NOTE

Throughfall chemistry beneath *Quercus rubra:* atmospheric, foliar, and soil chemistry considerations

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Concentrations of inorganic ions were measured in bulk rainfall and bulk throughfall collected beneath northern red oak *(Quercus rubra* L.) trees growing in fertile, limestone-derived soil and less fertile sandstone/shale-derived soil. Rainfall passing through the crowns at both sites was enriched with SO_4^{2-}, PO_4^{3-}, Ca^{2+}, Mg^{2+}, K^+, Mn^{2+}, and Fe^{2+}, but lost NH_4^+ to the crowns. Concentrations of Na^+ and H^+ in incident rainfall were higher at the infertile site. There was no difference in ionic leaching from red oak crown leaves exposed to simulated acid rain solutions. Rainfall volume, leaf area, and large particle dryfall ionic concentrations were all larger at the fertile rather than the infertile site. Concentrations of Ca^{2+}, Mg^{2+}, NH_4^+, Mn^{2+}, and SO_4^{2-} measured in throughfall at the fertile site were higher than those of the infertile site. These differences in throughfall chemistry are likely due to site differences in *(i)* the ability of tree crowns to capture dry deposition, *(ii)* dry deposition chemistry and occurrence, and *(iii)* rainfall volume.

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Les concentrations d'ions inorganiques ont été mesurée dans la précipitation totale et dans la precipitation au sol recueillie sous un couvert de chênes rouges *(Quercus rubra* L.) croissant sur un sol fertile dérivé de calcaire et un sol moins fertile dérivé d'ardoise et de grb. La précipitation passant à travers les cimes aux deux endroits était enrichie de SO_4^{2-}, PO_4^{3-}, Ca^{2+}, Mg^{2+}, K^+, Mn^{2+} et Fe^{2+}, mais il y avait une perte de NH_4^+. Les concentrations de Na^+ et H^+ dans la précipitation totale Ctaient supérieures à I'endroit du sol peu fertile. On n'a décelé aucune différence dans le lessivage ionique des feuilles des cimes du chêne rouge exposées à des solutions simulées de pluies acides. Le volume de précipitation, la surface foliaire ainsi que les concentrations ioniques des grosses particules sbches Ctaient tous plus grands à l'endroit du sol fertile qu'à celui du sol peu fertile. Des concentrations plus fortes de Ca^{2+}, Mg^{2+} NH_4^+, Mn^{2+} et SO_4^{2-} ont été mesurées dans la précipitation au sol à I'endroit du sol fertile qu'à celui du sol peu fertile. Ces différences dans la chimie de la précipitation a sol sont vraisemblablement dues à des différences dans *(i)* la capacité des cimes des arbres de capter les dépôts secs, *(ii)* la prévalence et la chimie des dépôts secs et *(iii)* le volume de la précipitation.

[Traduit par la revue]

Introduction

The forest canopy is the initial point of contact between rain influenced by air pollutants and forest ecosystems. The chemical composition of throughfall differs from rainfall. Hardwood and coniferous forest throughfall is generally higher in K, Ca, and Mg, and may be higher or lower in NO; and SO_4^{2-} content than incident precipitation (Eaton *et al.* 1973; Cronan and Reiners 1983; Mollitor and Raynal 1983; Lindberg *et al.* 1986). Hardwood throughfall during the growing season usually has higher pH than rain; conifer throughfall often has equal or lower pH than rain (Nihlgard 1970; Mollitor and Raynal 1983; Cronan and Reiners 1983). Stand structure (Olson *et al.* 1981) and canopy structure (Reiners and Olson 1984) also influence throughfall chemistry.

Washing of large-particle dryfall and leaching of substances from leaves are dominant processes in throughfall chemical enrichment (Parker 1983). Foliar absorption of nutrients in low supply (e.g., N) may also occur. Foliar nutrient status has been increased with fertilizers and found to affect throughfall enrichment (Khanna and Ulrich 1981). Other studies suggest that foliar nutrient status is an indicator of throughfall enhancement (Eaton *et al.* 1973; Henderson *et al.* 1977).

The objective of this study was to examine the influence of foliar nutrient status on inorganic ion concentrations of throughfall. Northern red oak (Quercus *rubra* L.) growing on sites with either low or high soil fertility were used to test this relationship. Rainfall and throughfall at each site were collected and chemically analyzed. Northern red oak leaves were treated with simulated acid rain solutions to determine differences in ion leaching between sites.

Materials and methods

Study sites

Two forested sites were chosen because of their different soil nutrient status. One site was located in the Shenandoah National Park (SNP), Greene County, Virginia (38°21 'N latitude, 78°32'W longitude). Soils of the SNP are classified as dystrochrepts (USDA 1980), i.e., either loamy-skeletal, mixed, mesic rupticalfic (Catoctin series), or loamy-skeletal, mixed, mesic typic (Parker series). These

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TABLE 1. A comparison of surface soil horizon chemistry for Shenandoah National Park (SNP), Greene Co., VA, and Fernow Experimental Forest (FEF), Tucker Co., WV

Variable”	SNP	FEF
N (ppm)	0.07	**0.21***[b]
P (ppm)	3.50	5.00 ns
K (ppm)	37.38	**56.00***
Ca (ppm)	12.00	**138.00***
Mg (ppm)	5.88	**22.00***
Zn (ppm)	1.13	**2.38***
Mn (ppm)	7.75	**79.25***
A1 (ppm)	379.75	301.75 ns
Organic matter (%)	7.45	**10.20***
pH	4.48	4.38 ns

Total N, extractable P, K, Ca, Mg, Zn, Mn, and Al.

[b] Student's t-test results; * indicates a significant site difference at $P \leq 0.05$, and ns indicates no site difference.

soils are derived from sandstone and shale parent material of the Swift Run and Pedlar geologic formations (Gathright 1976). The site supports an upland oak community which includes northern red oak, chestnut oak (*Quercus prinus* L.), white oak (*Q. alba* L.), and eastern white pine (*Pinus strobus* L.). The SNP site is on the west-southwest facing side of a ridge with an average slope of 50%. The site has a mean elevation of 615 m and receives an average of 109 cm precipitation annually. Northern red oak site index was calculated to be 54 ft (16.46 m) (base age 50 years) using age and height measurements, and a published site index equation (Hahn and Carmean 1982).

The second site was located in the Fernow Experimental Forest (FEF), Tucker Co., West Virginia (39°3′N latitude, 79°40′W longitude) on a fine-loamy, mixed, mesic typic hapludalf of the Belmont soil series (USDA 1980). This soil is derived from calcareous shale, sandstone, and limestone parent material (Auchmoody 1972). The FEF site supports northern hardwood forest species including northern red oak, chestnut oak, yellow-poplar (*Liriodendron tulipifera* L.), and sugar maple (*Acer saccharum* Marsh.). The site faces north northeast, with an average slope of 30%, mean elevation of 725 m, and receives 147 cm of mean annual precipitation. Northern red oak site index at base age 50 years is 85 ft. (25.91 m) (Yawney 1964).

Soil sampling method

Eight samples were collected from the A soil horizon (defined as 0-20 cm in depth) of each site using a 2.5-cm punchtube. Soil was sampled beneath four randomly selected codominant trees of each of two species on both sites to determine the representative soil chemistry. Soil was sampled at the SNP beneath four eastern white pine and four northern red oak trees during the 1982 growing season. FEF soil was sampled during the 1983 growing season beneath four black cherry (*Prunus serotina* Ehrh.) and four northern red oak trees. Prior to chemical analyses, soils were dried at room temperature and passed through a 2-mm screen.

Leaf collection and leaching methods

Northern red oak leaves were sampled from three randomly selected codominant trees on each site, at three 6-week intervals beginning on June 25, 1984. Trees were climbed and, with a pole pruner, leaves were removed from six to eight positions representative of the lower to middle portions of the crown. Leaves were kept in plastic bags, on ice, in an insulated cooler no longer than 48 h while in transit to the laboratory. Leaves were refrigerated an additional 48-72 h prior to experimental treatments. Leaf areas were measured with a Licor model Li-3000 portable leaf area meter.

TABLE 2. Leaf areas and average concentrations of 11 nutrient elements in northern red oak leaves collected three times during the 1984 growing season at Shenandoah National Park (SNP), Greene Co., VA and Fernow Experimental Forest (FEF), Tucker co., WV

Nutrient’ element	June 25-26		Aug. 6-7		Sept. 26-27	
	SNP	FEF	SNP	FEF	SNP	FEF
S (%)	0.12	**0.16***[b]	0.13	0.14 ns	0.13	0.14 ns
N (%)	2.45	**2.86***	2.32	**2.71***	2.34	**2.43***
P (%)	0.13	**0.18***	0.19	**0.23***	0.22	0.22 ns
K (%)	1.44	**1.71***	1.15	**1.33***	0.92	**1.07***
Ca (%)	0.56	**0.67***	0.78	**0.90***	0.80	**0.84***
Mg (%)	0.19	**0.13***	0.20	**0.14***	0.20	**0.13***
B (ppm)	18	**22***	16	**22***	36	**53***
Zn (ppm)	24	**28***	27	**31***	32	31 ns
Mn (ppm)	637	**808***	782	**1075***	684	**754***
Cu (ppm)	9	**12***	4	**7***	6	7 ns
Fe (ppm)	56	**49***	58	57 ns	62	58 ns
LA (cm’)	111	**136***	112	**120***	99	**130***

‘Nutrient elements are in total forms and are pooled from leaves treated and untreated with rain solutions. LA, leaf area.

‘Significant site differences at $P \leq 0.05$ are indicated by *; ns indicates no site difference.

Leaves from each site were exposed to a simulated rain solution of pH 4.3 following the three harvests. Simulated rain solutions contained inorganic ions in proportions measured in rainswhich occurred in southwestern Virginia (Chevone *et al.* 1984). Approximately 6 g of leaves, selected from the total collection at each site, were placed in 18 x 20 cm clear, plastic-zippered bags containing 250 mL of the simulated rain solution. Rain solution was placed in bags without leaves to serve as controls. Following a 2-h treatment, leaves were removed from the bags and prepared for chemical analyses. Leaves not exposed to simulated rain solutions were also chemically analyzed. Solution pH was measured before contact with leaves, and immediately after leaves were removed from bags, Inorganic ion concentrations were measured in control and treatment rain solutions. Prior to chemical analyses, leaves were dried at 70°C for 24 h, and ground to pass through a l-mm screen.

Atmospheric deposition sampling methods

Bulk throughfall and bulk rainfall were collected weekly for 16 weeks beginning June 5 and ending September 25, 1984. Collectors consisted of a 20-cm diameter polyethylene funnel and a 250-mL polyethylene bottle connected by 75 cm of clear Tygon tubing. Collectors were mounted 1.8 m above the ground on metal posts. Bulk rainfall collectors were placed in a clearing near each site. Bulk throughfall collectors were located beneath the same three trees on each site from which the leaves were collected. Glass wool, rinsed in deionized water to remove trace elements, was placed in the neck of each funnel to exclude contaminants. Each week collectors were cleaned with deionized water and new glass wool was placed in the funnels.

Chemical analyses

A double acid extraction procedure (University of Georgia Soil Testing and Plant Analysis Laboratory, Athens, Georgia) was used to determine concentrations of P, K, Ca, Mg, Mn, Zn, and Al in soil. Soil extracts were analyzed by a Jarrell-Ash model 955 Atom Comp argon plasma emission spectrometer. Total N was determined by the macro-Kjeldahl procedure. A glass pH electrode and reference electrode were used to determine soil pH in a 1: 1 mixture of soil and deionized water. Percent organic matter in soil was determined by wet oxidation with potassium dichromate.

Nutrient element concentrations of leaves collected in June and August were analyzed by the Virginia Tech Soil Testing and Plant Analysis Laboratory, Blacksburg, Virginia. Concentrations of total

Ca, Mg, Mn, Zn, Cu, and Fe were measured by atomic absorption spectrophotometry. Leaf total P and total B were determined colorimetrically, and total K was determined by flame photometry. Nutrient element concentrations of leaves harvested in September were determined by a Jarrell-Ash model 750 arc spark emission spectrograph, at the University of Georgia Soil Testing and Plant Analysis Laboratory, Athens, GA. Both labs used a micro-Kjeldahl technique to measure leaf total N, and a Leco sulfur analyzer to measure leaf total S. There were no differences in nutrient element concentrations (t-tests, $P \leq 0.05$) in the chemical analyses of 10 duplicate samples sent to each laboratory.

Concentrations of total P, K, Ca, Mg, Mn, Fe, B, Mo, and Na, in rainfall and throughfall samples were determined by a Jarrell–Ash model 955 Atom Comp argon plasma emission spectrograph. Concentrations of ammonium and nitrate (following reduction to ammonium) were determined by distillation of basic ammonium and titration to an end point (University of Georgia Soil Testing and Plant Analysis Laboratory). Sulfate concentrations were determined by the turbidimetric method (Anonymous 1983) using a model 900-3 Klett-Summerson photoelectric colorimeter. Rainfall and throughfall pH were determined using a Beckman combination pH electrode with an Ag/AgCl reference element. Electrical conductivity was measured using a Thomas model 275 linear conductance/resistance meter.

Results

Site characterization comparisons

Soil chemistry

Student's t-tests were used to compare nutrient element concentrations in soil between sites. FEF soil had greater concentrations of total N, extractable P, K, Ca, Mg, Mn, Zn, and greater percent organic matter than SNP soil (Table 1). Concentrations of Al and pH of the two soils did not differ.

Northern red oak crowns

One sample of leaves representing the total collection from each tree per harvest was exposed to rain solutions and used for chemical analyses. Chemical variables in leaves and in simulated rain solutions were compared between sites for each harvest using analysis of variance and a 2 (sites) x 3 (trees/site) design. Concentrations of total N, P, K, Ca, Mn, Zn, S, B, and Cu were greater in FEF than SNP leaves (Table 2). However, SNP leaves had higher concentrations of Mg and Fe than FEF leaves. Differences in N, K, Ca, Mg, B, and Mn between sites were significant ($P \leq 0.05$) at all harvests. The magnitude of nutrient ion exchange was similar between simulated rain solutions and leaves with high (FEF) and low (SNP) nutrient element concentrations (Table 3).

Rain chemistry

Student's t-tests were used to compare nutrient element concentrations in bulk rainfall. Average concentrations of Ca^{2+}, Mg^{2+}, K^+, NH_4^+, NO;, SO_4^{2-}, PO_4^{3-}, $B(OH)_4^-$, Zn^{2+}, Mn^{2+}, Fe^{2+}, MoO_4^-, and conductivity did not differ between the sites (Table 4). The average concentration of Na^+ in rain at SNP was more than twice that at FEF ($P < 0.05$). Rainfall pH at the FEF (pH 4.69) was lower than the SNP (pH 5.57). Chemical variables of wet-only rainfall from National Atmospheric Deposition Program/National Trends Network (NADP/NTN) preliminary data for both sites were compared by Student's t-tests. Rainfall at SNP, FEF, and NADP/NTN sites were collected contemporaneously. The NADP/NTN sampler WV 18 is about 5 km from the FEF site and NADP/NTN sampler VA 28 is about 24 km from the SNP site. Rain collected at NADP/NTN sampler VA 28 had higher concentrations of K^+, and NH_4^+, and higher pH ($P < 0.05$) than rain at NADP/NTN WV 18.

TABLE 3. Exchange of eight nutrient ions (mequiv./m^2 leaf area) between simulated rain solutions of pH 4.3 and northern red oak leaves harvested three times during the 1984 growing season at the Shenandoah National Park (SNP), Greene Co., VA and the Fernow Experimental Forest (FEF), Tucker Co., WV

Nutrient variable	June 25-26		Aug. 6-7		Sept. 26-27	
	SNP	FEF	SNP	FEF	SNP	FEF
Ca^{2+}	-0.10	-0.05	0.02	0.02	0.16	0.07
Mg_{2+}	0.02	0.01	0.03	0.01	0.08	0.02
K^+	0.57	0.82	0.17	0.23	0.42	0.40
NH_4^+	0.00	0.04	-0.15	-0.06	-0.07	-0.04
Mn^{2+}	0.00	0.00	0.00	0.01	0.02	0.01
PO_4^{3-}	0.01	0.11	0.10	0.06	0.04	0.04
so:-	0.15	0.10	0.02	0.05	-0.02	-0.04
NO;	-0.40	-0.34	0.00	0.16	0.00	0.00

NOTE: Positive values indicate ions leached from leaves, negative values indicate ions retained by leaves. Site differences were all nonsignificant (F-tests, $P \leq 0.05$).

Site process comparisons

Rainfall to throughfall transformation

A one-way analysis of variance was used to compare chemical variables in bulk rainfall and throughfall within each site for 16 weeks. Concentrations of Ca^{2+}, Mg^{2+}, K^+, Mn^{2+}, and Fe^{2+} were higher in throughfall than rainfall at SNP (Table 4). Throughfall at the FEF had greater concentrations of Ca^{2+}, Mg^{2+}, K^+, Mn^{2+}, Fe^{2+}, SO_4^{2-}, and PO_4^{3-} than rainfall. Concentrations of MoO_4^- were higher in rainfall than throughfall at SNP and FEF. Throughfall pH (5.30) was higher than rainfall pH (4.69) at FEF. Rainfall (pH 5.57) and throughfall (pH 5.21) pH did not differ at SNP. Rainfall and throughfall had similar conductivities and concentrations of NH_4^+, Na^+, Zn^{2+}, NO_3^-, and $B(OH)_4^-$ at both sites, and similar concentrations of SO_4^{2-} and PO_4^{3-} at the SNP.

Throughfall chemistry

Bulk throughfall chemical variables were compared between sites using nested analysis of covariance and a 2 (sites) x 3 (trees/site) design. No measurable rainfall occurred during weeks 1 and 14 at SNP or week 1 at FEF, thereby reducing sample sizes for statistical comparison to 14 and 15, respectively. Data for each chemical variable were tested for normality and found to be skewed. Natural log transformations resulted in normal distributions. All variables measured in rainfall were significant covariates in the analysis of transformed throughfall data. Throughfall at FEF had greater concentrations of Ca^{2+}, Mg^{2+}, Mn^{2+}, NH_4^+, SO_4^{2-}, $B(OH)_4^-$, and conductivity than at SNP (Table 4). Concentrations of K were greater in FEF throughfall than SNP throughfall, but this difference was not significant due to high variability in the data. There was no difference in pH between sites.

Discussion

There are three sources of chemical input that could account for differences in throughfall chemistry between SNP and FEF. First, the chemistry of rain falling on the sites could differ, and second, the amounts of leachable

TABLE 4. Average weekly chemistry of rainfall (RF) and throughfall (TF) at the Shenandoah National Park (SNP), Greene Co., VA and the Fernow Experimental Forest (FEF), Tucker Co., WV from June 12, 1984 to September 25, 1984

Chemical variable	Bulk rainfall		RF vs. TF		Bulk throughfall[b]			
	SNP	FEF	SNP	FEF	SNP		FEF	
Ca^{2+} (ppm)	0.65	0.74 ns[a]	*	*	1.12	(1.04)	1.39	(1.44)**
Mg^{2+} (ppm)	0.18	0.13 ns	*	*	0.28	(0.31)	0.33	(0.30)**
K^+ (ppm)	0.51	0.20 ns	*	*	2.53	(2.46)	3.30	(3.39) ns
Na^+ (ppm)	1.41	0.62*	ns	ns	1.02	(1.31)	1.17	(1.27) ns
NH_4^+ (ppm)	0.73	0.48 ns	ns	ns	0.40	(0.39)	0.46	(0.46)*
Zn^{2+} (ppm)	0.05	0.03 ns	ns	ns	0.04	(0.04)	0.03	(0.03) ns
Mn^{2+} (ppm)	0.00	0.01 ns	*	*	0.06	(0.05)	0.10	(0.11)**
Fe^{2+} (ppm)	0.01	0.01 ns	*	*	0.02	(0.02)	0.02	(0.02) ns
PO_4^{3-} (ppm)	0.09	0.06 ns	ns	*	0.15	(0.15)	0.18	(0.18) ns
SO_4^{2-} (ppm)	4.09	4.19 ns	ns	*	4.36	(4.13)	6.79	(6.83)**
NO_3^- (ppm)	0.32	0.44 ns	ns	ns	0.42	(0.36)	0.39	(0.44) ns
$B(OH)_4^-$ (ppm)	0.73	0.58 ns	ns	ns	0.66	(0.66)	0.80	(0.73)*
MoO_4^- (ppm)	0.03	0.02 ns	*	*	0.02	(0.02)	0.00	(0.00) ns
pH	5.57	4.69**	ns	*	5.21	(5.46)	5.30	(5.07) ns
Conductivity (μmho/cm)[c]	28.1	32.3 ns	ns	ns	29.7	(28.6)	34.7	(35.8)*

[a] Variable means were compared by F-tests; ns indicates no site difference; * and ** indicate site differences at $P \leq 0.05$ and $P \leq 0.01$, respectively.

[b] Analysis of covariance (ANCOVA) means, the ANCOVA procedure used the natural log of adjusted means. Values in parentheses are means of volume weighted average concentrations among three replicate samples.

[c] 1 mho = 1S.

nutrients within canopy leaves could differ. Third, the amounts and composition of dry material deposited on the canopies from atmospheric sources could differ.

Site characterization

Atmospheric, soil, and foliar chemistry

Average chemical concentrations of bulk rainfall were similar for SNP and FEF except for Na^+ and pH (Table 4). Differences in pH between FEF (4.69 = 20 mequiv.) and SNP (5.57 = 3 mequiv.) are small in terms of chemical equivalents. Bulk rainfall data and NADP/NTN rainfall data both indicate few statistical differences between SNP and FEF.

Soil fertility and site productivity are greater at the FEF due to differences in the chemical nature of soil parent material, and amounts of rainfall occurring at each site. Long-term records show that FEF receives 26% more average annual precipitation than SNP. This would tend to increase weathering and depth of soil at the FEF site. Higher clay and organic matter content in FEF soil suggest a higher cation exchange capacity than at SNP. Greater total N in FEF soil indicates that biological turnover of N, and mineralization rates of nutrients in general, may be greater at FEF.

Leaves at FEF had higher nutrient element concentrations through the growing season than at SNP, except for Mg. Low levels of Mg^{2+} in FEF leaves may be due to a high extractable calcium to extractable magnesium ratio (6.3:1) in FEF soil. The Ca/Mg ratio of FEF leaves was 6.0: 1 over the growing season. The Ca/Mg ratio was 2.0: 1 in SNP soil and 3.6:1 in SNP leaves. High levels of extractable Ca^{2+} in soil can inhibit absorption of extractable Mg^{2+} by roots (Barber 1984). The absence of between-site differences in nutrient losses from leaves despite large differences in nutrient concentrations suggests that leaf surfaces remained impermeable to simulated rain solutions. Differences in leaf nutrient concentrations may be due to nutrients in the tissue not being readily subject to leaching.

Site processes

Throughfall comparisons between sites

Rainfall was enriched chemically at both sites. The distance between sites may confound wet and dry deposition conditions with soil fertility. Comparisons of bulk rainfall to wet-only rainfall may be confounded by differences in collector type and deposition conditions. Still, these data indicate that the chemical nature of rainfall was similar at both sites. Thus, wet deposition is not likely to cause the differences in throughfall chemistry between the SNP and FEF sites. The simulated rain experiments show that foliar leaching of nutrients contributes to chemical alterations of rainfall. They also suggest that leaching is not the sole cause of between-site differences in ion concentrations in throughfall. Large particle dry deposition data for NADP/NTN sites WV 18 and VA 28 during May to September of 1982, 1983, and 1984 suggest that dryfall may be greater at FEF than SNP. The occurrence and chemical nature of dry deposition at the sites needs to be studied thoroughly in order to verify this assertion.

The physical nature of tree crowns can affect throughfall chemistry (Reiners and Olson 1984). Average leaf area was 16% greater at FEF than SNP (Table 2). Larger leaves at FEF provide a larger area for capturing dryfall. More rainfall (26%) at FEF would affect greater leaching of chemicals from leaves, since leaves need only to be wet for leaching to occur (Tukey 1971). Thus, differences in throughfall chemistry between SNP and FEF may be caused by differences in *(i)* the ability of northern red oaks to capture dryfall, *(ii)* dryfall chemistry and occurrence, and *(iii)* rainfall volume.

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