

# Characterization and spatial distribution of particulate and soluble carbon and nitrogen from wildfire-impacted sediments

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## Abstract

**Purpose** The heavily forested Cache la Poudre (CLP) watershed in northern Colorado, USA, was impacted by the High Park wildfire in 2012. The wildfire burned land and vegetation immediately adjacent to the CLP River where blackened, ashy sediment samples were collected from five sites upstream of the City of Fort Collins drinking water intake to evaluate the spatial distribution and characteristics of burned sediments, along with quantifying and characterizing soluble compounds following a leaching experiment.

**Materials and methods** At each site, samples were collected from three locations: (1) the edge of the bank adjacent to the water edge (downbank), (2) 1 m upslope of location

1 (midbank), and (3) 2 m upslope of location 1 (upperbank). All solid sediment samples were analyzed for elemental composition, and a subset of solid sediment samples were analyzed with <sup>13</sup>C solid-state nuclear magnetic resonance spectroscopy. Sediments were mixed with the background CLP River water collected from upstream of the wildfire and allowed to leach for 6 and 24 h to determine the quantity and quality of water-soluble constituents. Filtered samples were analyzed for dissolved organic carbon (DOC), iron, manganese, and inorganic nutrient concentrations, by optical properties, and for disinfection byproduct (DBP) formation.

**Results and discussion** Percent carbon and nitrogen content of the solid sediments were good predictors of leachate DOC concentration. The mean fluorescence index was higher for wildfire-impacted sediment leachates (1.50) compared to the background CLP River water (1.37), which may be due to changes in DOM molecular weight and oxidation of organic matter. All sediment leachates showed consistently higher haloacetonitrile and chloropicrin yields (DBP concentration/DOC concentration) compared to background CLP River water, whereas carbonaceous DBPs did not.

**Conclusions** The collected sediments showed that burned material accumulated downstream near the river and was composed of inputs from burned soil and biomass along with the mobilization of unburned terrestrial material. The leachates of these sediments have different characteristics compared to the background CLP River water, indicating that DOM leached from sediments following a wildfire may increase aquatic DOC concentrations and N-DBP formation.

**Keywords** DBP · Disinfection byproduct formation · Dissolved organic matter · DOM · Fluorescence · NMR · Sediments

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## 1 Introduction

Wildfire frequency has been increasing over the last several decades due to changes in seasonal precipitation patterns, climate, droughts, and forest disease outbreaks (Brown et al. 2004; Westerling et al. 2006). Many communities rely upon forested watersheds to provide high-quality source water supplies, which may be imperiled following a wildfire. In addition to impacting water quality, wildfires have been reported to change soil characteristics as a result of heating and combustion processes (Bodi et al. 2014). These changes to soil properties include water repellency (DeBano 2000; Specht and Frimmel 2000), nutrient content (Prieto-Fernandez et al. 1998; Prieto-Fernandez et al. 2004; Homann et al. 2011), and organic carbon content (Knicker 2007; Homann et al. 2011). Depending on heating temperatures, the effects of thermal alteration to soil samples can vary with changes to both physical properties and organic matter characteristics (Araya et al. 2016a, b). In a field study of prescribed burns and wildfires in pine forests in Oregon, wildfire severity was positively related to C and N losses in soil as a result of consumption of the organic soil horizon immediately following high severity fire conditions (Homann et al. 2011). However, Homann et al. (2011) also reported C and N deposition onto the soil surface during the year following the wildfire primarily from heat-killing needles from the overstory. This freshly deposited material was reported to have much lower C/N ratios than typical litterfall, potentially having implications for the recovery of a soil microbial community and vegetation (Homann et al. 2011). In addition to altering the concentration of C and N in soils, wildfires have been reported to alter the speciation of C, N, and P (reviewed in Knicker 2007).

Soil properties are known to impact dissolved organic carbon (DOC) concentration and character in nearby water bodies (Sanderman et al. 2009; Dick et al. 2015). Therefore, wildfire-induced changes to soils are likely involved in the water quality impacts observed in streams following wildfires. Soils mobilized by surface runoff and entrained in flowing water are referred to as sediments, which can settle out along the banks of streams, in slow-moving pools, or in lakes. For a watershed impacted by a prescribed burn, the eroded quantity (tons per hectare) of the sediment increased by one to three orders of magnitude for 2 years following the burn and up to 3 years for some areas of the watershed (Shakesby et al. 2015). Rainstorm events may be especially important for the transport of terrestrial material and soil organic matter to aquatic systems (Clark et al. 2007; Inamdar et al. 2011, 2012). Rainstorms following a wildfire in the Rocky Mountains mobilized more DOCs in a burned watershed than in an unburned reference watershed, but concomitant increases in DOC were not reported for high stream flows during spring snowmelt (Murphy et al. 2012; Writer et al. 2012).

The increased quantity of suspended sediments in streams following wildfires, especially during precipitation events, likely facilitates the dissolution of sediment-derived organic matter following wildfires and prescribed burns (Murphy et al. 2012; Writer et al. 2012; Vieira et al. 2015). In addition to hydrologic factors, soil and sediment characteristics may influence the amount and type of DOC mobilized. The C/N ratio of soils has been reported to explain most of the variability in riverine DOC flux for several biomes with increasing soil C/N ratio correlating with increasing riverine DOC flux (Aitkenhead and McDowell 2000). To date, this relationship has not been confirmed for wildfire-impacted watersheds with altered C/N ratios. However, for sediments collected after a prescribed burn, increases in N content and organic content were reported, suggesting that there may be increased mobilization of N-containing organic compounds following burning (Shakesby et al. 2015).

Wildfires are known to directly alter watershed-scale geochemical cycling and sediment transport (Williams and Melack 1997), often causing increases in nitrate and phosphate export, changes in stream water DOC concentrations, and mobilization of metals (Gresswell 1999; Writer et al. 2012), which are presumably derived from burned sediments (Doerr and Santin 2016; Santin and Doerr 2016). Wildfire effects on DOC concentrations in simulated laboratory studies and from field studies are mixed, likely due to differences in the precursor material burned in natural settings versus in laboratory settings. Revchuk and Suffet (2014) reported evidence for increased DOC export following a wildfire based on laboratory leaching experiments of recently charred material and 2-year-old aged char collected from field locations. However, following a fire in Glacier National Park, DOC concentrations in a stream draining a burned area showed “minimal” increase in DOC relative to an unburned area, indicating that changes in DOC concentration may vary depending on the wildfire and watershed (Mast and Clow 2008). In addition to altering ecologically relevant water quality parameters, e.g., nutrients and turbidity, wildfires have been reported to influence the formation of disinfection byproducts (DBPs) (Wang et al. 2015; Hohner et al. 2016), which form when natural waters are disinfected prior to distribution as finished drinking water (Liang and Singer 2003; Krasner et al. 2006; Hua and Reckhow 2007). DOC quantity and quality can have a significant impact on the concentrations of DBPs, which are regulated by the US Environmental Protection Agency, thus making control of their formation a priority for drinking water utilities (Singer et al. 1995).

To help solve this apparent disconnect between DOC concentrations from field and laboratory studies and to better understand how wildfire changes to water quality will affect the drinking water treatment processes, such as the formation of DBPs, it would be useful to conduct both field and laboratory studies on the same wildfire to link the two conditions. In

this study, riverbank sediments were collected from a wildfire-affected watershed and leached in river water, to mimic water quality responses to wildfires. The aim was to evaluate the sediment characteristics along the lateral and longitudinal length of a reach of wildfire-impacted river and potential changes in water quality and DBP formation following a wildfire. Sediment leachates were filtered and characterized for general water quality parameters including DOC concentration, nutrients, and optical properties. In addition, a subset of the leachates was chlorinated at bench scale and analyzed for DBP formation. The characterization of the leachates represents an integrated signal from wildfire-induced chemical changes to burned soil, litter, and biomass, along with physical changes that likely resulted in the mobilization of an unburned terrestrial material to the river. Studying the solid material and the water-soluble material together provides a more comprehensive picture of the potential impacts of wildfires on drinking water sources and may be useful for understanding and predicting watershed recovery.

## 2 Materials and methods

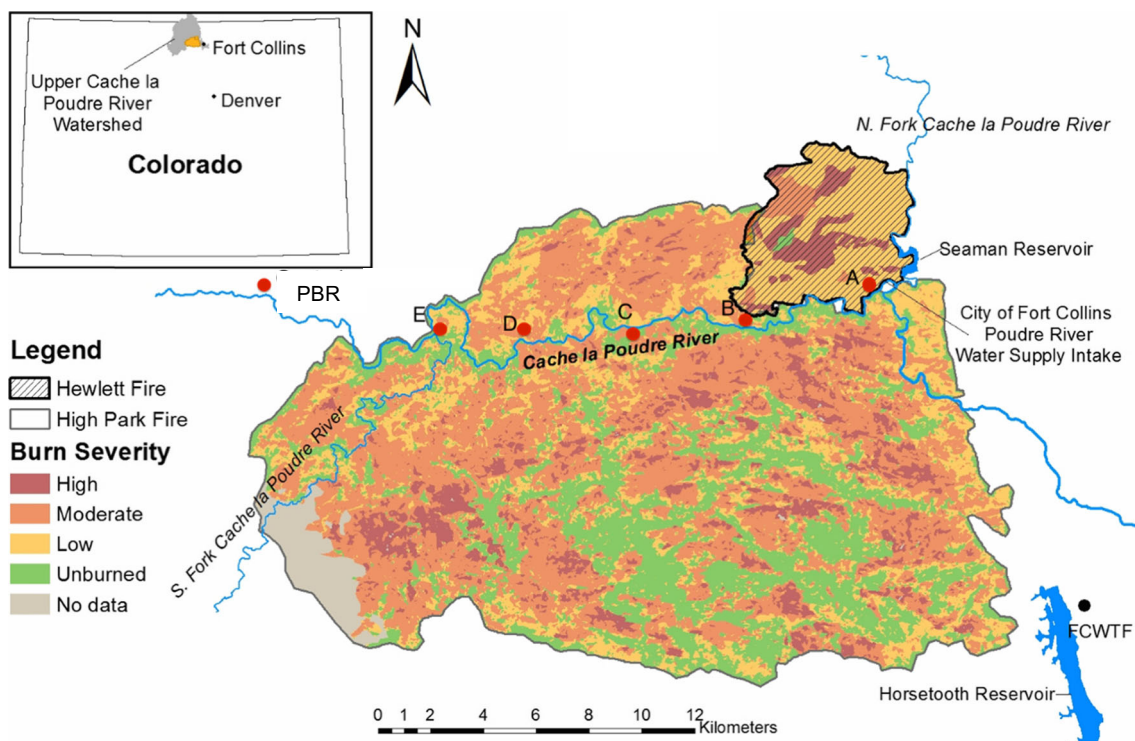
### 2.1 Sediment collection following the High Park fire

The High Park fire began on June 9, 2012, and was fully contained on July 2, 2012. The wildfire severity varied spatially from low to high (Fig. 1). Four rainstorms took place

between June and October 2012: June 27, July 6, July 19, and July 25, 2012. Rainstorms were evidenced by changes in stream discharge from less than 10 cfs to greater than 700 cfs, and likely deposited wildfire-impacted soils and sediments along the banks of the Cache la Poudre (CLP) River.

Sediments were collected on October 31, 2012, from five sites along the CLP River (Fig. 1) within the burned area. The sediments were likely transported downstream after being entrained in the river before being deposited along the bank, possibly resulting in “pre-leaching” of highly soluble compounds prior to their collection. The most downstream sampling location, A, is immediately upstream of the City of Fort Collins drinking water utility intake. At each site (A–E; Fig. 1), sediments were collected from three riverbank locations (upperbank, midbank, and downbank) perpendicular to the stream bank spaced approximately 1 m apart. The “downbank” samples were collected at the edge of the river and the “upperbank” samples were collected furthest from the river.

The accumulation of black, charred sediment deposits along the riverbank was visibly distinct (i.e., loose black fines) from the native soil profile (i.e., fairly compact mineral soil). The sediment deposits consisted of a well-mixed layer of dark material with a discernible smoky odor, which was not present in upstream, unburned areas. The sediments were collected in bulk, ranging from 6 in.–2 ft deep. The samples contained a mixture of loose materials, including ash, charred organic material, and sediments, which were likely to be easily resuspended during a rise in river elevation during higher flow



**Fig. 1** Sediment collection sites (A–E) are shown along the CLP River within the High Park fire burn area. CLP River water for the leaching experiments was collected from the PBR site upstream of the burned area

events. An unburned soil sample was not collected for comparison, as the native riverbank soil from an upstream site would not represent the same precursor starting material of the organic-rich sediment deposits collected from the riverbank in the wildfire-impacted area. Rather, the sediment leachates are compared to baseline water quality and reactivity from a water sample collected upstream of the wildfire-impacted area of the watershed.

## 2.2 Laboratory sediment leaching

The sediment samples were dried first at room temperature (~3–4 days) and then in a furnace at 105 °C for 2 h. The sediments were sieved through a 0.991-mm sieve, which represents fine silt and clays in the American Society for Testing Methods (ASTM) classification, and kept in the freezer until the leaching experiments were performed. For each riverbank location (upperbank, midbank, downbank), 200 g of sediment was placed in a 2-L Pyrex glass beaker and mixed with the CLP River water collected from a site upstream of the wildfire (Poudre Below Rustic (PBR); Fig. 1) for 2 min with a glass rod. The water used for leaching was a portion of a large (55 gal) sample collected in October 2013 from upstream of the wildfire that was stored in a large walk-in cooler. Thus, the water used to leach the sediments had the same characteristics and differences in the water chemistry of the leachates can be attributed to differences in sediment characteristics rather than differences in water quality prior to leaching. The samples were kept in the dark at room temperature for the allocated leaching duration (6 and 24 h). Leaching times represent high river flow (6-h leaching duration) and low river flow (24-h leaching duration). At the end of the leaching periods, the samples were decanted without disturbing the settled sediment, filtered (0.70 µm GF/F), and stored at 4 °C.

## 2.3 Analytical methodology

### 2.3.1 Solids analysis

Total carbon and nitrogen of the sediments were determined on a LECO Tru-Spec CN analyzer (Leco Corp., St. Joseph, MI, USA). Measurements were calibrated using a standard soil sample. Five sediment samples with high carbon concentrations were analyzed using  $^{13}\text{C}$  NMR spectroscopy. It was not possible to collect spectra for the samples with lower carbon concentrations. All samples were ground prior to NMR analysis. A 300-MHz Varian instrument with an operating  $^{13}\text{C}$  resonance of 75.4 MHz and  $^1\text{H}$  resonance of 299.9 MHz equipped with a 7.5 mm HX probe was used for all analyses housed at the Environmental Molecular Science Laboratory (EMSL) at the Pacific Northwest National Laboratory (Richland, WA). Samples were packed in a zirconia rotor and analyzed using a variable amplitude cross-polarization

magic angle spinning coupled with total suppression of spinning sidebands (CPMAS/TOSS) pulse sequence with a spinning speed of 5 kHz. The pulse sequence begins with a hard  $^1\text{H}$  radiofrequency pulse followed by a variable amplitude CP pulse of 1 ms and the four pulse-toss sequence. The power for these toss pulses was optimized to minimize the sidebands. A 62.5-kHz  $^1\text{H}$  TPPM decoupling field was applied during the toss sequence with a 10-ms acquisition time. The number of scans was between 30,000 and 100,000 and varied according to the amount of C present in the sample. Recycle delay between acquisitions varied between 1 and 2 s and was optimized for each sample by analyzing each sample's proton signal. The data were processed with zero filling to 32 k and 200 Hz line broadening. The carbon chemical shift was calibrated using hexamethylbenzene as the external standard. Chemical shift regions were integrated according to broad functional group assignments: 0–25 ppm for terminal methyl, 25–45 ppm for methylene in rings/chains, 45–60 ppm for *O*-alkyl/*N*-alkyl, 60–90 ppm for carbohydrate/ether/alcohol, 90–160 ppm for aromatic/alkenes, and 160–190 ppm, for carboxyl/carbonyl/amide groups.

### 2.3.2 CLP River monitoring sample collection

As part of a previous study, surface water samples were collected biweekly through the first spring snowmelt (2013) following the High Park fire, including a site upstream of the burned area (PBR) (Fig. 1). CLP River water for the leaching experiments was also collected from the PBR sampling site. Starting in August, the samples were collected monthly through October. Samples were filtered through pre-combusted GF/F glass fiber filters and were stored at 4 °C until analysis, as described in Sect. 2.2.

### 2.3.3 CLP River monitoring and leachate sample analysis

DOC and total dissolved nitrogen (TDN) were measured with a TOC- $V_{\text{SCH}}$  (Shimadzu Corp., Japan) analyzer.  $UV_{254}$  was measured using a UV-Vis spectrophotometer (Cary 100, Agilent Technologies) with a 1-cm path length quartz cuvette. Specific ultraviolet absorption at 254 nm ( $SUVA_{254}$ ) was determined by dividing  $UV_{254}$  absorbance by the DOC concentration (Weishaar et al. 2003). Total dissolved phosphorous (TDP) analysis followed Lachat QuikChem 8500 spectrophotometric flow injection methods. Nitrate and nitrite analyses were done with an analytical flow solution IV spectrophotometric analyzer (OI Analytical, USA). Ammonium was measured using a BioTek Synergy 2 Microplate Reader. Dissolved organic nitrogen (DON) was determined by the difference between TDN and the sum of all inorganic nitrogen species (nitrate, nitrite, and ammonium). Fe and Mn concentrations were measured at the University of Colorado Laboratory for Environmental and Geological Studies (LEGS) using ICP-



AES. The samples for the metal analysis were made by mixing 1.5 g of solid material with 100 mL of CLP River water and filtering after 24 h.

Size distribution of the molecules in the solution was determined using a high-pressure liquid chromatograph equipped with a protein-pak column (Waters), UV detector (280 nm), and polystyrene sulfonate standards (210, 1000, 4300, 6800, and 17,000 Da). A phosphate buffer solution (pH = 6.8) was used as the mobile phase at a flow rate of 0.7 mL/min. Larger molecular weight compounds travel through the column more quickly than smaller molecular weight compounds due to their exclusion from small pores within the column. Therefore, retention time is negatively related to the log of the molecular weight of the molecules in solution.

Fluorescence excitation emission matrix (EEM) plots were recorded using a John Yvon Horiba FluoroMax-4 spectrofluorometer over excitation wavelengths of 240 to 450 nm in 10-nm increments, and fluorescence intensity was measured at emission wavelengths from 300 to 600 nm in 2-nm increments. Slit widths of 5 nm and integration times of 0.25 s were used for both excitation and emission wavelengths. The signal was collected in  $S_c/R_c$  mode to incorporate instrument-specific correction factors. EEMs were corrected for primary and secondary inner filter effects using the corresponding UV-Vis absorbance spectrum. EEMs were blank subtracted and Raman-normalized based on the Raman peak area for 18.2 M $\Omega$  MilliQ water collected at an excitation wavelength of 350 nm. Corrected EEMs are presented in Raman units (RU). Lamp scans and cuvette contamination checks were performed daily. EEMs were corrected and analyzed using MATLAB software. The fluorescence index (FI) is reported as the ratio of the emission at 470 nm to that at 520 nm with an excitation at 370 nm. The expected range for the FI lies between 1.2 and 1.9. Lower values are associated with DOM from terrestrial origin, and higher values are associated with DOM from microbial sources (McKnight et al. 2001). More recently, the FI has been shown to relate to shifting of the peak location, which may indicate other factors than DOM precursor material influencing the FI value (Korak et al. 2014).

Eight leachates were selected for bench-scale chlorination tests and DBP analysis based on water quality and DOM properties (e.g., DOC, SUVA<sub>254</sub>; Table S1, Electronic Supplementary Material). Chlorination methods followed uniform formation conditions (UFCs) (Summers et al. 1996), which mimic typical chlorination conditions in US drinking water treatment plants. Samples were chlorinated at room temperature with a buffered sodium hypochlorite dosing solution (pH 8) at 20 °C. A borate buffer was added to the samples to maintain a pH of 8 ( $\pm 0.3$ ). Chlorine doses were selected from a preliminary demand test to target a chlorine residual of 1.0 mg/L ( $\pm 0.4$  mg/L). Chlorinated samples were incubated in the dark for 24 h ( $\pm 1$  h) at room temperature (20  $\pm$  1.0 °C).

Chlorine residuals were measured with the *N,N*-diethylphenylenediamine (DPD) colorimetric method (SM4500-Cl G) and quenched with ammonium chloride immediately following 24 h prior to DBP analysis.

Chlorinated samples were analyzed for DBPs following EPA Method 551.1 (1995) for total trihalomethanes (TTHM) and haloacetonitriles (HAN). EPA method 552.2 was followed for analysis of haloacetic acids (HAAs). An Agilent 6890 Gas Chromatography System with an electron capture detector was used. Statistical analyses, including pairwise comparisons of DBP yields and DOM quality, were performed in JMP Pro 11.0 software using a threshold of 0.05 for *p* values. TTHM species include chloroform, bromodichloromethane (DCBM), chlorodibromomethane (CDBM), and bromoform. HAN<sub>4</sub> species include dichloroacetonitrile (DCAN), trichloroacetonitrile (TCAN), dibromoacetonitrile (DBAN), and bromochloroacetonitrile (BCAN). HAA species include chloroacetic acid (MCAA), bromoacetic acid (MBAA), dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), and dibromoacetic acid (DBAA).

Burned watershed areas for each classified wildfire severity (low, moderate, and high) draining to each sediment collection site were determined using the ArcMap 10.3 software. A 10-m resolution digital elevation map (DEM) of Larimer County, CO, a shapefile containing the Burned Area Emergency Response (BAER) wildfire severity for the High Park fire, and GPS coordinates from a Garmin handheld GPS unit were used for the GIS analysis. Briefly, the CLP watershed boundary and stream network were determined using a flow accumulation method and the watershed areas draining to each sediment sampling location were calculated using the watershed tool and geometry calculator.

## 3 Results and discussion

### 3.1 Sediment site burn severities and drainages

The percent drainage area burned (all severities) decreased from downstream, site A, to upstream, site E. At site A (Fig. 1), 1.7, 7.3, and 3.6% of the watershed area was burned at high, moderate, and low severity, respectively (87.4% unburned). Similarly, at site B, 1.7, 7.2, and 3.5% of the watershed area was burned at high, moderate, and low severity, respectively (87.6% unburned). Site C had a lower percent burned area with 0.95, 4.3, and 2.3% of the drainage burned at high, moderate, and low severity, respectively (92.5% unburned). At site D, 0.74, 2.9, and 1.4% of the watershed area was burned at high, moderate, and low severity, respectively (94.9% unburned), and at site E, 0.63, 2.4, and 1.1% of the watershed area was burned at high, moderate, and low severity, respectively (95.9% unburned). Overall, the total area draining the sediment collection sites was primarily unburned. However,

the sediments along the riverbank had clear indications of being wildfire impacted, such as a dark black color and smoky smell. These evident impacts are likely due the proximity of the fire to river and the increased erosion potential of burned land (Moody and Martin 2001; Writer et al. 2012).

### 3.2 Solid-state characterization

Elemental analysis of the sediments showed that the downbank locations (nearest stream) had the lowest percent C and N, whereas the midbank locations generally had the highest percent carbon (C) (Table 1). Erosion at the downbank location may have washed high carbon sediments downstream, contact with flowing water may have “pre-leached” soluble carbon compounds, or initially, the sediments had lower carbon content. The midbank site would have had less contact with water to erode or leach sediments. There was likely limited deposition of wildfire-impacted sediments at the upperbank location because it was not reached by the river water level following deposition. There was also a noticeably higher percent C and N at site A, located furthest downstream and draining the largest burned area which is indicative of wildfire impacts and consistent with the finding that wildfires can increase soil C content, attributed to recalcitrant-burned biomass and re-colonization of burned land with N-fixing vegetation (Raison 1979; Johnson and Curtis 2001; Heath et al. 2015). Site A sediments also had a higher percent Fe compared to all of the other sites. A general longitudinal decreasing trend in percent Fe moving upstream was observed.

Thus, the increased Fe concentrations measured at site A are likely due to heterogeneity in the watershed where geologic minerals may be the source of Fe and the physical changes following the High Park fire that destabilized watershed areas mobilizing Fe-containing minerals from unburned soil (Pereira et al. 2014).

<sup>13</sup>C NMR analysis was performed on the five samples with the highest percent C from the preliminary elemental analysis. The selected samples for the analysis included site A (upperbank, midbank, and downbank), site B (upperbank), and Site C (midbank) (Table 2). Many of the other solid samples had very low C content and obtaining meaningful <sup>13</sup>C NMR spectra were not possible. For these five sites, the spectra were dominated by aromatic C compounds (Table 2, spectra shown in Fig. S1, Electronic Supplementary Material), which could be representative of condensed (160–90 ppm region) combustion products from wildfire, commonly referred to as black carbon (Knicker et al. 2008; Knicker et al. 2012; Merino et al. 2014) and charred material derived from pulp, leaves, and sawdust (Knicker et al. 2008). The presence of these heteroatoms is likely a result of incomplete combustion of precursor soil and biomass material (Knicker et al. 2008). These types of aromatic organic molecules are also found in DBP precursor material (Chang et al. 2001; Yang et al. 2008). The content of carbohydrates and other aliphatic compounds was low for all of the soil samples that were analyzed (Table 2). These compounds are often found in higher abundance in unburned soils and are likely destroyed during wildfires leading to lower abundances detected in this study (VanBergen et al. 1997; De la Rosa et al. 2013).

**Table 1** Elemental composition of solid sediments (%C, %N, %Fe, and C/N) and water quality (WSOC, DON, SUVA<sub>254</sub>, and FI) following extraction (6 h) in the Cache la Poudre (CLP) water collected from a site upstream of the burned area (100 g of sediment per 1 L of CLP water)

Sample name	Solids analysis				Leachate analysis			
	%C	%N	%Fe	C/N	WSOC 6 h (mg <sub>C</sub> /g)	DON (mg <sub>N</sub> /L)	SUVA <sub>254</sub> (L/(mg m))	FI
Site A upperbank	9.3	0.58	2.8	19	0.146	0.66	4.0	1.51
Site A midbank	14.7	0.78	2.7	22	0.142	0.92	4.3	1.51
Site A downbank	7.3	0.43	2.8	20	0.115	0.75	4.0	1.45
Site B upperbank	2.5	0.23	2.2	13	0.073	0.37	4.8	1.56
Site B midbank	2.7	0.18	2.1	17	0.069	0.54	4.7	1.48
Site B downbank	1.7	0.14	2.7	14	0.053	0.52	4.4	1.42
Site C upperbank	1.6	0.14	1.4	14	0.047	0.07	3.9	1.41
Site C midbank	13.4	0.69	2.2	23	0.098	0.44	4.1	1.49
Site C downbank	0.42	0.06	2.3	7.7	0.044	0.22	3.9	1.45
Site D upperbank	1.0	0.10	1.8	13	0.062	0.07	4.5	1.40
Site D midbank	1.5	0.11	1.7	15	0.057	0.25	4.4	1.46
Site D downbank	0.61	0.07	1.5	10	0.056	0.29	3.9	1.45
Site E upperbank	3.4	0.23	1.9	17	0.053	0.13	4.3	1.47
Site E midbank	2.9	0.20	1.9	17	0.058	–	4.2	1.48
Site E downbank	2.8	0.17	1.7	19	0.049	0.09	4.4	1.47

Elemental analysis for solid samples (%C, N, and Fe). Detailed results for all samples, duplicates, and leaching times can be found in Table S3

**Table 2** NMR integration regions for the samples analyzed

Structural group (observing C in each)	Region (ppm)	Site A upperbank	Site A midbank	Site A downbank	Site B upperbank	Site C midbank
Carboxyl/carbonyl/amide	190–160	15.9	17.2	23.4	17.9	11.5
Aromatic, alkene	160–90	53.2	52.0	64.1	59.4	52.7
Carbohydrate, ether, alcohol	90–60	9.7	10.0	1.5	8.1	9.3
O-alkyl, N-alkyl	60–45	3.9	4.2	1.0	8.0	5.1
Methylene in rings/chains	45–25	9.3	10.0	2.9	5.1	10.5
Terminal methyl	25–0	8.8	7.3	6.8	1.9	10.9

Five sediments with the highest carbon content were analyzed. Site A is the most downstream site with B and C further upstream

For site A, an NMR spectrum was obtained for all three riverbank locations due to the high percent C, facilitating a discussion regarding the possible mobilization distance/washing effect of fire-impacted soil organic matter (SOM) along the banks of the river. The total contribution of aromatic structures to the spectrum was highest (64.1%) for the downbank location compared to the midbank (52.0%) and the upperbank (53.2%) locations, indicating that a greater amount of fire-impacted SOM was deposited closest to the river, and was spread approximately evenly 1 (midbank) and 2 m (upperbank) from the river. In contrast, an opposite relationship, higher relative abundance for the upperbank and midbank locations, was determined for the signal derived from carbohydrates in hexose, some alcohols, and amino acids (90–60 ppm). Differences in the signals between bank locations reflect the impact of distance from the river upon SOM composition with the downbank location showing more recalcitrant, less soluble carbon structures consistent with wildfire impacts. Comparing site A midbank to site C midbank, we observe very similar contributions from each structural entity. However, site A and B upperbank SOM compositions were more different and reasons for this remain unclear. Heterogeneity in SOM source material or variability in accumulation and erosion may explain these differences. Compared to a  $^{13}\text{C}$  NMR study of whole soil and extracts collected from the Colorado montane environment, the aromatic carbon content of the High Park fire sediments (upperbank, midbank, and downbank) was higher than for soils that have not been affected by a wildfire recently (Keeler et al. 2006).

### 3.3 Concentration and character of water-soluble constituents

Leaching experiments were performed by mixing the sediments with CLP River water collected from the PBR site, upstream of the burned area. Sediments were leached for 6 or 24 h, and then filtered for relevant source water quality analyses. Dissolved metals, among other solutes, can be a concern for drinking water utilities, and elevated levels have been observed in stream water, soils, and vegetation following

wildfires (Costa et al. 2014; Mitic et al. 2015). The concentrations of water-extractable Mn were in general highest for the downbank samples compared to the midbank and upperbank samples collected from the same site (Table S2, Electronic Supplementary Material). There was also an increase in the average Mn concentrations of the leachates with distance downstream. Fe concentrations were higher at site B compared to all of the other sites except for site A, which also had high Fe concentrations compared to the more upstream sites (Table S2, Electronic Supplementary Material). The mobilization of metals following wildfire was hypothesized to be from entrained sediment (Costa et al. 2014), and the leaching study here supports that conclusion. Thus, efforts to prevent excessive erosion of soils into surface waters following a wildfire may help to avoid elevated concentrations of metals.

Nutrient levels in surface waters may enhance algal growth and contribute to the pool of DBP precursors. CLP River water samples for the year following the wildfire showed elevated levels of nutrients at a wildfire-impacted site relative to a site upstream of the burned area (Hohner et al. 2016). It was hypothesized that the increases in surface water nutrient concentrations resulted from the leaching of eroded, burned material. Using the leaching studies to test this hypothesis, no discernable spatial trends were found for total dissolved phosphorous (TDP) related to the upstream/downstream sediment sites (Table S3, Electronic Supplementary Material), with values that varied from one another, but were quite patchy. However, laterally from the river TDP was the lowest for the downbank locations, while the upperbank and midbank locations did not show a trend, with values that varied between sites and were higher than the downbank values. At sites A and C, the midbank location had the highest TDP concentrations, whereas at sites B and E, the upperbank location had higher TDP concentrations compared to the other locations. This finding is somewhat unexpected, as water quality studies (Murphy et al. 2006; Miller et al. 2013) and soil studies (Norouzi and Ramezani 2013; Xue et al. 2014) have both reported increased concentrations of phosphorus in wildfire-impacted areas. However, a study of the effects of fire severity on ash chemical properties reported a decrease in TP with increasing fire severity (as estimated using ash color)

indicating that fire severity may play an important role in soil chemical properties and water-extractable constituent concentrations (Pereira et al. 2012). Based on the BAER severity map for the High Park fire (Fig. 1), the fire impacts were very patchy in severity throughout the watershed, which may have led to the variability in the TDP concentrations leached from sediment samples collected from different areas. It is also possible that there were wildfire impacts on phosphorus (P) concentrations that were not captured in this study because in the short time between the High Park fire and collection of the sediments, P was abiotically removed (e.g., leached by stream water or sorbed to sediments) or biologically assimilated, and wildfire impacts on P were short lived in this system (Son et al. 2015).

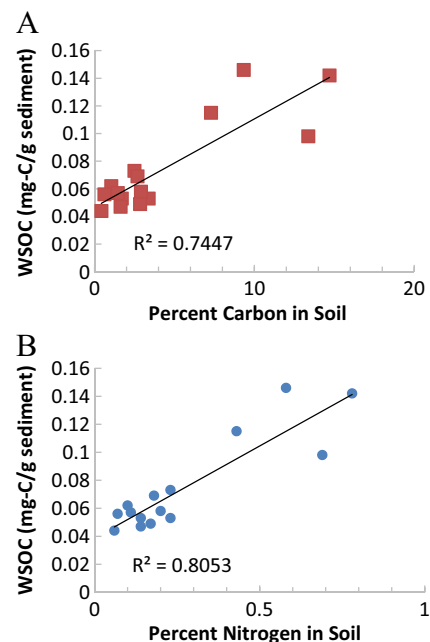
Similar to TDP, the leachates showed no consistent TDN trend from the downstream to upstream sediment collection sites (Table S3, Electronic Supplementary Material). Site E had higher  $\text{NO}_2^- + \text{NO}_3^-$  than all other sites, except for site B. It is possible that forest ecology may play an important role in stream water nutrient concentrations for the area (Rhoades et al. 2013).  $\text{NH}_4^+$  concentrations were higher for site A leachates, the most downstream site, than for any of the other sites (Table S3, Electronic Supplementary Material). Site B had the lowest  $\text{NH}_4^+$  concentration. Similarly, the DON values at site A were higher than any of the other sites. Lee and Westerhoff (2005) reported that when DON is less than 40% of the TDN, calculating by difference can lead to inaccurate results when compared to samples pretreated with dialysis. For most of the leachates, DON was less than 40% of TDN making the calculation of DON potentially erroneous. The form of nitrogen found in the heated material has been shown in laboratory experiments to depend on heating duration (Knicker et al. 1996). Thus, water-soluble N content may be driven by wildfire intensity and/or duration, which is unknown for the environmental samples. However, there was a general increasing trend in DON and  $\text{NH}_4^+$  concentrations with distance downstream, consistent with wildfire impacts mobilizing organic N and  $\text{NH}_4^+$  that have been reported for soils following wildfires (Covington et al. 1991; Certini 2005).

### 3.4 Characterization of DOM leached from sediments

Another concern following a wildfire is the potential for increased mobilization of terrestrial DOM, resulting in higher DOC concentrations and DBP formation during drinking water treatment. Water-soluble organic carbon (WSOC) concentrations were calculated as the mass of carbon released into a solution divided by the mass of the sediments leached (Table 1). The yields of organic carbon in the solution per unit sediment after 6 h were the greatest for site A (mean 0.134 mg-C/g-sediment) with a decreasing trend upstream from site B (mean 0.065 mg-C/g-sediment) to site E (mean 0.053 mg-C/g-sediment). The spatial trend is likely influenced

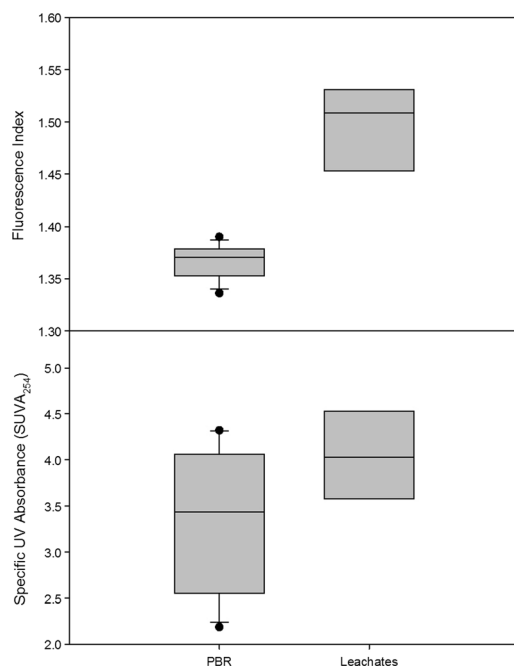
by a combination of hydrologic transport of wildfire-impacted sediments to the downstream sites, and the higher percentage area burned draining to sites A and B. Without a time-series dataset, the influence of hydrology and spatial distribution of wildfire severity cannot be decoupled. In addition to the higher percentages of C and N in the solid material, sites A and B leached more carbon into the solution when mixed with river water. The WSOC concentration showed a strong positive relationship with the percent nitrogen and carbon in the sediment samples as determined by elemental analysis (Fig. 2). The higher %N content of the solid sediments is characteristic of wildfire impacts and may enhance the solubility of carbon containing compounds, specifically aromatic ones that may result from burned biomass (Knicker et al. 2005; Knicker 2007). It is also possible that the wildfire resulted in the addition of carbon and nitrogen from partially combusted biomass to the soils, with a fraction soluble in the form of WSOC (Johnson and Curtis 2001).

The average  $\text{SUVA}_{254}$  values of the leachates showed an increasing trend with increasing lateral distance from the river, with the less wildfire-impacted upperbank locations having higher  $\text{SUVA}_{254}$  values than the midbank or downbank locations (Table S3, Electronic Supplementary Material). A longitudinal trend was also observed, with the downstream site A leachates having the lowest  $\text{SUVA}_{254}$  values (mean  $\text{SUVA}_{254}$  3.8) and site B and D leachates (mean  $\text{SUVA}_{254}$  4.4 for both) having the highest average  $\text{SUVA}_{254}$  values. These trends do not correlate with the elemental analysis of the solid samples, indicating that the C or N content did not simply drive the aromaticity of the leachates. Higher  $\text{SUVA}_{254}$  values



**Fig. 2** Water-soluble organic carbon (WSOC) concentrations show an increasing trend with the percent carbon (*panel A*) and nitrogen (*panel B*) in the solid sediment samples





**Fig. 3** Box plots of optical properties including FI and  $SUVA_{254}$  values for the CLP River water (PBR site) and sediment leachates. The centerline of the box represents the mean with the top and bottom representing the upper and lower quartiles. For the CLP River water, 95% confidence intervals are represented by the whiskers and the dots are the highest and lowest values

following a wildfire were observed in a previous study (Writer et al. 2012); however, it is unclear whether the increase is attributed to enhanced erosion of unburned soil following the wildfire or due specifically to the mobilization of burned soil and biomass material. When the sediment leachates are compared to the upstream CLP River monitoring site, PBR, clearly higher  $SUVA_{254}$  values were observed for the leachates (Fig. 3). Based on the leaching experiments, it appears that the erosion and leaching of unburned terrestrial material may contribute to some of the changes to water quality in combination with burned material following a wildfire.

The FI is another optical parameter used to understand the DOM precursor material and chemical characteristics

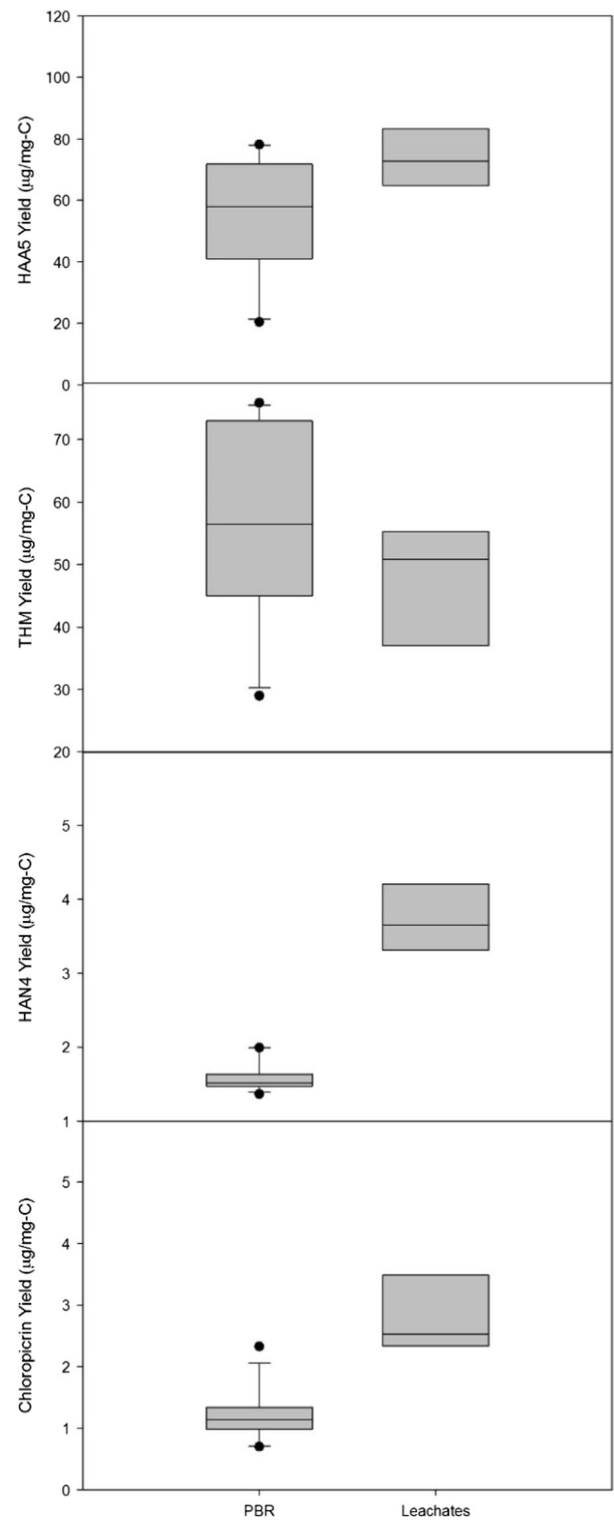
(McKnight et al. 2001; Korak et al. 2014). The FI values were not substantially different between the 6- and 24-h leachates (Table S3, EEMs of site A are presented in Fig. S3, Electronic Supplementary Material). The data obtained for different sediment sites along the river and at different lateral riverbank locations did not show clear trends (Table S3, Electronic Supplementary Material). However, the leachates had higher FI values compared to the background CLP River water (Fig. 3). Although the FI values can be skewed by Fe interactions, the metal concentrations measured for the leachates were not high enough to significantly influence the FI values (Poulin et al. 2014). The FI seems to be a particularly sensitive metric for wildfire-impacted DOM, and it is unlikely that the FI strictly represents the microbial to terrestrial continuum described by McKnight et al. (2001), as wildfires are likely not selectively destructing terrestrial material or adding microbial material causing the increase in FI.

Thus, molecular weight changes that shift fluorescence spectral characteristics may partially explain the higher FI associated with the wildfire-impacted sediment leachates. Using preparative scale size exclusion chromatography (SEC), a general trend of increasing FI with decreasing molecular weight was reported for the surface water samples (Romera-Castillo et al. 2014). A blue shift of the fluorescence, caused by increased oxidized functional groups following fire, could also explain the higher FI values (Coble 1996; Korak et al. 2014), which relates well with the primarily aromatic composition of the sediments from the NMR analysis. There were no significant differences in the MW estimates of the five sediment sites despite considerable variability (Table S2, Electronic Supplementary Material). It is possible that the sediments contained larger molecular weight, fire-derived compounds that were not soluble in river water and were thus not captured by the SEC. Wildfire may produce low-solubility, larger molecular weight aromatic organic compounds and/or degrade higher molecular weight compounds to lower molecular weight compounds, water-soluble compounds. To date, there are no other studies of wildfire-impacted sediments leachates that have evaluated DOM molecular weight using SEC.

**Table 3** DBP yields (DBP concentration/DOC concentration) for a subset of the leachates

Sample name	TTHM yield ( $\mu\text{g}/\text{mg}_C$ )	HAA5 yield ( $\mu\text{g}/\text{mg}_C$ )	HAN4 yield ( $\mu\text{g}/\text{mg}_C$ )	Chloropicrin yield ( $\mu\text{g}/\text{mg}_C$ )
Site A upperbank (24 h)	37.5	80.0	3.6	3.7
Site A midbank (24 h)	31.2	54.8	3.7	2.5
Site A downbank (24 h)	36.8	70.0	5.1	4.7
Site B upperbank (24 h)	51.5	71.1	3.5	2.3
Site C midbank (24 h)	51.6	84.4	4.4	3.0
Site C downbank (6 h)	56.5	74.3	3.2	2.0
Site D upperbank (24 h)	76.0	103.8	3.1	2.6
Site E midbank (24 h)	50.1	63.1	3.7	2.4

**Fig. 4** Box plots of the DBP yields for CLP River water (PBR site) and the sediment leachates. The *centerline of the box* represents the mean with the *top* and *bottom* representing the upper and lower quartiles. For the CLP River water, 95% confidence intervals are represented by the *whiskers* and the *dots* are the highest and lowest values



### 3.5 DBP reactivity of wildfire impacted sediment leachates

Eight of the leachates were chlorinated and analyzed for DBP formation based on water quality and DOM properties (e.g., DOC, SUVA<sub>254</sub>; Table S1, Electronic Supplementary Material). DBP yields were calculated (DBP concentration/

DOC concentration) in order to gain an understanding of the reactivity of the precursor material leached from the sediments on a per unit carbon basis. DBP yields for carbonaceous DBPs (C-DBPs; TTHMs and HAA5s) and nitrogenous DBPs (N-DBPs; HAN4 and chloropicrin) are presented in Table 3. Site D showed the highest TTHM and HAA5 yields, appearing to

have the greatest C-DBP reactivity compared to the other sites. The site A downbank leachate (24 h) showed the highest HAN4 and chloropicrin yields overall. Assuming that the sediments from site A were the most wildfire-affected, these findings suggest that the burned material may be more reactive for forming N-DBPs and is enriched in N-DBP precursors compared to carbonaceous precursors, consistent with other work (Wang et al. 2015). The higher nitrogen content of the more downstream, wildfire-impacted sediments, specifically at site A, also supports the higher N-DBP reactivity. The leachates had higher HAA5 yields and slightly lower TTHM yields than the background CLP River water, although they were not substantially different (Fig. 4). In addition, both HAN4 and chloropicrin yields were higher for the leachates compared to the background water (Fig. 4). The mobilization of the N-DBP precursors may be one of the greatest impacts of soluble compounds from the burned sediments compared to the background CLP River water.

#### 4 Conclusions

Overall, this study confirms that water-soluble compounds leached from sediments following a wildfire contributed measured changes to water quality, reported and suggested elsewhere, but not directly compared to surface water quality changes in the same area (Revchuk and Suffet 2014; Wang et al. 2015). In addition, we found evidence that spatial distribution of the sediments may be hydrologically influenced with the more downstream site, which drains a larger burned area, showing greater wildfire impacts in the form of higher C and N content in the sediments. This study also highlights that changes in stream water quality characteristics are likely altered due to a combination of inputs from burned soil and biomass along with the mobilization of unburned terrestrial material that was not heated, but rather had increased mobility following the wildfire. Hydrology and erosion impart different characteristics on sediments and may account for spatial variability in solid sediments and leachate characteristics. However, it is clear that the leachates of these wildfire-impacted sediments have different characteristics compared to the background CLP River water, indicating that utilities may need to alter their treatment processes to handle increased sediment loads, raised nutrient and TOC concentrations, and to control N-DBPs.

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