Dear Antonio Jordán (Topical Editor),

Please find attached: (1) a revised version of the manuscript with marked changes, followed by (2) a report with detailed answers to both reviewers' comments.

Sincerely,

Samuel Araya (on behalf of all authors)

Thermal alteration of soil organic matter properties: a systematic study to infer response of Sierra Nevada climosequence soils to forest fires

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Abstract

Fire is a major driver of soil organic matter (SOM) dynamics, and contemporary global climate change is changing global fire regimes. We conducted laboratory heating experiment of soils from five locations across the western Sierra Nevada climosequence to investigated thermal alteration of SOM properties and determined temperature thresholds for major shifts in SOM properties. Topsoils (0 to 5 cm depth) were exposed to a range of temperatures that are expected during prescribed and wild fires (150, 250, 350, 450, 550 and 650 °C), with increase in temperature, we found that the concentrations of C and N decreased in a similar pattern among all five soils that varied considerably in their original SOM concentrations and mineralogies. Soils were separated into discrete size classes by dry sieving. The C and N concentrations in the larger aggregate size fractions (2-0.25mm) decreased with increase in temperature that at 450 °C temperature, the remaining C and N were almost entirely associated with the smaller aggregate size fractions (<0.25mm). We observed a general trend of ^{13}C enrichment with temperature increase. There was also ^{15}N enrichment with temperature increase followed by ^{15}N depletion when temperature increased beyond 350 °C. For all the measured variables, the largest physical, chemical, elemental, and isotopic changes occurred at the mid-intensity fire temperatures, i.e. 350 and 450 °C. The magnitude of the observed changes in SOM composition and distribution in three aggregate size classes, as well as the temperature thresholds for critical changes in physical and chemical properties of soils (such as specific surface area, pH, cation exchange capacity) suggest that transformation and loss of SOM are the principal responses in heated soils. Findings of this systematic investigation of soil and SOM response to heating are critical for predicting how soils are likely to be affected by future climate and fire regimes.

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Keywords : Thermal alterations, Soil Organic Matter, Fire, Climosequence.

1. Introduction

Fire is a common, widespread phenomenon globally (Bowman et al., 2009) that controls dynamics of soil and soil organic matter (SOM). Vegetation fires burn an estimated 300 to 400 million hectares of land globally every year (FAO, 2005). In the US alone, over 80,000 fires were reported in 2014–including about 63,000 wildland fires, and 17,000 prescribed burns that burned over 1.5 million and 970,000 ha of land, respectively (National Interagency Fire Center, 2015). In the Sierra Nevada, vegetation fires have a major influence on the landscapes, Ecological functions such as plant regeneration, habitat revitalization, biomass accumulation and nutrient cycling, are influenced by fires (McKelvey et al., 1996). Historically most fires were caused by lightning fires and vegetation fires play important role in maintaining the health of many ecosystems around the world (Harrison et al., 2010). In recent decades, anthropogenic activities have become major causes of vegetation fires (Caldararo, 2002), Moreover, climate and climatic variations exert strong control on the distribution, frequency, and severity of fires (Harrison et al., 2010). Significant changes in global fire regimes are anticipated because of climate change including increased frequency of fires in the coming decades (Pechony and Shindell, 2010; Westerling et al., 2006). However, our understanding of how climate change and changes in fire regimes will interact to influence topsoils in fire affected ecosystems is limited.

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In addition to combustion of aboveground biomass and alteration of vegetation dynamics, fires also affect the physical, chemical and biological properties of soils (Certini, 2005; González-Pérez et al., 2004; Mataix-Solera et al., 2011). The degree of alteration caused by fires depends on the fire intensity and duration, which in turn depend on factors such as the amount and type of fuels, properties of above ground biomass, air temperature and humidity, wind, topography, and soil properties such as moisture content, texture and soil organic matter (SOM) content (DeBano et al., 1998). The first-order effects of fire on soil are caused

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by the input of heat causing extreme soil temperatures in topsoil (Badía and Martí, 2003b; Neary et al., 1999) resulting in loss and transformation of SOM, changes in soil hydrophobicity, changes in soil aggregation, loss of soil mass, and addition of charred material and other combustion products (Albalasmeh et al., 2013; Araya et al., 2016; Mataix-Solera et al., 2011; Rein et al., 2008; Santos et al., In press).

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The duration of burning regulates the amount of energy transferred through the soil. Fires with longer residence time and lower temperature typically impact the soil and SOM more than fires with shorter residence time that burn at a higher temperature (Frandsen and Ryan, 1986; González-Pérez et al., 2004). Penetration of heat down a soil profile depends on intensity and duration of fire as well as the thermal conductivity of the soil (Steward et al., 1990). Soil has a low thermal conductivity that in fires only the top few centimeters of soil experiences extreme temperature, For example, in short duration or low severity fires temperatures typically reach only 100 – 150 °C at 5 cm depth with no significant change of temperature at 30 cm depth (DeBano, 2000; Janzen and Tobin-Janzen, 2008).

Fire has multiple, complex effects on carbon (C) dynamics in soil. Wildfires alone lead to the release of up to 4.1 Pg C yr^{-1} to the atmosphere in the form of carbon dioxide, with an additional 0.05 to 0.2 Pg C yr^{-1} added to the soil as black or pyrogenic carbon ash (Singh et al., 2012). The changes in SOM characteristics due to combustion include reduced solubility of OM due to loss of external oxygen containing functional groups; reduced chain length of fatty acids, alcohols and other alkyl compounds; higher aromaticity due to transformation of carbohydrates and lipids; production of pyrogenic carbon; formation of heterocyclic nitrogen (N) compounds; and macromolecular condensation of humic substances (González-Pérez et al., 2004). In the long term, fires can affect soils by altering and removing vegetation and topsoil biomass, and increasing soil erodibility (Carroll et al., 2007; DeBano, 1991),

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subsequently leading to a shift in plant and microbial populations (Janzen and Tobin-Janzen, 2008).

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The aim of this study is to determine the effects of heating temperatures on important SOM properties. We used a laboratory heating experiment on five soils from a well-characterized climosequence in the western Sierra Nevada mountain range (Dahlgren et al., 1997). We analyzed changes in SOM quantity and quality following heating treatment with the aim to: (1) determine magnitudes of change in SOM properties associated with different fire heating temperatures; (2) identify critical thresholds for these changes; and (3) infer the implications of changing climate on topsoil SOM properties that might experience changing fire regime. This study aims to contribute to the systematic evaluation and development of ability to predict the effect of different intensity fires on soil properties under changing climate and fire regimes.

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2. Materials and methods

Following laboratory heating of five soils from western Sierra Nevada to temperatures ranging from 150 to 650 °C, we analyzed changes in SOM quality and quantity. We measured the changes in C and N concentration in the soil and changes in the distribution of C and N to different aggregate size classes. We also measured changes in isotopic composition of ^{13}C and ^{15}N in the soils and in the different aggregate size classes. Changes in SOM quality was analyzed using Fourier-transform infrared (FTIR) spectroscopy of soils. Description of the study site is given in Section 2.1 and details of the methods used are given in Sections 2.2 to 2.4.

2.1. Study site and soil description

For this study, we collected soils from five sites across an elevation transect along the western slope of the central Sierra Nevada, California (Figure 1); the sites were previously characterized by Dahlgren et al. (1997). We selected four forested sites that are likely to experience forest fires and a fifth lower elevation grassland site. The thermal alterations of bulk soil physical and chemical properties from the same study soils was previously reported in Araya et al. (2016).

All the sites have a Mediterranean climate characterized by warm to hot dry summers and cool to cold wet winters. Mean annual air temperature ranges from 16.7 °C at the lowest site located at 210 m to 3.9 °C at the highest elevation site which is at an elevation of 2865 m. Annual precipitation ranges from 33 cm at the lowest site to 127 cm at the highest site (Dahlgren et al., 1997; Rasmussen et al., 2007) (Table 1).

The lower elevation woodlands of Sierra Nevada experience less frequent fires than further upslope and the fires are often fast moving and lower severity (Skinner and Chang, 1996). At the middle-elevation zone of Sierran forest, the mixed conifer zones, frequent fires are low to moderate severity at lower altitudes but fire frequency generally increases with altitude towards the upper elevation of the mixed conifer forest (Caprio and Swetnam, 1993). Fires are infrequent and low severity within the high altitude, Subalpine, zone of Sierra (Skinner and Chang, 1996).

Soils from the lowest elevation site, Vista Series soils (210 masl), fall within the oak woodland zone (elevations < 1008 m). This is the only soil in our study that does not have an O-horizon, the soil has dense annual grass cover, however, and the A-horizon SOM originates mainly from root turnover. The Musick Series soils (1384 masl) lie within oak/mixed-conifer forest (1008—1580 masl) and mixed-conifer forest (1580—2626 masl). These soils receive the highest litter fall biomass. The Shaver and Sirretta Series soils (1737 and 2317 masl, respectively) fall within the mixed-conifer forest range zone while the Chiquito Series soils (2865 masl) lies within the subalpine mixed-conifer forest range

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(2626—3200 masl). These soils have lower litter fall compared to the lower elevation soils (van Wagtendonk and Fites-Kaufman, 2006).

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The western slope of central Sierra Nevada presents a remarkable climosequence of soils that developed under similar granitic parent material and are located in landscapes of similar age, relief, slope and aspect (Trumbore et al., 1996) with significant developmental differences attributed to climate. The soils at mid-elevation range (1000 to 2000 masl) tend to be highly weathered while soils at high and low elevations are relatively less developed (Dahlgren et al., 1997; Harradine and Jenny, 1958; Huntington, 1954; Jenny et al., 1949). Among the most important changes in soil properties along the climosequence include changes in soil organic carbon (SOC) concentration, base saturation, and mineral desilication and hydroxyl-Al interlayering of 2:1 layer silicates. Soil pH generally decreases with elevation and the concentrations of clay and secondary iron oxides show a step change at the elevation of present-day average effective winter snowline, i.e. 1600 m elevation (Tables 1 and 2) (California Department of Water Resources, 1952-1962; Dahlgren et al., 1997).

2.2. Experimental design and sample collection

Triplicate samples (0 to 5 cm depth) were collected at the five sites, approximately 10 m apart from each other. Any overlaying organic layer was removed prior to sampling so that only mineral soil was collected. The soils were air-dried at room temperature and passed through 2 mm sieve. Prior to furnace heating, the soils were oven dried at 60 °C overnight. Soil bulk density and field soil moisture were determined from separate undisturbed core samples collected from each site (Table 2).

Sub-samples from each soil were heated in muffle furnace to one of six selected maximum temperatures (150, 250, 350, 450, 550 and 650 °C). To ensure uniform soil heating and reduce formation of heating gradient inside, the soils were packed 1 cm high in a 7 cm diameter porcelain flat capsule crucibles. Oxygen supply was not limited during the heating,—the volume of soil sample to volume air in furnace was approximately 1:50. Furnace temperature was ramped a rate of 3 °C min^{-1} and soils were exposed to the maximum temperature for 30 minutes. Once cooled to touch, soils were stored in in air-tight polyethylene bags prior to analysis.

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The six heating temperatures were selected to correspond with fire intensity categories that are based on maximum surface temperature (DeBano et al., 1977; Janzen and Tobin-Janzen, 2008; Neary et al., 1999), that is, low intensity (150 and 250 °C), medium intensity (350 and 450 °C), and high intensity (550 and 650 °C). These fire intensity classes generally correspond with thresholds for important thermal reactions in soils observed by differential thermal analyses (Giovannini et al., 1988; Soto et al., 1991; Varela et al., 2010). Heating rate of 3 °C min^{-1} is preferred in laboratory fire simulation experiments (Giovannini et al., 1988; Terefe et al., 2008; Varela et al., 2010), the slow heating rate prevents sudden combustion when soil’s ignition temperature is reached at about 220 °C (Fernández et al., 1997, 2001; Varela et al., 2010). The samples were exposed to the maximum set temperature for a period of 30 minutes. This length of time ensures that the entire sample is uniformly heated at the set temperature and is in keeping with wide majority of similar laboratory soil heating experiments (for example Badía and Martí, 2003a; Fernández et al., 2001; Giovannini, 1994; Varela et al., 2010; Zavala et al., 2010). The duration of soil heating under vegetation fires is highly varied and not uniform across landscape (Parsons et al., 2010). The same heating procedure was used for all the soils so that it would be possible to compare how the soils from different climate regimes are likely to respond to the fires.

2.3. Laboratory analysis

Dry-aggregate size distribution was measured by sieving. Samples were dry sieved into three aggregate size classes: 2–0.25 mm (macro-aggregates), 0.25–0.053 mm (micro-aggregates) and <0.053 mm (silt and clay sized particles or composites). These aggregate size classes were selected to enable comparison with other studies that investigated the effect of different natural and anthropogenic properties on soil aggregate dynamics and aggregate protected organic matter (Six et al 2000).

C and N concentrations and stable isotope ratios were measured using an elemental combustion system (Costech ECS 4010 CHNSO Analyzer, Costech Analytical Technologies, Valencia, CA, USA) that is interfaced with a mass spectrometer (DELTA V Plus Isotope Ratio Mass Spectrometer, Thermo Fisher Scientific, Inc, Waltham, MA, USA). For the analyses, air-dried soil samples were ground to powder consistency on a ball-mill (8000M MiXer/Mill, with a 55 ml tungsten Carbide Vial, SPEX SamplePrep, LLC, Metuchen, NJ, USA) and oven dried at 60 °C for over 36 hours. This lower temperature and longer duration oven-drying was used to avoid possible heating related C or N changes that might occur if drying was done 105 °C (Kaiser et al., 2015). The C and N concentration results were corrected for moisture by oven-drying subsamples at 105 °C overnight. The C and N concentration results were corrected by adjusting for moisture as: $W_{adj} = W \times (100 - W_m)$. Where W_{adj} is the adjusted percent concentration, W is the concentration before moisture adjustment and W_m is the percent moisture content. All concentration changes resulting from moisture adjustment were a decrease of less than 1% of the value. The stable isotope ratios are presented using the δ notation (per mill, ‰) as $\delta\ ^{13}C$ and $\delta\ ^{15}N$ calculated as: $\delta = \left[\left(R_{sample} - R_{standard}\right)/R_{standard}\right] \times 1000‰$; where R is ratio of $^{13}C/^{12}C$ for $\delta\ ^{13}C$, and $^{15}N/^{14}N$ for $\delta\ ^{15}N$. The standards used for analyses are atmospheric N_2 $\delta\ ^{15}N$ and Vienna Pee Dee Belemnite (VPDB) $\delta\ ^{13}C$.

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Bulk soil organic matter composition was analyzed using Fourier-transform infrared (FTIR) spectroscopy on a Bruker IFS 66v/S vacuum FT-IR spectrometer (Bruker Biosciences Corporation, Billerica, MA, USA). We used diffuse reflectance infrared Fourier-transform (DRIFT) technique (Ellerbrock and Gerke, 2013; Parikh et al., 2014). Powder samples were dried overnight at 60 °C and scanned in mid-IR from 4000 to 400 cm^{-1}. In this study, we used Non-KBr diluted samples were used after preliminary analyses revealed that dilution is not necessary. KBr dilution is not required for soils with low (<10%) organic matter concentrations (Ellerbrock and Gerke, 2013; Reeves III, 2003). The FTIR spectrum was collected using KBr background and was baseline corrected using to the Rubberband correction method with the default 64 baseline points that is part of the OPUS software (Bruker Corporation, 2009).

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2.4. Statistical Analysis

All quantitative results are expressed as means of three replicates ± standard error, unless otherwise indicated. Differences of means were tested by Analysis of Variance (ANOVA) and pairwise comparison of treatments done using Tukey’s HSD test at $p<0.05$ significance level. The normality of the data and the homogeneity of variances was checked using Shapiro-Wilk’s and Levene’s tests respectively. All statistical analysis were performed using R statistical software (R Core Team, 2014). The Pearson correlation coefficient was used to examine relationships between C concentration and changes in soil properties.

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3. Results

3.1. Carbon and nitrogen concentration

The initial concentration of C ranged from 1.5% (Vista soil, 210 masl) to 7.7 % (Musick soils, 1384 masl). Soil C concentration continuously decreased with increasing temperature. The largest decrease occurred between temperatures of 250 and 450 °C. At 450 °C, all soils

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lost more than 95% of their original C. C concentration changes with heating above 450 °C were small and not statistically significant at $p<0.05$. The C:N ratio ranged from 10 (Vista soils, 210 masl) to 29 (Musick soils, 1384 masl). Following a similar pattern to C concentration changes, the C:N ratio decreased with an increase in heating temperature (Figure 2).

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The loss of C and N from soils due to heating showed a similar response among all five soils (Figure 2). After 250 °C, all the soils lost more than 25% of their initial C (except Shaver soils that lost only about 10%). At 350 °C all soils lost 50 to 70% of C. Heating at 450 °C led to the loss of more than 95% of their initial C for all soils in this study. However, the rate of loss of N was lower than that of C. At temperatures greater than 550 °C there was 5 to 15% of soil N still remaining. Consequently, we observed a decrease of C:N ratio with increased heating temperature. All soils continued to lose about 15% soil N for every 100 °C increase and maintained more than 60% of their N at heating temperatures up to 350 °C. After heating at 450 °C, all soils lost more than 60% of their original soil N and 85% by 550 °C.

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3.2. Carbon and nitrogen stable isotopes

The $\delta^{13}C$ composition of all soils was indicative of C-3 vegetation. Soil $\delta^{13}C$ composition was most negative at about -28‰ for the lowest elevation Vista site (210 m) and the value got consistently less negative with an increase in elevation reaching -24‰ for the highest two sites (i.e. >2317 m elevation). For all soils, there was a general trend of $\delta^{13}C$ enrichment with temperature increase (Figure 2). The largest change (2.5 to 3.0‰) occurred at heating temperature between 250 and 450 °C for the lower elevation soils and between 150 and 450 °C for the two highest elevation soils. For the two highest elevation soils, there was a significant ($p<0.05$) depletion above that temperature. For all soils, except Musick (1384 m) and Shaver (1737 m), the maximum enrichment occurred at 450 °C. All soils showed a

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similar pattern δ^{15}N composition change with temperature. The soils were increasingly δ^{15}N enriched with temperature increase up to 350 °C. At temperatures above 350 °C, the soils got more δ^{15}N depleted with the most negative δ^{15}N occurring at 650 °C (Figure 2).

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3.3. Carbon and nitrogen distribution in aggregate size fractions

C and N concentrations, as well as ^{13}C and ^{15}N stable isotope ratios were measured for individual soil aggregate size class. The analysis was done on samples heated up to a temperature of 450°C. The concentration of C and N in samples heated above 450 °C was too low to measure significant changes in C distribution in the different aggregate size classes.

The distribution of C in the three aggregate sizes fractions followed the same general pattern with increase in the heating temperatures. The macro aggregate size fraction (2-0.25 mm) had the least C concentration and silt-clay size particles (<0.053 mm) had the largest concentration of C (Figure 3). N concentration for the macro size aggregates was below the detection limit at 450°C for Chiquito and Sirretta. The change in C and N concentration across heating temperature was similar for all soils

The distribution of C and N in different size aggregates did not change noticeably except at 450 °C where concentration in all three fractions converged to zero. The distribution of N in the three aggregate sizes fractions was similar to that of C and followed a similar pattern across all the heating temperatures. Similarly, the macro aggregate size fraction (2-0.25 mm) had the least amount of N concentration and silt-clay size particles (<0.053 mm) had the largest concentration of N. For Shaver (1737 m), Sirretta (2317 m) and Chiquito (2865 m) soils, the macro size aggregate N concentration was too low and could not be detected (Figure 3). The atomic C:N ratio generally stayed the same for all soils through the temperatures. C:N ratio was highest in macro size aggregates, which had lowest C and N concentrations, followed by micro and by silt-clay sizes for all soils.

The stable isotope composition of ^{13}C was very similar between aggregate sizes with silt-clay size aggregates being slightly more enriched except for Shaver (1737 m), which had slightly more enriched macro aggregates. On the other hand, the δ^{15}N values showed clear differences among aggregate fractions even though the measured values of δ^{15}N did not change notably with combustion temperatures. δ^{15}N was highest in silt-clay size particles and lowest in macro size aggregates with the micro size aggregates showing intermediate values. The pattern of change in δ^{15}N across combustion temperatures did not affect this order of δ^{15}N values among aggregate fractions. Most of the C and N in the soils was associated with the larger, macro and micro, aggregate size fractions. With the exception of Vista (210 m) soils, the distribution changed with increase in heating temperature where the concentrations in macro aggregates decreased markedly that the remaining C and N concentrations were distributed between the smaller aggregate fractions (Figure 4). At 450 C, most of the C and N of the higher altitude soils (Shaver, Sieretta and Chiquito) was now associated with the silt-clay sized fractions.

3.4. FTIR spectroscopy

Changes in chemical composition of SOM due to heating were analyzed by infrared spectroscopy using Diffuse reflectance infrared fourier transform (DRIFT) technique. The spectra and peaks after contrasting levels of thermal treatments exhibited qualitative similarities among the different soils. FTIR spectra for the soils are shown in Figure 5. One notable changes that occurred in the functional group composition of SOM with heating is the lowered absorbance intensity of aliphatic methylene groups (as represented by the aliphatic C–H stretching peak that appear at bands between 2950 – 2850 cm^{-1}) at >250 °C in all soils. When comparing intensity of peaks at 2910 – 2930 and 2853 cm-^{1} wave numbers (from aliphatic methyl and methylene groups, band A) with those at 1653 and 1400 cm^{-1} (oxygen containing carboxyl and carbonyl groups, band B), the decrease in prominence in the

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Deleted: combustion at different temperatures aliphatic C-H peak occurs early in the heating sequence while the C=O band shows little relative change. In addition, after heating at a temperature of 550 °C all soils lost the O–H stretching peaks (between 3700 – 3200 cm^{-1}). In a pattern that is more prominent for the Musick soil that had the highest concentration of OM, the aromatic C=C stretch around 1600 cm^{-1} gets more resolved with increase in heating temperature. This pattern in the C=C is visible, but less well resolved in the rest of the soils, especially the Vista soil that showed the least resolved aromatic C=C stretch peak at this region.

4. Discussion

4.1. Changes in SOM concentration, distribution and composition

Our results show significant effects of combustion temperature on concentration, distribution, and composition of SOM on topsoils that experience the most intense heating during vegetation fires. Topsoils have relatively high OM and low clay content that render them more sensitive to heating as the SOM experiences significant changes during heating. In our study system, the effect of fire heating on SOM ranged from slight distillation (volatilization of minor constituents) typically at temperatures below 150°C, to charring which typically starts at temperatures above 350°C and complete combustion, consistent with findings of previous studies (Badía and Martí, 2003b; Certini, 2005). Our findings also confirmed that regardless of the differences our soils had in mineralogy and other soil physical and chemical properties, the heating treatments (as proxy for wild fires) led to consistent decrease in concentration of soil C. This was in agreement with previous studies that showed decrease in soil C concentration in topsoil after fires (for example Badía et al. (2014); Certini (2005)). However, this loss of C is expected to be restricted to topsoil while it is expected that the C concentration in subsoil is likely to remain unchanged or may even increase (for example

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We observed significant changes in concentration, distribution, and composition of SOM with increasing heating temperature. The steep decline in concentration of C in soil that we observed between this study is consistent with decrease of about 25% C at 250°C and an almost 99% loss at 450°C (Figure 6). The magnitude of C loss with heating we observed is similar to the findings of (Terefe et al., 2008; Ulery and Graham, 1993) that investigated changes in soil C using artificial heating experiment. Similarly, Giovannini et al. (1988) also found OM decrease started at 220 °C with about 15% loss of OM and about 90% OM loss at 460 °C; while Fernández et al. (1997) reported 37% of SOM loss at 220 °C and 90% at 350 °C. Furthermore, along with the change in C concentration; between 150 °C and before almost total loss of C above 450 °C, the SOM went through significant qualitative changes that included decrease in C:N ratio, enrichment in $\delta^{13}C$ isotope, changes in $\delta^{15}N$ isotope, and changes in FTIR spectra. Loss in N after fire heating is the result of combustion and volatilization (Fisher and Binkley, 2000). In this study, we observed that N is not as significantly reduced until 350°C with about 75% N remaining as opposed to greater than 50% loss of C concentration at the same temperature (Figure 6). Previously studies had showed that moderate to high intensity fires convert most organic-N into inorganic forms of N, such as Ammonium (Certini, 2005; Huber et al., 2013). Ammonium is the immediate combustion product that contributes to formation of nitrate (NO_3^-) by nitrification reactions in weeks or months after fire. Other studies have showed that a considerable amount of N is transferred into pyrogenic OM products, to black N (de la Rosa and Knicker, 2011; Knicker, 2010), which would also explain the decrease of the C:N. Decrease in C:N ratio with fire heating has previously been observed in both laboratory and field fire studies (Badía and Martí, 2003a; Certini, 2005; Fernández et al., 1997; González-Pérez et al., 2004) .

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SOM has a C isotopic composition that reflects the δ^{13}C signature of native vegetation. Plants are depleted in δ^{13}C relative to atmosphere. The δ^{13}C composition for our soils indicated that the dominant source of OM in all soils is C3 plant biomass that had average δ^{13}C of -27%, with the higher elevation soils having more positive δ^{13}C than the low elevation. Enrichment of ^{13}C with heating is consistent with the loss of plant derived C. In addition, the fact that lipids (that have relatively more δ^{13}C depleted than the woody materials) are combusted at lower temperatures than woody materials (such as cellulose and lignin), might contribute to the enrichment of δ^{13}C with heating (Czimczik et al., 2002). The stable C and N isotope composition of our soils showed significant fractionation with temperature. δ^{13}C values became more positive (enriched in δ^{13}C) up to 450 °C where up to 99% of C was lost (Figure 6). At higher temperature there was a less uniform pattern among the soils. For the last <1% C, Sirretta and Chiquito soils continued to be more negative (depleted in δ^{13}C) at higher temperature while for the rest of the soils there was a slight depletion at 550°C followed by a slight enrichment at 650 °C (Figure 2). The depletion of δ^{13}C at 550 and 650 °C we found in this study is likely a result of SOM charring as there was little or no decrease in C concentration between these temperatures. In a wood charring experiment (non-oxygen atmosphere) at 150, 340 and 480 °C, Czimczik et al. (2002) observed an enrichment of δ^{13}C at 150 °C where there was no C concentration change but a depletion of δ^{13}C at 340 and 480 °C with charring where the C concentration increased over 50% due to charring.

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Fires tend to lead to enrichment of ^{15}N, this is particularly observed in soils immediately in the aftermath of fires (Boeckx et al., 2005; Grogan et al., 2000; Herman and Rundel, 1989; Huber et al., 2013), but there is limited information available on the exact temperature ranges that cause specific levels of ^{15}N enrichment. In this study, we observed enrichment of ^{15}N up to 350 °C and depletion after 350 °C for all soils (Figure 2). It is likely that the continued ^{15}N enrichment with heating is the result of fractionation due to combustion and volatilization of

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organic matter which discriminate against ^{15}N. However, the exact mechanism behind continued depletion of ^{15}N when heated above 350 °C remains unclear. One potential explanation for the ^{15}N depletion at higher temperatures could be indiscriminate removal of N as higher temperatures cause the combustion and volatilization process to happen instantly, compared to charring of OM at lower temperatures. In a post fire-analysis of δ^{15}N on a sub-alpine ecosystem in Australia, Huber et al. (2013), found that the ^{15}N enrichment of bulk surface soil (from unburnt leaves) was higher than that of the charred OM, which was again higher than that of the ash. They attributed this difference in enrichment level to be the result of the lower heating intensity experienced by the bulk soil which provided slower processes for greater fractionation, while higher heat intensity experienced in by the ash result in full combustion of plant material providing little opportunity for isotopic discrimination. The temperature range where we observed the depletion of ^{15}N in our experiment corresponds with the range where steep decline in N concentration happened (Figure 6), which would be consistent with the explanation.

4.1.1. Implication of SOM changes with heating

The alterations and loss of SOM is likely more important cause of soil property changes rather than alterations to soil minerals. SOM is vulnerable to temperatures while soil minerals are only affected at much higher temperatures (Araya et al., 2016). In addition, all of the soils in our study are characterized by low clay content and low concentration of reactive minerals, but high concentration of SOM especially in topsoil leading to strong relationships between SOM concentrations and soils' physical properties.

Degradation of lignin and hemicellulose begins between 130 and 190 °C (Chandler et al., 1983); and carbohydrate signal is completely removed from ^{13}C NMR spectra by 350 °C. Furthermore, Knicker observed loss of stable alkyl C and carboxyl C at 350 °C leading to

Deleted: is dependent on duration

Deleted: intensity of heating .

Deleted: 0 – 5 cm

Deleted: enriched in ^{15}N (approximately 3.3‰) while

Deleted: enriched to a lesser extent (approximately 0.5‰) and

Deleted: to an even lesser extent (approximately -0.6‰).

Deleted: , that is, lower heat intensity

Deleted: (observed in bulk soil),

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Deleted: in ash). The

Deleted: observed after 350 °C

Deleted: our findings of

Deleted:) further supporting

Deleted: that the enrichment prior to 350 °C is likely due to fractionation during combustion and volatilization whereas at higher temperatures reversal of pattern towards depletion is likely a result of the indiscriminate removal of N

Deleted: *4.1.1*

Deleted: conducted a SOM and plant residue heating experiment in which she observed degradation

Deleted: beginning at

Deleted: , respectively; and completely removal of enrichment of aromatic functional groups in the remaining residue, consistent with what would expected from incomplete combustion of OM during fires, leading to transformation and production of charred products (Almendros et al., 2003; Knicker et al., 1996). FTIR analyses from our work showed that the aliphatic O−H stretch peak (bands 3700 – 3200 cm^{-1}) disappeared at temperatures above 550 °C for all soils accompanied by nitriles or methanenitrile C≡N stretch (2300 – 2200 cm^{-1}) at temperature above 450 suggesting condensation of aromatic functional groups.

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Loss of OM from soil due to combustion has multiple implications on soil physical and chemical properties. Simple linear correlation between C concentration changes and other soil physical and chemical changes that we observed with heating (reported here and in Araya et al. (2016)) show that more than 80% of the variability in mass loss, aggregate strength, SSA, pH, CEC and N concentrations is associated with changes in C concentration at the different heating temperatures. Table3 summarizes the correlation coefficients of soil property changes with change in C concentration. Analyses of associations between C concentration and several soil properties showed linear association between: C and N (R^2 >0.8), mass loss (R^2 >0.8, except for Vista and Sirretta soils), pH (R^2 >0.8, except for Shaver and Sirretta), CEC (R^2 >0.7, except for Chiquito). Linear association between C concentration and aggregate strength (R^2 >0.7, except for Musick and Chiquito which had R^2 ~0.7). Specific surface area showed relation with C (R^2 >0.7 except for Vista and Musick).

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In this study, the greatest changes in SOM occurred between temperatures 250 and 450 °C and we found that temperatures below 250 °C had little effect on the quality and quantity of SOM. This implies that lower intensity fires, such as typical prescribed fires, where soil surface temperatures do not exceed 250 °C (Janzen and Tobin-Janzen, 2008) have minimum impact on SOM.

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4.2. Climate Change Implications

Investigation of the response of climosequence soils to different heating temperature in this study enables us to infer how, in the long-term, changes in climate are likely to alter the effect of fires on topsoil physical and chemical properties. Along our study climosequence, we observed critical differences in response of topsoils based mostly on concentration OM in soil and soil development stages of each soil. Soil OM concentration and composition in particular has been shown to respond to changes in precipitation amount and distribution, as is expected in the Sierra Nevada (Berhe et al., 2012b). Consequently, changes in soil C storage associated with climate change are expected to lead to different amounts of C loss due to fires. This is evidenced by the observed highest total mass of C loss from the mid-elevation Musick soil that had the highest carbon stock, compared to soils in either side of that elevation range. Anticipated changes in climate in the Sierra Nevada mountain ranges are expected to include upward movement of the rain-snow transition line exposing areas that now receive most of their precipitation as snow to rainfall and associated runoff (Arnold et al., 2015, 2014; Stacy et al., 2015). Upward moving of the rain-snow transition zone under anticipated climate change scenarios and associated more intense weathering at higher elevation zones can render more C to loss during fires. More than 80% of the variability in mass loss, aggregate strength, SSA, pH, CEC and N concentrations is associated with changes in C concentration (Table 3). Hence, as the vulnerability of these ecosystems to increased fire frequency increases, due to climate change (Westerling et al., 2006), we can expect more soil C loss with fires, along with associated changes in soil chemical and physical properties. In particular, our findings of important changes in soil physical and chemical properties occurring between 250-450 ºC are important for recognizing that critical transformations of topsoil SOM are likely to occur when, as a result of climate change, systems that are adapted to low severity fires experience medium to high severity fires.

Deleted: (and associated changes in soil properties)

Deleted: --both variables that are expected

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The different responses of soil aggregation in our climosequence to the treatment temperatures also suggest potential loss and transformation of the physically protected C pool in topsoil. Degradation of aggregates during fire (Albalasmeh et al., 2013) is likely to render aggregate-protected C to potential loses through oxidative decomposition, leaching and erosion. Moreover, in systems such as the Sierra Nevada which are dominated by steep slopes, movement of the rain-snow transition zone upward is likely to increase proportion of precipitation that occurs as rain. The kinetic energy of raindrops and the observed increase in hydrophobicity of soils after fires (Johnson et al., 2007; Johnson et al., 2004) can lead to higher rates of erosional redistribution of especially the free light fraction or particulate C that is not associated with soil minerals (Berhe et al., 2012a; Berhe and Kleber, 2013; McCorkle et al., 2016; Stacy et al., 2015).

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Deleted: and organic matter-rich topsoils dominate

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5. Conclusion

Previously, considerable amount of work had been published to demonstrate how fires affect OM concentration and composition in biomass. This study fills critical gaps by determining how and to what extent OM in soil experiences changes due to heating. The findings of this study also showed that changes in soil properties during heating are closely related to changes in C concentrations in soil. The temperatures most critical to C loss and alteration were found to be 250 °C, where charring of organic matter starts and 450 °C where most of the SOM is combusted. Most soil properties exhibited a steep change in this temperature range. SOM exhibited largest change, i.e. soils became enriched in ^{13}C and ^{15}N isotopic composition until approximately 90% of C and N was lost, at higher temperatures slight depletion of ^{13}C and steep depletion of ^{15}N is observed. FTIR spectroscopy showed the reduction and disappearance of aliphatic OH functional groups with temperature increase and accumulation of aromatic carbon groups.

Deleted: Finally, with changes in climate it is anticipated that fires will increase in severity . Our findings of important changes in soil physical and chemical properties occurring between 250-450 ºC are important for recognizing that critical transformations of topsoil SOM are likely to occur when, as a result of climate change, systems that are adapted to low severity fires experience medium to high severity fires.¶

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This study presented the effects of heat input on topsoil properties. The study is necessary to understand thermally induced changes on soil properties in isolation from other variables that accompany vegetation fires such as the addition of pyrolysis products from plants and ash, and the fire induced soil moisture dynamics. Findings from this study will contribute towards estimating the amount and rate of change in carbon and nitrogen loss, and other essential soil properties that can be expected from topsoil exposure to different intensity fires under anticipated climate change scenarios.

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Figures

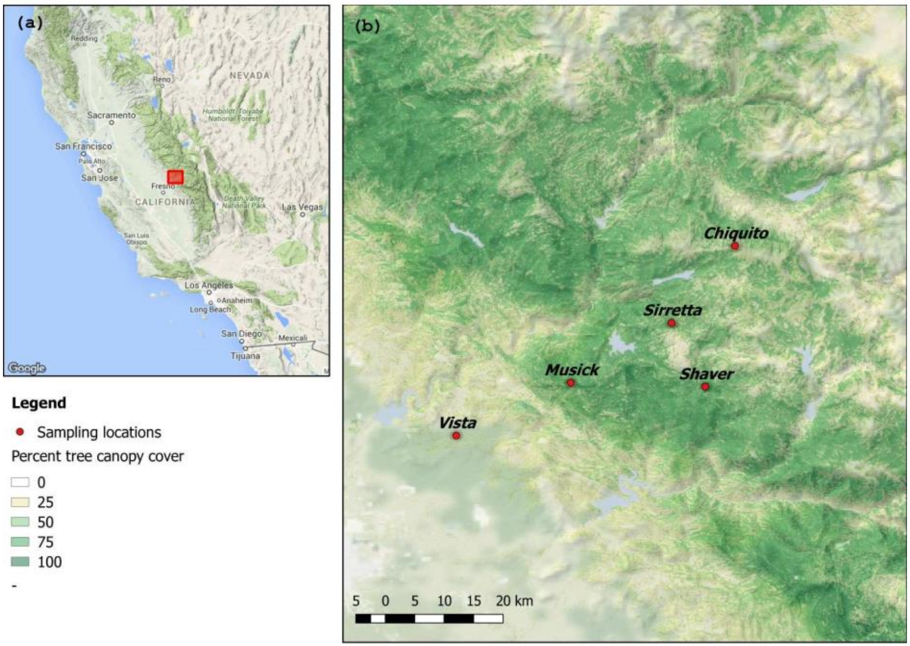

Figure 1: (a) Location of the sampling site on the western slopes of the Sierra Nevada, California, and (b) map of the five sampling locations and percent tree canopy cover (U.S. Geological Survey, 2014).

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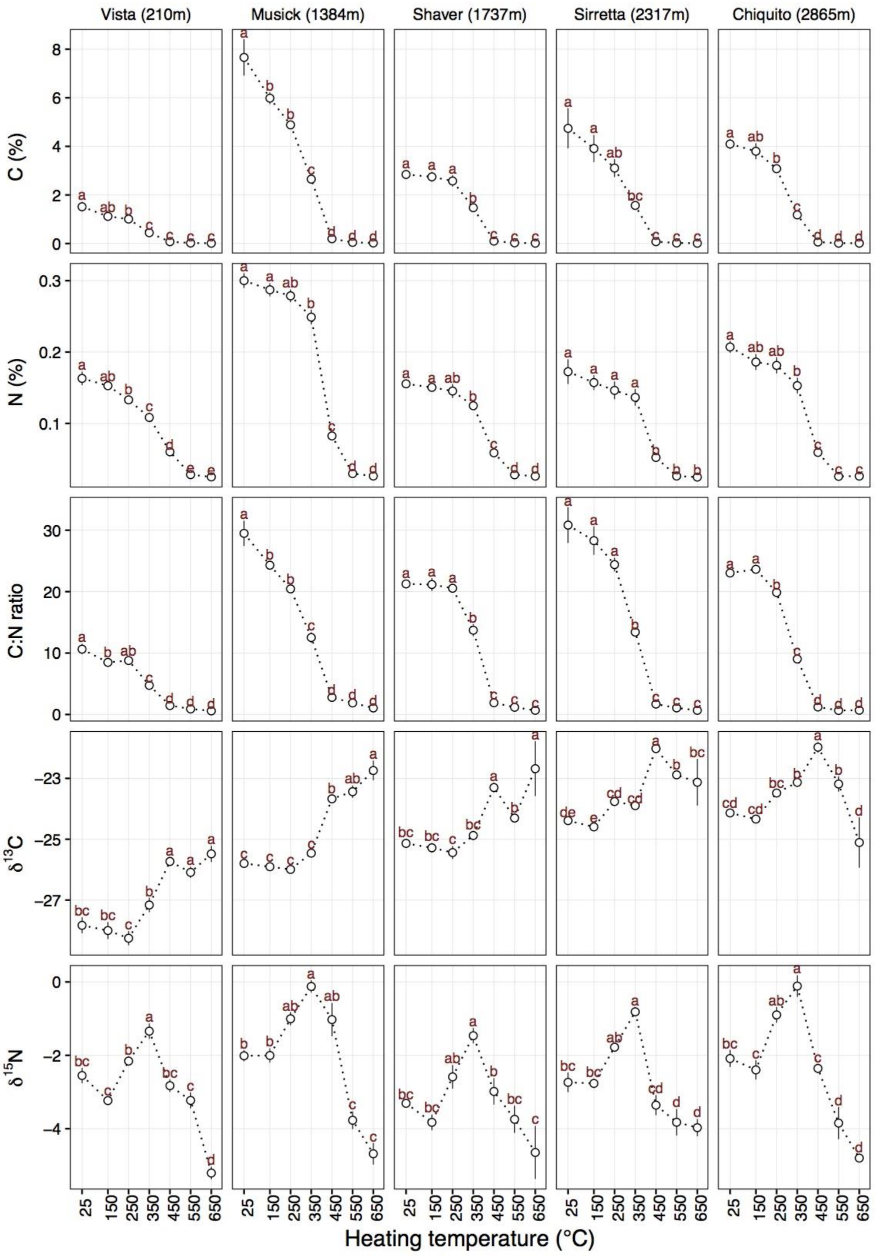

Vista (210m)
Musick (1384m)
Shaver (1737m)
Sirretta (2317m)
Chiquito (2865m)
C (%)
0
2
4
6
8
a ab b c c c c
a b b c d d d
a a a b c c c
a a ab bc c c c
a ab b c d d d
N (%)
0.1
0.2
0.3
a ab b c d e e
a a ab b c d d
a a ab b c d d
a a a a b b b
a ab ab b c c c
C:N ratio
0
10
20
30
a b ab c d d d
a b b c d d d
a a a b c c c
a a a b c c c
a a b c d d d
$\delta^{13}C$
−27
−25
−23
bc bc c b a a a
c c c c b ab a
bc bc c bc a b a
de e cd cd a b bc
cd cd bc b a b d
$\delta^{15}N$
−4
−2
0
bc c b a bc c d
b b ab a ab c c
bc bc ab a b bc c
bc bc ab a cd d d
bc c ab a c d d
25
150
250
350
450
550
650
Heating temperature (°C)

1

Figure 2: Bulk soil Carbon and Nitrogen concentrations, C:N atomic ratio, and $\delta^{13}C$ and $\delta^{15}N$ isotope (‰) changes with increase in heating temperature. Error bars represent standard error where n=3. Different letters represent significantly different means ($p<0.05$) at each temperature after Tukey’s HSD testing.

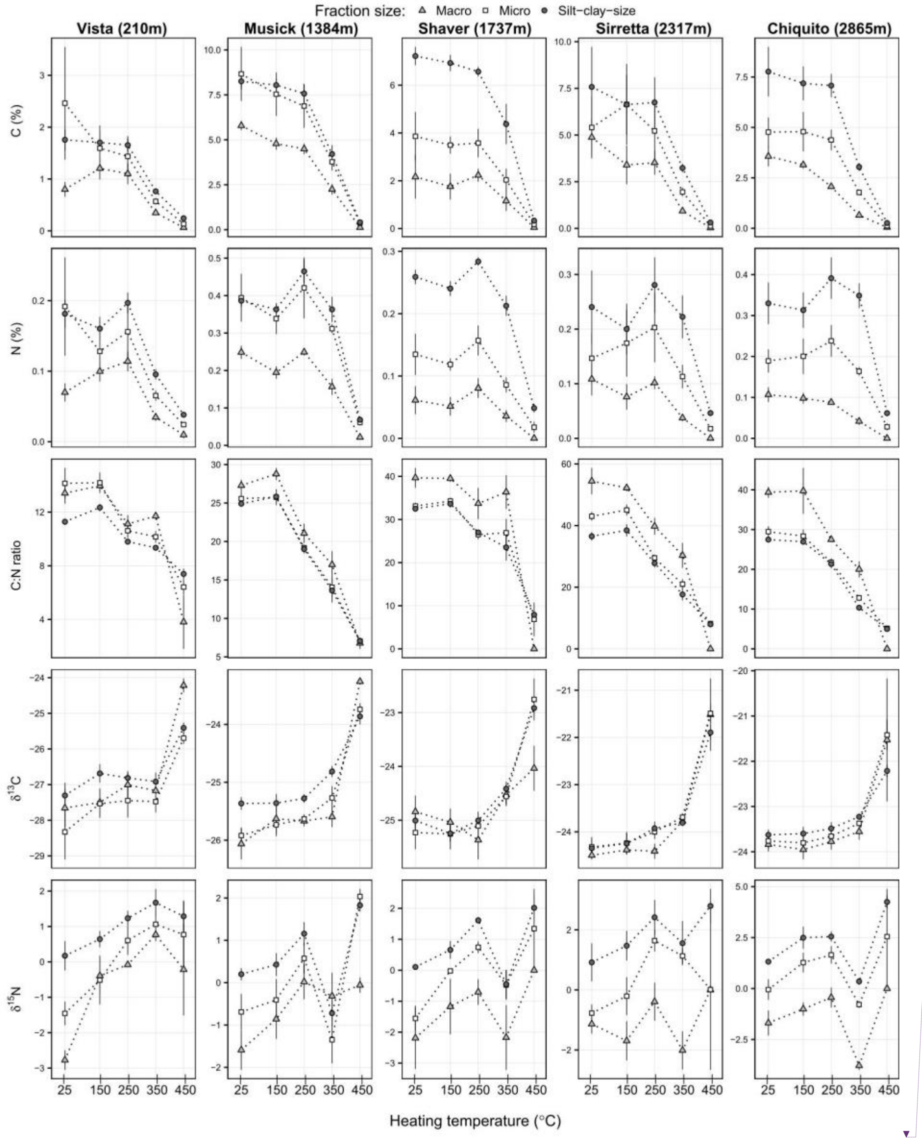
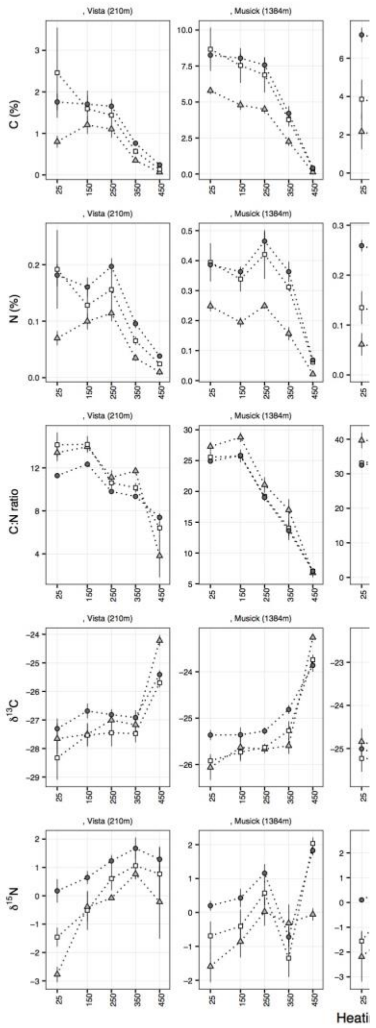


Figure 3. C and N concentrations, C:N atomic ratio, and δ^{13}C and δ^{15}N isotope (‰) changes in macro (2-0.25 mm), micro (0.25-0.053 mm) and silt-clay sized (<0.053 mm) aggregates with increase in heating temperature. Error bars represent standard error where n=3.



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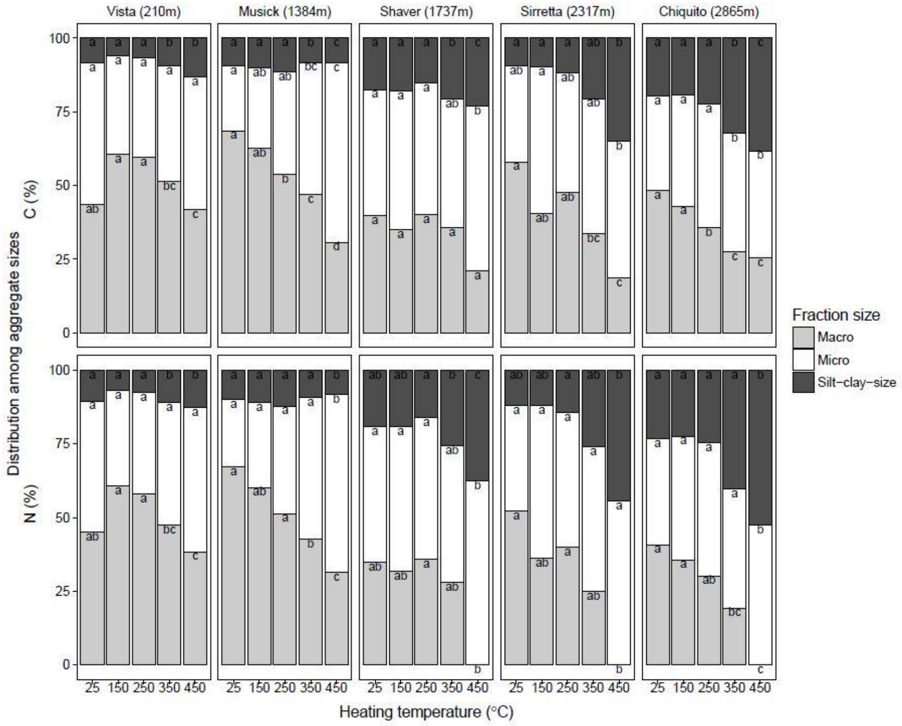
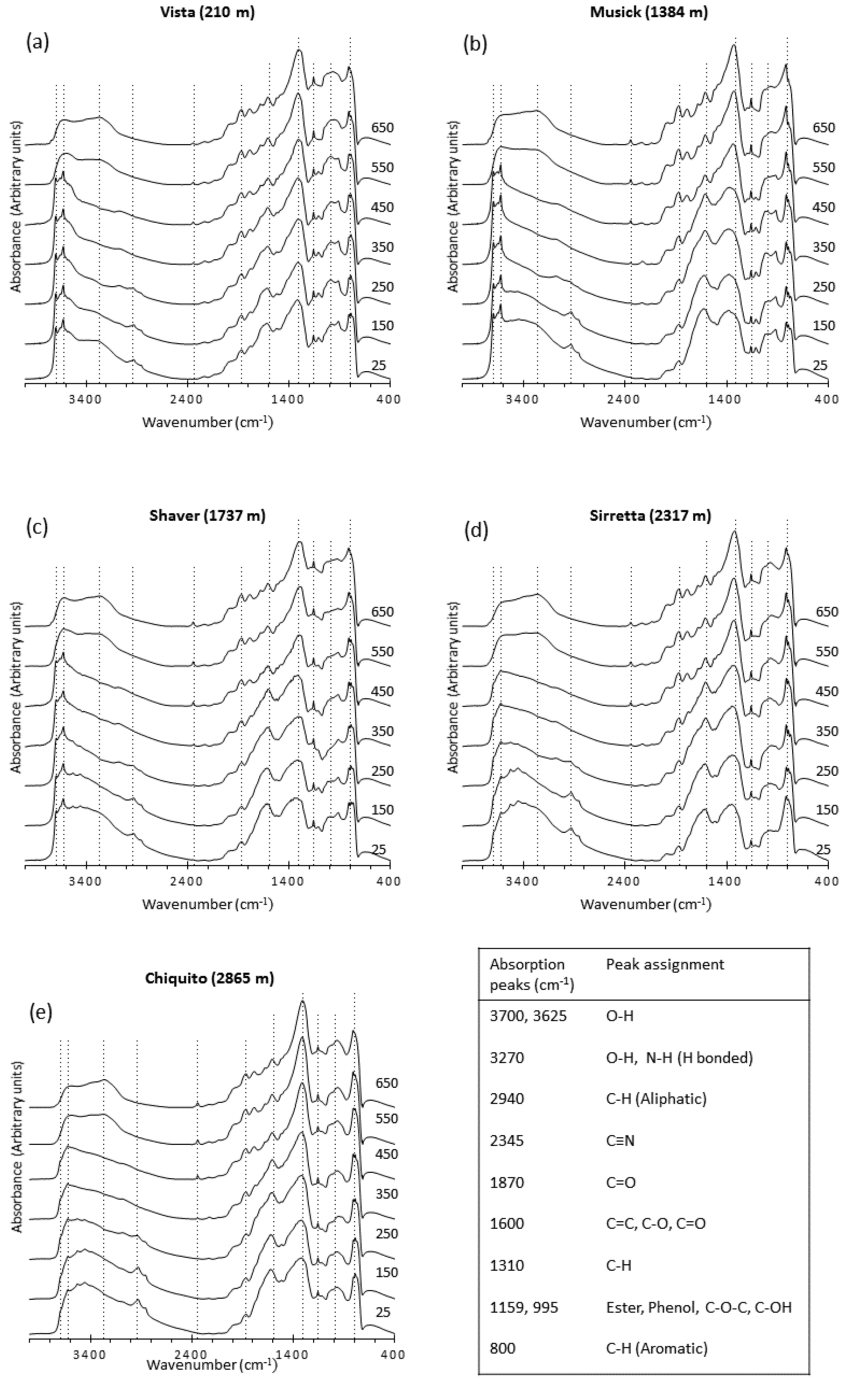


Figure 4: C and N distributions in macro (2-0.25 mm), micro (0.25-0.053 mm) and silt-clay sized (<0.053 mm) aggregates.



Absorption peaks (cm^{-1})	Peak assignment
3700, 3625	O-H
3270	O-H, N-H (H bonded)
2940	C-H (Aliphatic)
2345	C≡N
1870	C=O
1600	C=C, C-O, C=O
1310	C-H
1159, 995	Ester, Phenol, C-O-C, C-OH
800	C-H (Aromatic)

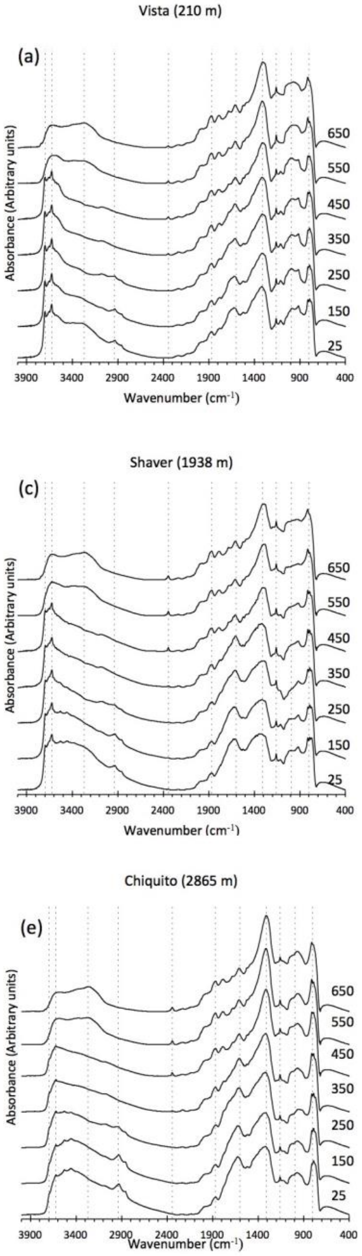


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Figure 5: FTIR spectra of the five soils at the different heating temperatures. Heating temperatures, in Celsius, are shown to the right of each spectrum.

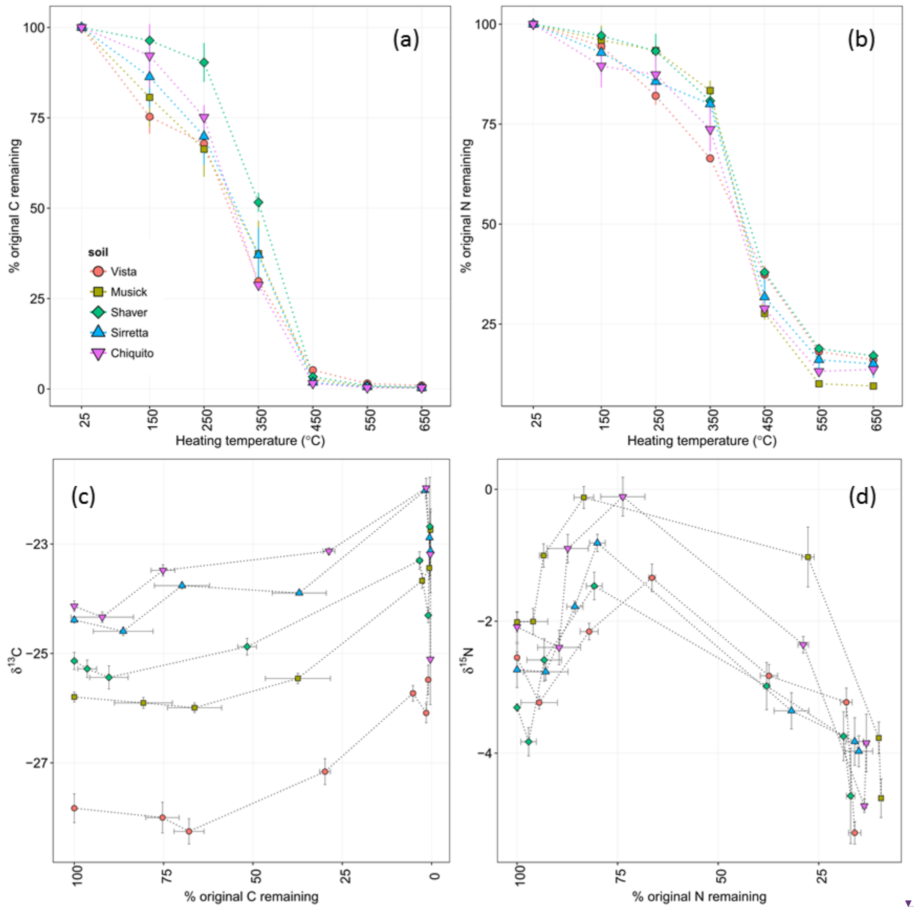

Figure 6: (a) Percentage of C and (b) N loss with heating; and (c) change in δ^{13}C and (d) δ^{15}N versus percent of total C and N lost from soils (error bars represent standard error where n=3).

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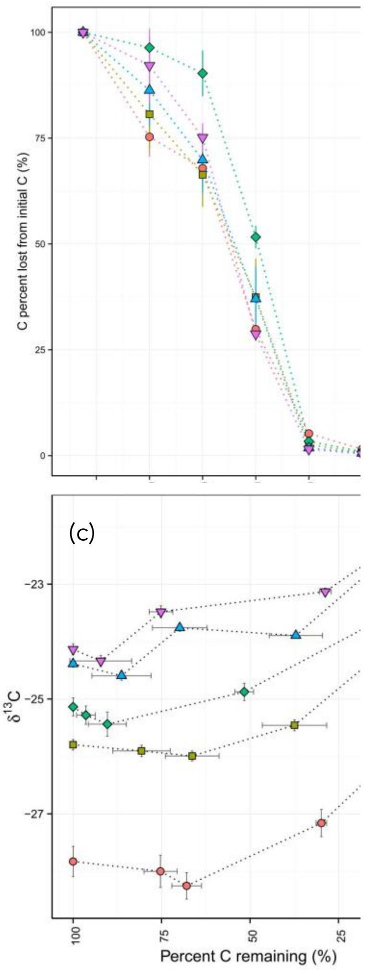

Tables

Table 1 Soil classification and site description for the five sites along elevational transect in the western slopes of the Sierra Nevada (adapted from Dahlgren et al., 1997)

Field Code Changed

Soil Series	**Elevation (m)**	**Ecosystem**	**MAT[a] (°C)**	**MAP[b] (cm)**	**Precip[c]**	**Dominant vegetation (listed in order of dominance)**	**Soil taxonomy (family)**
Vista	210	Oak woodland	16.7	33	Rain	Annual grasses; *Quercus douglasii*; *Quercus wislizeni*	Coarse-loamy, mixed, superactive,thermic; Typic Haploxerepts
Musick	1384	Oak/mixed-conifer forest	11.1	91	Rain	*Pinus ponderosa*; *Calocedrus decurrens*; *Quercus kelloggii*; *Chamaebatia foliolosa*	Fine-loamy, mixed, semiactive, mesic; Ultic Haploxeralf
Shaver	1737	Mixed-conifer forest	9.1	101	Snow	*Abies concolor*; *Pinus lambertiana*; *Pinus ponderosa*; *Calocedrus decurrens*	Coarse-loamy, mixed, superactive, mesic; Humic Dystroxerepts
Sirretta	2317	Mixed-conifer forest	7.2	108	Snow	*Pinus jeffreyi*; *Abies magnifica*; *Abies concolor*	Sandy-skeletal, mixed, frigid; Dystric Xerorthent
Chiquito[d]	2865	Subalpine mixed-conifer forest	3.9	127	Snow	*Pinus contorta murrayana*; *Pinus monticola*; *Lupinus* species	Sandy-skeletal, mixed; Entic Cryumbrept

[a] Mean annual air temperature, calculated from regression equation of Harradine and Jenny (1958); [b] Mean annual precipitation; [c] Dominant form of precipitation; [d] Tentative soil series

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Table 2 Bulk density, water content, pH, C concentration, cation exchange capacity (CEC), specific surface area (SSA) and particle size distribution for the five soils (mean ±standard error, n=3)

Soil series and elevation (m)	**Bulk density (g/cm^3)**	**Gravimetric water content (%)**	**pH (CaCl$_2$)**	**Carbon (%)**	**CEC (cmol$_c$/kg)**	**SSA (m^2/g)**	**Particle size distribution[a] (%)**		
							Sand	**Silt**	**Clay**
Vista (210)	1.26 ±0.07	0.7 ±0.0	5.53 ±0.0	1.51 ±0.2	8.40 ±1.1	1.75 ±0.2	79	11	10
Musick (1384)	0.90 ±0.06	9.3 ±1.6	4.67 ±0.1	7.66 ±0.8	25.20 ±2.0	4.98 ±0.3	60	27	15
Shaver (1737)	0.98 ±0.06	8.3 ±1.1	4.85 ±0.3	2.84 ±0.2	10.67 ±2.1	3.08 ±0.3	80	15	5
Sirretta (2317)	0.61 ±0.09	9.9 ±2.2	4.54 ±0.1	4.74 ±0.8	12.23 ±2.6	6.63 ±0.8	80	15	5
Chiquito (2865)	1.17 ±0.03	6.1 ±1.9	3.96 ±0.1	4.10 ±0.2	6.03 ±1.8	1.00 ±0.04	80	16	4

[a] Particle size distribution of top soil profile from Dahlgren et al. (1997), Vista (0 – 14 cm), Musick (0 – 29 cm), Shaver (0 – 4 cm), Sirretta (0 – 6 cm) and Chiquito (0 – 6 cm)

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Table 3 Linear correlation coefficients of changes in soil properties with changes in C concentration. All correlation coefficients have p-values < 0.01 unless otherwise indicated.

Soil	Correlation coefficient (r^2) values					
	Mass loss	**SSA**	**Aggregate Stability**	**pH ($CaCl_2$)**	**CEC**	**N concentration**
Vista	0.74	0.73	0.21[a]	0.77	0.78	0.89
Musick	0.89	0.58	0.77	0.89	0.96	0.83
Shaver	0.82	0.58	0.68	0.74	0.78	0.93
Sirretta	0.60	0.34[b]	0.47	0.67	0.87	0.86
Chiquito	0.82	0.62	0.78	0.88	0.44	0.87

[a] p = 0.078; [b] p = 0.035

Author responses to Referee # 1 Comments

(doi:10.5194/soil-2016-57-RC1, 20160)

The referee comments are followed by our responses. We have indicated location of texts in the manuscript by a combination of page number and line number (page#,line#).

General Comments:

The authors first begin describing OM "quality and quantity" in the Discussion, so a brief statement much earlier in the manuscript is needed to introduce and identify the metrics the authors are using to asses SOM quality.

> **Author response:** We have added statements earlier in the manuscript that describe the metrics of SOM quality assessments. We added a sentence in the introduction (P5, L5-6). We also added a brief description of the methods, including SOM quality, in the Materials and Methods section (P5, L14-21)

My major critique is that the authors have not presented the full results of their statistical tests, yet they discuss increases, decreases and correlations.

> **Author response:** We have decided to include the full results of statistical tests as an appendix to the manuscript. In addition, we have indicated p-value for correlation coefficients (r^2) reported in Table 3.

Additional editing for English language is needed, particularly in the Discussion (many of missing articles; misuse of "while" instead of the correct "whereas" when making contrasts; disagreement in verb use and plurality).

> **Author response:** done

Specific comments and technical corrections:

P2, L2: Can you revise the first statement to provide a more engaging beginning to your manuscript?

> **Author response:** done

P2, L3 (5): Remove capitalization on Climosequence; specify laboratory heating experiment.

> **Author response:** done

P2, L10-13: confusing/clarify Many places: "increase" should be "increased" so check English language usage throughout manuscript.

> **Author response:** done

P3, L8: such as?

> **Author response:** We have added a sentence with examples (P3, L8-9).

P4, L5(2) and elsewhere in the manuscript: I think you should remove self-citation of the information you are presenting in the current article for review, even in "published" previously as a Discussion article. However, I will defer to the Editor's recommendation on this issue.

> **Author response:** The citation in question (Araya et al. 2016) is from a separate publication which has some important data on physical and chemical changes that result from heating.

P4, L7: it determines in part (not exclusively!). Also, replace "longer durations" with "longer residence time" or "durations at a particular location"

> **Author response:** done.

P4, L8-9 (4-6): this statement is confusing because you're comparing duration, temperature and rate of spread. I personally understand what you're trying to say but it is not clear as currently written. Clarify this statement.

> **Author response:** done.

P4, L18-20 (15-17): clarify awkward statement.

> **Author response:** done.

P4, L22 (19): "used"

> **Author response:** done.

P4, L23 (19)-P5, L3: English language needs revision in objectives statements. E.g., "to determine (2) identify" and "to determine (3) infer" does not make sense. Just move the "determine" to after the (1) to fix the problem here.

> **Author response:** done.

P5, L11 (9-10): grassland sites also experience fires in this region

> **Author response:** This statement was meant to highlight that the grassland site is not likely to experience burn temperatures at the high temperatures (>350 C) and at the residence times (30 min) that we used in this study. But, we do understand the reviewer's concern, and for the sake of clarity, we have removed 'for comparison" from that sentence (P6 L2).

P5, L13: complete citation or remove duplicate

> **Author response:** done.

P6, L3-11: clarify in the text that Vista, Musick, etc. soils are soil Series names. Also, what do you mean by "soils receive the highest biomass"? From what do they receive biomass? Do you mean they support the greatest aboveground biomass and receive the greatest annual litterfall? Are you talking about aboveground biomass or microbial biomass? Please clarify.

> **Author response:** done.

P7, L2: Were O horizon(s) included in your samples, or did you first remove the overlying O horizons to collect only the 0-5cm depth of the mineral soils? This is very important to clarify your sampling approach here.

> **Author response:** done.

P7, L23: clarify here what part of the soil ignites at 220C

> **Author response:** Explicitly, the soil organic matter ignites. However, since it is part of the soil, we feel the general statement is sufficient.

P8, L5: yes, but the duration of heating experienced by soils during fires in the environment is typically very short (just a few seconds) except where the soils are overlain by a lot of fuels that have potential to provide sustained heating into the mineral soil.

> **Author response:** We agree with the reviewer here. In deed this is why we decided not to prescribe heating times, and treat all the soils to the same heating time.

P9, L3: "by adjusting"

> **Author response:** done.

P9, L11-22: add description about what was used as the background spectrum, and the approach used for scaling and baseline adjustments, etc.

> **Author response:** done (P10 L7-P11).

P13, L12 (5): reference error. Also, consider replacing "combustion" with "contrasting levels of thermal treatments"

> **Author response:** done.

Where are the results from the statistical tests? Letters are indicated on Figure 2, but nowhere are F or p-values reported for any of the tests. Correlation coefficients are reported in Table 3, but without associated p-values. Except for a few places, the results text also does not state whether the observed "differences" are statistically significant or not. Much more detail in needed about the statistical results to be able to adequately interpret the significance of the author's observed patterns. Report the full results (all coefficients) from the simple linear regressions.

> **Author response:** done. We have now decided to include the full report of statistical tests as an appendix to the manuscript. The statistical tests we performed were described in Section 2.4. In the manuscript we have used the term statistically significant to mean where p-values from Tukey's HSD test are < 0.05. We have also indicated the p-values to the correlation coefficients in Table 3.

P14, L5 (P13, L22): Here the authors state "significant effects" but have not provided sufficient detail about the results of their full set of analyses.

> **Author response:** done. We address this comment by including of the results of ANOVA tests in the appendix. We think this might be too much information to publish

as supplementary materials, but we will let the editors make that decision.

P15, L23-P16, L1 (L13-16): Here it is not clear whether you are talking about the combustion of specific types of organic compounds (lipids versus cellulose and lignin) or combustion of types of materials (X? versus woody materials). Please revise to make your meaning more clear.

Author response: done (P16, L5-8).

P16, L14: Revise: awkward sentence L17: observed L24: revise punctuation to: "...heating intensity; that is, lower..."

Author response: done.

L23-P16, L3 (P16, L10-17): I don't follow your meaning here. Also, it's unclear whether this information refers to heat intensity or fire (fireline) intensity. Relating intensity to slowness of a process is a questionable analogy. Check sources and revise language to clarify your meaning.

Author response: We have re-written the entire paragraph to clarify the meaning. Specifically, what was meant by 'slowness' of the process is the charring of OM which happens more at lower temperature heating in our experimental setting as opposed to total combustion (P17, L1-5).

P18, L15 (L3): replace "most significant" with "greatest" (especially because all the statistical results have not been reported in this version).

Author response: done.

P18, L18: revise to clarify your meaning, or omit "below".

Author response: done.

L23: omit the parenthetical clause because it is redundant with the information that follows.

Author response: done.

P19, L20 (L7): I don't think that 2% to 8% C can be called organic-matter rich (they are only relatively rich in SOM, compared to subsoils). Omit this part of the sentence.

Author response: done. (P20, L6)

P20, L13: replace "got" with "was" or "became"

Author response: done.

Figure1: Assign letters to figure panels. Revise caption to state which panel shows the basemap of tree canopy cover.

Author response: done.

Figure 2: Very nice figure that shows clear trends across the temperature treatments.

Author response: Thank you for the comment.

Figure 3: Revise to agree with the format used for Figure 2: show only one set of column labels and one set of x-axis labels to be able to increase the size of the panels. Currently it is much too small to read. Move the legend symbols into the figure caption for better use of space.

Author response: done.

Figure 5: Provide more detail in figure caption, for example, state that heating temperature is shown to the right of each spectrum

Author response: done.

Figure 6: Increase the font size used for axis labels and axis titles. Statistical results?

Author response: We have increased font size as suggested. We have not indicated statistical significance in the plot because such information for the different soils would overcrowd the plots to the point where it would be difficult to interpret.

Table 3: add p-values for all correlation coefficients.

Author response: done.

We appreciate the thoughtful comments from the reviewer. Thank you.

Author responses to Referee # 2 Comments

(doi:10.5194/soil-2016-57-RC2, 2016)

The referee comments are followed by our responses. We have indicated location of texts in the manuscript by a combination of page number and line number (page#,line#).

General Comments:
The present work describes research performed to investigate the impact of fire temperature on the organic matter composition of soils from a climosequence derived from the Sierra Nevada (USA). It was intended to infer the implications of changing climate on topsoil SOM properties that might experience changing fire regime. This subject is of course of interesting for the readers of SOIL and deserves a proper treatment. In order to reach their objectives, the authors conduct a systematic study to examine the changes of topsoil organic matter from the selected soils occurring during their heating under laboratory conditions at increasing temperatures. Those experiments show that major changes occur at temperatures > 250°C and that those changes are expressed mainly in a loss of C and N and the formation of aromatic C. This approach has also been used in many other studies which came to the same results, although they used other materials. Taking this into account, the study is not really a novel idea or concept, but confirms already existing data. The authors try to put their results into the context of climatic change. Well, the climate change may change the C contents in soil, the quality of the litter and the fire frequency, but the chemistry occurring during combustion will not be altered. The climate change will also not have a considerable direct impact on fire intensity, since the latter is mostly determined by the available fuel and its moisture (OK, if there is more fuel due melting of permafrost soils, yes there is an indirect impact of climate on fire intensity. If the climate turns drier there are only more fires if the fuel density does not change etc.). I think, it is difficult to perform the relationship which was done by the others which turns some of the conclusions into opinions which can be obtained without conducting the present work. I think also, that it is difficult to conclude that climatic change lead to more fires with temperatures above 250°C. If the right fuel is present, the temperatures of a forest fire can easily increase to 1000°C and more - even without climatic change. Of course not much charcoal will accumulate under those conditions. The produced accumulated charcoal, on the other hand, is the material which enters the soil and will change the chemical composition of its SOM. This impact will be higher than the transformation of SOM in the mineral phase itself, since the low thermal conductivity of the latter prevents easy increase of soil temperatures and thus major heat-induced alterations of its SOM. Considering further that mineral soils were analyzed, I strongly suggest to include a part explaining the temperature profile of mineral soils during fire in the introduction. Summarizing my concerns together, it is not clear to me, how the used experimental design can add to a better understanding of the impact of climate change on the transformation of topsoil SOM due to fire. I think if the authors present their data as a relationship between soil properties and their alterations due to the impact of fire and the content of SOM (without trying to include climate change), the paper would still be of interest but less speculative.

> **Author response:** We appreciate the reviewer's comments and summary. We have revised the introduction section overall for clarity and to address comments from both reviewers. Specifically, we have added a brief explanation of vertical temperature profile during fires in the introduction (P4, L 9-14).

Specific comments:

P4, L12: sugars cannot be aromatic. I suggest, . . .higher aromaticity due to transformation of carbohydrates and lipids (actually, the latter may rather transform into smaller units)

> **Author response:** done. (P4, L 20).

Mention which Sierra Nevada was studied (USA).

> **Author response:** done. (P5, L 24).

The whole description of the study site is very extensive which is not necessary to be able to follow the approach. It should be considerably shortened.

> **Author response:** We understand the reviewer's concern, but we believe shortening this section would make the methods, site information and justification too brief for audiences that are not familiar with the study area. We have left it as is for now, and would be happy to revisit this discussion if the reviewer and editor think it is a must.

I missed the information if the litter layer was included in the sampling or if only mineral soils was used. This question is of importance since the litter represents the fuel.

> **Author response:** Only the mineral soil was used in the study. We have added a sentence in the methods to explicitly state that that O horizon was not used (P7, L17).

Why was oxygen supply limited? In natural above-ground fires, the fire commonly stops under oxygen depletion. In addition, it has to be considered that pyrolysis allows to go to higher temperatures before complete combustion occurs.

> **Author response:** We have edited the statement regarding oxygen supply during heating, the intended meaning was that oxygen supply was not limited. The statement in section 2.2 is meant to express that oxygen was not limited since volume of furnace was relatively large (P8, L1).

P8, L4: At the used temperatures no alteration of the mineral phase is expected. In addition, dry soils were used, thus higher moisture due to colder climate is not probed. Therefore, the only differences in soil properties which are relevant for the effect of fire temperature is the quality and the quantity of organic matter which can be related to climate regime but may also be altered by other environmental conditions (Whereas the quantity of C has been considered for the discussion of the data, the quality was largely neglected). Thus, in my opinion, it is difficult to relate unbiasedly climate regime to impact of fire on SOM.

> **Author response:** While mineral transformation is generally associated with extremely high temperatures, we have observed some mineralogical changes in the soils (for which the results were reported Araya et al. 2016, SOIL, doi: 10.5194/soil-2-351-2016). In addition, the properties of the mineral phase, including the physical and chemical properties, might affect the response of SOM to heating. Change in the quality of SOM was explored by FTIR and C and N isotopic composition.

Page 10: With respect to the concomitant loss of C and N, see also early work of (Knicker et al., 1996; Almendros et al., 2003)

> **Author response:** done. We have now added these useful citations to the discussion

> section as they are very useful to support the arguments for why we see the pattern of C and N loss with fire

Page 11: A fact, which has to be considered with stable isotopic data interpretation that different chemical compound classes don't exhibit the same resistance against heat. For example, carbohydrates transform into furans at temperatures at which lignin polymers are only cleaved and only some side chains are released as CO_2 or water (Sharma et al., 2001; Sharma et al., 2004). Since different biomolecules have different stable isotopic ratios, this behavior can result in the shift of the ratios for the whole sample.

> **Author response:** We agree that the different sensitivity of organic compounds to heating might also explain the shift in isotopic composition. We have now elaborated on this idea in Discussion (P16, L5-8).

Page 13: I suggest to include FTIR spectra of the unburnt soils which would allow to relate the initial quality of the SOM (most likely affected by the climate and vegetation cover) to changes induced by the fire.

> **Author response:** done. We have added description to the caption of Figure 5 to clarify.

P14, L6: The authors are certainly not discussing the physico-chemical properties of a soil, but the physical (pore size etc.) and chemical (pH, chemical composition etc.) properties.

> **Author response:** We have changed the wording as physical and chemical properties. We have also edited the paragraph to clarify the meaning (P14, L18,20, and P18, L8).

P14, L8: In the reference "Knicker et al." it is stated that fire can lead to increase or decrease of organic C in the soil, depending upon the intensity of the fire and the amount of produced char derived from the vegetation cover and litter layer.

> **Author response:** done. The reference Knicker et al (2005) was misplaced, it has now been moved to the following statement (P 15, L 2).

Page 15, line 4: Other studies showed that a considerable amount of N is transferred into the so called Black N (Knicker, 2010; de la Rosa and Knicker, 2011), which would much better explain the decrease of the C:N ratio with temperature (This does not mean that ammonium is not formed, but not all N turns into inorganic forms..)

> **Author response:** We have included black N as a possible explanation (P15, L21-23)

Page 17, line 5: The reference represents a review and does not show studies in which the degradation of lignin and hemicellulose starts at 130°C. However, studies of the same author showing complete removal of carbohydrate at 350°C are (Almendros et al., 1994; Knicker et al., 1996; Knicker et al., 2008).

> **Author response:** done. We have updated it with the reference for the original research (P17, L22).

As mentioned above, I have problems with the part Climate Change implications. For example, the statement of line 16 to 23 on page 18 is very general and has only very few relation to the results of the data. Actually, if the rain-snow transition zone is moving higher, considerable losses will occur due to increased SOM degradation. If less SOM is present, this may give less fuel to the fire (only a mind game...)

> **Author response:** We have edited this section for clarity and to address the concerns of the reviewer.

Page 19: Fires in the range of 250 to 450˚C (topsoil temperature) are in the range of low to medium severity fires. The respective mineral topsoil is expected to have temperatures which are still below 200˚C. High severity fires are hotter than 510˚C and are recognized by the fact that mostly ash remains on the soil.

> **Author response:** We understand the reviewer's point here. But, this particular statement is referring to systems experiencing low-severity fires experiencing medium to high severity fire in the future. The text in this section reads: "Our findings of important changes in soil physical and chemical properties occurring between 250-450 ºC are important for recognizing that critical transformations of topsoil SOM are likely to occur when, as a result of climate change, systems that are adapted to low severity fires experience medium to high severity fires."

We appreciate the thoughtful comments from the reviewer. Thank you.