimated by titrating part of the sample to pH 4.2 using 0.01 M HCl. After adding a few grains of citric acid to prevent precipitation of metal ions, color at 450 nm was measured for colorimetric background correction. The samples were stored in iodated polyethylene bottles (100 mL) at -28 °C until further analysis. The concentration of free sulfide was determined in a 10 mL subsample fixed immediately after collection with sulfide antioxidant buffer containing NaOH, Na-EDTA, and ascorbic acid (Van Gemerden, 1984). A sulfide ion-specific Ag electrode and a double junction calomel reference electrode were used (Roelofs, 1991). The concentrations of ortho-phosphate (SRP, soluble reactive P), NO_3^- (and NO_2^-), NH_4^+ , and Cl^- (used as a tracer for solute concentration due to dehydration) in the pore water samples were measured colorimetrically with Technicon AA II systems, using ammonium-molybdate (Henriksen, 1965), hydrazine sulfate (Technicon, 1969), salicylate (Kempers and Zweers, 1986) and ferriammoniumsulfate (Technicon, 1969), respectively. The data were corrected for color caused by humic acids. Potassium was determined by flame photometry (Technicon Flame Photometer IV). Total concentrations of Ca, Mg, Fe, Al, Mn, Zn, and S in the moisture samples were determined by inductively coupled plasma emission spectrometry (Jarrell Ash IL Plasma-200).

A repeated measures analysis was used to examine the response to treatments (Potvin *et al.*, 1990), using the SAS procedure GLM, model one-way ANOVA, for repeated measures (SAS, 1989). Data were log-transformed to make the variances less dependent on the sample means and to fit a normal distribution. Differences at a given time were analyzed by *a posteriori* Tukey tests at the 0.05 confidence limit. For clarity of presentation, the means and standard errors presented in the figures represent the non-transformed data.

3. Results

During desiccation, soil redox potential (E_h) increased from values around -150 mV to 400 mV, without an effect of SO_4^{2-} treatment (Figure 2; Table II). In the middle of the dry period, between weeks 15 and 20, no pore water samples could be taken because of dehydration. During the initial waterlogging, soil pH in both SO_4^{2-} -treated soils was higher than the control value ($p < 0.05$), and after water the table reduction to -10 cm it decreased

slightly. There were, however, no significant differences from the control treatment. At the beginning of the dry period, the pH of both SO_4^{2-} treatments dropped to values of 4.5-5. This persisted even two weeks after the waterlogged situation had been restored (significantly lower than [0]). The pH of the controls, however, barely decreased, fluctuating between 6.4 and 5.9. During the initial waterlogging, there was a significant difference between the alkalinities of the soil moisture for the three groups, with values being higher for the SO_4^{2-} -treated soils. After reducing the water table to -10 cm, alkalinity rapidly declined in all groups. During the initial phase of complete desiccation, alkalinity was below $100 \mu\text{eq L}^{-1}$ (Figure 2). After renewed waterlogging, alkalinity recovered so that by week 29, the value for [S4] was significantly higher than for the control.

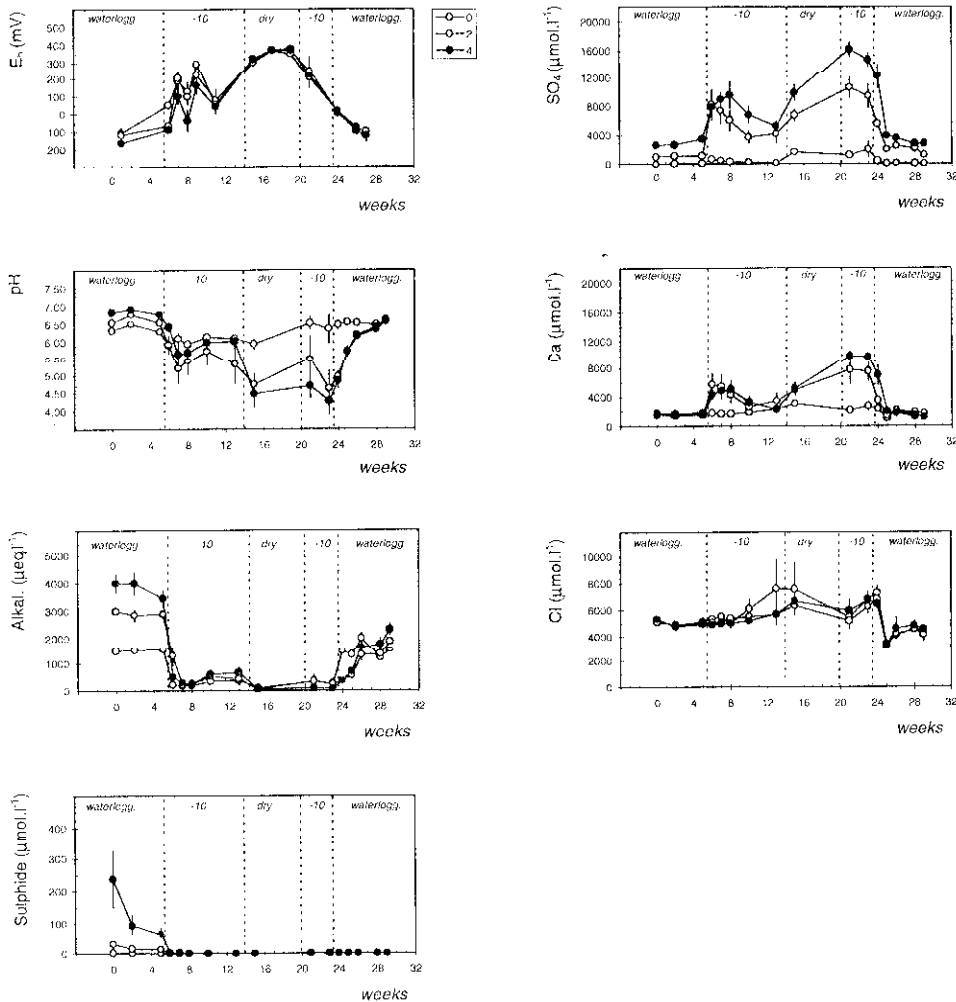


Fig. 2a. Soil pore water characteristics (means \pm standard errors; $n = 6$), during consecutive waterlogging (waterlogg.), water level reduction to 10 cm below soil surface (-10), complete desiccation (dry), restored water table at -10 cm (-10), and renewed waterlogging (waterlogg.) with either 0, 2 or 4 mmol L^{-1} SO_4^{2-} (indicated 0, 2, 4; white, grey, and black markers, respectively).

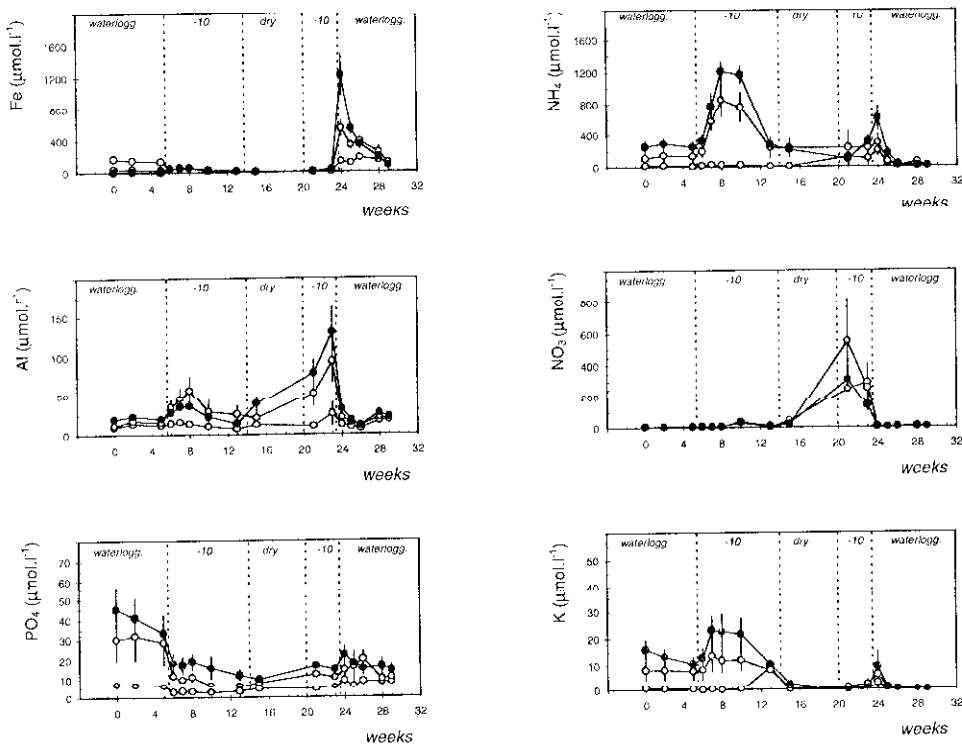


Fig. 2b. Soil pore water characteristics (means \pm standard errors; $n = 6$), during consecutive waterlogging (waterlogg.), water level reduction to 10 cm below soil surface (-10), complete desiccation (dry), restored water table at -10 cm (-10), and renewed waterlogging (waterlogg.) with either 0, 2 or 4 mmol L^{-1} SO_4^{2-} (indicated 0, 2, 4; white, grey, and black markers, respectively).

Chloride, being relatively uninfluenced by soil biogeochemistry, was used as a tracer for solute concentration by dehydration. Figure 2 and Table II show that there were no significant differences between the three groups. Chloride concentrations rose from 5,000 to 7,000 $\mu\text{mol L}^{-1}$ during the period in which sampling was possible. After rehydration of the soils, Cl concentrations returned to pretreatment levels after a brief decline (Figure 2).

TABLE II

Repeated measures analysis of variance (general linear models) of the effects of SO_4^{2-} addition on several pore water characteristics, as shown in figure 1 and 2 (except for Mg, Mn, Zn). For treatment $df = 2$, and for time $df = 15$, except for E_h for which $df = 11$. F -values are given, with their level of significance: ^a $p \leq 0.01$, ^b $p \leq 0.001$, ^c $p < 0.0001$; ns, not significant.

Dependent variable	Independent variable		
	SO_4^{2-} -treatment	Time	Interaction
Alkalinity	6.96 ^a	23.51 ^b	2.73 ^a
pH	8.43 ^a	17.14 ^c	4.75 ^c
SO_4^{2-}	898.45 ^c	56.47 ^c	5.6 ^c
E_h	2.45 ns	59.25 ^c	1.24 ns
Cl	0.50 ns	15.51 ^c	0.62 ns
Sulfide	68.46 ^c	230.61 ^c	50.75 ^c
ortho-P	18.53 ^b	9.45 ^c	2.67 ^a
NH_4^+	21.46 ^c	13.5 ^c	4.27 ^c
NO_3^-	0.12 ns	90.5 ^c	0.84 ns
K	10.79 ^a	17.28 ^c	7.30 ^c
Fe	0.45 ns	89.74 ^c	10.18 ^c
Ca	12.16 ^a	16.52 ^c	3.47 ^c
Mg	8.28 ^a	28.90 ^c	6.54 ^c
Al	26.42 ^c	10.72 ^c	1.31 ns
Mn	15.36 ^b	6.52 ^c	5.24 ^b
Zn	54.01 ^c	42.61 ^c	5.60 ^c

During waterlogging, SO_4^{2-} concentrations in the pore water of [S2] and [S4] were 1,000 and 2,500-3,000 $\mu\text{mol L}^{-1}$, respectively (Figure 2; Table II). After water table reduction, the concentrations rapidly increased to about 8,000 $\mu\text{mol L}^{-1}$ for both [S2] and [S4], after which they decreased again. During complete desiccation and subsequent restoration of the water table to -10 cm, SO_4^{2-} concentrations rose to about 10,000 and 16,000 $\mu\text{mol L}^{-1}$, respectively (both significantly higher than for [0]). In the controls, lowering of the water table to -10 cm and the subsequent complete desiccation also increased SO_4^{2-} concentrations. Renewed waterlogging restored the values to their pre-drought concentrations for all groups. The concentration of sulfide was significantly enhanced by the SO_4^{2-} treatment during the first waterlogging period, with significant differences between the three groups (Figure 2, Table II). After lowering the water table and subsequent desiccation, no detectable amounts of sulfide (i.e., concentration $< 1 \mu\text{mol L}^{-1}$) were noted. Even after renewed waterlogging, free sulfide could not be detected.

Calcium concentrations for [S2] and [S4] showed a significant peak of 6,000 $\mu\text{mol L}^{-1}$ after the first reduction of the water table, and increased to 7,500 and 10,000 $\mu\text{mol L}^{-1}$ after complete desiccation and the restoration of the water table to -10 cm, respectively

(Figure 2). The same trend was observed for magnesium, with concentrations rising significantly to 1,500 and 2,000 $\mu\text{mol L}^{-1}$, respectively (data not shown).

Iron concentrations in pore water were significantly depressed by both SO_4^{2-} treatments during the initial waterlogged situation (Figure 2). During desiccation, the values decreased (to less than 5 $\mu\text{mol L}^{-1}$) and then differences disappeared. After renewed waterlogging, iron concentrations showed a peak, with values significantly higher for both [S2] and [S4]. The concentrations of Al did not follow the same pattern. Compared to controls, significant peaks of Al were noted after reducing the water table and complete desiccation for [S2] and [S4]. The control soils did not show this strong response to desiccation, although a small peak of the Al concentration was detected after complete desiccation (Figure 2). The same trend holds for Mn and Zn concentrations (data not shown). Manganese in [S2] and [S4] soils increased from 6 to 40-50 $\mu\text{mol L}^{-1}$ after reducing the water table to -10 cm, and further to 30-70 $\mu\text{mol L}^{-1}$ following complete desiccation. Zinc concentrations in these groups rose from 0.7 $\mu\text{mol L}^{-1}$ to 5-10 $\mu\text{mol L}^{-1}$ during -10 cm, and to 20 $\mu\text{mol L}^{-1}$ after complete desiccation.

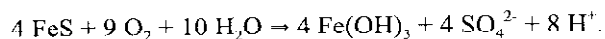
The dynamics of the major nutrients showed distinct patterns (Figure 2). Ortho-P concentrations were significantly higher for [S2] and [S4] during initial SO_4^{2-} treatment. Desiccation decreased the concentrations and there were few significant differences between the groups. Ammonium concentrations were also higher ($p < 0.05$) in the SO_4^{2-} -treated soils during waterlogging. Lowering the water table to -10 cm significantly raised NH_4^+ concentrations in [S2] and [S4] soils, as compared to controls. The control soils did not show such a response. During complete desiccation and rehydration the differences disappeared, although [S4] showed a peak. Nitrate concentrations markedly increased after restoring the water table to -10 cm after desiccation (no significant differences between groups). Waterlogging rapidly returned concentrations to levels as low as 5 $\mu\text{mol L}^{-1}$. Potassium concentrations were significantly higher for [S2] and [S4] during waterlogging and water table reduction. During complete desiccation and the subsequent period, K concentrations decreased to low levels in all treatments.

4. Discussion

The pretreatment of increased SO_4^{2-} supply during waterlogging stimulated sulfate reduction, as indicated by SO_4^{2-} disappearance from the medium, alkalinity generation and production of free sulfide (Figure 2). Sulfate treatment had a dose-dependent effect on the mobilization of ortho-P, NH_4^+ , and K. This type of nutrient mobilization, without input of nutrients, is termed internal eutrophication so as to distinguish it from eutrophication by allochthonous nutrients (Roelofs, 1991). Free Fe concentrations were depressed strongly as a result of precipitation with sulfide (see also discussion in Lamers *et al.*, in press).

The initial step of reducing the water table to -10 cm was taken to evaluate the effect of slight desiccation, as often occurs in wet meadows (including the one from which the sods were collected) during summer, on soil biogeochemistry. Soil redox potential rose by 200-250 mV, comparable to field data during water table drawdown to -10 cm (data not shown), and SO_4^{2-} was rapidly generated in [S2] and [S4], to levels twice the maximum SO_4^{2-} treatment concentration of 4,000 $\mu\text{mol L}^{-1}$. Chloride concentrations showed that this

could not be attributed to concentration effects (Figure 2), but must have been caused by oxidation of FeS and/or FeS₂:



Iron ion concentrations remained low as a result of iron oxidation to iron hydroxides. After rehydration, these Fe compounds were reduced again, showing peaks after one week for both SO₄²⁻-treated groups. The highest peak was found for [S4], for which the source of oxidized Fe originating from FeS_x was highest because of a higher sulfate reduction rate during pretreatment (Lamers *et al.*, in press). The responses of the three groups were contrary to those during the beginning of the pretreatment, showing lower peaks for [S2] and [S4] due to rapid FeS_x precipitation (Lamers *et al.*, in press).

For the control, the effects on pH by acid production due to FeS_x oxidation were limited, but the bulk of soil moisture alkalinity (almost entirely caused by HCO₃⁻) was rapidly consumed. Nitrate concentrations did not increase sufficiently to account for the observed consumption of alkalinity by proton generation through nitrification. However, the increase in SO₄²⁻ concentration from 50 to 650 μmol L⁻¹ for the control does explain the observed HCO₃⁻ consumption (see equation above). In [S2] and [S4] soils, not only was HCO₃⁻ consumed, but also there was mobilization of Ca and Mg, indicating cation exchange against protons at the soil adsorption complex and dissolution of CaCO₃.

During desiccation, HCO₃⁻ was consumed completely in all three groups by the oxidation of FeS_x and the absence of alkalinity generating processes like denitrification. Whereas SO₄²⁻ concentrations increased to 2,000 μmol L⁻¹ for controls, high values up to 16,000 μmol L⁻¹ were measured in [S2] and [S4]. These values are similar to those measured in drainage water originating from desiccated peatlands (Heathwaite, 1990). The observed drought-related SO₄²⁻ production and alkalinity consumption also has been described for poorly buffered moorland pools (Vangenechten *et al.*, 1981; Schuurkes *et al.*, 1988; Van Dam, 1988) and mineral rich fens (Kemmers and Jansen, 1988; Boeye and Verheyen, 1994).

In the minerotrophic peaty soils of the mesocosms, acid neutralization by cation exchange at the soil adsorption complex (as indicated by further mobilization of Ca and Mg) was inadequate to prevent soil acidification down to pH 4.5 in the SO₄²⁻-pretreated soils. This induced the mobilization of metals like Al, Zn, and Mn to potentially toxic concentrations. In the controls, however, no effects of desiccation on soil pH could be detected and Al, Mn, and Zn concentrations remained low. Base cations were mobilized, but to a much lesser extent than in the SO₄²⁻-pretreated soils. Apparently, the ANC of minerotrophic soils, not subject to SO₄²⁻ pollution, was adequate to prevent pH changes and the subsequent severe mobilization of metals. By definition, however, there was acidification because ANC decreased (van Breemen *et al.*, 1983). Van Haesebroeck *et al.* (1997) also found no significant effect of drought on the pH of pore water in rich fen soils, although the pH of water-extracted fresh soil at the end of the drought period had slightly decreased.

The fact that NH₄⁺ and K concentrations showed a peak for [S2] and [S4] during water table reduction to -10 cm (Figure 2) presumably indicated exchange of both monovalent cations against protons at soil binding sites and/or a temporal stimulation of the mineralization rate. For freshwater marshes, an increase of the mineralization rate during moderate

desiccation has been shown (De Groot and Van Wijck, 1993). The decrease in K concentrations for all treatments during complete desiccation may suggest jarosite ($\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$) formation (van Breemen, 1976).

Nitrification was stimulated only after complete desiccation, as shown by the strong increase in NO_3^- concentrations of all soils after rehydration. Subsequent waterlogging probably suppressed nitrification and stimulated denitrification, leading to low NO_3^- concentrations (Barendse *et al.*, 1994). The decrease in ortho-P concentrations during desiccation most probably can be explained by increased binding of phosphate to oxidized Fe(III) compounds, such as iron phosphate, iron(hydr)oxide-phosphate and humic-iron-phosphate complexes (Patrick and Khalid, 1974; Khalid *et al.*, 1977; Richardson, 1985; Golterman, 1984, 1988).

5. Implications for Wetland Management

From the outcome of this experiment it may be concluded that the present S pollution in many freshwater wetlands is a threat not only by inducing internal eutrophication, but also by increasing the sensitivity of wetlands to desiccation, another general threat to wetland functioning and biodiversity. The increased pool of FeS_x will stimulate drought-related soil acidification, influencing both ANC and pH, and will cause an increase of potentially toxic metals like Al in pore water. The combination of increased S loading with a growing risk of the occurrence of desiccation is therefore an important cause for concern. Furthermore, the high SO_4^{2-} concentrations generated by desiccation and the subsequent runoff following rehydration will inevitably cause internal eutrophication in reduced peaty soils and sediments where the water is discharged.

The extent to which drought sensitivity increases depends on soil ANC, the pre-drought levels of both SO_4^{2-} and reducible organic compounds (regulating sulfate reduction rates), and on the amount of Fe available for sulfide precipitation. Particularly in areas that possess a large Fe pool in the soil (e.g., because of actual or former groundwater discharge), SO_4^{2-} pollution will seriously enhance FeS_x storage and the related response to desiccation. Therefore, for management and restoration of wetlands, much importance should be devoted to the preservation or restoration of the original hydrology and hydrochemistry of deteriorating wetlands.

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