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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 investigated thermal alteration of SOM properties by exposing five different topsoils (0 to 5 cm depth) from the western Sierra Nevada Climosequence to a range of temperatures that are expected during prescribed and wild fires (150, 250, 350, 450, 550 and 650 °C), and determined temperature thresholds for major shifts in SOM properties. 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 increase temperature whereas there was ^{15}N enrichment with increase in temperature only up to 350 °C followed by ^{15}N depletion as 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.

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

1. Introduction

Fire is a common, widespread phenomenon in many ecosystems around the world (Bowman et al., 2009). 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 (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 an increase in 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.

In addition to combustion of aboveground biomass and alteration of vegetation dynamics, fire also significantly affects 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 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). The duration of burning impacts soil properties because it determines the amount of energy transferred through the soil. Fires with longer durations typically have greater impact on soil properties and SOM than higher temperature fires if they are fast-moving (Frandsen and Ryan, 1986; González-Pérez et al., 2004).

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 of sugars and lipids; production of pyrogenic carbon; formation of heterocyclic nitrogen (N) compounds; and macromolecular condensation of humic substances (González-Pérez et al., 2004). Furthermore, by altering and removing vegetation and topsoil biomass, and increasing soil erodibility (Carroll et al., 2007; DeBano, 1991), subsequently leading to a shift in plant and microbial populations (Janzen and Tobin-Janzen, 2008).

The aim of this study is to investigate effects of heating temperatures on important SOM properties. We use a laboratory heating experiment on soils from a well-characterized climosequence in the western Sierra Nevada mountain range to determine: (1) 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

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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.

2. Materials and methods

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 (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 for comparison. The thermal alterations of bulk soil physical and chemical properties from the same study soils was previously reported in Araya et al. 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).

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Soils from the lowest elevation site, Vista 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. Musick soils (1384 masl) lie within oak/mixed-conifer forest (1008—1580 masl) and mixed-conifer forest (1580—2626 masl). These soils receive the highest biomass and litter fall. Shaver and Sirretta soils (2317 masl) fall within the mixed-conifer forest range zone while Chiquito soils (2865 masl) lies within the subalpine mixed-conifer forest range (2626—3200 masl). These soils have lower biomass and litter fall compared to the lower elevation soils (van Wagtendonk and Fites-Kaufman, 2006).

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. 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 during the heating was limited by the availability of space, and 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.

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;

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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 for moisture by and 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) $\delta\,^{13}C$.

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}. Non-KBr diluted samples were used after preliminary analyses revealed that dilution is not necessary for soils with low (<10%) organic matter concentrations (Ellerbrock and Gerke, 2013; Reeves III, 2003). Furthermore, using non-diluted samples was also favored because, even though dilution has the advantage of increased spectral quality, non-KBr diluted DRIFT has advantage in that it reduces sample preparation to a minimum, reduces possible interference by absorbed matrix hydration, maintains higher sensitivity, and the use of relatively larger samples provide better representation of sample heterogeneity (Janik et al., 1998).

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 ordinary linear regression technique was used to examine relationships between soil properties.

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 decreased with increase in temperature with the largest decrease occurring between temperatures of 250 and 450 °C. At 450 °C, all soils had lost more than 95% of their initial C. At temperatures above 450 °C, C concentration changes were small and we did not find statistically significant changes at $p<0.05$. The C:N ratio ranged from 10 (Vista soils, 210 masl) to 29 (Musick soils, 1384 masl). The C:N ratio decreased with increase in heating temperature in a similar pattern to the C concentration (Figure 2).

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 increase in 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 the initial soil N and 85% by 550 °C.

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

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 $\delta^{15}N$ values showed clear differences among aggregate fractions even though the measured values of $\delta^{15}N$ did not change notably with combustion temperatures. $\delta^{15}N$ 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 $\delta^{15}N$ across combustion temperatures did not affect this order of $\delta^{15}N$ 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.

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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. Absorption band functional group assignments that were used in this study are given in **Error! Reference source not found.**. The spectra and peaks after combustion at different temperatures 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 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, for all the soils that varies in mineralogy and other soil physico-chemical properties, the heating treatments (as proxy for wild fires) led to consistent decrease in concentration of soil C in topsoil as was previously observed by Badía et al. (2014); Certini (2005); and Knicker et al. (2005). As topsoil experiences most significant changes, it is expected that the C concentration in subsoil is likely to remain unchanged or may even increase (for example Dennis et al. (2013); Kavdır et al. (2005)) due to incorporation of necromass from surface biomass (Almendros et al., 1990).

We observed significant changes in quantity and quality 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

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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, specifically Ammonium (NH_4^+) (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. 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) .

SOM has a C isotopic composition that reflects the $\delta^{13}C$ signature of native vegetation. Plants are depleted in $\delta^{13}C$ relative to atmosphere. The $\delta^{13}C$ composition for our soils indicated that the dominant source of OM in all soils is C3 plant biomass that had average $\delta^{13}C$ of -27%, with the higher elevation soils having more positive $\delta^{13}C$ than the low elevation. Enrichment of ^{13}C with heating is consistent with the loss of plant derived C. The enrichment of $\delta^{13}C$ with heating is also likely enhanced because the relatively more $\delta^{13}C$ depleted lipids are combusted at lower temperatures relative to woody materials (cellulose, lignin) which are typically combusted at higher temperatures (>300°C) (Czimczik et al., 2002). The stable C and N isotope composition of our soils showed significant fractionation with temperature. $\delta^{13}C$ values became more positive (enriched in $\delta^{13}C$) 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 $\delta^{13}C$) 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 $\delta^{13}C$ 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 $\delta^{13}C$ at 150 °C where there was no C concentration change but a depletion of $\delta^{13}C$ at 340 and 480 °C with charring where the C concentration increased over 50% due to charring.

Fires tend to lead to enrichment of ^{15}N was observed in soils, especially 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 obxerved enrichment of ^{15}N up to 350 °C and depletion after 350 °C for all soils (Figure 2) likely due to isotopic fractionation of ^{15}N during combustion and volatilization, the extent of which is dependent on duration and intensity of heating (Huber et al., 2013). In a post fire-analysis of $\delta^{15}N$ on a sub-alpine ecosystem in Australia, Huber et al. (2013) found that bulk soil (0 – 5 cm) was enriched in ^{15}N (approximately 3.3‰) while charred OM was enriched to a lesser extent (approximately 0.5‰) and ash to an even lesser extent (approximately -0.6‰). They attributed this difference in enrichment to be the result of heating intensity, that is, lower heat intensity provided slower processes for greater fractionation (observed in bulk soil), while higher intensity fires result in full combustion of plant material providing little opportunity for isotopic discrimination (observed in ash). The depletion of ^{15}N observed after 350 °C corresponds with our findings of steep decline in N concentration (Figure 6) further supporting the explanation 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.

4.1.1. 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

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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.

Knicker (2007) conducted a SOM and plant residue heating experiment in which she observed degradation of lignin and hemicellulose beginning at 130 and 190 °C, respectively; and completely removal of carbohydrate signal from ^{13}C NMR spectra by 350 °C. Furthermore, Knicker observed loss of stable alkyl C and carboxyl C at 350 °C leading to 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. 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.

Loss of OM from soil due to combustion has multiple implications on soil physico-chemical properties. Simple linear regressions 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).

In this study, the most significant 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 below 250 °C (Janzen and Tobin-Janzen, 2008) have minimum impact on SOM.

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 (and associated changes in soil properties) 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 -- both variables that are expected to respond to changes in climate (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). Moving of the rain-snow transition zone higher and promotion of more intense weathering at higher elevation zones then is likely to render more C to loss during fires. As we found in this study, 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 (Table 3).

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 where steep slopes and organic matter-rich topsoils dominate, 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 observed increase in hydrophobicity of soils post-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).

Finally, with changes in climate it is anticipated that fires will increase in severity (Westerling et al., 2006). 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.

5. Conclusion

The findings of this study 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. soil got 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.

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

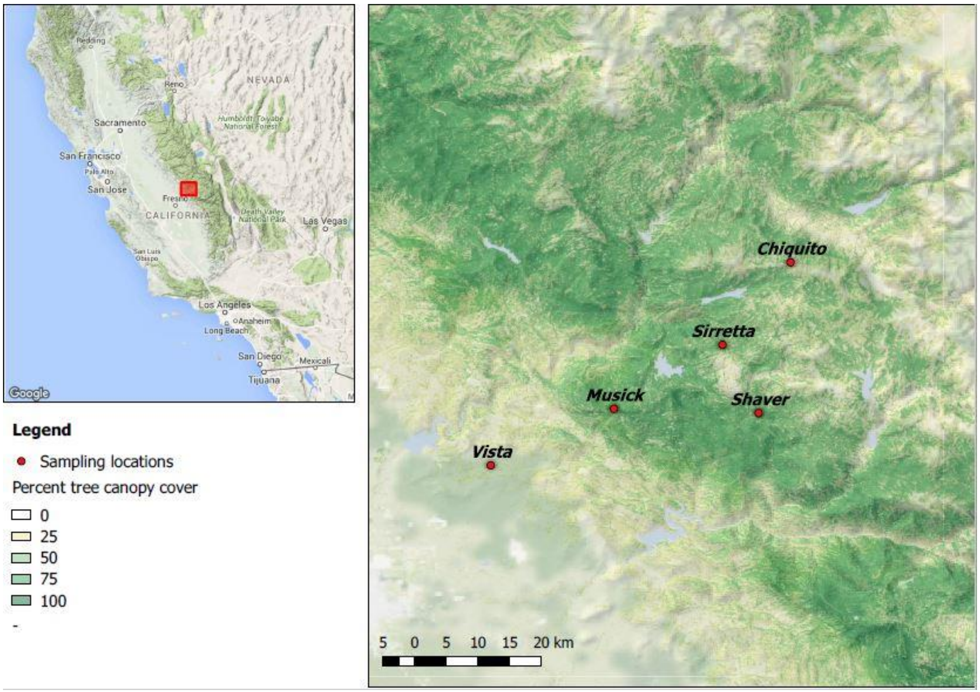

Figure 1: Map of the five sampling sites along elevational transect in the Western Sierra Nevada, California. Base map: percent tree canopy cover (U.S. Geological Survey, 2014).

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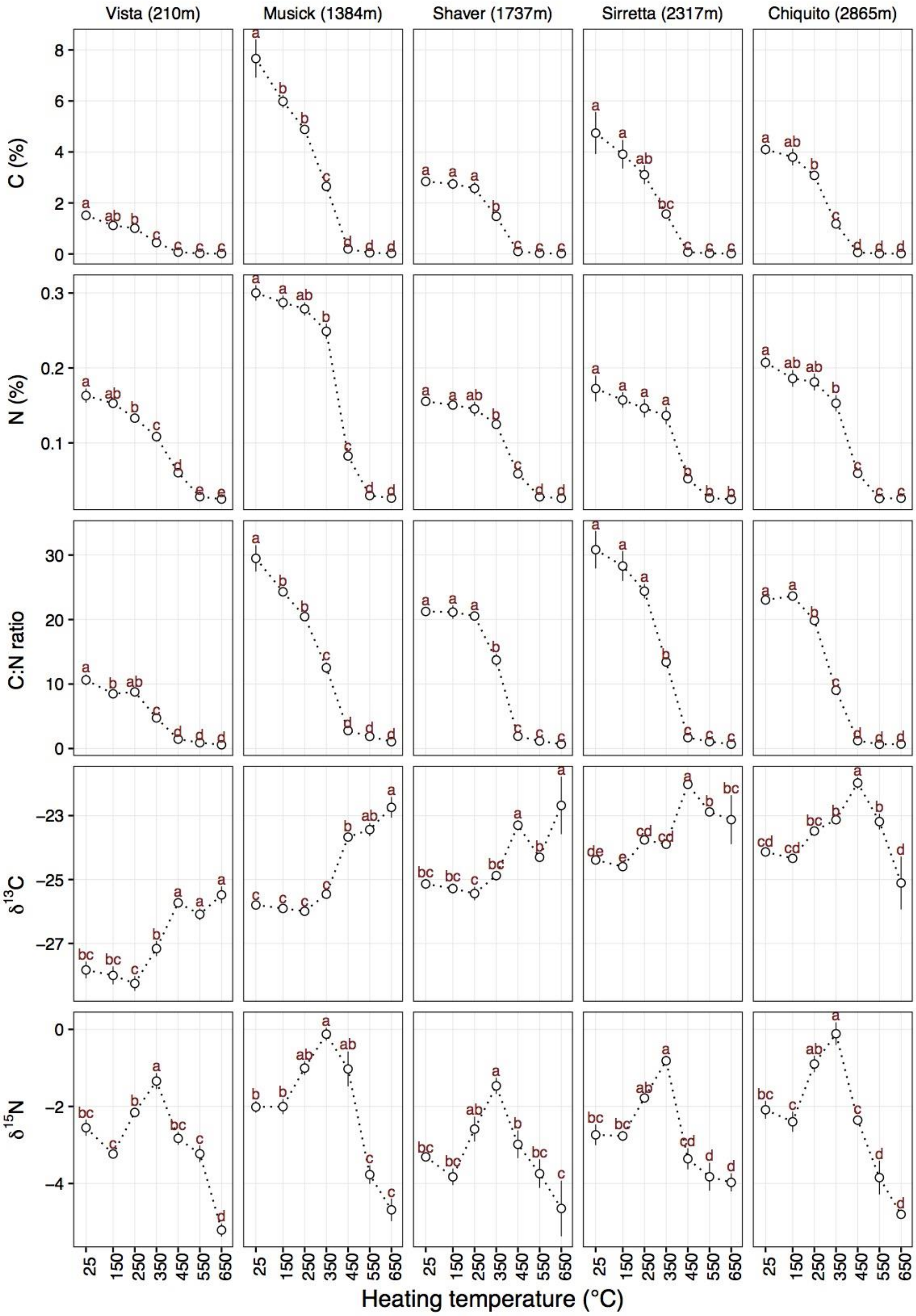

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Figure 2: Bulk soil Carbon and Nitrogen concentrations, C:N atomic ratio, and $\delta^{13}C$ and $\delta^{15}N$ isotope (‰) changes with increase in heating temperature. Error bars represent standard error where n=3. Different letters represent significantly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

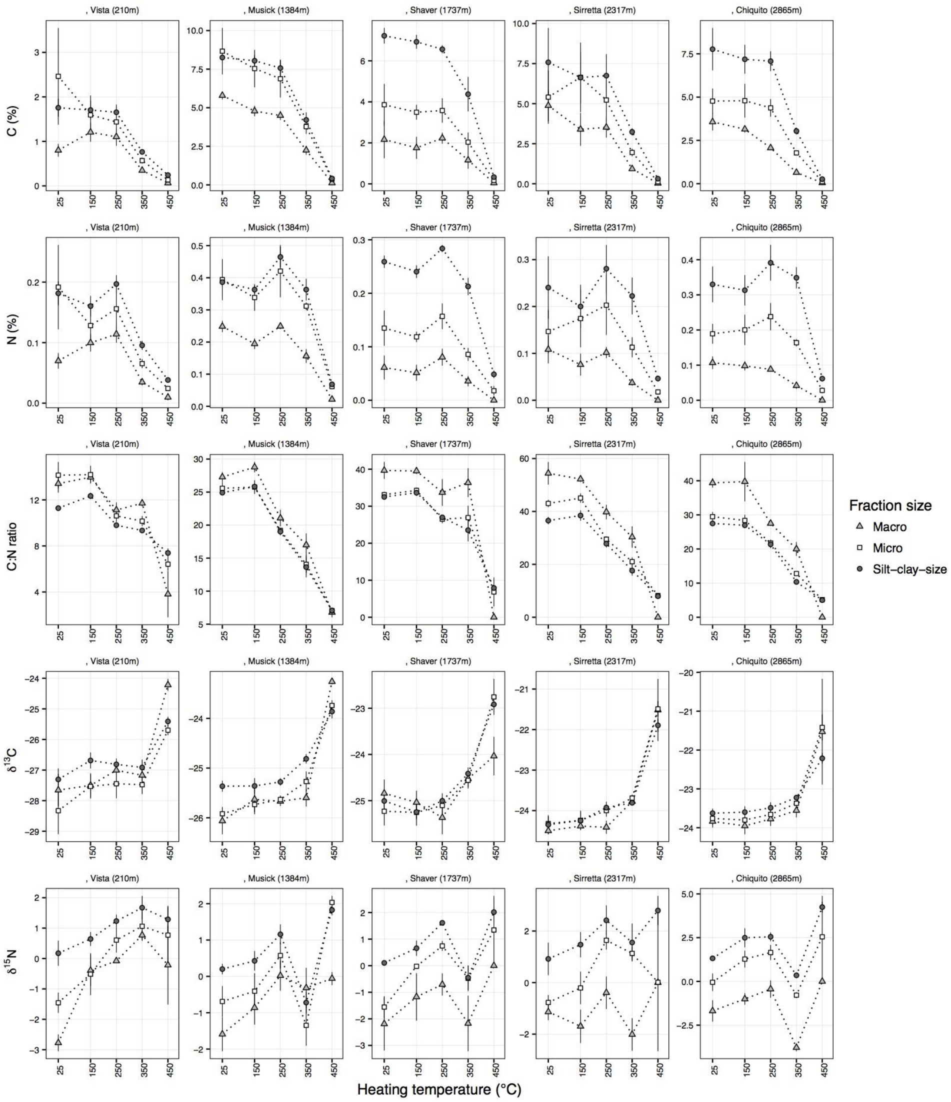

Figure 3. C and N concentrations, C:N atomic ratio, and $\delta^{13}C$ and $\delta^{15}N$ isotope (‰) changes in macro (2-0.25 mm), micro (0.25-0.053 mm) and silt-clay sized (<0.053 mm) aggregates with increase in heating temperature. Error bars represent standard error where n=3.

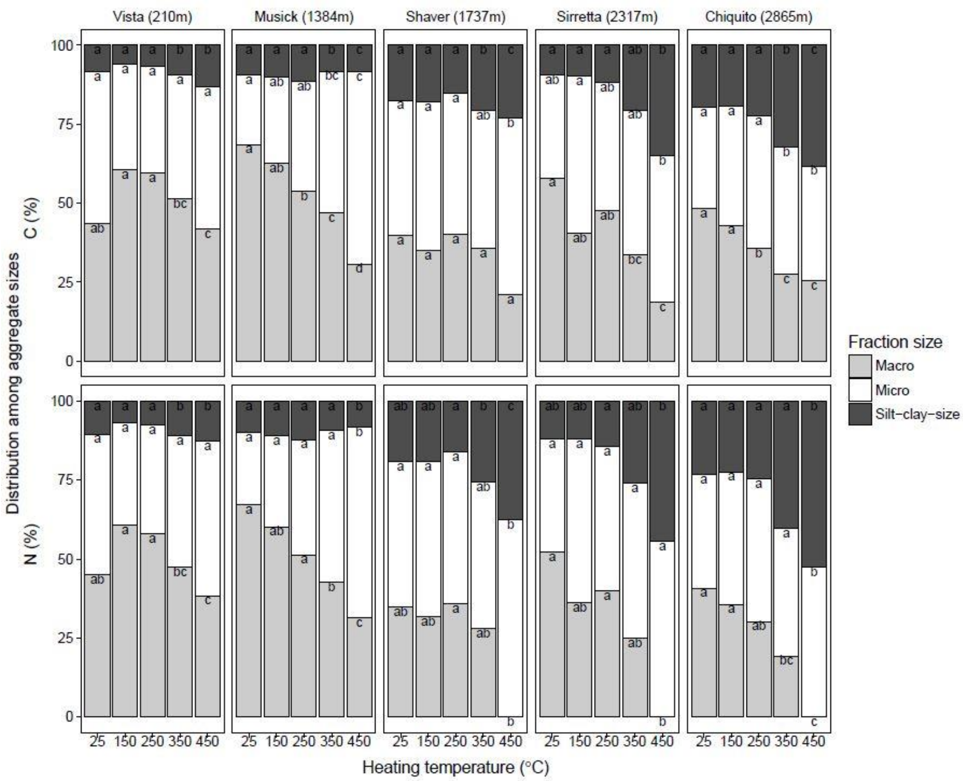

Figure 4: C and N distributions in macro (2-0.25 mm), micro (0.25-0.053 mm) and silt-clay sized (<0.053 mm) aggregates.

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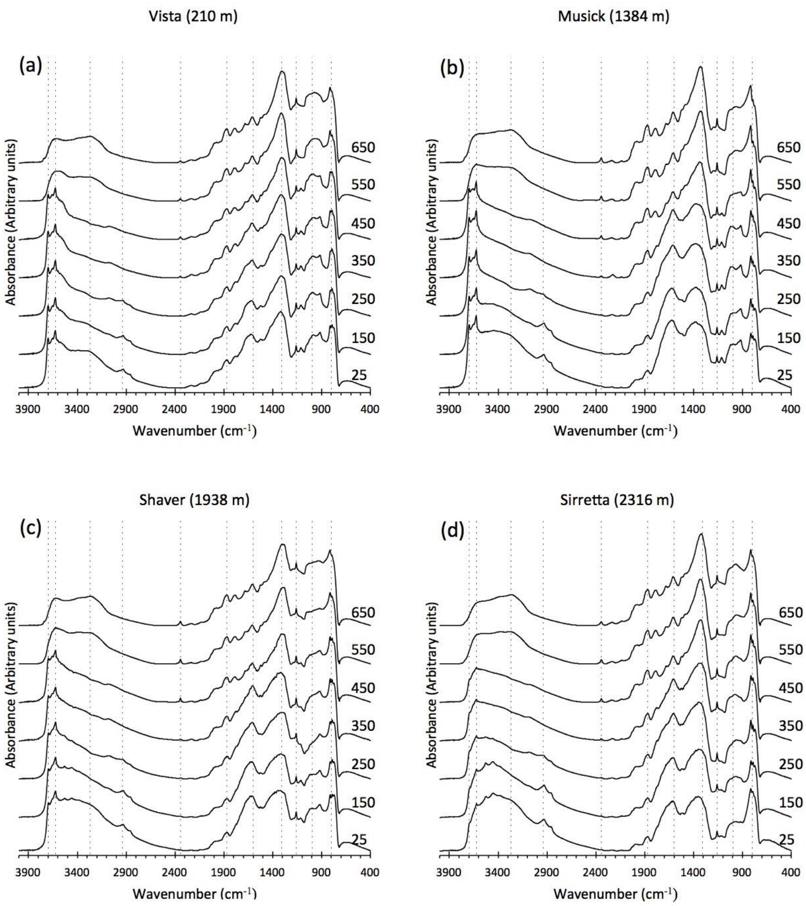

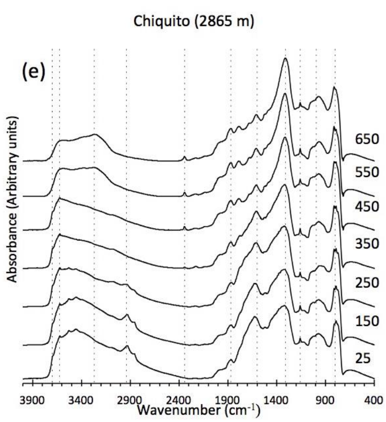

Absorption peaks (cm^{-1})	Peak assignment
3700, 3625	O-H
3270	O-H, N-H (H bonded)
2940	C-H (Aliphatic)
2345	C≡N
1870	C=O
1600	C=C, C-O, C=O
1310	C-H
1159, 995	Ester, Phenol, C-O-C, C-OH
800	C-H (Aromatic)

Figure 5: FTIR spectra at the different heating temperatures.

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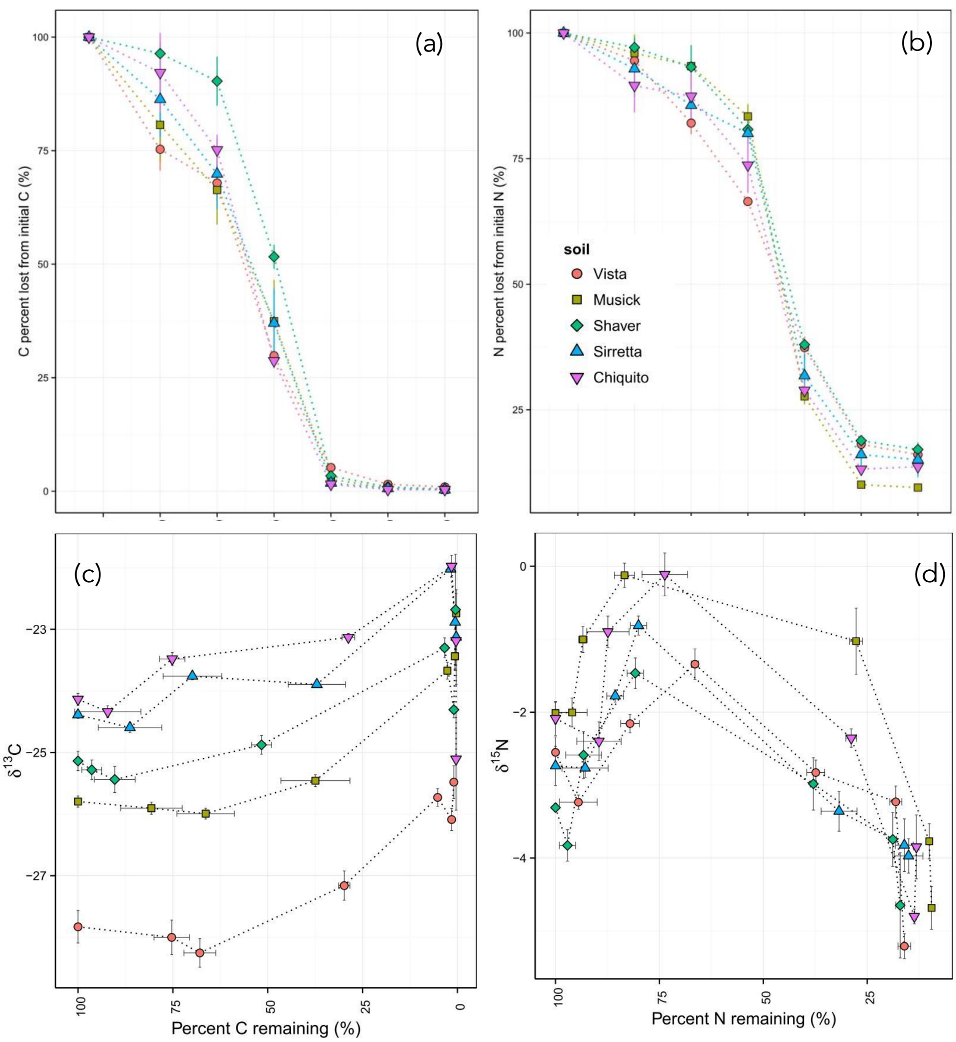

Figure 6: (a) Percentage of C and (b) N loss with heating; and (c) change in $\delta^{13}C$ and (d) $\delta^{15}N$ versus percent of total C and N lost from soils (error bars represent standard error where n=3).

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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)

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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2 Table 3 Linear correlation coefficients of changes in soil properties with changes in C
3 concentration

Soil	Correlation coefficient (r^2) values					
	Mass loss	**SSA**	**Aggregate Stability**	**pH ($CaCl_2$)**	**CEC**	**N concentration**
Vista	0.74	0.73	0.21	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	0.47	0.67	0.87	0.86
Chiquito	0.82	0.62	0.78	0.88	0.44	0.87

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