RETENTION AND RELEASE OF S FROM A FRESHWATER WETLAND

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ABSTRACT. A freshwater wetland at the Experimental Lakes Area in northwestern Ontario stored most of the  $S0<sub>4</sub><sup>2</sup>$  received annually from precipitation, runoff and experimental additions. The S budget was determined for a small fen spray irrigated with H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub>. Annual S retention was greatest during the first year of experimental addition of H<sub>2</sub>SO<sub>4</sub> (73% of input in 1983). Retention was lowest (22%) in 1984, a year of lower than average precipitation, with a long hot summer. During years with hot, dry summers,  $50<sub>4</sub>$ was produced from the reoxidation of reduced S compounds in the peat and released to surface waters. The autumn  $S0<sub>4</sub><sup>2</sup>$  pulse was accompanied by the release of Ca and Mg but was not accompanied by a H- release as has been detected in eastern Ontario and southern Norway, areas which receive more acidic precipitation.

## I. INTRODUCTION

Many freshwater wetlands appear to retain S on an annual basis. Braekke (1981) found a 35% retention of SO $_4$ <sup>2</sup> in the peatland-dominated Storgama watershed in S. Norway during dry years and only a 4% retention during wet years. Urban et al. (1986) found retention of  $50<sub>4</sub><sup>2</sup>$  averaged  $56%$  in Marcell Bog in northern Minnesota, an area relatively unaffected by anthropogenic atmospheric deposition. Calles (1983) found a 25 to 64% retention of  $SO_4^2$  in Swedish watersheds which contained from 8 to 33% bogs. Hemond (1980) calculated that approximately 77% of the input  $S0<sub>4</sub><sup>2</sup>$  was retained in Thoreau's Bog in the northeastern US, in

spite of high atmospheric S deposition.<br>Other studies have documented the decrease in SO<sub>4</sub><sup>2-</sup> concentration as water passes through a wetland (Vitt and Bayley, 1984; Kerekes et al., 1985; Wieder and Lang, 1984). Wieder and Lang observed that acidic, sulfate-rich mine drainage was neutralized as it passed through a Sphagnum-dominated bog in West Virginia.

Retention of  $SO_4^2$  in wetlands would be expected to be lower in areas with a large snowpack and subsequent rapid springmelt or in

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wetlands with high rates of water flow, because there would be little time for the water and  $S0<sub>4</sub><sup>2</sup>$  to remain in contact with the reduced substrate. Lazerte and Dillon (1984) found that a ten in<br>Dorset, Ontario released high concentrations of SO4<sup>2—</sup> during the springmelt but retained  $50<sub>4</sub><sup>2</sup>$  during the summer. It appears that  $S0<sub>4</sub><sup>2</sup>$  is retained more effectively when the hydrologic conditions maximize the contact time between the water, vegetation and anaerobic peat.

In contrast to most wetlands studied, terrestrial watersheds in acid sensitive areas release most of the  $\mathtt{SO}_4{}^+$  received in precipitation (Braekke, 1981; Likens et al., 1977; Bayley and Schindler, 1986; Schindler et al., 1976; Wright, 1983). Wetlands located at the outflow of terrestrial watersheds can modify the acid pulse and total  $50<sub>4</sub><sup>2</sup>$  received by streams and lakes even in acid impacted areas (Braekke, 1981). However, during hot dry seasons, the water level in many wetlands drops, allowing  $0<sub>2</sub>$  to penetrate further into the peat and reduced sulfides are oxidized to sulfate. With subsequent rainstorms,  ${\rm SO}_4$   $^-$  is flushed from the wetland to downstream waters. This autumn  $\mathtt{SO}_4$  pulse has been observed at ELA (this study), Norway (Braekke, 1981), Dorset, Ontario (Lazerte and Dillon, 1985), England (Rippon et al., 1980; Brown, 1980), and West Virginia (Wieder, 1985).

This study presents  ${SO}_4$ <sup>-</sup> budgets under natural conditions as well as under a regime of increasing  $S0<sub>4</sub><sup>2-</sup>$  additions. We present evidence that a poor fen in northwestern Ontario retains  $SO_4^2$  on an annual basis, even with a 50% increase in  $S0<sub>4</sub><sup>2</sup>$  loading from experimental acidification. Secondly, we present evidence that wetlands can lose a large portion of the stored  $S0<sub>4</sub><sup>Z</sup>$  if the hydrologic conditions favor oxidation of reduced S compounds. The data show that  $504^{2-}$  release is accompanied by release of Ca and Mg while the  $H^+$  concentration does not increase. We hypothesize that the autumn  $504^{2-}$  pulses in wetlands are not accompanied by a decrease in pH in the early stages of acid deposition (prior to Ca and Mg exhaustion), or in wetlands with high cation loading.

While the previously mentioned  $\texttt{S04}^{\texttt{c}}$  -mass balance studies of wetlands have documented  $SO_4^+$  retention, some of these studies have methodological problems which this study does not have. This study area has very low dry deposition of  $\mathtt{SO}_+$   $\check{\phantom{a}}$  (Barrie, 1982) and good control of the hydrology. The basin is bound by granitic bedrock and there is little ground water seepage in or out of the basin. The low  $S0<sub>4</sub><sup>2</sup>$  deposition coupled with known experimental addition of  $H_2SO_4$  permitted greater accuracy in measurement of  $504^{2}$  retention than is possible under circumstances where the major input is atmospheric only.

## 2. MATERIALS AND SITE DESCRIPTION

The experimental wetland (3.67 ha) (Figure i) is part of the Northeast watershed (10.58 ha) of L.239, located at the Experimental Lakes Area (ELA) in northwestern Ontario, Canada (49°40'N and

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93°44'W). The upland portion of the watershed (6.91 ha) is covered with a thin layer of mineral soil derived from the underlying granite bedrock. The terrestrial portion of the watershed burned in 1980 and has been quickly re-vegetated with Pinus banksiana, Populus tremuloides and other boreal species. The peatland is dominated by black spruce (Picea mariana) and three Sphagnum species, S. magellanicum, S. angustifolium and S. fuscum. See Vitt and Bayley (1984) for a detailed vegetation description. The peatland is composed of an oligotrophic center area and a more minerotrophic edge area (Figure i). Runoff from the watershed flows directly into the peatland. The single outflow stream from the peatland is gauged with a weir.

Water samples were collected weekly during the ice free season at various stations within the fen during 1984. Outflow samples were collected weekly since 1971 but the outflow data used in the mass balance calculations is from 1981 through 1984. Precipitation was collected after each event.  $SO_4$ <sup>2</sup> concentration was measured with an ion chromatograph (Dionex) using filtered samples. Ca and Mg concentration were measured by atomic absorption (Stainton et  $a1., 1977$ .

Sulfate budgets for the fen were calculated using the approach outlined in Schindler et al. (1976). Annual runoff volume into the fen was calculated from the water balance of the fen (i.e Runoff  $=$ Outflow + Evapotranspiration - Precipitation on fen  $\pm \Delta$  Storage). Evaporation was derived from the traditional mass transfer relationship as described by Newbury and Beaty (1980) for small ELA lakes. Storage in the fen was assumed to be zero, based on water level measurements. Concentrations of  $SO_4$ <sup>2</sup> from a pool in the upland watershed were multiplied by the runoff volume to calculate  $S0<sub>4</sub>$ <sup> $\sim$ </sup> loading to the fen due to runoff.

Total S and total inorganic S in peat cores were measured as described in Behr (1985). Total S was measured on a LECO sulfur analyser, model SC-132. Total inorganic S was measured as the sum of acid volatile sulfides (AVS), elemental  $S(S^{\circ})$  and pyrite  $(Fes_2)$ . AVS released from sediment by HCL was trapped with a zinc acetate trap and the ZnS precipitate measured by iodine titration.  $S^{\circ}$  and FeS<sub>2</sub> were measured by the chromium (II) reduction technique (Howarth and Merkel, 1984).

In 1983, experimental acidification of the fen began. The purpose of the experiment was to simulate lowering the average pH of precipitation falling on the fen from pH 5 to pH 4. To achieve this,  $\rm H_2SO_4$  and  $\rm HNO_3$  (on a equinormal basis) were added to lake water (pH 6.56; Ca 127  $\mu$ Eq L  $\hat{}$  ) and sprayed once each month during the ice-free season. An experimental part of the fen received the acid plus lake water (2.66 ha) while an upslope part of the fen (0.85 ha) was used as a control and received only lake water (Figure i).

The fen can be divided into an edge or lagg zone, which is a minerotrophic area receiving runoff from the surrounding uplands and an oligotrophic center area (Figure i) which does not receive upland runoff. In this paper data from the minerotrophic pools and outflow



Figure i. Map of NE Fen showing acidified area and control area. Hatched area is the minerotrophic edge.

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as well as the whole fen mass balance are presented. The minerotrophic experimental site received acid and lake water applications and runoff from the surrounding watershed (which was not irrigated). The minerotrophic control pool received lake water applications plus runoff from the surrounding watersheds. For further details of the acidification procedure see Bayley et al. (1986).

## 3. RESULTS AND DISCUSSION

## 3.1. Wetlands as a Sink for S

On an annual basis, the fen at ELA was a sink for S. From 1981-1984,  $SO_4^2$  retention ranged from 22 to 73% of inputs (Table I). The annual storage ranged from 21 to 51 meq  $\mathrm{SO}_+$   $\mathrm{^-}$  m  $\mathrm{^-}$ yr  $^+$  (0.33 to 0.82 g S m  $^+$  yr  $^+$ ). Higher absolute and percent S storage was detected after experimental acidification in 1983 but not in 1984. Both were years of lower than average precipitation. Pakarinen (1981) and Pakarinen and Tolonen (1980) measured approximately 0.87 to 1.08 g S m ~ yr ' retained in South Finnish bogs. Brown (1985) calculated a rate of S accumulation in peat of 4.76 to 6.06 g m ~ yr \_in an area with high S deposition. Gaseous losses of S from the fen were not measured and would reduce calculated retention if these processes were important. However, Brown and Macqueen (1985) concluded that volatilization of H2S from peat cores was negligible.

TABLE [ Annual  ${SO_4}^{2-}$  budgets in the experimental wetland (1981-1984) in meq  ${SO_4}$  $m^{-2}$  yr<sup>-1</sup>.

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Atmospheric bulk deposition of SO<sub>4</sub><sup>2-</sup> at ELA has averaged 22.3 mEq  $50<sub>4</sub>$ <sup>2</sup> over the last 11 years (Linsey et al., 1986). While the deposition at ELA can be considered low, there has been sufficient input from the atmosphere and runoff for the peat to store up to 155  $\mu$ mol S  $g^{-1}$  dry matter (1.4%) in the 10 to 15 cm depths (Figure 2), and over 90% of the S was organic S (Behr, 1985). Pakarinen and Tolonen (1980) found peak concentrations of 109  $\mu$ mol S  $g^{-1}$  dry matter in a South Finnish bog.



Figure 2. Concentration of total and organic S in a peat core. Concentration of H2S-S in porewater on 29 June 1984. Average water table fluctuation is from 10-20 cm below moss surface. The minimum water level was at 32 cm during August 1985.

Because the fen retained a large percentage of SO  $4^{27}$  inputs, and because  ${SO_4}^{\mathcal{L}^-}$  reduction has been shown to occur there. (Behr, 1985), it seems reasonable to believe that the peak in S concentration at 10 to 15 cm was due to downward diffusion of  $SO_4^2$  and its reduction there. Also,  $SO_4^2$  reduction can lead to formation of organic S (Behr, 1985; Rudd et al., 1986), probably

as a result of  $H_2S$  reactions with organic molecules (Casagrande et al., 1979; Brown, 1986). The organic S formed in this way in lake sediments has been shown to be partly lost during winter when  $0_2$ penetrates further into the sediments (Rudd et al., 1986). Loss of metal sulfides due to oxidation is well known. Thus, any change in the 02 penetration depth in the peat (controlled in large part by changes in water level) could result in remobilization of reduced S from both inorganic and organic forms.

The water level in the fen generally fluctuated between I0 and 20 cm below the surface of the moss which is coincident with the zone containing the highest S concentration (Figure 2). Hydrogen sulfide concentration was also highest in that zone (Figure 2) indicating that  $0<sub>2</sub>$  was depleted to within a few cm of the H $<sub>2</sub>$ O</sub> surface, resulting in conditions suitable for  $SO_4^2$  reduction. During the 1984 summer drought the water level dropped 30 cm below the moss surface and  $0<sub>2</sub>$  probably penetrated to that depth, oxidizing the reduced S. Then, when autumn rain came, this oxidized S was flushed out as described below.

3.3. Autumn  $50<sub>h</sub>^{2-}$  pulse

With the first storms after the summer drought, there was an increase in the concentration of  $\text{SO}_4{}^+$  in the minerotrophic control pool starting 12 September 1984 (Figure 3a). The concentration increased from a summer low of 17 to 731  $\mu$ Eq L<sup>-1</sup> in mid September. The abrupt increase was detected after a 13 mm rainfall, followed by a 15 mm application of lake water. The concentration remained higher than spring and summer yalues for the remainder of the ice-free season (Figure 3). The  $SO_4$ concentration at the control station (Figure 3a) was higher than the  $S0<sub>u</sub><sup>2</sup>$  concentration in the minerotrophic experimental station  ${{50}_4}^{2-}$  concentration in the minerotrophic experimental station (Figure 3b) which had received 758  $\rm \mu Eq$  L  $^{-}$  of SO $\rm _4$   $^{-}$  as H  $\rm _2$ SO  $\rm _4$ during the acidification experiment. This may be because the control receives more runoff than does the experimental station. The concentration of  ${SO_4}^{\prime-}$  in the outflow of the fen also showed an increase after the summer drought (data not shown).

3.4. Oxidation of reduced S

We believe the sharp increase in  $S0<sub>4</sub><sup>2-</sup>$  concentration in September in the minerotrophic control pool (Figure 3a) was caused primarily by the oxidation of reduced S during the preceding dry period, not by runoff of SO $_{4}$  - from the upland watershed. This is suggested because the  $\mathrm{SO}_4$  - increase also occurred in August in the same pool with just the addition of lake water (Figure 3a). August 1984 was the driest August on record (Beaty, 1986) and there was no upland runoff.

Another line of evidence suggesting that oxidation of reduced S compounds caused the increase in surface water concentration was the decrease in total inorganic sulfides in peat cores from spring 1984 to autumn 1984 (Figure 4). There was an average 36% reduction in



Figure 3. a. Weekly concentrations of SO $_{\mathsf{L}}$  , Ca, Mg and H  $^{\mathsf{+}}$ during the 1984 ice-free season in the minerotrophic control site. b. Weekly concentrations of ions in the minerotrophic experimental site.

concentration per gram of peat over the dry summer with most of that loss in the I0 to 20 cm depths (Figure 4).

Another contributor to the observed autumn  $SO_4^2$  pulse might be enhanced mineralization of S from organic peat by heterotrophic decomposer activity in the dry aerobic peat.



Figure 4. Inorganic S concentration in peat cores collected 31 May 84, 2 June 84 (spring), 1 Oct 84 and 2 Oct 84. Data in  $\mu$ Eq g dry wt  $\pm$  SE,  $n = 4$ .

3.5. Release of Ca and Mg

The release of  ${50<sub>4</sub>}^2$  after summer drought was accompanied by the release of Ca and Mg in both control and experimental stations (Figures 3a and b). Ca and Mg (data not shown) also increased in the outflow of the fen. Sphagnum moss releases cations with the addition of anions (Bayley, 1986).

The higher autumn concentration of Ca, Mg and  $S0<sub>4</sub><sup>2</sup>$  could be due to evaporation, drying of the moss and concentrating of the water during the dry summer. With autumn rains, the Ca and Mg salts of sulfate would be dissolved and released in the water. Assuming that sodium sulfate would dry and redissolve in a similar

fashion as  $CaSO_4$  and  $MgSO_4$ , then Na can be used as a predictor to account for the evaporative drying of Ca, Mg and  $\text{SO}_4^2$ . The ratio of spring Na concentration to autumn Na concentration is assumed to represent evaporative concentration of all the cation salts of sulfate. Based on this ratio, the autumn concentrations of Ca,  $Mg$  and SO $4^2$  were much higher than that predicted based on evaporative drying alone. In fact, Ca and Mg concentrations were 2 times and SO  $_4^2$  5 times higher than what would be expected from evaporative drying.

It is unlikely that the Ca and Mg release could be from the lake water or acid application since lake water concentrations of Ca and Mg averaged 12/  $\mu$ Eq L  $^{-}$  and 58  $\mu$ Eq L  $^{-}$ , respectively. In September after the first rains and fresh water application, the Ca concentration reached 476  $\mu$ Eq L<sup>-1</sup> and Mg 375  $\mu$ Eq L<sup>-1</sup> in the minerotrophic control pool. While storm runoff into the fen could increase the Ca and Mg concentrations to some extent, it is unlikely that runoff was the immediate source of the increased Ca and Mg. Comparison of minerotrophic experimental surface pool Ca and Mg concentrations before acidification in August 1984 with concentrations\_after acidification show a similar release of Ca and Mg as the  $SO_4$ <sup>c</sup> concentration increased (Figure 5a), and since there was virtually no rainfall in August, the Ca and Mg must have come from the Sphagnum mosses and peat.

In September 1984 natural reoxidation of S in the control site produced higher concentrations of S than did acidification of the experimental site<sub>\_</sub>(Figure 5b and c). In these minerotrophic sites, increases in SO<sub>4</sub><sup>2</sup> were not accompanied by increases in  $H^{\dagger}$ concentration (Figure 3a and b, 5a, b and c).

In a fen in Dorset, Ontario, during a dry autumn, the SO  $4^{2-}$ rose to  $1000$   $\mu$ Eq L  $^-$ , Ca increased to 6/5  $\mu$ Eq L  $^-$ , while the H rose to 140 µeq L <sup>-</sup> (Lazerte and Dillon, 1985). Similarly, in the Storgama peatlands of Norway, Braekke (1981) detected a September increase in SO42-, Ca and H during dry years.

The most apparent difference in the ELA site and the two sites mentioned above is that the fen at ELA has received acid for only 2 yrs while the Dorset, Ontario and Norwegian sites have had high atmospheric loadings for decades. Another explanation for the lower  $H<sup>T</sup>$  concentration at ELA could be that the cation (primarily Ca and Mg) supply rates to the fen are higher than the cation supply at the Dorset, Ontario or Storgama Norwegian sites. However, Table II indicates that the ELA fen had lower cation supply in runoff than the Norwegian site (Braekke, 1981) and similar cation supply to the Dorset, Ontario site (NRC, 1981). Therefore, differences in cation supply are not likely to be causes of the lower H' concentrations at the ELA site. The release of H⊂ at the Dorset, Ontario and<br>Norwegian sites during the autumn SO4<sup>2-</sup> pulse could be due to reduction of Ca and Mg neutralization capacity under the longer and higher  $SO_4^2$  deposition conditions, however, both of those sites still release Ca and Mg during the  $\text{SO}_4^2$  pulse.



**Figure 5. Effect of autumn rains on ion concentrations, a. Concentrations of ions in the experimental site before and after acidification in August 1984 during the summer drought. b. Concentrations of ions in the control site before and after addition of lakewater in September 1984, after the summer drought. c. Concentrations of ions in the experimental site before and after acidification in September 1984, after the summer drought.** 

#### TABLE II

Runoff loadings into wetlands. Data in meq m $^{-2}$  yr $^{-1}$ .



•Total input at ELA is the sum of natural runoff plus the experimental input. Experimental input alone is in brackets, At Dorset and Storgama, the total input is runoff from the wstersheds.

## 4. CONCLUSION

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Annual  $\mathrm{SO}_4^-$  -retention by the fen is high (mean 51%) in the 4 yr  $^+$ of study, including 2 yr of elevated  $\mathrm{SO}_4^-$  inputs from experimental acid additions. Release of S04 was greatest during the first complete year of experimental acidification, however, the release of S04 also occurred in the control areas after the long dry summer, indicating that the acidification was not the dominant factor in the release of  $SO_4$ .

When the water table declined during dry summers, reduced S compounds were oxidized to  $50<sub>4</sub><sup>2-</sup>$  and flushed from the fen causing an autumn  $S0_4^2$  pulse. This pulse was locally important to downstream waters even though the fen still retained some of its  $SO_4^2$  inputs. The hydrology of the wetland (depth and frequency of flooding) and the total amount of precipitation may be the most important factors determining annual S retention.

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