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Soil humic-like organic compounds in prescribed fire emissions using nuclear magnetic resonance spectroscopy

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ABSTRACT

Here we present the chemical characterization of the water-soluble organic carbon fraction of atmospheric aerosol collected during a prescribed fire burn in relation to soil organic matter and biomass combustion. Using nuclear magnetic resonance spectroscopy, we observed that humic-like substances in fire emissions have been associated with soil organic matter rather than biomass. Using a chemical mass balance model, we estimated that soil organic matter may contribute up to 41% of organic hydrogen and up to 27% of water-soluble organic carbon in fire emissions. Dust particles, when mixed with fresh combustion emissions, substantially enhances the atmospheric oxidative capacity, particle formation and microphysical properties of clouds influencing the climatic responses of atmospheric aeroso. Owing to the large emissions of combustion aerosol during fires, the release of dust particles from soil surfaces that are subjected to intense heating and shear stress has, so far, been lacking.

1. Introduction

Particulate organic carbon (POC) in fire emissions has been regularly attributed to biomass combustion (Hoffer et al., 2006; Decesari et al., 2006) with water-soluble humic-like substances (HULIS) accounting for a large fraction of POC. Meanwhile, the resuspension of mineral particles in fires due to enhanced turbulent mixing near and after the burn front was previously observed (Clements et al., 2008) and some similarities with terrestrial organic matter were observed (Stone et al., 2009; Kavouras et al., 2012). When dust particles are mixed with biomass burning emissions, the overall oxidative capacity (i.e. reactions) and new particle formation potential of the smoke plume is enhanced influencing the formation of cloud condensation nuclei (DeMott et al., 2003). Fires have a detrimental effect on ecosystems and the atmosphere, near and far away from the area of the burn (Certini, 2005; Chen et al., 2009). More frequent, larger and more destructive wildfires due to recurrent and long-lasting droughts, longer fire seasons and changes in biomass have been observed over the last few decades (Gedalof et al., 2005; Keeley et al., 1999). Areas with the greatest fire frequency include the Arc of Deforestation in Brazil, sub-Saharan and southeastern Africa, Australia, southeastern and western US, southeast Asia, the southern plains of Siberia and deserts in Mongolia and China (Giglio et al., 2006). In these regions, the fire season lasts, on average, six months, although longer periods (8–12 months) were observed in the savannahs of Africa, eastern Australia, the US and Brazil. Many of these regions have also been characterized by soil textures that are prone to wind erosion contributing ~1500 Tg/yr of dust particles (Maria et al., 2004).

The heating of soil during a fire can modify the physical and chemical properties of soil textures by forming coarse soil particles through mineralization and by producing lipophilic layers due to pyrolysis of soil organic matter, increasing the potential for wind erosion (Atanassova and Doerr, 2011). These effects were more evident in deserts and shrublands as compared to grass and forest fires due to soil heating and differences in soil stability (Cruz et al., 2011). Studies of physical properties of soils before and after fires

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in arid and semi-arid areas often show an increase in soil bulk density, disaggregation of soil structure, and collapse of pores, at least in part a result of destruction of organic binding agents (Kennard and Gholz, 2001).

The heating in and around the fire front has been shown to generate sufficient turbulence and wind shear to trigger the suspension of loose dust particles into the atmosphere (Clements et al., 2008). In a recent study, we identified that soil minerals may contribute up to 10% of PM10 (particles with aerodynamic diameter less than 10 μm) emissions from prescribed fires (Kavouras et al., 2012). An organic carbon-to-organic mass (OM/OC) conversion factor of 3.1, comparable to OM/OC values for mineral soils in the US (Cambardella et al., 2001), was used to reconstruct aerosol mass when soil contribution was the highest. The conversion factor was associated with the increased content in humic and fulvic acids. These compounds have been found to modify the indirect aerosol climate forcing by changing the critical size and super-saturation of cloud condensation droplets (Kokkola et al., 2006). Aerosol containing humic acids was also shown to influence atmospheric oxidation such as the conversion of NO_2 to nitrous acid (HONO) (Wang et al., 2003). At the Gleason prescribed fire, impacts of the fire included progressive breakdown of soil structure over slightly more than one year that resulted in disaggregated soil particles at the soil surface and into the soil column that contributed to higher post-fire soil aeolian suspension potential; and decreased water infiltration potential (Chief et al., 2012).

This paper presents, for the first time, the *in-situ* chemical profiles of particulate water-soluble organic carbon (WSOC) emissions from a prescribed burn event and their relationships to soil organic matter and biomass combustion. By applying the similarity index and the chemical mass balance model, we calculate the contribution of soil and biomass combustion particles to prescribed burn WSOC emissions in order to identify which components of soil organic matter are present in the fire plume and whether turbulence in the fire front and/or temperature-induced structural changes enhance the resuspension of soil organic matter.

2. Materials and methods

The prescribed burn extended over an area of 1.55 km^2 covered by sagebrush (*Artemisia* sp.) and pinyon pine/juniper (*Pinus monophylla* and *Juniperus osteosperma*) plant communities in the Upper Gleason watershed within the Great Basin Desert in east-central Nevada (39°23′43″N, 115°03′57″W). The fire was ignited with a heavy drip-torch starting at the southern boundary of the plot (heading fire). It lasted for approximately 6 h with soil temperatures reaching 315 °C at a 1-cm depth under the sagebrush canopy. Measurements included: (*i*) the collection of time-resolved PM10 aerosol samples on Teflon and quartz filters during the prescribed burn event using the In-Plume Emission Test Stand (IPETS) (Nussbaum et al., 2009), corresponding to 0–2 h, 2–4 h and 4–6 h after the initial ignition (hereafter, Rx#1, Rx#2 and Rx#3, respectively); (*ii*) PM_{10} soil dust before and after the prescribed burn event using the Portable In-Situ Wind Erosion Laboratory (PI-SWERL) (Etyemezian et al., 2007); and (*iii*) PM_{10} aerosol samples from the combustion of ~2 m^3 of native biomass in a heavy-duty metal fire pit for 2 h (biomass combustion, hereafter). Samples were analyzed for elements, ions, elemental carbon, total organic carbon and WSOC. A portion (half) of the collected filters was extracted in H_2O and analyzed by NMR.

The IPETS sampling system was located on the north boundary of the prescribed burn plot (winds blowing from the south at less than 10 m/s). PM10 aerosol samples of resuspended soil dust (on both Teflon and quartz filters) were also collected from eight locations inside the prescribed burn plot before the event using the PI-SWERL system. The WSOC was determined by thermal optical reflectance method. A portion (half) of the quartz filter was sonicated in ultrapure H_2O for 1 h. The extract was dried and re-dissolved in 0.5 ml D_2O containing 30 μg of 3-(trimethylsilyl)propionic acid-d4 sodium salt (TSP-d4) internal standard and transferred to 5 mm diameter NMR tubes. ^1H NMR spectra were obtained on Bruker NMR spectrometers with operating frequencies of 500 and 600 MHz at 298 K using spin-lock, acquisition time of 2 s, relaxation delay of 1 s and 1 Hz exponential line broadening (8192 acquisitions). A suppression sequence using 90-degree selective pulses for water was applied to eliminate the water signal interference. The baseline was manually corrected and integrated using the ACD/NMR processor (Version 12.01 Academic Edition). The identification was done based on their chemical shift (δ) relative to that of TSP-d4 set at 0.0 ppm. Because of atmospheric H_2O residues in the sample, the segment from ~4.5 to ~5.0 ppm was removed from all NMR spectra. We applied the *icoshift* algorithm to align the NMR spectra (Savorani et al., 2010) and integrated the intensity of signals in 250 pre-defined bins with 0.04 ppm width. A portion of the NMR spectrum was excluded because of water suppression and low signal-to-noise ratios; thus, the number of variables was reduced to 236.

3. Results and discussion

NMR spectroscopy has recently been used to qualitatively determine the chemical content of the water soluble fraction of biogenic, marine and background organic aerosol (Decesari et al., 2006, 2011; Tagliavini et al., 2006; Ziemba et al., 2011). The NMR spectra of native soil (from samples collected prior to the burn) organic matter and biomass combustion are illustrated in Fig. 1. The

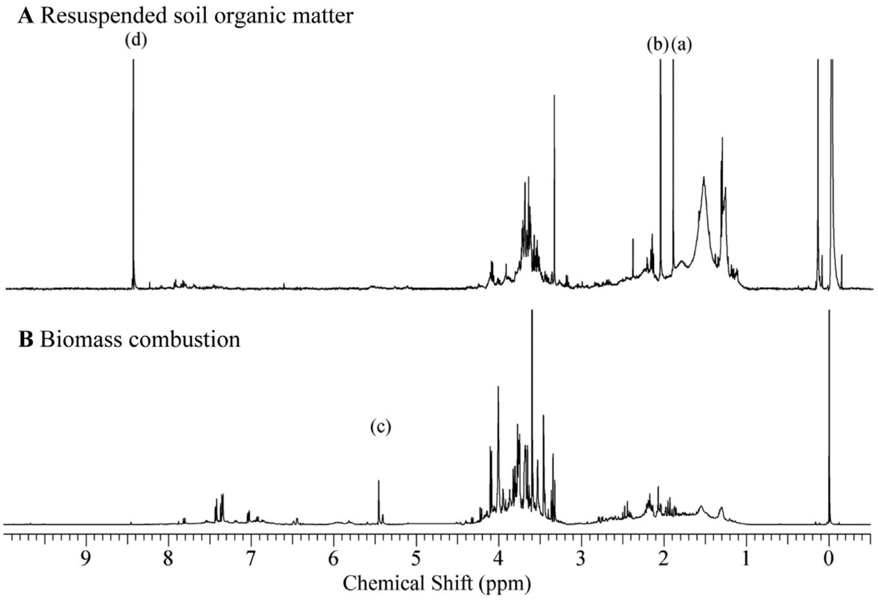

Fig. 1. 600 MHz ^1H NMR of resuspended (by PI-SWERL) soil organic matter (A) and biomass combustion (B). The segment from ~4.5 to ~5.0 ppm was removed from all NMR spectra due to H_2O residues. The peaks (a), (b) and (d) were uniquely identified in soil organic matter extract but not assigned to a specific compounds. The peak (c) was tentatively assigned to levoglucosan, a tracer of wood burning.

Table 1
H- and C-content of Water Soluble Organic Carbon (WSOC) extracted from soil organic matter, biomass combustion and prescribed fire aerosols. Rx#1: 0–2 h, Rx#2: 2–4 h and Rx#3: 4–6 h represent the prescribed burn periods after the initial ignition.

	Soil organic matter	Biomass combustion	Rx#1	Rx#2	Rx#3
Total H content (μmol/m^3)	1.30	8.40	1.75	5.00	4.10
% (R–**H**)	45.7	13.1	34.1	32.1	33.2
% (=C–C–**H**)	17.6	20.7	36.5	40.5	37.5
% (O–C–**H**)	23.4	56.3	23.9	16.7	17.7
% (O–C**H**–O and = C–**H**)	2.9	1.5	1.0	1.3	0.9
% (Ar–**H**)	10.3	8.3	4.4	9.4	10.7
WSOC (μmol/m^3)	7.11	21.35	2.73	7.40	6.07
H/C molar ratio	0.18	0.39	0.64	0.68	0.68

spectrum of soil showed convoluted strong signals in the 1.0–1.8 ppm range (aliphatic, R–H), consistent with the expected resonances of humic and fulvic acids (Havers et al., 1998; Francioso et al., 2000), and a limited number of sharp peaks in the 1.8–3.2 ppm range. The peaks in this range accounted for 70% of H-atoms in the soil NMR spectra (Table 1). In comparison, the spectrum of biomass combustion demonstrated two weak convoluted peaks in the aliphatic range and many sharp peaks in 1.8–3.2 ppm, accounting for 13.1 and 20.7% of the H-atoms, respectively. The two large peaks at 1.87 ppm (a) and 2.08 ppm (b) in the soil NMR were not observed in the biomass combustion. The peak at 1.87 ppm was previously assigned to acetate (Decesari et al., 2001; Matta et al., 2003). Both soil and biomass NMR spectra showed a mixture of convoluted and sharp peaks in the carbohydrate region (3.2–4.4 ppm; O–C–H) which was attributed to sugars and anhydrides, typically found in both soil organic matter and biomass burning (Simoneit et al., 2004). The peak at 5.40 ppm (c) in the biomass burning NMR was previously assigned to levoglucosan, a tracer of biomass burning (Tagliavini et al., 2006). This peak was not observed in the soil organic matter (Fig. 1a). The H-atoms in the carbohydrate range accounted for 24% of the soil organic matter total H-atoms and 57% of the biomass combustion total H-atoms. The aromatic range (6.2–9.0 ppm) of the soil organic matter was characterized by a strong single sharp peak at 8.49 ppm (d), previously assigned to formate (Decesari et al., 2001; Matta et al., 2003), and a few smaller peaks from aryl-H atoms in the 7.5–8.0 ppm. For biomass combustion, we did not observe the single peak at 8.49 ppm, but sharp peaks from H-atoms were identified in the 6.8–7.5 ppm range. The percentages of H-atoms in the aromatic range were 10.3% for soil organic matter and 8.3% for biomass combustion. The total concentrations of H-atoms were 1.30 and 8.40 μmol/m^3 for soil and biomass combustion, respectively (Table 1). The concentrations of the water-soluble organic carbon (determined by thermal analysis (Chen et al., 2009) were 7.11 and 21.35 μmol/m^3 for soil and biomass combustion, respectively, resulting in molar H/C ratios of 0.18 for soil organic matter and 0.39 for biomass combustion (Table 1). Overall, distinct differences between the NMR spectra of soil organic matter and biomass combustion were observed in terms of individual peaks as well as the relative contributions of the different types of H-atoms, raising the prospect of resolving the contribution of soil organic matter to aerosol samples collected during a prescribed burn event.

The NMR spectra of aerosol samples collected during the prescribed burn event demonstrated similarities with both the soil organic matter and biomass combustion NMR spectra (Fig. 2). Specifically, the convoluted peaks in the aliphatic range were observed in all prescribed burn samples. In the unsaturated range, the NMR pattern during Rx#1 was almost identical to that observed for soil organic matter (including the two peaks (a) and (b)), while Rx#2 and Rx#3 NMR profiles included most of the peaks observed in the biomass combustion NMR spectrum. We did not observe clear patterns in the carbohydrate fraction because of the large and overlapping number of peaks. However, the levoglucosan peak at 5.40 ppm (c) was observed in all prescribed burn samples with increasing intensity from Rx#1 to Rx#3. In the aromatic range, the peak at 8.49 ppm (d) was also observed in all prescribed burn samples but with decreasing intensity from R_x#1 to R_x#3. This peak (d) was more pronounced in the R_x#1, while the intensity of other aromatic-H atoms (found in biomass combustion in 6.5–8.0 ppm range) intensified in the R_x#2 and R_x#3 samples. The prescribed burn NMR spectra were dominated by peaks in the resonance range expected for carbohydrates (36.5%–40.5%) followed by aliphatic range peaks (32.1%–33.2%). The H/C ratios ranged from 0.64 for Rx#1 to 0.68 for Rx#2 and Rx#3. These qualitative analyses

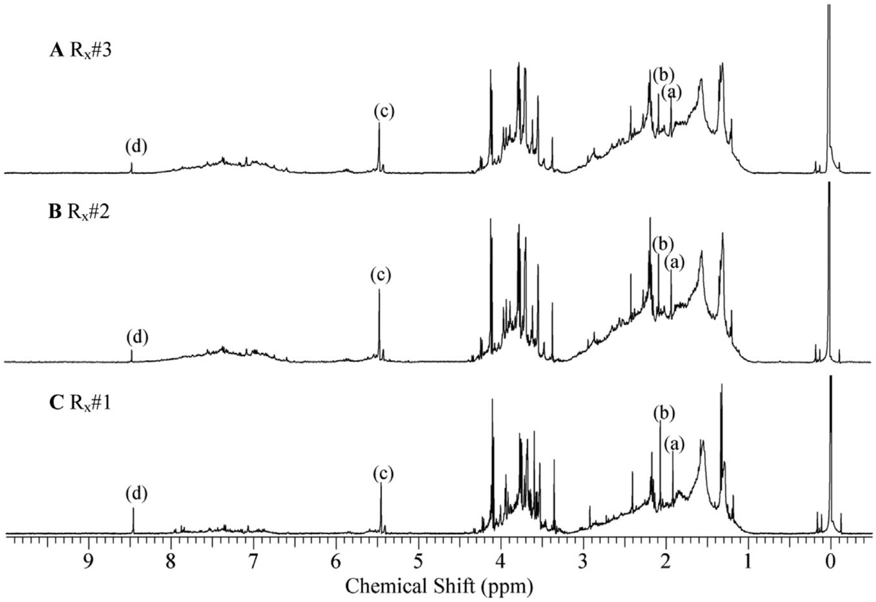

Fig. 2. 600 MHz ^{1}H NMR of prescribed burn particulate water soluble organic carbon. The prescribed burn periods were R_x#3: 4–6 h (A), R_x#2: 2–4 h (B) and R_x#1: 0–2 h (C) after the initial ignition.

indicated the presence of soil organic matter in prescribed burn emissions mostly in the Rx#1 and to a lesser extent in Rx#2 and Rx#3.

Additionally, we computed the similarity index (SI) using the bin method (Bodis et al., 2007). The SI values range from 0 to 1, with 0 being indicative of no similarities and 1 suggesting identical patterns. The bin method relies on bucketing of NMR spectra into *n*-bins and integrating signal intensities in each bin, allowing for the identification of similarities even if the positions of the signals differ by more than their widths. In addition, it enables the same signal processing (and thus reduced errors) as is used in the chemical mass balance analysis (described below). The SI values between the soil organic matter and the three prescribed burn samples were 0.41 for Rx#1, 0.41 for Rx#2 and 0.36 for Rx#3. For biomass combustion, the SI values were 0.61 for Rx#1, 0.73 for Rx#2 and 0.77 for Rx#3. While these values showed that biomass combustion during prescribed burns was a larger source of organic matter, but that soil organic matter was present in fire emissions, too.

We applied the chemical mass balance (CMB) model to estimate the contributions of soil organic matter and biomass combustion on prescribed burn emissions. The CMB has been extensively used in environmental applications to estimate the contribution of sources of pollutants at a receptor site when emission profiles are known (USEPA, 2004). Here, the emission profiles were the spectra of soil organic matter and biomass combustion, and the receptor profiles were the prescribed burn NMR spectra. We previously showed that the profiles of minerals (Al, Si, Fe, K, Ca and Ti) in Rx#1, Rx#2 and Rx#3 were identical to those observed for the aerosolized soil dust (Kavouras et al., 2012). In addition, the relative concentration distribution of OC fractions in the Rx#1 was similar to mineral soil. These similarities indicated that soil minerals and organic matter may be directly resuspended during prescribed fires, most likely by the turbulence generated close to the fire front (Clements et al., 2008).

Table 2 shows the percentage contributions of soil organic matter and biomass combustions on prescribed burn emissions. High R^2 (0.87–0.93) values indicated that soil organic matter and biomass combustion accounted for most of the variation in prescribed burn emissions. For Rx#1, soil organic matter and biomass combustion accounted for 41 ± 5% and 51 ± 8% of the H atoms, respectively (unexplained: 8 ± 9%). The influence of soil organic matter dropped to 24 ± 8% for Rx#2 and 29 ± 7% for Rx#3, while biomass combustion increased to 71 ± 4% and 74 ± 5%, respectively. Assuming the H/C ratio of 0.18 for soil organic matter and 0.39 for biomass combustion (Table 1), soil organic matter contributed 27%, 14% and 15% of WSOC in Rx#1, Rx#2 and Rx#3, respectively.

The associations of NMR spectra of soil organic matter and prescribed burn emissions provide, for the first time, evidence and quantitative estimates of the release of soil organic compounds into the smoke plume especially in the early phases of the burn. The estimated biomass burning emissions for POC in North Africa, South Africa, northern Asia and Australia were 10.064, 12.155, 10.999 and 3.2538 Tg/yr (with a global enrichment factor of 3.4). Assuming the 50% of emitted OC is water soluble (Timonen et al., 2008) and the estimates of this study represent the lower and upper limits of soil organic matter in biomass emissions (14 and 27%, respectively), then aerosolized soil organic matter in fires may account for 1.35, 1.64, 1.48 and 0.43 Tg/year (Kaiser et al., 2012). These emissions add up to 13% in North Africa, 35% in Asia and 100% in Australia of resuspended soil organic matter by wind erosion (assuming 1% of soil organic matter in savannah and desert soils) (Ginoux et al., 2001). In prescribed burns, wind speed does not exceed 10 m/s reducing the potential for wind erosion of soil drastically. As a result, we only examine the possible effect of the dynamics developed at and ahead of the fire. In fact, the magnitude of soil organic matter emissions during wildfires may be significant in arid regions (i.e. African savannahs and deserts in Australia, Asia) because of dry fuel loads and increased soil erosion. The latter is induced by strong winds that are often a key ingredient to large wildfires.

Table 2
Predicted H- and C-content. Percentage contribution (mean ± standard error) of soil organic matter and biomass combustion using the Chemical Mass Balance (CMB) model for H-atoms; and Water Soluble Organic Carbon (WSOC) assuming molar H/C ratios of 0.18 for soil organic matter and 0.39 for biomass combustion (from Table 1).

	Soil organic matter	Biomass combustion	Unexplained
H-atoms			
Rx#1	41 ± 5	51 ± 8	8 ± 9
Rx#2	24 ± 8	71 ± 4	5 ± 9
Rx#3	29 ± 7	74 ± 5	−3 ± 8
WSOC			
Rx#1	27	73	
Rx#2	14	86	
Rx#3	15	85	

4. Conclusions

We characterized the WSOC fraction of resuspended soil particle, vegetation combustion and prescribed burn emissions by NMR. The soil organic matter was dominated by strong signals in the saturated aliphatic (45.7%) and carbohydrate (23.4%) ranges. Formate and acetate were also present. The NMR spectra of vegetation combustion was characterized by strong convulated signals in the carbohydrate (56.3) and unsaturated aliphatic (20.7) ranges. Levoglucosan was also detected. The molar H/C ratios for soil and vegetation combustion WSOC were 0.18 and 0.39, respectively. The NMR spectra of aerosol samples collected during three phases of a prescribed burn showed strong contributions of aliphatic and carbohydrates H-atoms with molar H/C ratios from 0.64 to 0.68. Resemblances between the soil organic matter and Rx#1, and vegetation combustion and Rx#2 and Rx#3 were observed using the similarity index.

Taking into account that soil organic matter in prescribed fire emissions may be due to turbulence-induced resuspension of soil particles in front of the fire front, we quantified the contributions of soil and vegetation combustion on prescribed burn WSOC using the CMB model. As anticipated, organic matter associated with vegetation combustion dominated fire emissions; however, soil organic matter accounted up to 27% of WSOC in the early stages of the prescribed burn (Rx#1) and 14–15% in Rx#2 and Rx#3. These findings indicated that soil organic matter, composed mostly of humic and fulvic acids, may be present in biomass burning emissions.

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