

Molecular Weight Distribution of Soluble Organics from Laboratory-Manipulated Surface Soils

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ABSTRACT

Transfer of organic matter from solid to solution phase is the first step in the redistribution by leaching of organic C and associated elements in forest soil profiles. To examine the effects of disturbances on solubilization of organics, three A horizons and one O horizon were exposed to air drying, waterlogging, addition of urea, addition of ash, and addition of ash in combination with heating at 250 °C. Following incubation at 20 to 22 °C for 1 to 50 d, soluble organic C (SOC) was extracted with 0.5 mM SrCl₂. The SOC was 0.2 to 0.7% of total organic C in controls. Urea and heat-plus-ash treatments yielded up to 17 times more SOC than controls, and other treatments yielded up to three times more than controls. Distribution of SOC among molecular-weight classes, as determined by dialysis, was 21 to 32% in the <3500-dalton class, 8 to 20% in the 3500 to 14 000-dalton class, and 47 to 71% in the >14 000-dalton class. Urea treatment yielded a higher proportion of SOC in the >14 000-dalton class than in the controls, while air drying resulted in a lower proportion. Both the quantity and quality of soluble organics can be influenced by soil disturbances, but the specific response varies with the type of disturbance.

ORGANIC MATTER is commonly present in forest soil solutions (McKeague et al., 1986). These soluble organics contain complexed metals and nutrients such as N, P, and S (Pohlman and McColl, 1988; Dahlgren and Ugolini, 1989; Homann et al., 1990; Qualls et al., 1991). Their leaching from the surface to deeper horizons is important in redistributing elements in forest soils and, therefore, is significant to both pedogenesis and nutrient cycling (McKeague et al., 1986; Schoenau and Bettany, 1987).

The transfer of organics from the solid to the solution phase is the critical initial step in their redistribution in the soil profile by leaching. Solubilization of organics is influenced by many factors. Soluble organics generally increase with drying of soils (Bartlett and James, 1980). Their concentrations are affected by ionic strength, anionic composition, and acidity of applied solutions (Evans et al., 1988; Vance and David, 1989). They change during soil incubation (Qian and Wolt, 1990) and increase with incubation temperature (Buldgen, 1982).

The factors that control organic solubility can be altered by forest disturbances. As a consequence, organic concentrations in soil solutions are affected by

natural phenomena and by management practices, including deposition of volcanic tephra (Dahlgren and Ugolini, 1989), application of CaCO₃ (Simard et al., 1988), and urea fertilization (Ogner, 1975; Otchere-Boateng and Ballard, 1978). Other disturbances, e.g., fire (Khanna and Raison, 1986), also affect the soil chemical and biological properties, but the response of soluble organics to these changes has not been determined.

The properties of soluble organics are important in determining their behavior and function in soils. Characterization of soluble organics has included elemental composition and functional-group analysis (Dahlgren and Ugolini, 1989), analysis of specific compounds or groups of compounds (Pohlman and McColl, 1988), hydrophilic–hydrophobic and acid–base qualities (Pohlman and McColl, 1988; Vance and David, 1989), and MW (Buffle et al., 1978; Dawson et al., 1978, 1981; Candler and Van Cleve, 1982; Cole et al., 1984; Manley et al., 1987; Spiteller, 1987). Soluble organics of MW less than several thousand daltons include aliphatic acids, aromatic acids, amino acids, mono-saccharides, oligosaccharides, and low MW fulvic acids (Dawson et al., 1981; McKeague et al., 1986; Pohlman and McColl, 1988), while higher MW organics include complex substances of indefinite structure, such as high MW fulvic and humic acids.

Not only is MW an inherent property of soluble organics, and hence of interest in its own right, it is also correlated with organic behavior and function. Compared with fulvic acids, the higher MW humic acids are more easily coagulated, are less effective in solubilizing metal ions, and tend to form less soluble complexes with metal ions (Stevenson, 1982). Compared with fulvic and humic acids of lower MW, those with higher MW decreased the activity of pronase and carboxypeptidase enzymes (Butler and Ladd, 1971).

The MW of soluble organics from several soils has been investigated (Buffle et al., 1978; Dawson et al., 1978, 1981; Candler and Van Cleve, 1982; Cole et al., 1984; Manley et al., 1987; Spiteller, 1987), but little information is available concerning the influence of soil disturbances. Field applications of urea have enhanced soluble organics in forest soils, with preferential increase in higher MW substances (Ogner, 1975). In this study, we examined several disturbances to determine if they produced responses similar to those associated with urea fertilizers. Specifically, through laboratory manipulations, we tested the hypothesis that soil disturbances (simulated fire, drying, waterlogging, and urea fertilization) increase the

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quantity of soluble organics and shift their distribution to higher MW.

EXPERIMENTAL DESIGN

Samples

The study site was a vegetation-topographic transect at the Cedar Creek Natural History Area, Minnesota. Mean annual air temperature is 6 °C and annual precipitation is 660 mm (Grigal et al., 1974). Three sampling locations were selected: (i) field — Cedar Creek Natural History Area field no. 42, which was abandoned from agriculture in 1951 and is undergoing a secondary succession of grasses, forbs, and sedges (Inouye et al., 1987); (ii) summit — the adjacent closed-canopy forest, which is dominated by northern red oak (*Quercus rubra* L.) and red maple (*Acer rubrum* L.); and (iii) toeslope — located within the same forest but at the base of a slope. Soil at the field and summit locations is of the Zimmerman soil series (mixed, frigid Alfic Udipsamment; Grigal et al., 1974). The toeslope occurs at the transition between the upland Zimmerman series and the wetland Lupton series (euic Typic Borosaprist). An A horizon sample (0–10-cm depth) was collected in January 1990 from a single soil pit at each sampling location. An Oi plus Oe horizon sample (designated as *forest O*) was composited from the summit, toeslope, and an intermediate backslope position. The A horizon soil is >90% sand (Grigal et al., 1974). Other sample characteristics are presented in Table 1.

Treatments

The samples were collected when the soil was frozen. They were stored field moist at ≈ 1 °C for a period of 8 to 12 wk before being used in experiments. Since storage conditions can affect soluble organics (Ross and Bartlett, 1990), this preexperiment storage may have influenced the initial levels of soluble organics; however, it is unlikely to have influenced the magnitude of responses to the subsequent experimental treatments.

Samples from the O horizon were coarsely ground (4 mm) and those from the A horizons were sieved (2 mm). Aliquots of either field-moist O horizon equivalent to 2 g oven-dried mass or A horizon equivalent to 5 g oven-dried mass were incubated after receiving one of six treatments: (i) control — no additions; (ii) dried — no additions, but allowed to air dry during incubation; (iii) saturated — addition of distilled, deionized water to waterlog sample; (iv) urea — addition of 0.02 g of fine-granular urea; (v) ash — addition of 0.1 g of ash, which was produced by heating deciduous leaf litter at 450 °C for >24 h; (vi) heat plus ash — addition of 0.1 g of ash in combination with heating at

250 °C for 30 min, followed by adjustment to original moisture content.

The treated samples were incubated in plastic vials at room temperature (20–22 °C) for 1, 8, 30, or 50 d. The dried treatment was incubated uncovered to allow evaporation of water. Other treatments were loosely capped to allow air exchange but to minimize water loss; they were maintained close to original moisture contents by periodic water additions (Table 1). Three replicate samples were incubated and analyzed for each soil-treatment-time combination.

Following incubation, the samples were transferred to 60-mL plastic syringes containing water-rinsed filter pulp and extracted on a mechanical vacuum extractor (Centurion International, Lincoln, NE) with 55 mL of 0.5 mM SrCl₂. This solution has a low ionic strength typical of natural soil solutions and an equilibrium pH of 4.7 due to coordination of hydroxyl ions with Sr. Extraction time was typically 1 h, but some urea-treated samples required as long as 24 h to complete the extraction.

Chemical Analyses

Aliquots of unfiltered extracts were analyzed for pH. Separate aliquots were filtered through 0.4- μ m polycarbonate filters (Nuclepore Corp., Pleasanton, CA) that previously had been rinsed with 40 mL of deionized, distilled water. Dark-colored extracts were diluted with 0.5 mM SrCl₂ prior to filtration; subsequent analyses indicated that these extracts initially had organic C concentrations >100 mg C L⁻¹. Filtrates were analyzed for absorbance at 465 nm (1 cm pathlength), a wavelength commonly used for organic characterization (Stevenson, 1982). Organic C in filtrates was determined by ultraviolet-persulfate oxidation with infrared detection of CO₂ (Dohrmann DC-80 Total Organic Carbon Analyzer, Xertex Corp., Santa Clara, CA). Values were corrected for organic C in blanks run through the extraction procedure, and SOC was calculated as milligrams of C per kilogram of oven-dried soil. Remaining filtrates were frozen and stored at -20 °C in polyethylene bottles prior to MW fractionation. A preliminary experiment indicated freezing did not affect MW fractionation.

Molecular Weight Fractionation

The MW fractionation was performed by dialysis of composited filtrates. Filtrates were thawed and composited to yield two independent composites for each soil-treatment combination; typically, one composite contained filtrate from two 8-d replicates, one 30-d replicate, and two 50-d replicates, while the second composite contained filtrates from the remaining 8-, 30-, and 50-d replicates.

Dialysis bags were made of membranes (Spectra/Por 3 and 4 membranes, Spectrum Medical Industries, Los Angeles) with nominal MW cut-offs of 3500 and 12 000 to 14 000 daltons (the latter hereafter referred to as 14 000 daltons). Composites and diazylate (distilled, deionized water external to the dialysis bag) were adjusted to pH 4 with H₂SO₄, because pH can affect both molecular structure and aggregation (Gnosh and Schnitzer, 1980) and dialysis rates (Haumaier et al., 1990). Composites were dialyzed against a stirred diazylate maintained in an ice bath to minimize microbial activity. The ratio of composite to diazylate was 1:20. Diazylate was changed at 18- to 24-h intervals. Total dialysis duration was 66 h.

Organic C in initial and dialyzed composites was determined as described above for filtrates. Values were corrected for organic C in distilled, deionized water blanks run through the dialysis procedure. For each composite, distribution of organic C in three nominal MW classes was calculated: <3500, 3500 to 14 000, and >14 000 daltons.

To compare dialysis with ultrafiltration, a sample of humic

Table 1. Characteristics of soil samples used in study of soluble organics.

Sample and horizon	Organic C†	Total N†	pH‡	Moisture content		
				During incubation		
				Initial	Moist§	Dried¶
				kg kg ⁻¹ dry soil		
Field A	13	1.0	5.1	0.17	0.19	0.01
Summit A	41	3.1	5.2	0.28	0.32	<0.01
Toeslope A	105	3.5	4.3	0.90	0.85	0.04
Forest O	450	11.6	5.6	2.70	2.70	0.09

† Determined by combustion with Carlo-Erba CNS analyzer.

‡ Water/soil = 1:1 for A horizons, 5:1 for O horizon.

§ Average during 1–50 d of incubation for control, ash, heat plus ash, and urea treatments; moisture was maintained by frequent additions of water.

¶ Average during 8–50 d of incubation for air-dried treatment.

acid (sample C-HA Comp 200 NTNR 1-4 1989, previously characterized by Clapp et al. [1989]) was dissolved in water and adjusted to pH 4.0. Solution with an initial concentration of 50 mg C L⁻¹ was dialyzed with the Spectra/Por 4 membrane by the procedures described above. An Amicon PM-10 filter (nominal MW cut-off of 10 000 daltons) in an Amicon stirred cell (Model 52, Amicon, Beverly, MA) was used for ultrafiltration. The filter was soaked in distilled water and leached with 200 mL of distilled, deionized water to remove organic contaminants. Then a 30-mL aliquot of humic acid solution with an initial concentration of 25 mg C L⁻¹ was filtered under pressure of 0.2 MPa applied with N₂. The first 10 mL of filtrate were discarded and the next 10 mL were collected for organic C analysis. Procedures were performed in duplicate, and values were corrected for blanks.

Statistical Analysis

The SOC values were analyzed with a three-way fixed-factor ANOVA; values were log transformed to homogenize variances. The factors were soil, incubation time, and treatment, with three replicates per cell. The MW values were analyzed for each MW class with a two-way fixed-factor ANOVA. The factors were soil and treatment, with two replicates per cell. When ANOVAs indicated differences at $P < 0.05$, the Tukey honestly significant difference test was conducted to separate means, with an experiment-wise error rate of $P < 0.05$. Analyses were conducted with the SAS statistical package (SAS Institute, 1982).

RESULTS AND DISCUSSION

Soluble Organic Carbon

Statistical analysis indicated there were significant ($P < 0.0003$) effects of soils, treatments, incubation times, and all interaction terms on SOC. Therefore, the Tukey test was performed on all soil-treatment-time combinations. The SOC for each combination is presented in Fig. 1; any two points may be compared by using the illustrated critical difference from the Tukey test.

In the A horizons, dried, saturated, and ash treatments yielded slightly to moderately higher levels of SOC than controls, while heat-plus-ash and urea treatments yielded much higher levels (Fig. 1). In contrast, in the forest O horizon, dried and ash treatments had no effect and saturated, heat plus ash, and urea had a moderate effect. The enhanced SOC in urea-treated soils is consistent with previous field studies of fertilizer effects (Ogner, 1975; Otchere-Boateng and Ballard, 1978).

The apparent lower response of the forest O horizon to disturbance may be due to the less humified organic matter compared with the A horizons. Another factor is the high SOC in the forest O horizon controls. The SOC averaged 0.72% of total organic C for forest O horizon controls, compared with only 0.20% for toeslope A horizon, 0.35% for summit A horizon, and 0.19% for field A horizon. Yet, the highest level of SOC from treated samples was 2.4% of total organic C for forest O horizon, which lies within the range of 1.5% for toeslope A horizon, 3.7% for summit A horizon, and 4.9% for field A horizon. Therefore the apparent lower response of the forest O horizon is due in part to the high levels of SOC in its controls.

The control and ash treatments were relatively sta-

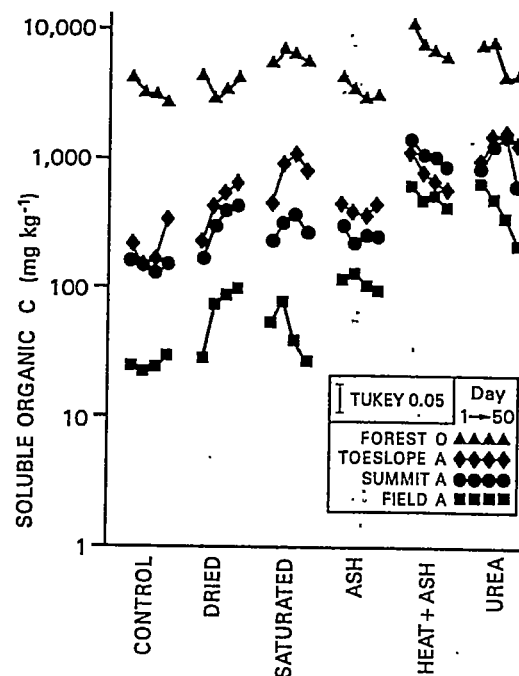


Fig. 1. Soluble organic C (log scale) in four soil samples affected by treatment and incubation time. Each set of four points is a time series of 1, 8, 30, and 50-d incubations.

ble over time, compared with the other treatments (Fig. 1). In the A horizons there was progressively more SOC with greater drying time, while the O horizon did not have this temporal pattern. The SOC in the heat-plus-ash treatment decreased with time for all soils. Saturated treatments showed peaks in SOC, while temporal patterns differed between soils for the urea treatment.

Some of the responses to treatments may be attributed to chemical factors. Higher pH results in higher SOC concentrations (Vance and David, 1989). Hydrolysis of urea yields NH₄OH and creates alkaline conditions in soils (Otchere-Boateng and Ballard, 1978). Initial hydrolysis of urea probably led to very high pH at microsites, causing solubilization of organics just as humic and fulvic acids are extracted with high-pH solutions (Stevenson, 1982). However, as pH dropped

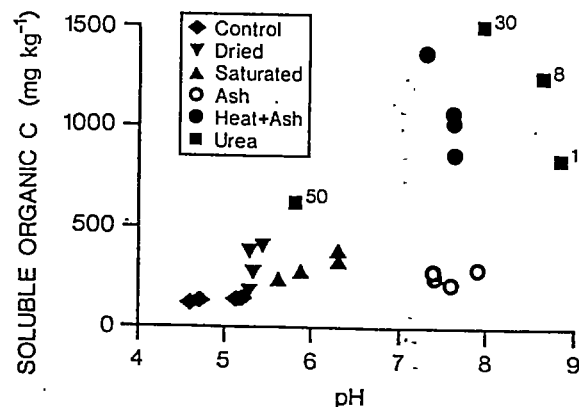


Fig. 2. Soluble organic C vs. extract pH for the forest summit A horizon sample. Numbers associated with the urea treatment are incubation time in days.

over time, SOC did not necessarily drop, as illustrated for the summit A horizon (Fig. 2). Therefore, other factors not examined in this study, such as microbial activity, were also important in controlling SOC.

Ash substantially increased pH (Fig. 2) and yielded slightly higher SOC in the A horizons (Fig. 1 and 2). Heat plus ash increased pH to the same extent as ash, but yielded much greater SOC. This suggests the heat had a much greater effect than the pH on solubilization of organics. Pyrolysis of soil organic matter (Schnitzer and Hoffman, 1964) or death of microorganisms with subsequent release of their organic components may have caused the response to heating.

In addition to the quantitative differences in SOC between treatments, qualitative differences were evident. In the summit A horizon extracts (Fig. 3), the urea treatment resulted in lower organic C per absorbance unit than the other treatments, especially the heat plus ash. The urea treatment also had the lowest ratios of organic C to absorbance in the other soils (data not shown). These results were not due to differences in solution pH, because solutions adjusted to pH 4 yielded similar results. The light-absorbance results suggest soluble organics produced by various treatments can be chemically different.

Molecular Weight Distribution

Concentrations of organic C in extracts from the field soil were too low to yield meaningful results with the dialysis method. Therefore, only extracts from forest summit, toeslope, and O horizon were assessed for MW distribution. In general, the >14 000-dalton class contained most of the organic C, followed by the <3500-dalton and 3500- to 14 000-dalton classes (Table 2). However, the MW distribution was affected by both soil and treatment. Toeslope A horizon had a higher fraction of SOC in the 3500- to 14 000-dalton class and a lower fraction in the >14 000-dalton class than summit A horizon and forest O horizon. Dried treatment had a higher fraction of SOC in the <3500-dalton class and a lower fraction in the >14 000-dalton class, while urea had an opposite effect on MW fractionation. The enhanced high MW fraction in the urea treatment is consistent with the results obtained by Ogner (1975) in a field study of the effects of fertilization.

Similarity of MW fractionation between the controls and in situ soil solutions indicates the laboratory observations in this study are relevant to field conditions. For soil solutions collected with ceramic cup samplers under northern hardwood (*Tilia americana* L. and *Acer saccharum* Marsh.), pine (*Pinus resinosa* Ait. and *P. strobus* L.), and cedar (*Thuja occidentalis* L.) stands at the Cedar Creek Natural History Area, organic C was distributed as follows: 37 to 43% in the <3500-dalton class, 7 to 16% in the 3500- to 14 000-dalton class, and 46 to 50% in the >14 000-dalton class (P.S. Homann and R. Bouabid, 1990, unpublished data). In both the soil solutions and the laboratory extracts, the highest percentage of organic C was in the high MW fraction and the lowest percentage in the medium MW fraction.

Different techniques of MW fractionation can yield different results (Wershaw and Aiken, 1985). Dialysis

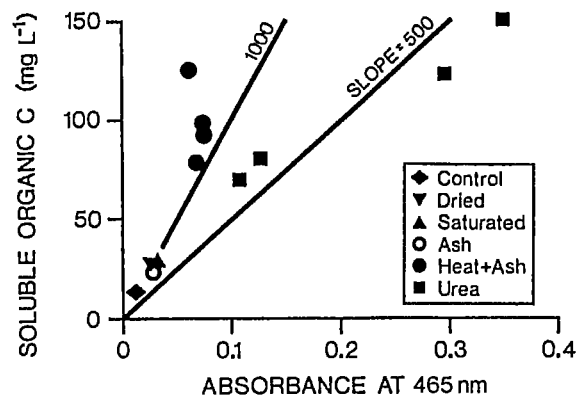


Fig. 3. Soluble organic C vs. absorbance at 465 nm (1-cm pathlength) for extracts from the forest summit A horizon sample. Each point for urea and heat-plus-ash treatments is a different incubation time, while the point for each of the other treatments is the mean of all incubation times.

and ultrafiltration were tested in this study to determine if the two methods yielded consistent results. Lower amounts of organics were expected to be retained by the dialysis membrane because of its greater nominal MW cutoff (14 000 daltons) compared with the Amicon PM-10 ultrafilter (10 000 daltons). This expected pattern was observed, with 87% of a humic acid characterized as >14 000 daltons by dialysis and 96% >10 000 daltons by ultrafiltration.

Therefore, results from dialysis using the Spectra/Por 4 membrane are consistent with results from ultrafiltration using the PM-10 membrane, and comparison of dialysis results from this study with PM-10 ultrafiltration results from other studies (Table 3) is valid. The MW distribution of SOC in controls from this study (47–71% >14 000 dalton; Table 2) corresponds to the large proportion of high MW organics in decomposed-leaf extracts (Table 3). Other soil solutions and soil extracts analyzed with PM-10 ultrafiltration had much lower percentages of high MW organics (Table 3), indicating the soluble organics were inherently different from those in this study.

Ultrafiltration studies using other membranes also show both large and small proportions of high MW organics (Table 3); but they cannot be directly compared with dialysis and PM-10 ultrafiltration, because different membranes may give different results (Wershaw and Aiken, 1985). Using gel filtration, Candler and Van Cleve (1982) concluded that most of the dissolved material in aqueous extracts from B horizons in aspen (*Populus tremuloides* Michx.) and birch (*Betula papyrifera* Marsh.) forests was <5000 daltons. Also using gel filtration, Dawson et al. (1978, 1981) found the major fraction of dissolved organics in soil solutions from a subalpine podsolized Cryandept and a Durohumult to have an average MW of <1000 daltons (Dawson et al., 1978, 1981). Because it is unclear if all initial SOC was recovered from gel filtration columns, however, comparisons of these values with those from dialysis and ultrafiltration may not be valid.

The different techniques used in evaluation of MW of soluble organics preclude quantitative comparison of results among all studies. But individual studies provide valuable information about factors that influ-

Table 2. Molecular weight (MW) distribution of soluble organic C (SOC) in extracts from three soil samples exposed to six treatments.

MW class	Soil sample and horizon	SOC content						\bar{x} for soil
		Control	Dried	Saturated	Ash	Heat + Ash	Urea	
daltons		% of total						
<3500	Summit A	23	35	23	33	28	12	26a†
	Toeslope A	32	49	38	30	32	11	32a
	Forest O	21	36	36	25	28	16	27a
	\bar{x} for treatment	25b	40a	33ab	29ab	29ab	13c	
3500–14 000	Summit A	15	10	12	13	10	2	10b
	Toeslope A	20	15	18	15	17	18	17a
	Forest O	8	7	3	10	9	0	6b
	\bar{x} for treatment	14a	10a	11a	13a	12a	7a	
>14 000	Summit A	62	55	64	54	62	86	64a
	Toeslope A	47	36	44	55	51	71	50b
	Forest O	71	57	61	65	63	84	67a
	\bar{x} for treatment	60b	50c	56bc	58bc	58bc	80a	

† For each MW class, data were analyzed with a fixed-factor two-way ANOVA and Tukey multiple-range test: Soil \times treatment interactions were not significant at $P < 0.2$. Means followed by the same letter are not different at $P < 0.05$; $n = 2$ for each soil-treatment combination; square root of the error mean square from ANOVA = 6.8 for <3500-dalton class, 5.0 for 3500 to 14 000-dalton class, and 5.2 for >14 000-dalton class.

ence the natural variation of organics. The findings of Dawson et al. (1978, 1981) indicate variation of MW distribution among horizons, while results of Candler and Van Cleve (1982) and Spiteller (1987) show the influence of forest type. These are complemented by the results of this study, which indicate that MW distribution of soluble organics also varies with landscape position (Table 2).

CONCLUSIONS

We tested the hypothesis that soil disturbances increase the quantity of soluble organics and shift their distribution to higher MW. Air drying, waterlogging, urea application, ash application, and heat in combination with ash enhanced soluble organics in A horizons, but only waterlogging, heat plus ash, and urea increased soluble organics in the O horizon. The magnitude of increase varied considerably among treatments and soils, with disturbances producing up to 17 times more SOC than controls. Urea shifted the MW distribution of soluble organics to higher weights, while air drying shifted the MW distribution to lower weights. Light-absorbance characteristics also indicated differences between soluble organics from different treatments.

These differences in quantity and quality of soluble organics are important in understanding organic matter dynamics in forest soils. Solubilization of organics is the first step in their redistribution within the soil profile by leaching. The results of this study indicate short-term events — fire, fertilization, waterlogging,

drying — can enhance solubilization. During fires in chaparral soils, organics are volatilized and transported in vapor phase deeper into the soil until they condense (Savage, 1974). The solubilization of organics and subsequent transport in solutions is another process of redistributing organics in the soil profile as a consequence of fire. Such processes must be considered in assessments of movement of organics and in models of organic matter dynamics in forest soils.

The MW and light-absorbance results show that organics solubilized by different disturbances can be chemically different. Although the specific consequences of these differences are not currently known, both the function and fate of soluble organics depend, in part, on their chemical characteristics. Due to their chemical differences, soluble organics produced by various disturbances may have different propensities to be mineralized by microorganisms, adsorbed on soil surfaces, and precipitated from solution. Further research is required to explore the relationship between disturbances and the production and fate of soluble organics in forest soils.

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Table 3. Percentage of soluble organic C (SOC) in >10 000-dalton class determined by ultrafiltration.

Sample	Upper size limit†	Ultrafiltration membrane	SOC	Reference
	μm		>10 000 daltons	
Decomposed-leaf extracts	0.2	Amicon PM 10	55–80	Buffle et al., 1978
Soil extracts	0.2	Amicon PM 10	8–19	Buffle et al., 1978
Soil solutions, hardwood forest B horizon	1.2	Amicon PM 10	12	Cole et al., 1984
Soil extracts, E horizon	0.22	Amicon YM 10	<50	Manley et al., 1987
Soil solutions, oak forest O horizon	0.45	Millipore	55–80	Spiteller, 1987
Soil solutions, spruce forest O horizon	0.45	Millipore	20–40	Spiteller, 1987

† Pore size of filter to separate SOC from suspended particulates.

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