Biological sulfur retention in surface soils as a predictor of ecosystem sensitivity to acidic precipitation

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ABSTRACT

Three processes affecting sulfate retention were measured for A horizon soils from eleven US forest sites. These were physico-chemical sulfate adsorption of ^{35}S-sulfate, biological incorporation of added ^{35}S-sulfate into organic matter, and subsequent mobilization (depolymerization followed by mineralization) of biologically formed ^{35}S-organic sulfur. The last two values were used to calculate the net biological sulfate retention for each soil. Values for the ecosystem wide leaching loss of the cationic plant nutrients Ca^{2+}, Mg^{2+}, and K^+, based on precipitation inputs and streamflow losses for each site, were used as indicators of ecosystem sensitivity to sulfates derived from acidic precipitation. Although many of the A horizon soils exhibited substantial amounts of physico-chemical sulfate adsorption, this process was not correlated with ecosystem wide cation leaching loss. Mobilization of organic sulfur was positively correlated to ecosystem wide cation loss. Both gross biological incorporation and net biological sulfur retention showed strong negative correlation with ecosystem wide cation loss. Thus, soils with high capacity for biological sulfate retention experience less ecosystem wide cation leaching loss and should be less sensitive to acidic deposition related sulfate inputs.

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INTRODUCTION

Much of the concern over potential impacts of acidic precipitation has focused on the effects of elevated sulfate loading in the affected soils. Sulfate is a predominant anionic constituent of acidic precipitation, and numerous investigations have confirmed a substantial depletion of soil cations associated with elevated sulfate inputs (Cronan 1980; Johnson et al. 1982, Kennedy 1986). This loss of nutrient base cations is due to ionic association with the incoming sulfate anion when it remains mobile and is leached out of the soil system (Foster et al. 1986, Reuss and Johnson - 1985). Although there is controversy concerning the relative roles of soil formation versus anthropogenic input with respect to soil acidification and nutrient leaching (Krug and Frink 1983), retention of incoming sulfate within the soil profile has been shown to reduce cation leaching loss at a variety of sites (Johnson et al. 1982, Rechcigl and Sparks 1985, Richter et al. 1983, Wilklander 1980). Therefore, the relative mobility of sulfate within a soil system is of considerable importance with respect to a site's sensitivity to the effects of acidic precipitation.

Sulfate retention by physico-chemical adsorption within subsurface soils has been widely documented, and several researchers have related physico-chemical sulfate adsorption in clay rich B horizon soils to decreased cation loss from forested ecosystems (Huete and McColl 1984, Reuss and

Johnson 1986, **Richter** et al. 1983, Singh et al.
1980). However, as pointed out by Task Group VI
of the National Acid Precipitation Assessment
Program, biological influences on sulfate retention
have been largely overlooked (Joyner 1987).
Biological incorporation of sulfate into organic
matter, a direct result of microbial activity
(Fitzgerald et al. 1982; Swank et al. 1984), which
has been addressed in relatively few studies (David
et al. 1987, David and Mitchell 1987, Fitzgerald et
al. 1985, Fuller et al. 1986), is an important but
largely ignored mechanism for the retention of
sulfate. The potential significance of biological
sulfate incorporation is emphasized by the
observation that many sites have larger organic than
inorganic sulfur pools throughout the soil profile
(**Autry** and Fitzgerald 1991a). In addition,
biological incorporation may represent a more
important long term sulfur storage mechanism than
adsorption, which tends to act as a temporary
retention mechanism (Autry and Fitzgerald 1991b).

In the past decade there have been numerous
attempts to formulate a predictive index of soil
sensitivity to acidic precipitation as indicated by
elevated levels of cation leaching (**Klopatek** et al.
1980; **McFee** 1980; Reuss and Johnson 1986).
Several proposed indices use estimates of sulfate
retention as predictors of cation leaching extent, but
generally **physico-chemical sulfate** adsorption has
been the only retentive process considered in these
estimates. **Rochelle** et al. (1987) compiled sulfur
retention estimates for a variety of US and
Canadian sites. In this **case** overall retention was
measured as input minus output, and biological vs.
abiotic processes were not examined separately.
The present study examines both abiotic (**physico-**
chemical adsorption) and biological (incorporation
into organic matter) sulfate retention and compares
these parameters to ecosystem-wide cation leaching
losses for eleven US forest sites. This is the first
multi-site examination of biological sulfate
retention in A horizon soils as a potential predictor
of ecosystem wide cation leaching loss.

MATERIALS AND METHODS
Site descriptions and sampling

The sites included in this study, along with major

vegetation type, precipitation, and soil
characteristics, are presented in Table 1. The study
sites are widely distributed across the US, with a
variety of soil types (**entisols, ultisols, inseptisols,**
etc.) represented. Major vegetation ranged from
alpine tundra within the Tesuque Watersheds of
New Mexico to a variety of deciduous and
hardwood forest **types.** Total Carbon (C) was
determined in triplicate for each soil using a
Hewlett Packard C analyzer, and total Sulfur (S)
was quantified by the **hypobromite** oxidation
method of **Tabatabai** and **Bremner** (1970).

Bulk soil samples from the A horizon were
obtained from each of the eleven described sites
during 1984 and used in laboratory determinations
of sulfate retention potentials. Root material was
removed by hand and all samples were stored in
sealed bags at 5°C prior to analysis. All analyses
were performed within one week of sample
collection.

Sulfate retention determinations

Field moist soil samples (1 g wet weight, not
sieved) were incubated for 48 h with 7.5 nmoles
$Na_2{}^{35}SO_4$ (3.3 x 10^{10} Bq mmole^{-1}, Amersham)
and then extracted according to the methods of
Fitzgerald et al. (1983) to recover salt **extractable**
and acid and base extractable fractions. The salt
extraction process has been shown to recover all
^{35}S-sulfate which has been adsorbed during
incubation (Fitzgerald et **al.** 1982). Following salt
extraction all ^{35}S remaining in the soil samples was
necessarily retained via **covalent** linkages which are
formed during sulfate incorporation into organic
sulfur. Treatment **with strong** acid and base
solutions released this fraction which is hereafter
referred to as biologically incorporated sulfate.
Separate experiments have confirmed that this
fraction is not merely strongly adsorbed sulfate
(Fitzgerald et al. 1985), but is organic sulfur
formed as a direct result of microbial activity
(Fitzgerald et al. 1982, 1983; Swank et al. 1984).
These two fractions were quantified by scintillation
counting and were normalized to yield retention
potentials per g soil dry weight (Table 2).

The rate of organic sulfur mobilization was
also assayed for each soil. Organic sulfur

Table 1 . Selected characteristics for A horizon soils included in study.

Site Description •	Soil Description	Dominant Vegetation	Precipitation (cm/yr)	Total Carbon (96)	Total Sulfur (mg/kg)
Coweeta Hydrologic Laboratory, Southern Appalachians, Otto, NC - WS2	Typic Dystrochrepts[a]	Mixed Hardwood[a]	180[a]	4.6f	283[f]
- WS18	Typic Hapludults[a]	Mixed Hardwood[a]	180[a]	Not available	295[f]
Hubbard Brook Experimental Station, White Mountain National Forest, **NH**	Haplorthods[b]	Mixed Deciduous / Hardwood[b]	120[b]	2.4[f]	72[f]
H.J. Andrews Experimental Forest, Cascade Mountains, OR	Typic Dystrochrepts[c]	Douglas Fir[c]	240[b]	6.4[f]	272[f]
Oakridge National Laboratory, Walker Branch, TN	Typic Paleudults[d]	Chestnut Oak[d]	151[b]	2.0[f]	206[f]
Tesuque Watersheds, Santa Fe National Forest, **NM** - WS15	Pergelic Cryoborolls[e]	Alpine Tundra[e]	76[e]	8.4[g]	1075[g]
- WS8	Dystric Cryochrepts[e]	Spruce Fir[e]	76[e]	4.7[g]	295[g]
- Aspen	Typic Cryochrepts[e]	Aspen[e]	76[e]	1.8[g]	134[g]
- WS2	Typic Ustorthents[e]	Ponderosa Pine[e]	76[e]	10.9[g]	4668
- PJ	Lithic Ustorthents[e]	Pinyon / Juniper[e]	76[e]	4.68	2758
North Inlet, Belle Baruch, SC	Aerie Haplaquods[h]	Loblolly Pine[h]	115[h]	4 8[h]	309[h]

[a] Swank and Crossley (1988), [b] Likens et al. (1977), [c] Sollins et al. (1980), [d] Johnson et al. (1980),
[e] Gosz personal communication, [f] Watwood and Fitzgerald unpublished data, 8 Watwood et al. (1986),
[h] Watwood et al. (1988).

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Table 2. Sulfate retention and organic sulfur mobilization potentials for the A horizon study soils. Values are site means ± standard error, with replicate number given in parentheses.

Site	Sulfate Adsorption	Biological Incorporation[a]	Organic Sulfur Mobilization[b]	Net Biological Retention[c]
Coweeta-WS2[d]	5.26 ± 0.73 (52)	1.94 ± 0.27 (52)	38.18 ± 7.09 (29)	1.20
Coweeta-WS18[e]	6.03 ± 0.20 (120)	1.67 ± 0.04 (120)	41.32 ± 2.00 (120)	0.98
Hubbard Brook[d]	2.96 + 2.08 (2)	0.75 ± 0.53 (2)	42.67 ± 31.36 (2)	0.43
H.J. Andrews[d]	8.36 ± 2.23 (14)	0.83 ± 0.23 (13)	43.44 + 3.82 (14)	0.47
Oakridge[d]	2.51 ± 0.89 (8)	0.84 ± 0.30 (8)	57.45 ± 16.74 (12)	0.36
Tesuque-WS15[f]	2.50 ± 0.23 (5)	2.80 ± 0.22 (5)	28.30 ± 3.11 (5)	1.79
Tesuque-WS8[f]	2.50 ± 0.23 (5)	2.50 ± 0.23 (5)	35.90 ± 3.95 (5)	1.60
Tesuque-Aspen[f]	4.90 + 0.44 (5)	1.60 + 0.13 (5)	50.10 ± 5.51 (5)	0.80
Tesuque-WS2[f]	1.40 ± 0.13 (5)	4.80 ± 0.38 (5)	23.80 ± 2.62 (5)	3.66
Tesuque-P₁[f]	1.20 ± 0.11 (5)	3.50 ± 0.28 (5)	19.70 + 2.17 (5)	2.81
North Inlet[g]	1.20 ± 0.10 (27)	0.70 + 0.10 (27)	55.60 ± 3.70 (27)	0.30

[a] Biological incorporation values are given as nmol S/48h/g dry weight, [b] Mobilization values are given as a percentage of ^{35}S-organic matter mobilized/24h, [c] Net retention = incorporation - (% mobilization x incorporation), [d] Watwood and Fitzgerald unpublished data, [e] Fitzgerald et al. (1988), [f] Watwood et al. (1986), [g] Watwood et al. (1988).

mobilization may lessen the retention effect of sulfate incorporation by releasing sulfate from recently formed organic sulfur moieties (Fuller et al. 1986, McLaren et al. 1985, Schindler et al. 1986), and must be applied to any estimate of biological sulfate incorporation in order to arrive at an accurate estimate of net sulfate retention. Mobilization potential was determined by allowing soil microorganisms to incorporate added 35s sulfate into organic matter over a 48 hr period and quantifying the subsequent release of ^{35}S-sulfate from the ^{35}S-organic matter over the following 24 hrs using the methods of Strickland et al. (1984). Net biological sulfate retention (Table 2) is expressed as: net retention = biological incorporation - (% mobilization x biological incorporation).

Statistical comparison with cation leaching data

Mean values for sulfate adsorption, gross biological sulfate incorporation, organic sulfur mobilization, and net biological sulfate retention for each site are presented in Table 2. Literature values for precipitation inputs and streamflow outputs were used to derive three indices of ecosystem wide leaching losses of Ca^{2+}, Mg^{2+}, and K^+ at each study site (Table 3). The first index of cation leaching loss used was simply the gross amount of streamflow output reported in the literature. Net cation leaching loss was calculated by subtracting amounts of precipitation input from this hydrologic output. In this case each resulting value was increased by an addition of 10.0 to prevent the presence of negative values in the data set. Finally,

Table 3. Cation leaching losses tor each study site. Gross losses reflect streamflow outputs (kg/ha/yr); net losses are corrected for precipitation inputs (kg/ha/yr. output-input + 10); loss ratios are output/input (no units).

Site	Ca^{2+}			Mg^{2+}			K+		
	Gross	Net	Ratio	Gross	Net	Ratio	Gross	Net	Ratio
Coweeta-WS2[a]	5.45	11..82	1.50	3.05	12.29	4.01	4.66	12.90	2.65
Coweeta-WS18[a]	7.03	13..03	1.76	3.49	12.64	4.11	4.93	12.95	2.49
Hubbard Brook[b]	13.90	21..70	6.32	3.30	12.70	5.50	2.40	11.50	2.67
H.J. Andrews[c]	123.10	129..50	34.19	8.60	17.40	7.17	9.50	18.60	10.56
Oakridge[b]	148.00	143.70	10.35	77.10	85.00	36.71	6.80	13.70	2.19
Tesuque-WS15[d]	11.47	14..67	1.69	2.63	1.1.93	3.76	2.07	11.20	2.38
Tesuque-WS8[d]	7.60	10..83	1.12	2.30	11.63	3.43	1.80	10.83	1.86
Tesuque-Aspen[d]	8.43	11..93	1.30	2.83	12.16	4.22	1.93	10.96	1.99
Tesuque-WS2[d]	3.57	8.54	0.71	1.97	11.44	2.51	0.'3	9.00	0.13
Tesuque-PJ[d]	5.17	10..77	1.18	1.73	11.10	3.13	0.i3	9.20	0.14
North Inlet[e]	205.64	210..34	38.80	17.62	25.69	9.13	5.95	14.60	4.41

Gross loss estimates derived from: [a] Swank and Waide (1988). [b] Likens et al. (1977). [c] Sollins et al. (1980), [d] Gosz (1980), [e] Blood unpublished data.

the ratio of cation output over input was used to indicate ecosystem sensitivity by expressing cation loss in the relative perspective of how much input a given site is experiencing. A low ratio indicates low sensitivity, and high values indicate high sensitivity due to large cation losses relative to inputs.

The relationships between sulfate retention processes and indices of ecosystem wide cation leaching loss were determined using Kendall's coefficient of rank correlation (Sokal and Rohlf 1981). This non-parametric test was used in lieu of parametric regression analysis because it could not be determined from the cation leaching loss data obtained from the literature whether these data met the parametric requirements of normality and homoscedasticity.

RESULTS

The forest sites in this study represented a wide range of soil and vegetation types, received varying amounts of yearly precipitation, and contained substantially different amounts of total C and total S (Table 1). It was therefore not unexpected that these sites exhibited considerably different levels of sulfate retention, both biological and abiotic. With

respect to nutrient cation inputs and outputs there was also considerable between site variability (Table 3), although most of the sites experienced a yearly net loss for each of the cations examined.

Sites differed significantly with respect to sulfate adsorption, biological incorporation, and organic sulfur mobilization (Table 2). Thus, these variables are site specific and any of them could potentially serve as indicator variables of ecosystem sensitivity. Kendall's coefficient of rank correlation was used to determine whether any of these variables had significant positive or negative relationships with ecosystem wide cation leaching loss. The results of these comparisons are shown in Table 4. Although many of the sites had substantial levels of sulfate adsorption in the A horizon, in only one case was there a significant rank correlation between sulfate adsorption and ecosystem wide cation leaching loss. This was between gross K^+ leaching loss and sulfate adsorption and the rank correlation was in the wrong direction. A negative rather than a positive rank correlation would be expected since higher levels of sulfate adsorption should lead to lower levels of cation leaching loss.

Biological sulfate retention indices,

Table 4. Rank correlations of sulfate retention parameters against ecosystem cation leaching loss. Leaching losses used are gross (total streamflow output), net (output-input + 10), and ratio (output/input).

Cation Loss	Sulfate Adsorption	Biological Incorporation	Organic Sulfur Mobilization	Net Biological Retention
Ca^{+2} Gross	-0.056[a]	-0.673**[b]	0.636**	-0.746**
Ca^{+2} Net	0.167	-0.709**	0.673**	-0.782**
Ca^{+2} Ratio	0.204	-0.709**	0.600**	-0.709**
Mg^{+2} Gross	0.426	-0.673**	0.782**	-0.746**
Mg^{+2} Net	0.352	-0.746**	0.782**	-0.782**
Mg^{+2} Ratio	0.278	-0.818**	0.855**	-0.891**
K^+ Gross	0.505*	-0.624**	0.618**	-0.624**
K^+ Net	0.426	-0.673**	0.564*	-0.673**
K^+ Ratio	0.389	-0.636**	0.455	-0.564*

[a] T values are from Kendall's coefficient of rank correlation (Sokal and Rohlf 1981).
[b] Significance at the 0.05 level is indicated by an asterisk; at the 0.01 level by two asterisks.

however, were significantly rank correlated to ecosystem wide cation leaching loss in all but one instance and in every case the rank correlation was in the direction expected (Table 4). Both gross biological sulfate incorporation and net sulfate retention were negatively correlated with cation leaching loss, and organic sulfur mobilization was positively correlated to cation loss. All three cation leaching parameters, gross loss, net loss, and output/input ratio, exhibited this pattern. Higher biological sulfate retention lead to lower ecosystem wide cation loss. Conversely, higher levels of organic sulfur mobilization (resulting in a re-release of sulfate) corresponded to significantly higher cation leaching loss.

DISCUSSION

During the past decade researchers have documented a strong correlation between physico-chemical sulfate adsorption in the B horizon and ecosystem wide sulfate retention (Johnson et al. 1982, Singh et al. 1980). Certainly this relationship is not unexpected due to the high amounts of adsorbing clays and metal sesquioxides found in many B horizon soils (Parfitt and Smart 1978, Singh 1984). Several studies have further documented that this type of sulfate retention reduces cation leaching, and that therefore the sulfate adsorption potential of a site's B horizon soil may act as a reliable predictor of ecosystem sensitivity to acidic precipitation (Johnson et al. 1980, Johnson et al. 1982). However, for the most part biological sulfate retention is not even mentioned in these reports (eg. Foster et al. 1986, Gobran and Nilsson 1988, Reuss and Johnson 1985, Von Freiesleben 1988). In fact, there are cases where researchers have debated the phenomenon of "permanent adsorption" without even considering that this may be due to biological sulfate incorporation (Johnson et al. 1979, Joyner 1987). This report is, to our knowledge, the first study to document the importance of biological sulfate retention processes as related to ecosystem sensitivity.

The results of this study clearly demonstrate that biological sulfate incorporation and retention in A horizon soils are directly related to nutrient cation movement through forest soil ecosystems. Sites with an A horizon capable of biologically incorporating substantial amounts of sulfate experienced less ecosystem wide cation leaching than those sites where biological sulfate incorporation in the A horizon was low or negligible. Although other studies have documented a similar relationship for physico-chemical sulfate adsorption and cation leaching loss (Johnson 1980, Rechcigl and Sparks 1985, Wilklander 1980), no such relationship was observed in the A horizon soils examined here in spite of the fact that there was a substantial amount of sulfate adsorption in these soils. The lack of a relationship is an unexpected result and may be due, at least in part, to the reversible nature of the ionic bonding involved in the adsorption process (Johnson 1980).

This information adds a new and important dimension to the classification of forest soil systems with respect to acidic precipitation sensitivity. This is because our results have clearly shown that microbially mediated sulfate incorporation operates very efficiently within the A horizon to retain incoming sulfate within the soil profile. The strong negative correlations between biological sulfate retention and cation loss solidly support the hypothesis that biological retention of sulfate retards charge associated leaching of nutrient cations. Conversely, organic sulfur mobilization releases water soluble sulfate into the soil profile, where it may be subject to leaching. Elevated levels of cation loss are observed in response to high levels of mobilization. Models of ecosystem sensitivity to acidic precipitation can no longer be considered complete unless they include these biological processes as variables.

These findings have specific implications with respect to site susceptibility to acidic precipitation. Surface soils, which have often been considered of minor importance with respect to acid precipitation "buffering" effects (Klopatek et al. 1980; Reuss and Johnson 1985) may be of considerable importance. Likewise it seems apparent that by regarding biological sulfate retention merely as "noise" in the adsorption database many researchers are making a false and dangerous assumption (Joyner 1987). As an index

of site susceptibility, net sulfate retention in A horizon soils appears to be very comprehensive, since it reflects not only gross incorporation, but organic sulfur mobilization capacity as well. Biological sulfate retention potentials measured under field conditions (Watwood and Fitzgerald 1988) have been found to correspond quite closely with laboratory derived results. The value of a biologically based index is further enhanced by the fact that once organic sulfur moieties have been formed, complete with covalent linkages, they become extremely recalcitrant (Strickland and Fitzgerald 1986). Thus, this soil organic sulfur fraction may be less susceptible to sulfate re-release than are many non-specific adsorptive ionic bonds, which can readily desorb sulfate depending on changes in environmental pH etc. (Johnson and Cole 1980).

The most thorough system of soil sensitivity classification would be one addressing both biological sulfate retention in the A horizon and physico-chemical adsorptive processes in the B horizon. However, for the sites included in this study, biological retention in the A horizon appears to be highly predictive of ecosystem cation loss. As more data regarding cation leaching estimates from additional forest sites becomes available, the current data analysis should be expanded to include many additional sites for which sulfate retention data have already been determined. An expansion of the current analysis will hopefully result in a normal data set so that parametric multiple regression techniques can be used to produce a truly predictive model relating biological sulfate retention to ecosystem sensitivity to acidic precipitation.

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