Soil processes and sulfate loss at the Hubbard Brook Experimental Forest

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Key words: Sulfur cycling, sulfate adsorption, clear cutting, stream chemistry

Abstract. The Hubbard Brook Ecosystem Study was designed to evaluate element flux and cycling in a northern hardwood forest and the effects of disturbance on these processes. In the original experiment, an entire watershed was deforested and regrowth was inhibited for three years using herbicides. Initial effects of the treatment included: elevated stream discharge, large increases in streamwater solute concentrations and elevated losses of those ions from the watershed. In contrast, streamwater concentrations and net ecosystem output of sulfate decreased in response to the treatment. During the post treatment period, the concentrations of most dissolved ions declined relative to a reference watershed while, again in contrast, sulfate concentrations increased relative to the reference. In this paper we develop a hypothesis which links acidification and sulfate adsorption processes in the soil to explain the observed trends in sulfate losses from the Hubbard Brook Experimental Forest.

Introduction

During November and December of 1965, Watershed 2 (W2) of the Hubbard Brook Experimental Forest (HBEF), New Hampshire, was clear cut. All vegetation was felled and left in place and regrowth was inhibited by periodic application of herbicides. Stream discharge increased, as did streamwater concentrations and efflux of many dissolved ions (Likens et al. 1970). In contrast, streamwater sulfate concentrations and export declined in response to the disturbance. Forest harvests by conventional methods (e.g. strip, clear-, and whole-tree cuttings) at the HBEF and elsewhere in New Hampshire also have resulted in reduced streamwater sulfate concentrations (Likens & Bormann 1974; Martin et al. 1984, 1986; Fuller et al. 1987). In this paper we develop a hypothesis which links acidification and sulfate adsorption processes in the soil to the observed trends in sulfate losses at HBEF.

Fig. **1. Annual volume weighted concentrations of dissolved substances by water year in streams draining experimental Watershed 2 (solid line) and reference Watershed 6 (dashed line). Shaded area indicates treatment years. (Data from the Hubbard Brook Ecosystem Study).**

Review of the W2 experiment

Deforestation of W2 produced two distinct periods of response. The treatment phase extended through the three water years from 1965-66 through 1967-68 (water years begin 1 June at HBEF; see Likens et al. 1977). During this first period the forest was cut and herbicide treatments applied. The post treatment phase, which was a period of vegetation regrowth, extends through the present.

The treatment phase was characterized by large increases in streamwater concentrations in all measured solutes except sulfate which declined to about half the pre-treatment levels during the second year of the experiment (Fig. 1). Nitrate concentrations demonstrated a particularly marked response, increasing from about $20 \mu\text{Eq} \cdot \text{L}^{-1}$ to concentrations greater than 1300 μ Eq \cdot L⁻¹ during the second year of the treatment (Likens et al. 1970). Concentrations of calcium, magnesium, potassium, and aluminum also reached peak values during the second year. Hydrogen ion concentrations increased at least 4-fold, resulting in a decrease in the weighted average pH from 5.1 during the 1965-66 water year to a low of 4.3 during 1968-69. Deforestation and the elimination of transpiration resulted in **39%** and 28% greater streamflow than expected relative to W6 during 1967-68 and 1968- 69 water years, respectively (Likens et al. 1970) (Fig. 1). The combination of increased annual discharge and elevated stream concentrations resulted

Fig. 2. Annual solute budgets (bulk precipitation inputs - stream outputs) by water year for W2 (solid line) and W6 (dashed line). (Data are from the Hubbard Brook Ecosystem Study).

in elevated ecosystem outputs for nitrate, hydrogen ion, calcium, magnesium, and potassium during the treatment period. In contrast to the response of other ions, declines in streamwater sulfate concentrations offset the increased streamflow and resulted in reduced annual sulfate efflux in W2 relative to the reference watershed (Fig. 2). This reduced sulfate output occurred despite the fact that over fifty percent of the forest floor was lost during the treatment phase (Dominski 1971) which should have resulted in extensive mineralization of sulfur pools.

During the post-treatment period, streamwater concentrations of previously elevated ions declined, while streamwater sulfate concentrations increased, and differences between annual streamflow from the experimental and reference watersheds were reduced (Fig. I). Annual weighted concentrations of nitrate and hydrogen ion declined sharply during the post-treatment phase and have remained lower than the corresponding concentrations in the reference watershed since 1972. Sulfate outputs became larger in W2 than W6 (Fig. 2) despite the fact that both organic matter and nitrogen were accumulating in the disturbed forest through regeneration of the forest floor and above ground biomass (Bormann & Likens 1979). Annual weighted sulfate concentrations increased and have remained higher than the reference watershed since 1970 (Fig. 1). Sulfate concentrations peaked at $170 \,\mu\text{Eq} \cdot \text{L}^{-1}$ in 1971–72, when the corresponding value for W6 was 130. Differences in streamwater sulfate concentrations between the two watersheds have narrowed with time. Both annual sulfate concentations and

yearly streamflow remain higher in W2 resulting in greater sulfate output relative to W6 (Fig. 2).

Concentrations of nitrate and sulfate were inversely correlated in W2 both during the treatment years and the water years preceding the treatment (Likens et al. 1970). Streamwater concentratins of the two anions have also shown a consistently negative correlation in the reference watershed (Nodvin 1983).

Mechanisms of accelerated export

Mechanisms responsible for the elevated stream concentrations in W2 during the treatment phase include accelerated mineralization of organic matter and the absence of nutrient uptake by vegetation (Likens et al. 1978). More importantly, the effects of the disturbance on the nitrogen cycle were principally responsible for the greatly increased export of solutes (Likens et al. 1970). Deforestation induced high net production of nitrate by mineralization and nitrification as evidenced by elevated populations of Nitrosomonas and Nitrobacter (Smith et al. 1968) and dramatically increased nitrate concentrations in the soil (Dominski 1971). Since nitrate is retained only weakly by soils at HBEF (Nodvin et al. 1986a) and plant uptake was absent during the treatment years, streamwater nitrate concentrations rose sharply (Fig. **l),** balancing net increases in streamwater cations and net decreases in other inorganic anions (Likens et al. 1969).

Sulfate retention

Adsorption reactions within soils can regulate aqueous sulfate concentrations in forest ecosystems (Johnson & Cole 1980). The adsorption of sulfate by soils is strongly influenced by solution pH (Chao et al. 1964; Parfitt 1978). We hypothesize that the following series of mechanisms acted to regulate streamwater sulfate concentrations in disturbed W2:

- acidification of soil during deforestation was driven by accelerated nitrification rates and increased net releases of nitric acid;
- soil acidification induced the protonation of surface adsorption sites thereby enhancing the ability of the soil to retain sulfate; and
- increased retention of sulfate contributed to reduced streamwater sulfate concentrations and outputs.

We also hypothesize that similar mechanisms act in the regulation of sulfate concentrations in undisturbed watersheds at HBEF.

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Methods

We evaluated the sulfate retention hypothesis through:

- a series of experiments on the pH dependent adsorption of sulfate by HBEF soil;
- an estimation of acidity produced by nitrification in W2 during the deforestation to compare with the amounts of acid required to affect sulfate adsorption in laboratory experiments; and
- an evaluation of seasonal trends in streamwater solution concentrations of sulfate, nitrate, and hydrogen ion in the reference watershed.

Laboratory adsorption experiments

Adsorption experiments were conducted using a composite sample of B horizon soil (20–60 cm depth), collected from a site adjacent to W6 at the HBEF. HBEF soil is described as a well drained Spodosol (Typic Fragiorthod) of the Becket series (Likens et al. 1977; Driscoll et al. 1985; Likens et al. 1985). The inorganic B horizon soil was studied since anion adsorption is primarily associated with two mineral components of soil: variable-charge clays and amorphous aluminum and iron oxide coatings (Parfitt 1978).

Experiments were conducted with soil suspension $(10g \text{ soil with } 100 \text{ ml})$ solution). Each suspension was equilibrated for $24 h$ at 20° C. Soil particles were removed from solution following equilibration by centrifugation. Four initial concentrations of sulfate, added as sodium sulfate, were used; 0, 42, 104, and 146 μ Eq·L⁻¹. The pH was modified by adding either nitric acid or sodium hydroxide to the solutions at the beginning of each experiment. Nitric acid was used to lower the pH since nitrate is non-specifically adsorbed by soils and should have little effect on sulfate adsorption (Nodvin et al. 1986a). Following each experiment, the equilibrium solution pH was measured using a glass electrode and the equilibrium sulfate concentration was measured using dual column ion chromatography (Tabatabai & Dick 1983).

Estimation of excess acid production

We estimated the of excess acid produced by nitrification in the experimental watershed from the amounts of nitrate output from the W2 in excess of the nitrate output from the reference watershed (Henderson et al. 1980). This procedure assumes that the excess net nitrate output is an estimator of induced nitrification and that one mol of acidity is produced for each mol of excess nitrate produced (Binkley & Richter 1987).

Seasonal trends in streamwater solution concentrations

Mean monthly weighted streamwater concentrations for sulfate, nitrate, and hydrogen ion from W6 at HBEF were obtained from the data base of the Hubbard Brook Ecosystem Study. Data were compiled for 19 water years from 1965-66 through 1983-84. Mean values and confidence intervals of the monthly weighted concentrations over the nineteen-year period were computed for use in a seasonal trend analysis.

Results and discussion

Laboratory adsorption experiments

The soil equilibration experiments demonstrated that changes in the acidbase status of mineral soil at HBEF significantly affected the ability of the soil to retain or release sulfate. Sulfate retention or release depended upon both the amount of sulfate initially added to the soil suspension and the pH of the equilibrated solutions (Fig. **3.).** (See Nodvin et al. (1986b) for a quantitative description of the results of these experiments). The mean pH of equilibrated solutions to which no acid or base had been added (the ambient pH) was 4.8. The ability of the soil to retain sulfate increased as the pH was decreased to pH 4.0 where maximum retention was evident (Fig. **3).**

Changes in the surface charge of free (non-silicate bound) aluminum and iron coatings were probably responsible for the increase in sulfate retention with decreasing solution pH. As soil pH is lowered, the net positive charge of the oxide surfaces increases and this process is believed to facilitate increased anion adsorption (Johnson & Cole 1980). Chao et al. (1964) demonstrated the importance of the composition of the surface coatings to sulfate adsorption and that aluminum oxide-coated soils show a maximum adsorption near pH 4, whereas iron oxide-coated soils show increasing adsorption with decreasing pH. At HBEF, insoluble sulfate is correlated with both iron and aluminum soil fractions but more strongly correlated with aluminum fractions (Fuller et al. 1985).

Estimation of excess acid production due to deforestation

We estimate that deforestation of W2 resulted in the production of 10.4 kmol per hectare of acidity associated with net nitric acid production during 1967-68, the water-year of peak nitrate discharge (Table 1). With this

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Fig. 3. Sulfate removal or release in response to initial sulfate added and equilibrium pH of solutions in the adsorption experiments. (Positive values indicate net removal and negative values indicate net release).

Water	Net budget of nitrate (Precip input-stream output)		Excess Nitrate produced	Estimated Acidity produced ¹
Year				
	W ₂	W ₆		
	$-eq \cdot ha^{-1}$			
1964-65	3	31	28	28
1965-66	149	188	39	39
1966-67	6939	236	7175	7175
$1967 - 68$	-10166	174	10339	10339
1968-69	7331	72	7403	7403
1969-70	6456	-204	6252	6252
1970-71	2648	49	2697	2697
$1971 - 72$	21	86	107	107
$1972 - 73$	366	144	-221	-221

Table 1. Excess nitrate and estimated excess net hydrogen ion produced in W2 above that in W6 at HBEF.

¹ Estimate assumes one mole of excess hydrogen ion produced per mole of excess nitrate output (see 'Methods').

Fig. **4. Titration curve of HBEF B horizon soil: equilibrium pH of soil solutions as a function of the amount of acid or base added. Results obtained from the sulfate adsorption experiments.**

estimation we can place the results of the sulfate adsorption experiments in perspective with the deforestation. Since there are approximately $173,000 \text{ kg} \cdot \text{ha}^{-1}$ of soil in the top 45 cm of mineral soil at HBEF (Bormann & Likens 1979), the amount of acidity produced during deforestation was equivalent to having added 60 microequivalents of acid per gram of soil to the surface mineral soil during that one year. If we assume similar pHbuffering mechanisms, the additions of this much nitric acid to soil in the sulfate adsorption experiments would have lowered the solution pH from 4.8 to 3.7 (Fig. 4), resulting in a marked increase in the ability of the soil to retain sulfate (Fig. 3; see Nodvin et al. 1986b).

Soil solution data are not available for the W2 experiment. However, the above analysis combined with the observation that stream water draining the W2 deforested catchment decreased by almost one pH unit during the treatment phase (Likens et al. 1970) suggest that deforestation substantially reduced the pH of the soil solution in W2. In a recent experiment at HBEF, the pH of the B-horizon soil solution was significantly lowered by as much as eight-tenths of a pH unit due to whole-tree harvesting of an experimental watershed (Fuller et al. 1987). We expect the change in soil solution pH due to deforestation of W2 should have been even greater due to the increased severity of the earlier experiment.

Fig. 5. Seasonal trends in streamwater chemistry in Watershed 6: nineteen year averages (water years 1965-66 through 1983-84) of monthly weighted concentrations and 95% confidence **intervals for sulfate, nitrate, and hydrogen ion. (Data from the Hubbard Brook Ecosystem Study).**

Seasonal trends in streamwater concentrations

There were significant seasonal trends in streamwater concentrations of hydrogen ion, nitrate, and sulfate in the reference watershed (W6) at HBEF based upon of 19 year mean weighted monthly concentrations (Fig. 5). The

Fig. *6.* **Trends in monthly weighted sulfate and nitrate concentrations in stream water and of** soil nitrate-nitrogen concentrations during 1973-74 and 1974-75 water years in forested **Watershed 6. (Stream data from the Hubbard Brook Ecosystem Study; soil data from Melillo, 1977).**

seasonal variations in the concentrations of hydrogen ion and nitrate were positively correlated, but the trends for these two ions negatively coincided with the seasonal trend for sulfate. Monthly sulfate and nitrate concentrations were negatively correlated within years but concentrations taken from any one month over the nineteen-year period were poorly correlated (Nodvin 1983). Therefore the seasonal variations in streamwater chemistry were responsible for the negative correlation between sulfate and nitrate. These trends suggest that seasonal variations in the biotic uptake, assimilation and release of nitrate and ammonium alter the acid/base chemistry of HBEF soils and may influence streamwater sulfate concentrations in forested watersheds at HBEF.

When W2 was deforested, nitrate and hydrogen ion concentrations increased in stream water whereas sulfate concentrations declined. In the nineteen year record of undisturbed W6, nitrate and hydrogen ion concentrations in the stream generally increased during the autumn and winter months and reached peak values in the early spring, at which time sulfate concentrations were lowest (Fig. 5). The concentrations of nitrate decreased precipitously in the late spring during the period in which both snowmelt and vegetation leaf-out occurred. Hydrogen ion also declined in late spring when sulfate concentrations began to increase. Peak sulfate concentrations were found in late summer when nitrate values were at a minimum and hydrogen ion concentrations were also low. Nitrate concentrations remained low during the summer and increased significantly between October and November after leaf-fall. Nitrate concentrations were therefore lowest during the growing seasons when hydrogen ion concentartions were also low and sulfate concentrations were high.

The seasonal trends for nitrate in stream water correspond to trends in the extractable nitrate content of the soil based upon data from Melillo (1977; Fig. 6). Melillo's data demonstrated that soil nitrate concentrations were at a minimum during the summer growing season when nitrification rates measured in his study were highest. It, therefore, appears that plant root uptake acted to maintain low nitrate concentrations in the ecosystem during the growing season. Continued soil nitrification activity in the winter coupled with minimal root uptake, allowed nitrate to accumulate within the soil. In May, leaching of the soil by snowmelt water and then root uptake probably served to reduce the nitrate concentrations in soil and drainage waters to minimum concentrations. During the winter months, the rate of nitrification exceeded the rate of nitrate uptake and acidity was produced resulting in an 'acid push' within the soil (Matzner & Ulrich 1985; Rascher et al. 1987). This accumulation of nitrate, as nitric acid, may have contributed to the production of acidic episodes in stream waters during snowmelt at HBEF as has been observed at HBEF and other surface waters in the northeastern United States (Johnson et al. 1969; Driscoll & Schafran 1984).

Small seasonal changes in hydrogen ion concentrations of streams may reflect seasonal variations in the acidity of surface soil solutions. The much smaller seasonal variations in the hydrogen ion concentrations in streams compared to that in precipitation (Likens et al. 1977, 1985) demonstrate the ability of the terrestrial ecosystem to moderate external influences. The more soil through which water passes, the more strongly buffered the water chemistry. For example, Johnson et al. (1981) found that the variance in mean streamwater pH measured over three years, decreased with decreasing elevation in an undisturbed stream at HBEF.

The linkage between nitrification and sulfate retention in the soil of undisturbed watersheds is complex. Nitrification rates were highest in the summer yet stream and presumably soil solution acidities were lowest at this season. These observations suggest that root uptake reduces nitrate concentrations in stream and soil water. For most plants, root uptake of anions from soils exceeds the uptake of cations (Noggle 1966). Electroneutrality is maintained in the plant either by the absorption of $H⁺$ or the excretion of either HCO₃ or OH⁻ (Davies 1973; Higinbotham 1973; Raven & Smith 1974). The absorption of nitrate by roots appears to contribute to the reduction in hydrogen ion concentrations within the ecosystem during the growing season. The seasonal trends in stream chemistry suggest that over an annual cycle, root uptake and nitrification processes interact to modify the acidity status of the soil and thereby regulate sulfate movement at HBEF.

Summary

Deforestation of W2 at HBEF led to increased streamwater concentrations and ecosystem losses of most dissolved substances. Increased losses were the result of elevated stream discharge and elevated streamwater concentrations. Increased mineralization, increased rates of nitrification, and decreased plant uptake all directly contributed to raising streamwater concentrations. Increased acid production in the soil due to the net release of nitric acid indirectly induced elevated streamwater concentrations through cation exchange and mineral dissolution. In contrast, the disturbance caused decreased stream concentrations and outputs of sulfate.

According to our hypothesis, sulfate concentrations and output were lowered in W2 through the interaction of several processes. Deforestation facilitated the net release of nitrate from the soil which in turn increased acid production. Soil acidification increased the net positive charge on soil surfaces thereby enhancing the ability of the soil to retain sulfate. Increased sulfate retention within the soil lowered solution concentrations in soil and

stream waters. The reductions in sulfate streamwater concentrations more than offset the elevated discharge rates during the treatment period in W2 causing a net reduction in sulfate output.

We demonstrated that acidification of soil at HBEF increased the ability of the soil to retain sulfate and that estimated acid production in W2 due to deforestation would have been large enough to decrease soil solution pH and increase sulfate retention by the soil. Seasonal trends in solution concentrations in W6 suggest that the mechanisms proposed are also relevant in undisturbed forested systems and function to regulate soil water and streamwater sulfate concentrations in those systems.

During the post-treatment period, hydrogen ion and nitrate concentrations declined and sulfate increased in W2 stream water. This observation suggests that a long-term effect of deforestation and regrowth may be to lower the acidity of the soil through strong nitrate retention and thereby decrease the ability of the soil to retain sulfate.

Ecosystems are complex and each of the myriad effects from a major perturbation such as the deforestation of W2 may not be the result of a single process or mechanism. Other mechanisms have been proposed for the response of sulfate to disturbance at HBEF (Likens et al. 1970). However the hypothesis presented here is the only proposed mechanism which is consistent with observations on soil and streamwater chemistry in both disturbed and undisturbed ecosystems at HBEF (Nodvin 1983; Fuller et al. 1987).

Acknowledgements

This is a contribution of the Hubbard Brook Ecosystem Study. Financial support was provided by a National Science Foundation Pre-Doctoral Fellowship to S.C. Nodvin and NSF grants to G.E. Likens.

We thank John Eaton for compiling long-term stream and precipitation data. We thank Robert Fuller, Dan Binkley, Dale Johnson, Jerry Melillo and an anonymous reviewer for many helpful comments.

The United States Department of Agriculture Forest Service, Broomall, Pennsylvania operates and maintains the Hubbard Brook Experimental Forest.

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