

Fire severity effects on soil organic matter from a ponderosa pine forest: a laboratory study

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Abstract. This study investigated the changes in soil organic matter composition by controlling fire severity of laboratory burns on reconstructed surface soil profiles (O, A1 (0–1 cm), and A2 (1–2 cm)). Laboratory burning simulated prescribed burns that would be typical in the understory of a ponderosa pine forest at low, moderate, and high–moderate severity levels. Soils were analysed for C, N and soil organic matter composition. Soil organic matter was fractionated into humin, humic acid, fulvic acid, soluble non-humic materials and other hydrophobic compounds. In the O horizon, low-, moderate-, and high-severity treatments consumed an increasing proportion of C and N. Carbon content of the mineral soil was unaffected by burning; however, N content of the A2 horizon decreased after the moderate- and high-severity treatments, likely as a result of N volatilisation. The proportion of non-soluble material in the O horizon increased with fire severity, whereas the proportion of humin C as total C of the A horizon decreased with fire severity. The decrease in humin was followed by an increase in the other hydrophobic compounds. The higher fire intensity experienced by the burning O horizon created recalcitrant materials while an increase in labile soil organic matter was observed in mineral soil. An increase in labile soil organic matter may cause elevated C and N mineralisation rates often seen after fire.

Additional keywords: black carbon, carbon, fire intensity, fulvic acid, humic acid, humin, nitrogen.

Introduction

Ponderosa pine (*Pinus ponderosa* Laws.) forests of the Pacific North-west historically, pre-European settlement, had a low-severity fire regime (most trees survive each fire occurrence). However, high-severity wildfires are now occurring more frequently owing to altered stand structure and accumulation of fuels caused by decades of fire suppression, timber harvesting, road building, and historical overgrazing (Agee 1994; Everett *et al.* 2000; Tiedemann *et al.* 2000). Low-severity prescribed fires are being used to reduce the threat of high-severity wildfires and return these forests to their historical stand structure and fire-return interval; however, even these fires have variable severity that depends on factors such as fuel loading and season (Thies *et al.* 2005; Hatten *et al.* 2008). The effect of fire on soils is largely through the alteration and combustion of soil organic matter (SOM). The degree to which fire influences this important soil component is likely dependent on fire severity, and is the focus of this paper.

Soil organic matter includes both humic and non-humic organic substances associated with organic and mineral soil. Non-humic substances are generally any materials that can be identified and classified whereas humic substances are refractory, dark-coloured, heterogeneous, organic compounds produced as by-products of microbial metabolism (Stevenson 1994). Another pool that may represent up to 45% of the SOM is black carbon

(BC) (Skjemstad *et al.* 2002). Black carbon is the heterogeneous, aromatic, and C-rich residue of biomass burning and fossil-fuel combustion that includes charcoal, soot, and graphite (Goldberg 1985). These classes of BC can be thought of as existing on a continuum that increases in aromaticity, condensation, C : H ratio, and C : O ratio with charcoal < soot < graphite.

Soil organic matter serves many physical and chemical functions in soil. In particular, it serves as a reservoir of available nutrients, particularly N, which can be mineralised by microorganisms and made available to plants (Stevenson 1994). The availability and accessibility of SOM to microorganisms is dependent on its nutrient content, chemical structure, and physical protection within the soil (Baldock and Skjemstad 2000; Baldock and Smernik 2002; Kelleher *et al.* 2006). Therefore, compounds such as lignin, waxes and highly condensed aromatic substances, such as charcoal, are less bioavailable than simpler molecules (e.g. saccharides and amino acids) (Baldock and Skjemstad 2000; Cheng *et al.* 2006; Kelleher *et al.* 2006). Further, soil organic matter can be responsible for 20–70% of soil cation exchange capacity (Stevenson 1994).

Operational definitions have been developed to define classes of SOM based on an alkaline extraction and subsequent solubility when subjected to an acidic solution. Typically, there are three fractions: humin, humic acid, and fulvic acid. Humin is insoluble in alkaline conditions and BC present in the soil is

often included in this fraction. The fulvic acid fraction contains those substances that are extracted by an alkaline solution and remain in solution after the pH is acidified. Humic acid is soluble in an alkaline extract, but precipitates in a subsequent acidic condition. The solubility of these fractions is dependent on the hydrophobic nature of the compound (humins > humic acid > fulvic acid), molecular size (humins > humic acid > fulvic acid), and carboxylic group content (fulvic acid > humic acid > humins) (Stevenson 1994).

Fire consumes and alters SOM at the surface of the soil through the process of combustion, which results in the volatilisation of C and other nutrients. A fraction of the energy produced from burning is able to penetrate into the soil and alter mineral-associated SOM. Organically bound elements such as N, P and base cations can be mineralised and become available for uptake by plants or leached from the soil (DeBano *et al.* 1998). In general, the chemical changes that occur in SOM after heating and combustion are an increase in aromatic structures and a disproportionate loss of H and O relative to C (Almendros *et al.* 1990, 2003; Baldock and Smernik 2002; Gonzalez-Vila and Almendros 2003; Gonzalez-Perez *et al.* 2004; Knicker *et al.* 2005). Reducing the oxygen content of the SOM has the effect of converting fulvic acid and humic acid fractions to humins and BC (Almendros *et al.* 1990; Fernández *et al.* 1997, 2001, 2004; Hernández *et al.* 1997). The creation of more recalcitrant forms of SOM has led some to believe that burning may be a mechanism by which soils accumulate recalcitrant forms of C (Kuhlbusch and Crutzen 1995), but limit long-term nutrient availability to organisms (Guinto *et al.* 1999).

In contrast to the creation of recalcitrant forms of C, soluble C has been seen to increase after heating mineral soil up to 500°C (Blank *et al.* 1994; Battle and Golladay 2003; Guerrero *et al.* 2005). These researchers attributed elevated levels of soluble C to destructive distillation of humic materials and destruction of microbial cells. The chemical characteristics of the fractions described above can impart different biologically relevant traits that are affected by fire and soil heating, making the operational definitions useful for exploring fire's effects on SOM. The impact of fire on ecosystem components is dependent on fire severity, which is likely controlling the alterations to SOM.

To determine the effect of fire severity on SOM, burns were conducted in the laboratory on columns of soil assembled from homogenised samples collected from a ponderosa pine forest. These burns were used to simulate the transfer of heat from a burning forest floor to the surface of the mineral soil. Treated soils were analysed for soil characteristics and quality of SOM to determine the effect of fire severity on SOM. Understanding the effect of fire severity on SOM will hopefully help natural resource managers utilise prescribed burning to preserve soil C and maintain benefits of SOM to soils.

Materials and methods

Laboratory burning

Laboratory burns were designed to represent prescribed fire in the understory of a ponderosa pine forest. Loam-textured soil samples were collected from the Dinkelman soil series, a Vitrandic Haploxeroll (Soil Survey Staff, Natural Resources Conservation Service, United States Department of Agriculture,

Official Soil Series Descriptions, see <http://soils.usda.gov/technical/classification/osd/index.html>, accessed 31 March 2008) beneath a ponderosa pine tree in a Douglas-fir (*Pseudotsuga menziesii* (Mirbel) Franco)–ponderosa pine forest located near Cashmere, Washington, on the east side of the Cascade Mountain Range (47°27'00"N, 120°37'54"W; 1070-m elevation). O and A horizons were collected and air-dried. O horizons were separated into Oi (unaltered pine litter) and Oe (slightly altered organic material or duff). Mineral soil (A horizon) was sieved to 2 mm, homogenised, and stored in air-tight containers.

A cylindrical form (36-cm diameter) was used to reassemble four replicated soil profiles per fire severity treatment. Sieved A horizon material was added to a depth of 2 cm and tamped to a bulk density of 0.8 g cm⁻³. The average bulk density of A horizons, at 14-cm average depth, collected from this area as part of another study was higher at 1.1 g cm⁻³ (J. Hatten, unpubl. data); this disparity was likely caused by the disruption of aggregates. However, the present study is examining the effects of burning on the surface 2 cm, which likely has a bulk density similar to our assembled columns.

Air-dried Oe horizon was placed on top of the mineral material to a depth of 4 cm at a bulk density of 0.09 g cm⁻³. Air-dried Oi horizon was placed on top of the Oe material to a depth of ~2 cm at a bulk density of 0.03 g cm⁻³. These depths and bulk densities were similar to the soil at the collection site. The contribution of fuel by O horizon represents much of a ponderosa pine forest's fuels as litter and duff represent ~25–80% of the mass of downed, dead fuels in ponderosa pine forests of Oregon and Washington (Page-Dumroese and Jurgensen 2006; McIver and Ottmar 2007).

Flaming combustion was induced with the short (~15 s) application of a propane torch to the surface of the O horizon. An O horizon with 18% moisture content was used for the low-severity treatments. To achieve the moderate- and high-severity levels, the fuels needed to be dried (using a convection oven at 40°C) to a <9% fuel moisture content. For the high-severity burns, a heat gun was positioned 30 cm above the surface of the O horizon and aimed at the centre of the circular form. The heated air increased the amount of oxygen reaching the combustion area, thereby increasing the severity and temperature of burning. Heated air was supplied until completion of flaming combustion (~30 min).

A heat-resistant, insulated circular form was used to stabilise and insulate the soil and fuelbed during burning. The temperature of the burns was monitored with three thermocouples connected to a datalogger that recorded temperature once every minute. The thermocouples were placed on the surface of the O horizon at the interface of the O horizon and mineral soil (0 cm) and 2 cm into the mineral soil. After burning, the soil columns were disassembled into O, A1 (0–1 cm), and A2 (1–2 cm). To control for additions of organic matter (OM) from the O horizon into the A horizon, control columns were also assembled, but not burned, and then disassembled similarly to the burn treatments.

Soil analysis

The pH of all samples was measured using the saturated-paste method (Van Miegroet *et al.* 1994). Subsamples of each horizon were ground and analysed for C, H, and N concentration using a

Perkin Elmer 2400 CHN analyser (Perkin Elmer, Waltham, MA) (Nelson and Sommers 1996). All C:N and C:H ratios were corrected for atomic weight. Carbon, H, and N concentrations were corrected for moisture content present in the air-dry samples. Only C:H from samples without a mineral matrix (e.g. O horizon) were considered owing to H-containing minerals. A linear regression (not shown) between the mass-normalised C and N found that the intercept was not significantly different from zero ($P > 0.05$), suggesting that the majority of C and N in these samples is organic and the C:N ratios describes organic matter composition with little to no influence by inorganic forms of these elements.

Soil organic matter was extracted from each horizon (O, A1, and A2) using a method modified from Schnitzer (1982). Ten grams of mineral and 1 g of O horizon were extracted with 100 mL of 0.1 M NaOH under N_2 . Samples were shaken overnight and the solution was separated from the non-soluble residue using a centrifuge at a force of 6635g for 10 min at room temperature (22°C). Although the residue from the A horizon was predominantly humin, the non-soluble residue from the O horizon (NS) was probably dominated by insoluble organic compounds such as lignin and cellulose. The supernatant solution and one rinse of the residue, containing the fulvic acid fraction and humic acid (HA), were collected for further processing. The residues were dried in a convection oven at 50°C, weighed and prepared for C, H, and N analysis. Ratios of C:N were calculated for all fractions, although C:H was not calculated for humin from the A horizons owing to the presence H-containing minerals.

The supernatant containing the fulvic and humic acid fractions was acidified to pH 1 using 6 M HCl and allowed to stand overnight to precipitate humic acid. The fulvic acid fraction was separated from humic acid by centrifuging at a force of 6635g for 10 min at room temperature (22°C). Humic acid was freeze-dried in a benchtop lyophiliser, weighed, and stored in a light-protected desiccator.

The fulvic acid solution was purified of salts and non-humic substances by passing it over 60 mL of non-ionic DAX-8 resin (Supelco SupeliteTM; methyl methacrylate ester, WGK, Germany) (Malcolm 1990; Swift 1996). Fulvic acid adsorbs to the resin at a pH of 1, whereas the soluble non-humic fraction (NH) was passed through the column along with three bed-volumes of deionised H_2O as a rinse, and was collected. Fulvic acid was desorbed by eluting the resin with two bed-volumes of 0.1 M NaOH. Other hydrophobic materials (HO) remained adsorbed to the column after elution with 0.1 M NaOH. The eluate containing fulvic acid was collected and passed over a column containing Dowex H^+ exchange resin (Dowex H^+ , JT Baker, Phillipsburg, NJ) to remove Na^+ . The salt- and acid-free solution of fulvic acid was freeze-dried, weighed, and stored in a light-protected desiccator. Prior to C, H, and N analysis, fulvic acid and humic acid samples were placed in preweighed tins and dried at 50°C for 48 h to drive off most of the residual water.

Only one sample from each horizon and severity level (12 samples) was collected and analysed for NH and HO fractions. The solution containing NH materials was analysed on a Shimadzu TOC/TN total organic carbon/nitrogen analyser (Shimadzu Scientific Instruments, Columbia, MD). The content

of HO C and N was determined by difference using the following equation:

$$HO = T - R - HA - FA - NH$$

where HO is the amount of other hydrophobic C or N that does not desorb from the DAX-8 resin column, T is the total soil C or N, R is C or N in the humin fraction (mineral soil) or non-soluble fraction (O horizon), HA is C or N from humic acid, FA is C or N from fulvic acid that desorbed from the DAX-8 resin column on elution with 0.1 N NaOH, and NH is C or N from the soluble non-humic fraction.

O and A horizons were analysed for thermally resistant (375°C for 20 h) organic matter (e.g. BC) using a method adapted from the CTO375 method outlined by Gustafsson *et al.* (1997). Loss on ignition (LOI; 550°C for 6 h) was used to determine the amount of thermally resistant OM remaining in each sample. Carbon content of 10 selected O and 10 selected A1 horizon samples was analysed after 20 h in the muffle furnace at 375°C to develop two ratios that would convert $LOI_{375-550}$ to BC. Further details of the analysis can be found in Hatten and Zabowski (2009).

Statistical analysis

O horizon and burn characteristics from four replicated burns and controls were tested with a one-factor ANOVA using burn severity as the independent factor. Tukey's test for honestly significant difference (HSD) was used to delineate homogeneous subsets.

A two-factor ANOVA was used to test the effect of burn severity level and A horizon depth on soil characteristics. Burn severity and horizon (A1 or A2) were the independent factors. If severity or depth was found to be a factor that was significantly different (P_s or $P_d < 0.05$ respectively), then Tukey's HSD was used to define homogeneous subsets. If there was a significant interaction between horizon depth and severity level ($P_{s \times d} < 0.05$), then the interaction was decomposed using pairwise contrasts. Pairwise contrasts were made on data between each depth at every burn severity level (four contrasts) and each severity level at each depth (12 contrasts). Bonferroni's procedure was used to reduce the chance of making a type I error below the α value of 0.05 when making simultaneous multiple comparisons. Statistical analysis was conducted using SPSS[®] (version 10.1.3 for Windows).

Results and discussion

Laboratory burning

The three burn severities showed increasing maximum temperature at the surface of the O and A1 horizon, and at 2 cm depth in the A2 horizon (Fig. 1). The moisture content of the low-severity burn treatments caused the fires to burn more coolly and extinguish on their own more quickly than the moderate- and high-severity burn treatments. Heated air applied to the high-severity burn treatments caused higher maximum temperatures, but it also consumed fuel (O horizon) more quickly than the moderate-severity treatment. Each of the four moderate-severity burns smouldered for a long period of time, reaching high temperatures at varying points, resulting in an

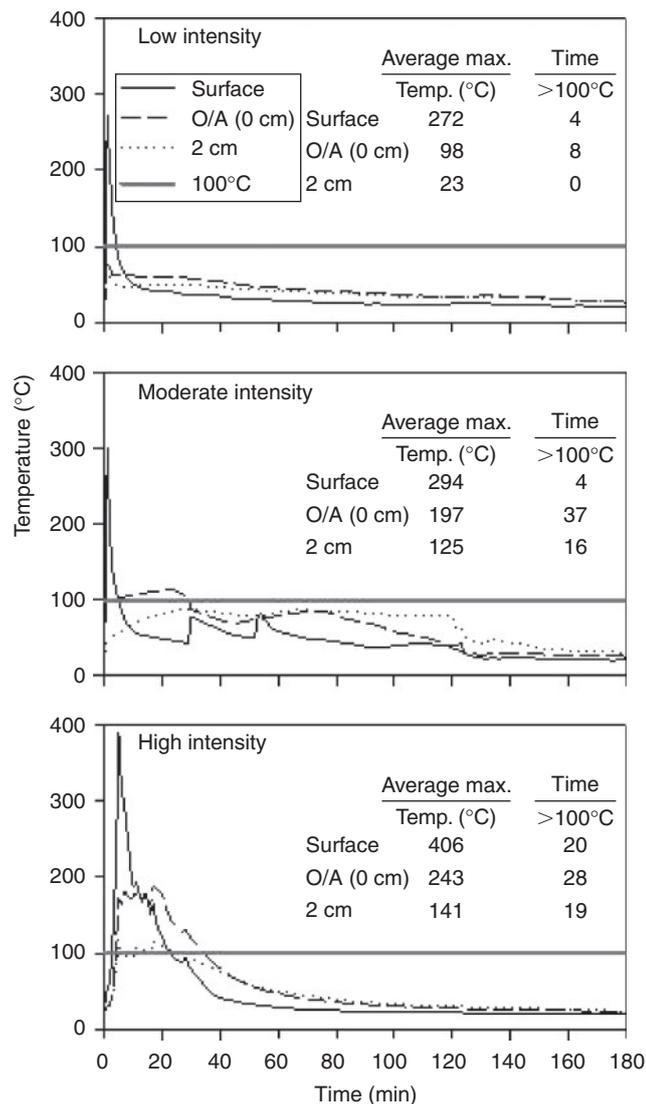


Fig. 1. Temperature profiles of soils burned at low, moderate, and high severity in the laboratory. Profiles are average values calculated at each time step (1 min) from four replicated burns at each severity. Temperatures were measured at the surface of the O horizon (O), interface of the O and A horizons (O/A), and 2 cm into the A horizon.

inconsistent temperature profile. The average maximum temperature of the mineral soil did not exceed 300°C, a temperature threshold at which structural changes to SOM are hypothesised to take place (Gonzalez-Perez *et al.* 2004).

The mass of O horizon consumed increased with burn severity so that nearly 80% was consumed by the high-severity burn treatments (Table 1). Burning at moderate- and high-severity levels caused the surface of the mineral soil to be darkened by changes to the SOM as well as addition of ash and char from the burnt O (Table 2). Post-fire assessments of fire severity are commonly made using qualitative guides such as the Composite Burn Index (CBI) (Key and Bensen 2006) and the classification system defined by Neary *et al.* (1999). Using both of these systems, the low- and moderate-severity treatments

Table 1. Proportion of mass, C and N consumed and C:N of combusted soil in the O horizon (mean \pm standard deviation) at three levels of fire severity ($n = 4$)

P_s values are from a one-factor ANOVA (severity as factor); significant values are in bold ($\alpha = 0.05$). Letters indicate similar subsets (columns) as determined by Tukey's honestly significant difference (HSD)

	Mass of O horizon (g g ⁻¹ initial mass, C, or N)	Carbon	Nitrogen	C:N
Low	0.32 \pm 0.13 ^a	0.37 \pm 0.15 ^a	0.27 \pm 0.19 ^a	99 \pm 35
Moderate	0.67 \pm 0.08 ^b	0.78 \pm 0.07 ^b	0.62 \pm 0.12 ^b	76 \pm 8
High	0.80 \pm 0.04 ^b	0.93 \pm 0.00 ^b	0.88 \pm 0.03 ^c	63 \pm 2
P_s	0.000	0.000	0.000	0.090

would be classified as low- and moderate-severity burns. Both methods would classify the highest-severity treatment between a moderate- and high-severity burn. If the mineral soil had been reddened and oxidised, then both methods would have classified the highest-severity treatment as a high-severity burn. A limitation of this study is that we did not achieve a very high severity burn that many forests experience during wildfires; however these laboratory burns simulated fire severities of prescribed burns that would be typical in the understorey of a ponderosa pine forest where there are few shrubs or coarse woody debris (e.g. not under a slash pile or under a smouldering log) at low, moderate, and high-moderate severity levels.

O horizon

Carbon volatilises and is combusted at lower temperatures than organic forms of N; therefore, at lower fire intensities and severities, C will be preferentially consumed relative to N (DeBano *et al.* 1998; Baldock and Smernik 2002). Accordingly, maximum temperature at the mineral-soil surface was significantly correlated with the C:N ratio of the combusted O horizon material ($r = -0.950$, $P \leq 0.05$). This led to the C:N ratio of the material combusted at low and moderate severity to be higher than that of the unburned O horizon (Tables 1, 2). At high severity, however, the rate of C and N consumption was roughly equal, so that the C:N ratio of the consumed material was not significantly different from the C:N ratio of the unburned O horizon.

Although combustion reduced the total content of all SOM fractions, SOM from the O horizon is dominated by non-soluble (NS) forms of C and N (Table 3). Carbon and N from the NS fraction increase in proportion to the other fractions with increasing fire severity (Fig. 2). The reduction of fulvic acid and humic acid C concentration of the O horizon is approximately equal with each step in severity, suggesting that combustion and transformation reactions did not discriminate between fulvic acid and humic acid. By and large, the O horizon was consumed and the remaining materials were converted to recalcitrant non-soluble materials.

Higher C:H ratios are typically associated with a higher degree of aromaticity and condensation, which may ultimately lead to lower bioavailability. Similarly, C:N ratios are also thought to influence the bioavailability of substrates, high C:N being indicative of low bioavailability (Colman *et al.* 2004). The

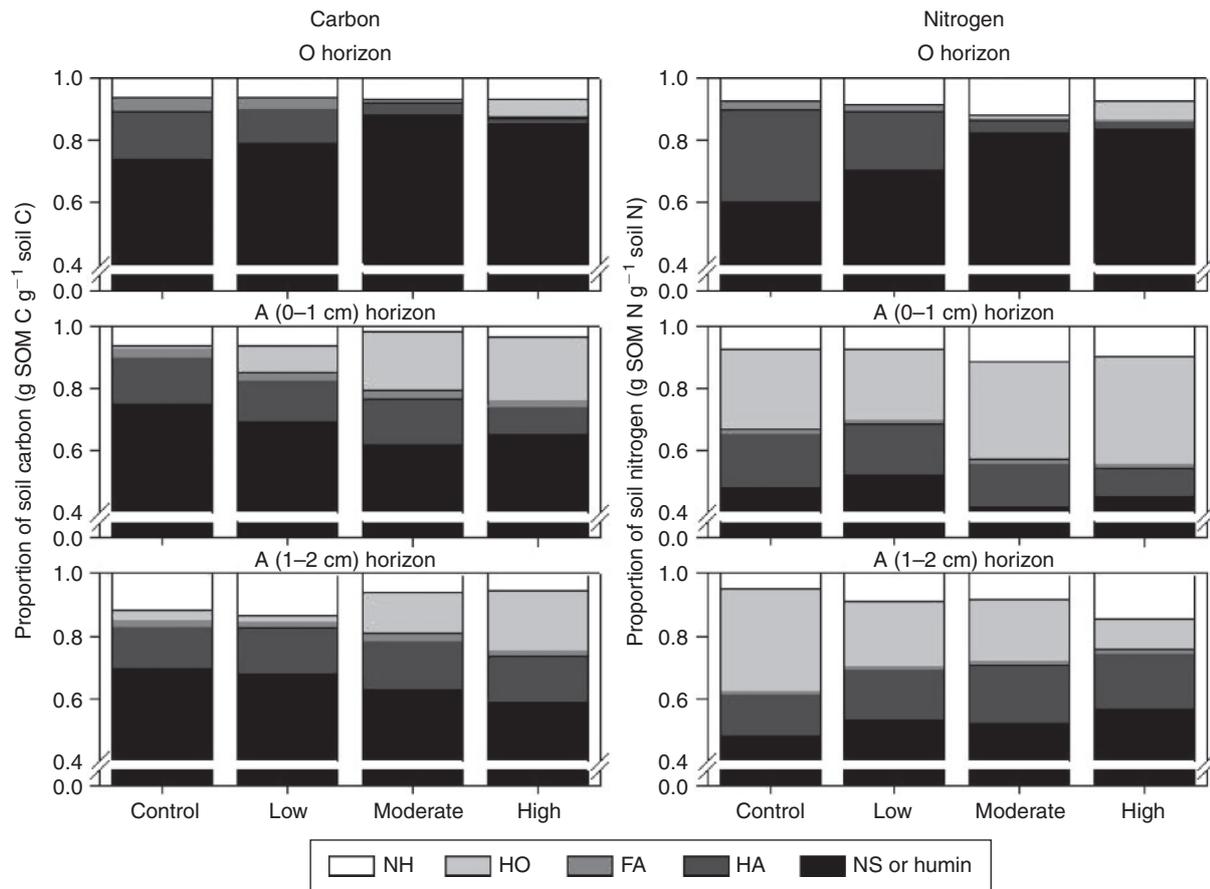


Fig. 2. Proportion of SOM (soil organic matter) C or N of O and A horizon C or N with three levels of burn severity and an unburned control ($n = 4$). Abbreviations are: NS, non-soluble (O horizon); HA, humic acid; FA, fulvic acid; HO, other hydrophobic materials; NH, soluble non-humic.

Table 4. Ratios of C : N and C : H of SOM (soil organic matter) fractions (mean \pm standard deviation) from O horizons burned at three levels of fire severity and an unburned control ($n = 4$, unless NH or HO, then $n = 1$)

P values are from a one-factor (severity (P_s)) ANOVA; significant values are in bold ($\alpha = 0.05$). Letters indicate similar subsets (rows) as determined by Tukey's HSD (a, b). Abbreviations are: NS, non-soluble; HA, humic acid; FA, fulvic acid; HO, other hydrophobic materials; NH, soluble non-humic; ND, not detectable

	Control	Low	Moderate	High	P_s
C : N NS	86 \pm 17 ^a	68 \pm 9 ^a	42 \pm 3 ^b	42 \pm 6 ^b	0.000
C : N HA	37 \pm 5	38 \pm 7	37 \pm 6	39 \pm 22	0.990
C : N FA	123 \pm 23 ^a	99 \pm 19 ^a	54 \pm 8 ^b	44 \pm 13 ^b	0.000
C : N HO	ND	ND	ND	35	
C : N NH	38	43	21	33	
C : H NS	0.8 \pm 0.1	0.8 \pm 0.1	0.8 \pm 0.0	0.9 \pm 0.1	0.294
C : H HA	0.8 \pm 0.04 ^a	0.9 \pm 0.2 ^a	1.0 \pm 0.1 ^a	1.9 \pm 0.6 ^b	0.002
C : H FA	0.9 \pm 0.02	0.9 \pm 0.1	1.0 \pm 0.1	0.9 \pm 0.1	0.206

350°C, humic acid was more susceptible to transformations affecting its solubility.

During burning, the surface of most O horizons achieved 300°C, except in two low-severity treatments, the threshold temperature suggested by Gonzalez-Perez *et al.* (2004) at which SOM polymerisation reactions take place. These reactions are converting labile materials to the NS fraction, which is likely

more recalcitrant, owing to high hydrophobicity and low solubility. The increase in the proportion of recalcitrant forms of C and N with increasing burn severity may allow recalcitrant materials to accumulate in the soil. Recalcitrant forms of C produced by fire may contribute to C sequestration (Kuhlbusch and Crutzen 1995) whereas recalcitrant forms of N may limit N availability (Guinto *et al.* 1999).

Table 5. Total soil C and N concentration of each SOM (soil organic matter) fraction (mean \pm standard deviation) from A horizons burned at three levels of fire severity and an unburned control ($n = 4$, unless NH or HO, then $n = 1$)

P values are from a two-factor ANOVA (severity by depth (P_s , P_d , and $P_{s \times d}$)); significant values are in bold ($\alpha = 0.05$). Letters indicate similar subsets (rows) as determined by Tukey's HSD (a, b, c, d) or pairwise contrasts (e, f for A1). A superscript δ indicates A1 was significantly different from A2 at that treatment level (used only when interaction was significant, $P < 0.05$). Abbreviations are: Hu, humin; HA, humic acid; FA, fulvic acid; HO, other hydrophobic materials; NH, soluble non-humic

		Control	Low	Moderate	High	P_s	P_d	$P_{s \times d}$	
		(g C kg ⁻¹ soil)							
Hu	A1	35.5 \pm 3.1 ^{ab}	36.5 \pm 4.3 ^a	28.8 \pm 1.8 ^b	28.8 \pm 5.0 ^{ab}	0.016	0.054	0.447	
	A2	32.1 \pm 2.6 ^{ab}	32.1 \pm 3.8 ^a	29.7 \pm 2.2 ^b	29.4 \pm 2.8 ^{ab}				
HA	A1	7.2 \pm 1.1 ^c	7.1 \pm 1.2 ^c	6.8 \pm 0.9 ^c	4.4 \pm 1.0 ^{fδ}	0.028	0.192	0.001	
	A2	6.2 \pm 1.1	6.7 \pm 0.4	7.2 \pm 0.4	7.1 \pm 0.3 ^{δ}				
FA	A1	1.3 \pm 0.2	1.4 \pm 0.4	1.3 \pm 0.2	1.1 \pm 0.1	0.108	0.002	0.528	
	A2	1.0 \pm 0.1	1.0 \pm 0.1	1.1 \pm 0.1	1.0 \pm 0.2				
HO	A1	5	4	9	10				
	A2	1	2	6	10				
NH	A1	3	4	1	2				
	A2	5	6	3	3				
		(mg N kg ⁻¹ soil)							
Hu	A1	1224 \pm 113	1334 \pm 200	1216 \pm 168	1310 \pm 126	0.417	0.303	0.855	
	A2	1380 \pm 279	1393 \pm 140	1220 \pm 119	1357 \pm 216				
HA	A1	437.4 \pm 43.9 ^c	425.4 \pm 76.8 ^c	405.9 \pm 68.8 ^c	255.8 \pm 63.9 ^{fδ}	0.016	0.063	0.002	
	A2	385.1 \pm 65.8	418.9 \pm 29.3	440.0 \pm 25.4	427.9 \pm 22.7 ^{δ}				
FA	A1	36.3 \pm 6.6 ^a	36.2 \pm 10.7 ^{ab}	47.3 \pm 7.3 ^{ab}	41.3 \pm 4.0 ^b	0.006	0.383	0.629	
	A2	29.2 \pm 4.8 ^a	29.1 \pm 2.3 ^{ab}	32.5 \pm 1.8 ^{ab}	36.4 \pm 14.8 ^b				
HO	A1	810	580	930	1000				
	A2	930	600	470	230				
NH	A1	189	191	335	292				
	A2	135	244	196	348				

A horizon

A horizon depth and severity interacted significantly to affect soil pH. The A1 horizon showed an increase in pH with burn severity, especially at moderate and high severity, that could be due to incorporation of extremely alkaline materials from the burned O horizons or oxidation of organic matter, neither of which appeared to occur in the A2 horizon (Table 2). Additionally, these soils were never exposed to precipitation, so that the Na and K hydroxides that have been shown to be responsible for high pH in ash immediately after burning would still have been present at the time of measurement (Ulery *et al.* 1993).

The C concentration of both A horizons was unaffected by severity of burn (Table 2). This suggests that any changes occurring to SOM composition in the mineral horizons will be due to SOM transformations and not consumption of C-containing materials.

A horizon depth and burn severity interacted significantly to affect N concentration and C : N ratio of both A1 and A2 horizons (Table 2). After the moderate- and high-severity burn treatments, there was a significant decrease in N concentration of the A2 horizon due to volatilisation. This agrees with the view that N is generally lost from mineral soils when heated or burned, after additions from the O horizon and litterfall are accounted for (DeBano *et al.* 1998; Neary *et al.* 1999; Certini 2005).

The proportion and total HO C increased with increasing severity whereas humin C decreased ($P_s = 0.119$) (Table 5 and Fig. 2). The A1 horizon humic acid C and N content of the high-severity burn also decreased whereas the A2 horizon remained

unaffected, causing a significant interaction with severity and depth ($P_{s \times d} = 0.002$). Increasing fire severity decreased the amount and proportion of humin C in both A1 and A2 horizons and the proportion of humic acid C and N of the A1 horizon of the high-severity burn. It appears as if SOM of the A1 horizon is becoming more labile with a concurrent decrease in the more recalcitrant SOM fractions, humic acid and humin.

We hypothesise that increased levels of HO materials may be due to thermal fragmentation, or destructive distillation, of long-chain aliphatic compounds that may have been fractionated as humic acid or humin in an unburned soil. In support of this is the work conducted by Almendros *et al.* (1988), who report a decrease in soil lipid chain length after wildfire. Additional authors found that fulvic and humic acids that remain adsorbed to a column of DAX-8 resin after elution with 0.1 N NaOH (similar to this study's HO fraction) have been shown to have a large content of aliphatic groups and may be somewhat hydrophobic (Yonebayashi and Hattori 1990; Dai *et al.* 2006). In the current study, the fragmented aliphatic molecules may have been extracted from the soil by 0.1 N NaOH and hydrophobic enough to have been removed from the fulvic acid fraction by DAX-8 resin. Further, Dai *et al.* (2006) found that these substances had a clear amide presence in the Fourier-transform (FT)-IR spectra, which corresponds with the current study's finding that these substances provide a major proportion (10–34%) of total soil N (Fig. 2).

These HO compounds may be similar to the hydrophobic neutral compounds defined by Leenheer (1981) using non-ionic

Table 6. Ratios of C : N and C : H of SOM (soil organic matter) fractions (mean \pm standard deviation) from A horizons burned at three levels of fire severity and an unburned control ($n = 4$, unless NH or HO, then $n = 1$)

P values are from a two-factor ANOVA (severity by depth (P_s , P_d , and $P_{s \times d}$)); significant values are in bold ($\alpha = 0.05$). Letters indicate similar subsets (rows) as determined by Tukey's HSD (a, b) or pairwise contrasts (e, f for A1). Superscript δ indicates A1 and A2 horizons are significantly different at that fire-severity level. Abbreviations are: Hu, humin; HA, humic acid; FA, fulvic acid; HO, other hydrophobic materials; NH, soluble non-humic

		Control	Low	Moderate	High	P_s	P_d	$P_{s \times d}$
C : N Hu	A1	34 \pm 4	32 \pm 1	28 \pm 5	28 \pm 2	0.123	0.005	0.198
	A2	28 \pm 4	27 \pm 3	28 \pm 1	26 \pm 3			
C : N HA	A1	19.2 \pm 1.0	19.6 \pm 0.3	19.7 \pm 0.8	20.0 \pm 0.5	0.075	0.004	0.949
	A2	18.6 \pm 0.3	18.8 \pm 0.5	19.1 \pm 0.3	19.4 \pm 0.3			
C : N FA	A1	42 \pm 3 ^a	44 \pm 6 ^a	33 \pm 2 ^{ab}	30 \pm 2 ^b	0.000	0.196	0.222
	A2	42 \pm 5 ^a	42 \pm 2 ^a	40 \pm 1 ^{ab}	35 \pm 10 ^b			
C : N HO	A1	6	8	10	10			
	A2	1	3	13	42			
C : N NH	A1	17	19	2	7			
	A2	40	26	15	8			
C : H HA	A1	0.8 \pm 0.0 ^c	0.8 \pm 0.1 ^c	1.0 \pm 0.0 ^{δb}	1.0 \pm 0.0 ^{δb}	0.000	0.000	0.002
	A2	0.8 \pm 0.1	0.8 \pm 0.1	0.8 \pm 0.0 ^δ	0.8 \pm 0.1 ^δ			
C : H FA	A1	1.0 \pm 0.1	1.0 \pm 0.1	1.0 \pm 0.1	1.0 \pm 0.1	0.673	0.908	0.064
	A2	1.0 \pm 0.1	1.1 \pm 0.1	0.9 \pm 0.0	1.0 \pm 0.1			

XAD-8 resin to fractionate dissolved organic matter (DOM) in surface waters. Nitrogen-containing hydrophobic neutral compounds that may also be hydrophobic include chlorophyll and humic substances with <1 carboxylic group per 13 C atoms; however, it is not known whether any of these substances are volatile when heated (Thurman 1985; Qualls and Haines 1991; Doerr *et al.* 2000). In addition to thermal fragmentation and destructive distillation of humin and humic acid, these labile substances may also have been derived from lysed microbe cells (e.g. cell walls and nucleic acids) as suggested by Serrasolsas and Khanna (1995).

Labile compounds after burning may cause a flush of C and N mineralisation from mineral soils as has been observed after fire (Fernández *et al.* 1999; Choromanska and DeLuca 2002). Serrasolsas and Khanna (1995) report increased dissolved organic N (which may encompass HO N) after heating a soil to 120°C. Soluble C (which may include HO C) has been seen to increase after heating mineral soil up to 500°C (Battle and Golladay 2003; Guerrero *et al.* 2005). These authors attribute the release of soluble C and N to the release of microbial cell contents and destructive distillation of humic materials caused by heating (Serrasolsas and Khanna 1995; Battle and Golladay 2003; Guerrero *et al.* 2005). The increase in labile C, possible at the expense of humin and humic acid, may increase post-fire mineralisation and further reduce soil C content.

Heat-induced polymerisation of materials such as fulvic acid has been shown to decrease their solubility through the reduction of O-containing functional groups and ring formation (Almendros *et al.* 1990, 2003; Fernández *et al.* 1997, 2001; Gonzalez-Vila and Almendros 2003; Gonzalez-Perez *et al.* 2004); however, the amount of fulvic acid C was not affected by fire severity (Table 5). Fulvic acid polymerisation has been shown to be followed by an increase in humic acid or humin (Almendros *et al.* 1990). Compared with the current study, previous research has used very severe heating or wildfire conditions. Burning at low- to moderate-severity levels may

not be high enough severity to cause reactions that create recalcitrant materials. There may be a threshold fire severity, above the severity achieved during the current study, at which mineral-associated SOM is predominantly being altered by polymerisation reactions and combustion.

Humin and humic acid C : N ratio was significantly affected by depth owing to O horizon additions in the A1 of the control, whereas the C : N ratio of the A1 horizon from the moderate- and high-severity burns decreased as a result of fire severity (Table 6). However, either N was converted to fulvic acid or fulvic acid was able to preserve N, which appears to have decreased its C : N ratio and possibly improved its quality as a substrate for microbe growth. Similarly to Almendros *et al.* (1990), fulvic acid C and N appear to be more resistant to fire severity than humin and humic acid C and N in A horizons. In general, higher burn severity appears to be associated with an increase in labile material and an increase in the bioavailability of fulvic acid, which may have increased post-fire mineralisation and further reduced C storage in the soil (Hatten and Zabowski 2009).

Black carbon

The LOI₃₇₅₋₅₅₀ had a significant correlation ($P < 0.001$) with BC from A horizons, with r^2 values of 0.971. Black carbon from A horizons were found to make up $37.2 \pm 6.9\%$ (95% confidence interval, CI) of LOI₃₇₅₋₅₅₀. Depending on soil type, C may account for 40 to 58% of SOM (Nelson and Sommers 1996), which agrees well with the ratio we developed for our soil to convert mineral soil LOI₃₇₅₋₅₅₀ to BC. For the O horizons, LOI₃₇₅₋₅₅₀ had a significant relationship ($P < 0.000$) with BC with an r^2 value of 0.935. Black carbon from the O horizon was found to make up $51.4 \pm 19.4\%$ (95% CI) of LOI₃₇₅₋₅₅₀.

Using this method, Vertisol and Mollisol BC standards from Australia were found to have a BC concentration of 6.1 and 3.6 g kg⁻¹ soil respectively. Hammes *et al.* (2007) found that these same soils had very low BC concentrations of 0.8 ± 0.4

Table 7. Black carbon (BC) and black nitrogen (BN) for O and A horizons (mean \pm standard deviation) treated to three levels of fire severity and an unburned control ($n = 4$)

P values are from a one- (severity (P_s)) or two- (severity by depth (P_d and $P_{s \times d}$)) factor ANOVA; significant values are in bold ($\alpha = 0.05$). Letters indicate similar subsets (rows) as determined by Tukey's HSD (a, b, c)

		Control	Low	Moderate	High	P_s	P_d	$P_{s \times d}$
BC (g kg ⁻¹ soil)	O	0.9 \pm 0.6 ^a	2.6 \pm 1.0 ^b	4.8 \pm 0.2 ^c	5.1 \pm 0.6 ^c	0.000		
	A1	5.3 \pm 1.1	4.2 \pm 0.7	4.7 \pm 0.4	4.4 \pm 0.8	0.474	0.899	0.946
	A2	4.4 \pm 0.8	4.1 \pm 0.8	4.7 \pm 0.2	4.4 \pm 0.6			
BN (g kg ⁻¹ soil)	O	0.02 \pm 0.01 ^a	0.07 \pm 0.03 ^b	0.12 \pm 0.01 ^c	0.13 \pm 0.01 ^c	0.000		
	A1	0.53 \pm 0.11	0.42 \pm 0.07	0.46 \pm 0.04	0.44 \pm 0.08	0.474	0.899	0.946
	A2	0.44 \pm 0.08	0.41 \pm 0.08	0.47 \pm 0.02	0.44 \pm 0.06			

(95% CI) and 0.8 \pm 0.5 (95% CI) g kg⁻¹ soil for the Vertisol and Mollisol respectively when tested by the CTO375 method (Gustafsson *et al.* 1997). The adapted method overestimated the BC concentration for the mineral-soil standards, probably as a result of mineral dehydration occurring in the Australian samples between muffle furnace temperatures of 375° and 550°C. When C analysis was conducted directly on the Vertisol and Mollisol standards heated to 375°C, BC concentration was determined to be 1.7 and 1.3 g kg⁻¹ respectively. The difference between the BC contents calculated by the adapted method and measured directly suggests that the conversion ratio is soil-type specific and must be developed for each soil. Charred-grass and wood standards prepared according to Hammes *et al.* (2006) were found to have BC concentrations of 5.4 and 0.6 g kg⁻¹ sample respectively using the method described above. Hammes *et al.* (2007) found these organic samples to have BC concentrations of 9.0 \pm 7.0 g kg⁻¹ soil (95% CI) and 0.0 \pm 0.0 for the grass and wood samples respectively (Hammes *et al.* 2007). Thus, the adapted method agreed well with the results obtained for the organic samples when the 95% CI are taken into account.

The BC content of the O horizons increased with burn severity and was positively correlated with the mass of O horizon consumed ($r = 0.800$; $P \leq 0.05$) (Table 7). Black carbon contents of the A horizons were not affected by burning or depth. The mineral soil, including control, had an average BC content of 4.5 g BC kg⁻¹ soil and a black nitrogen (BN) content of 0.45 g kg⁻¹ soil. Materials were converted to BC in the O horizon so that the proportion of total C as BC increased from 0.2% in the unburned control to 2.3% in the highest-severity treatment. Black carbon was not produced in the A horizon, averaging 9.3% of the total C, likely as the result of lower temperatures compared with the O horizon during burning. The BC content of the mineral soil is likely indigenous, produced by fires that have occurred in these fire-prone forests (Everett *et al.* 2000; Hatten *et al.* 2005).

The low- and moderate-severity burns produced BC at a rate of 21.7 and 9.0 kg ha⁻¹ respectively; high-severity burns consumed much of the O horizon, and had an undetectable production of BC in the entire profile. This production rate is much lower than that reported for a range of fire types, 61 to > 20 000 kg BC ha⁻¹ (Preston and Schmidt 2006) and likely the result of a low amount of woody material burned during this experiment.

Preston and Schmidt (2006) hypothesise that wood may be the principle source of most BC.

Conclusions

The effects of fire on soil organic matter are dependent on fire severity, which varies across the landscape and seasons in ponderosa pine–Douglas-fir forests. Fires at varying severity were reproduced in the laboratory and represented low- to moderate-severity level prescribed burns in the understorey of a ponderosa pine–Douglas-fir stand. Increasing fire severity increased the proportion of recalcitrant materials, e.g. charcoal, in the O horizon, similarly to other findings in the literature. The A horizon content of humin and humic acid decreased while the hydrophobic, alkaline-extractable and acid-soluble fraction increased. Additionally, fulvic acid from the A horizons appears to be more bioavailable as the C : N ratio is lower. These results contrast with those of other studies that have examined high-severity heating or wildfire effects on SOM, as most observed an increase in recalcitrant materials. Other studies used high-severity treatments that may have crossed a threshold at which mineral soil organic matter is predominantly being altered by polymerisation reactions and combustion. The severity that these laboratory burns experienced may have destructively distilled mineral soil humic materials or released the contents of microbial cells. Polymerisation reactions that could convert labile materials to more recalcitrant fractions did not dominate in the mineral soil owing to the low- to moderate-severity fires. The increase in labile soil organic matter may be the source of increased C and N mineralisation rates of the mineral soil often seen after fire. However, elevated levels of recalcitrant materials may protect the remaining C from decomposition.

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