Dear Antonio Jordan (Topical Editor),

Following is the revised version of manuscript with track-changes mark-up. We have included all the comments.

We appreciate the thoughtful comments.

Thank you,

Samuel Araya (on behalf of all authors)

Thermal alteration of soil physico-chemical properties: A systematic study to infer response of Sierra Nevada climosequence soils to forest fires.

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Abstract

Fire is a common ecosystem perturbation that affects many soil properties. As global fire regimes continue to change with climate change, we investigated thermal alteration of soils' physical and chemical properties after they are exposed to a range of temperatures that are expected during prescribed and wildland fires. For this study, we used topsoils collected from a climosequence transect along the Western slope of the Sierra Nevada that spans from 210 to 2865 m.a.s.l. All the soils we studied were formed on a granitic parent material and had significant differences in soil organic matter (SOM) concentration and mineralogy owing to the effects of climate on soil development. Topsoils (0 - 5 cm depth) from the Sierra Nevada climosequence were heated in a muffle furnace at six set temperatures that cover the range of major fire intensity classes (150, 250, 350, 450, 550 and 650 °C). We determined the effects of heating temperature on soil aggregate strength, aggregate size distribution, specific surface area (SSA), mineralogy, pH, cation exchange capacity (CEC), and carbon (C) and nitrogen (N) concentrations. With increasing temperature, we found significant reduction of total C, N and CEC. Aggregate strength also decreased with further implications for loss of C protected inside aggregates. Soil pH and SSA increased with temperature. Most of the statistically significant changes ($p < 0.05$) occurred between 350 and 450 °C. We observed relatively smaller changes

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at temperature ranges below 250 °C. This study identifies critical temperature thresholds for significant physico-chemical changes in soils that developed under different climate regimes. Our findings will be of interest to studies of inferences for how soils are likely to respond to different fire intensities under anticipated climate change scenarios.

Keywords

Chemical properties, Climate change, Climosequence, Fire intensity, Physical properties, Soil heating

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). 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.

Even though humans are responsible for causing a substantial proportion of vegetation fires (Caldararo, 2002), vegetation fires are also natural phenomena with an important role in maintaining the health of many ecosystems around the world (Harrison et al., 2010). In the Sierra Nevada, vegetation fires have a major influence on the landscapes (McKelvey et al., 1996).

In addition to alteration of vegetation, 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; Mataix-Solera et al., 2011; Rein et al., 2008). Soil temperature thresholds for some important soil transformations are illustrated in Figure 1.

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). Fires also impact soil by altering and removing above-ground 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 soil properties. Here we aim to determine how the same input of energy from fires affects topsoils that vary significantly based on carbon content, mineralogy, and associated soil physical and chemical properties. The inferences derived from this work are essential for determining how changing climate regimes (and associated changes in vegetation dynamics and soil properties) are likely to influence the response of topsoil to wild- and prescribed-fires. 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 soil physico-chemical properties associated with different fire heating temperatures; (2) identify critical thresholds for major changes in soil-physico-chemical properties for soils that significantly vary based on organic matter properties, texture, mineralogy, and other properties.; and (3) infer the implications of changing climate on topsoil physico-chemical 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 scenarios.

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 2); 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.

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).

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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, 1995). 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 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).

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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

To investigate the effect of heating temperature on physico-chemical properties of soils with significantly different carbon contents, mineralogy, and overall development, we collected top soils (0 to 5 cm depth) from five sites. Triplicate samples, approximately 10 m apart, were collected from each site. 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).

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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 only limited by the availability of space, the furnace was not sealed 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; 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

Soil color was measured using the Munsell Color Charts. Dry color was measured from air-dried samples and moisture was added to same sample for moist color measurement. 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). 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).

Water-stable aggregate percent was measured by wet-sieving methods of Nimmo and Perkins (2002). For this procedure, a wet-sieving apparatus with 0.25mm mesh size sieve is used (Eijkelkamp Agrisearch Equipment, Giesbeek, The Netherlands). Four grams of soil sample was placed into a sieve and pre-wetted slowly from a moist tissue paper placed beneath the sieve. The soil sample was then wet-sieved with an up-down motion at a rate of 35 cycles per minute and a vertical distance of 1.3 cm. Mass of the soil passing through the sieve was recorded (M_1) after evaporating the supernatant water in oven. The remaining soil sample was subjected to a second round of wet-sieving using a dispersing solution (2 gL^{-1} of sodium hexametaphosphate for the soils with pH >7 and 2 gL^{-1} NaOH for the soils with pH <7) until all particles smaller than the sieve screen opening pass. Mass of the soil that passed through the sieve in the second round of sieving (M_2) was also determined by evaporating supernatant solution in oven and subtracting the weight of the dispersing-agent. The water-stable aggregate (WSA) fraction was calculated as:

$$WSA = \frac{M_2}{M_1 + M_2} \times 100\%.$$

Specific surface area was measured using automated N_2-BET analyzer (Micromeritics Tri-Star 3000, Micromeritics Instrument Corporation, Norcross, GA, USA). For this procedure, approximately 1 g of soil was oven dried at 60 °C for 36 hours and out-gassed for another 30 minutes using a flow of N_2 gas with outgassing station mantle set to a temperature of 105 °C. Measurement was done using ultra-high purity N_2 gas and the instrument was set to use seven-point N_2-BET adsorption isotherm measurement from which the Micromeritics software calculated specific surface area.

Soil mineralogy was measured using X-ray diffraction analysis (XRD) using PANalytical Xpert Pro diffractometer (PANalytical Inc., Westborough, MA, USA). We used the PANalytical Xpert Pro software for identification of mineral phases and Rietveld refinement for quantification (Rietveld, 1969; Schulze and Dixon, 2002). Soil samples were ground to a fine powder consistency using a ball-mill (8000M MiXer/Mill, with 65 ml stainless steel grinding vial set, SPEX SamplePrep, LLC, Metuchen, NJ, USA) and oven dried at 60 °C for over 36 hours. Samples were scanned at generator setting of 45 mA by 40 kV. Scan start position was set to 5° 2θ and end position was set to 120 2θ. Scan step time was set to 10 seconds with a step interval size of 0.0170° 2θ. At least two replicate measurements were run for each sample and samples were measured in random order.

Soil pH was measured using 1:2 solid:solution ratio mixtures in a deionized water and 0.01 M $CaCl_2$ solution. Five grams of soil was mixed by shaking with 10 ml of solution and allowed to stand for 30 minutes, stirring every 10 minutes. The pH reading was taken by placing electrodes directly in the sediment slurry immediately after stirring (Thomas, 1996).

Cation exchange capacity (CEC) was measured by the barium exchange method. Barium was used to quantitatively displace soil exchangeable cations, and excess barium was removed by four deionized water rinses. A known quantity of calcium is then exchanged for barium and excess solution calcium is measured in order to determine CEC by the difference in the quantity of the calcium added and the amount left in the resulting solution. The method has a detection limit of 2.0 cmol$_c$/kg (Rible and Quick, 1960).

Elemental concentrations of carbon (C) and nitrogen (N) 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, the soil samples were ground to a powder consistency using 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 and to determine moisture content. 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.

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). ANOVA was implemented using aov() function and Tukey's HSD test subsequently implemented on the aov fit by TukeyHSD() function. The ANOVA tables and Tukey's HSD comparison of means tables are given in supplementary material. The ordinary linear regression technique was used to examine relationships between soil properties.

3 Results

3.1 Soil color

We observed a marked soil color change, as inferred using the Munsell color system, with increasing heating temperature (Figure 3). As the heating temperatures increased, the soils initially got darker, reaching their darkest color at mid temperatures (250 - 350 °C when measured in dry soil, and 250 - 450 °C when soils were moist). At higher temperatures, the soils became markedly lighter and became increasingly reddish in color (with hue changing from 10YR to 7.5YR at temperatures above 550 °C). Color change patterns were similar for both, dry and moist soil measurements except for the marked color change occurring at 450 °C when dry and at 550 °C when moist. Across the heating temperature range, Vista (210 masl) soils showed the least pronounced increase in darkness at 350 ºC while Shaver (1737 masl) soils showed the most pronounced darkening at that temperature range (from dry color of 10YR 5/2 unburned to 10YR 3/3 by 350 ºC). At higher temperatures, Musick soils (1384 masl) showed the largest change in dry soil color going from 10YR 2/2 at 350 ºC to 7.5YR 6/6 at 650 ºC.

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3.2 Mass loss

Mass loss was proportional to heating temperature in all the soils. For the high and low elevation soils, statistically significant mass loss, compared to unburned soils, was observed above 350 ºC. In contrast, significant mass loss was observed for the two mid elevation soils of Musick (1384 masl) and Shaver (1737 masl) starting 250 ºC. There was no significant mass loss at temperatures above 450 ºC for all soils. For all our soils, the steepest mass loss was observed between temperatures of 250 and 450 °C (Figure 4). Vista (210 masl) soils showed the lowest mass loss with heating while Musick soils (1384 masl) showed the highest mass loss with heating.

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3.3 Aggregate stability and size distribution

Aggregate stability generally decreased with temperature for all soils. While aggregate stability decreased in an almost uniform manner with increase in temperature for the lower to mid elevation soils, the higher elevation Sirretta (2317 masl) and Chiquito (2865 masl) soils showed a stepwise decrease in aggregate stability at 250 °C and 350 °C respectively. At higher

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temperature heating aggregate stability for the two soils showed only a small decrease from these two temperatures. Statistically significant decrease in aggregate strength, compared to unburned samples, was observed only at higher temperatures above 350 °C for Sirretta (2317 masl) and Chiquito (2865 masl) soils, and above 450 °C for Musick (1384 masl), Vista (210 masl) and Shaver (1737 masl) soils (Figure 4).

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Although not statistically significant, all soils showed a decrease in macro-aggregate fraction accompanied by increase in micro-aggregate and silt-clay sized fractions (Figure 5). For the two lower elevation soils (Vista and Musick) the decrease in macro-aggregate fraction was over 10% and less than 5% for all the other soils. Only Musick (1384 masl) soils showed a statistically significant decrease in macro-aggregate fraction between 150 and 350 °C temperatures.

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3.4 Specific surface area

For all soils, we observed a stepwise increase in specific surface area (SSA) for samples were heated to between 250 to 450 °C (Figure 4). Changes in SSA between soils heated below 250 °C and those heated above 450 °C were statistically significant at $p<0.05$ for all soils, except the high elevation soils Sirretta (2317 masl) and Chiquito (2865 masl). Sirretta soils showed a lot of variability and did not show any significant change in SSA throughout the temperature range while the Chiquito soil showed statistically significant increase between low temperature 150 – 250 °C and higher temperature 350 – 550 °C range. The pattern of change in SSA with temperature was similar for all soils. The lowest SSA was recorded for all soils when soils were heated at 250 °C, and highest SSA was observed at 350 °C (for Musick and Chiquito soils) or 450 °C (Vista and Shaver soils).

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3.5 Soil mineralogy

The bulk soil XRD results of changes in soil mineralogy in response to heating are presented for basic mineral groups as: feldspar (microcline and orthoclase); plagioclase (albite and oligoclase); amphibole; mica/illite (biotite); kaolinite; gibbsite; and expandable phyllosilicate (montmorillonite and vermiculite). We identified vermiculite with low confidence, since we did not correct with oriented clay treatments, hence it is not certain if the identified peaks are indeed representative of vermiculite, chlorite, or both. The XRD diagrams showed some significant transformations in soil mineralogy with heating, with shifted peaks at higher temperatures suggesting transformation of clay minerals. Layer silicates appeared to collapse structurally, possibly due to dehydration and the removal –OH (Figure 6). Summary mineral composition changes identified from XRD analysis using Rietveld method are presented in Figure 7. From the minerals identified, the concentration of kaolinite was the most affected by heating. In all soils, concentration of kaolinite showed a decrease when heated to 550 ºC and above. Gibbsite was also not be detected in soils heated to > 450 ºC. Furthermore, mica/illite, plagioclases and amphibole mineral groups changed consistently with increasing temperature. The largest change in soil mineralogy with heating was observed for the mid-elevation Music (1384 masl) and Shaver (1737 masl) soils that are also among the most developed soils with highest proportions of the 1:1 clay minerals (kaolinite).

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3.6 Soil pH

With increase in temperature, all soils showed a similar pattern of increase in pH (Figure 8). For all soils the largest increase in pH (2.5 – 5 units) occurred between 250 and 450 °C. All the soils started out with slightly to moderately acidic pH and with the exception of Chiquito (2865) soils. Yet, all soils became alkaline at temperatures above 450 °C. The largest increase in pH was observed for the Musick (1384 masl) soils which reached a pH of 10 at temperatures above 550 °C.

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3.7 Cation exchange capacity

The CEC of studied soils ranged from an average of 6 cmol$_c$/kg for Chiquito (2865 masl) soils to 25 cmol$_c$/kg for Musick (1384 masl) soils. With increase of heating temperature all soils showed continued decrease of CEC. With the exception of Musick (1384 masl) soils, CEC eventually dropped to below our detection limit (2 cmol$_c$/kg) at temperature above 550 °C (Figure 8). For the poorly weathered Chiquito (2865 masl) soils, CEC was below 2 cmol$_c$/kg at temperatures 250 °C and above. For the rest of the soils, statistically significant changes in CEC ($p<0.05$) occurred at 450 °C with the exception of Musick (1384 masl) soils which showed statistically significant drop at 250 °C and again 350 °C. At 350 °C, all the soils except Musick

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(1384 masl) showed a slightly higher CEC than at 250 °C thus interrupting continuous pattern of CEC decrease with increase in temperature.

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3.8 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 (Figure 9) 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 9).

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The loss of C and N from soils due to heating showed a similar response among all five soils. 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. 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 ºC. After heating at 450 °C, all soils lost more than 60% of the initial soil N and 85% by 550 °C.

4 Discussion

The topsoil layer is most affected by extreme temperature during vegetation fires. Our results show significant changes in soil properties as a result of temperature exposure. Our findings demonstrate that alterations and loss of SOM in topsoil because of heating, rather than alterations to soil minerals, was the most important driver for the observed changes in soil physico-chemical properties. Our XRD analysis shows notable changes in soil mineralogy only after the soils were heated to about 450 to 550 °C (Figure 7). In upland ecosystems, such as the Sierra Nevada Mountains, the soils typically have low clay content and low concentration of secondary minerals (Neary et al., 1999; Ubeda and Outeiro, 2009). In addition, these upland temperate ecosystems also tend to have relatively high concentration of SOM, including a fairly well developed O-horizon. Consequently, strong relationships are observed between SOM concentrations and the soils' physical and chemical properties. Simple linear regression analyses between C concentration changes and other soil physical and chemical changes for our study soils shows that more than 80% of the variability in mass loss, aggregate strength, SSA, pH, CEC and N concentrations are associated with changes in C concentration at the different heating temperatures. Table 3 summarizes the correlation coefficients of soil property changes with changes in C concentration.

The changes in soil color observed were consistent with the charring of SOM which leads to darkening of the brownish color of the soils. At temperatures over 450 °C, the near complete removal of SOM by combustion and the addition of ash products likely explains the observed lighter color of soils. The increase in Munsell chromas and reddening of soils at these high temperatures has been noted in previous works (e.g. Giovannini et al., 1988; Ketterings and Bigham, 2000; Ulery and Graham, 1993) and is likely a result and the transformation of iron oxides that occurs during heating.

The extent of mass loss in topsoil layers due to vegetation fires is strongly correlated to SOM combustion (Rein et al., 2008). In all of our soils, statistically significant mass loss ($p<0.05$) occurred within the temperature ranges of SOM combustion, between 250 ºC to 450 ºC. Proportion of soil mass loss with temperature was proportional to initial C concentration of the soils. For example, Musick (1384 masl) soils which had the highest initial C concentration (7.7%) had the steepest soil mass loss and lost 15% of mass at 550 ºC, while Vista (210 masl) which had less than 2% C concentration showed the smallest mass loss losing less than 5% mass at even at the highest temperature. Mass loss at temperatures below 250 ºC is likely driven by dehydration processes rather than by loss of SOM. On the other hand, mass loss at temperatures above which SOM combusts, >450 ºC, is likely driven by charring, ashing and volatilization processes.

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Most studies report significant soil aggregate stability reduction with fire heating (e.g. Arcenegui et al. (2008); Zavala et al. (2010)), however, contrasting findings are also reported in other studies. Mataix-Solera et al. (2011) explains that increase in aggregate strength with heating is possible in clay-rich soils where the main cementing agents are inorganic minerals such as calcium carbonates and metallic oxides which would fuse under fire heating increasing aggregate stability. Another possibility is, soils which initially had weak hydrophobicity may show increased aggregate stability due to increase in hydrophobicity, in such cases however aggregate strength would decline at higher severity fires as hydrophobicity is destroyed.

Aggregate stability in all our soils generally decreased with increase in heating temperature. Although we did not find a statistically significant difference, it is worth noting, that the lowest elevation soil (Vista, 210 masl) showed a trend of aggregate stability increase up to 350 °C while the high elevation soils, Sirretta (2317 masl) and Chiquito (2865 masl), showed a slight increase in aggregate stability at 150 °C. An increase in hydrophobicity at these temperatures is the most likely cause, substantial hydrophobicity was apparent with Chiquito soils (2865 masl) heated at 250 °C where resistance to slaking was remarkably evident during the aggregate stability test.

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Soil specific surface area (SSA) is an important soil property that affects soil adsorption, ion exchange capacity, reactivity, aggregation and porosity (Feller et al., 1992). SSA of soil is largely dictated by clay-size particles and SOM (Carter et al., 1986). The increase in SSA with heating that we observed in this work is most likely the result of physical disintegration and charring of SOM, especially at temperatures below 500 °C. Changes in soil mineralogy are not likely to be responsible for the changes in SSA we observed. XRD analysis showed notable mineralogical changes only at temperatures above 450 °C where the kaolinite peak disappeared at 550 °C and the phyllosilicate peaks were diminished or disappeared at 450 – 550 °C (e.g. Figure 6). Because of combustion, pyrolysis and thermal dehydration, larger organic matter particles are likely to have fragmented and reduced in size with an increase in heating temperature leading to increase in bulk soil SSA. Furthermore, thermal alteration and the removal of organic matter from mineral surfaces may have increased surface area by reducing the size of particles. At higher temperatures (>450 °C), XRD spectra showed some collapse of mineral complexes through dehydration and de-hydroxylation of clay minerals that may reduce mineral particle size and increase surface area. These changes in mineralogy might have a significant effect on SOM. A study by Rosa et al. (2013) found that soils released more organic compounds during pyrolysis when the soils were treated with HF acid, suggesting that mineral complexes play a role in protecting organic compounds from pyrolysis. The collapse of mineral complexes we observed and the decrease in aggregate strength is likely to have enhanced thermal oxidation of SOM. However mineralogical changes would play a more important role affecting soil properties at these high temperatures since SOM has been almost completely removed.

Soil pH generally increases with fire heating (Badía and Martí, 2003b; Chandler et al., 1983; Ubeda and Outeiro, 2009). In a soil heating experiment similar to our study, Fernández et al. (1997) observed a pH increase of 1.7 at 350 °C and 2.35 at 490 °C. Such increase in soil pH has been attributed to the denaturation of organic acids, release of base cations from combustion (K- and Na-hydroxides, Mg- and Ca-carbonates), deposition of ashes, and loss of hydroxyl groups from clays (Badía and Martí, 2003b; Certini, 2005; Ulery et al., 1993). In our soils, the higher elevation soils (Shaver, Sirretta and Chiquito) showed a statistically insignificant decrease of 0.3 to 0.5 pH units (measured in water) at 250 °C. The change in pH in our high elevation soils was consistent with previous results of Badía and Martí (2003b); Terefe et al. (2008) that found similar initial decrease. Terefe et al. (2008) hypothesized that this may be due to the combined effect of desiccation and heating effect which favor proton-reducing oxidation reactions. And the fact that this initial increase occurs below the temperature for the start of combustion of organic acids means contribution of SOM to this pH increase (organic acid denaturation and ash liming effect) was absent at this temperature. In a similar heating experiment Badía and Martí (2003b) found an increase in electric conductivity and soluble Ca along with decrease in pH at 250 °C. Such increase in soluble cations might explain our findings where we observed a decrease in pH when measured in water but not in $CaCl_2$ suggesting that the decrease in pH might have to do with increase of soluble salts with heating up to 250 °C.

The capacity of soil to exchange positively charged ions between soil and soil solution (CEC) decreased with increasing temperature. CEC of soils is a result of surface charges associated with secondary clay minerals and SOM (Sparks, 2005), and in our study soils, Dahlgren et al. (1997) had previously reported a strong relationship of CEC with soil organic carbon and clay concentrations. Different authors have attributed loss of CEC during heating mainly with loss of SOM (Fernández et al., 1997; Ubeda and Outeiro, 2009) partly because CEC loss starts to be observed at temperatures above 200 °C with little or no decrease at lower temperatures where SOM is not affected (Nishita and Haug, 1972; Soto and Diazfierros, 1993). The slight increase of CEC we observed at 350 °C may be due to the steep increase of specific surface area at that temperature (Figure 4). The additional surface for cation adsorption might have to an extent compensated for the loss of SOM at that temperature. Furthermore, the contribution of surface oxidation of char products has been shown to increase CEC per unit C (Liang et al., 2006) because of the almost complete loss of C at temperatures above 450 °C and very little loss at temperatures below 250 °C. The soils most likely had highest concentration of charred SOM at 350 °C temperature.

4.1 Importance of the 250 – 450 °C range

Taking maximum surface temperature as a metric of fire intensity, low intensity fires reach surface temperatures of up to 250 °C, medium intensity fires reach surface temperatures of 400 °C, and high intensity fires reach surface temperatures above 675 °C (Janzen and Tobin-Janzen, 2008). In this study, the most significant changes of soil chemical properties occurred between 250 and 450 °C. Figure 10 illustrates the changes between unburned and 650 °C burned soils and the amount of change that occurred within 250 to 450 °C heating temperature for a range of the variables discussed above. In all cases, the change in the 250 – 450 °C range accounts for most of the total change observed during our heating treatments. Among the variables we investigated in this study, we observed changes along two general lines: (1) mass loss, SSA and pH which showed a progressive increase with heating temperature, and (2) %C, %N, C:N ratio, CEC, and wet aggregate stability that showed a progressive decrease with increase in temperature (Figure 10), with the most significant changes in all cases being recorded in all soils between 250 – 450 °C.

Temperatures below 250 °C are very critical for many processes, water is lost at 95 °C and this has a significant effect on soil heat conduction and soil biota (Janzen and Tobin-Janzen, 2008). However, we found that temperatures below 200 °C have very little effect on quality or quantity of SOM. This implies that lower intensity fires, such as typical prescribed fires, where soil surface temperatures do not exceed below 250 °C temperature contribute little to soil C loss. Similarly, temperatures above 500 °C do little to SOM, which already has been lost or transformed into a pyrogenic product. The effect on soil inorganic particles starts at high temperature but the significance of change on minerals is not as large (Figure 1). Hence, we found that the most important soil changes occur the 250 – 450 °C range.

Important modifications of fire conditions that still allow for comparison of responses of different types of soils have to be adopted to conduct the type of heating experiments that we undertook in this work. A heating rate of 3 °C min^{-1} is common in laboratory soil heating studies, often because of technical considerations and because such a slow rate prevents sudden combustion which otherwise would happen as soil's ignition temperature is reached at about 220 °C (Fernández et al., 1997, 2001; Varela et al., 2010). However, it is important to recognize that during vegetation fires the rate of temperature increase experienced by the topmost layer of soil that is exposed to fire can be significantly higher. The rate of heating alone might have additional significant effects on soil properties beyond what we observe here. For example, Albalasmeh et al. (2013) found that slow rate of heating underestimates soil aggregate destruction of moist soils due to a slower buildup of pore-pressure.

4.2 Climate Change Implications

Investigation of the response of climosequence soils to different heating temperature in this study enables us to infer how changes in climate (and associated changes in soil properties) are likely to alter the effect of fires on topsoil physical and chemical properties. The overall pattern of soil property changes with increase in heating temperature were similar for all soils, however, the magnitudes of change were variable. Hence, these findings lead us to conclude that climate change is likely to alter the response of topsoil properties to different fire regimes. 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., 2012). 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). Improving our understanding of how topsoil properties are likely to respond to changes in climate becomes even more critical when we recognize that C concentration in soil is likely to respond quickly to changes in climate, compared to other soil physical and chemical properties (Berhe et al., 2012). Furthermore, the long-term fate of soil carbon in fire-affected ecosystems is also likely to be accompanied by changes in microbial community composition and OM decomposition kinetics (Holden et al., 2015; Tas et al., 2014) which are likely to have further implications for nutrient availability post-fire (Johnson et al., 2007b; Johnson et al., 1997).

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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 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., 2007a; 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 (Stacy et al., 2015). Moreover, the important differences in changes in pH, mineralogy, CEC in response to heating at different temperatures that we observed for soils along the climosequence suggest that changes in temperature are likely to lead to different effects on soil chemical properties in soils after fires.

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 physical and chemical properties 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. Soil aggregate stability, CEC, and C and N concentrations significantly decreased with increase in heating temperature while soil pH and SSA significantly increased. The most important effect of heating on soil mineralogy as observed by XRD analysis was the disappearance of kaolinite, which was undetectable at temperatures above 500 °C.

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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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
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

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 topsoil 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)

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

Figures

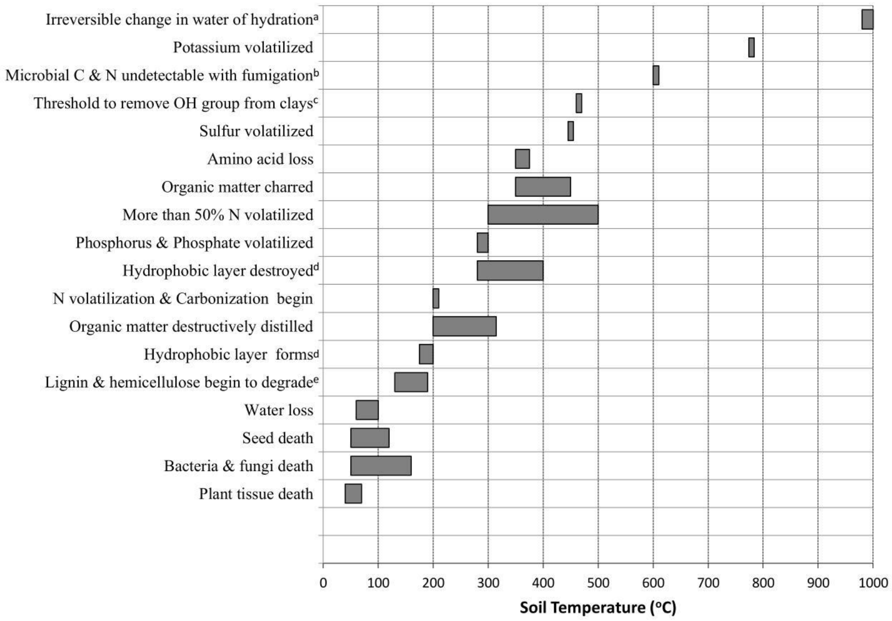

Figure 1: Temperature thresholds and ranges associated with fire heating. Figure adopted and expanded from Massman et al. (2010). ([a] DeBano et al. (1977), [b] Diaz-Ravina et al. (1992), [c] Giovannini et al. (1988), [d] DeBano (2000), and [e] Knicker (2007))

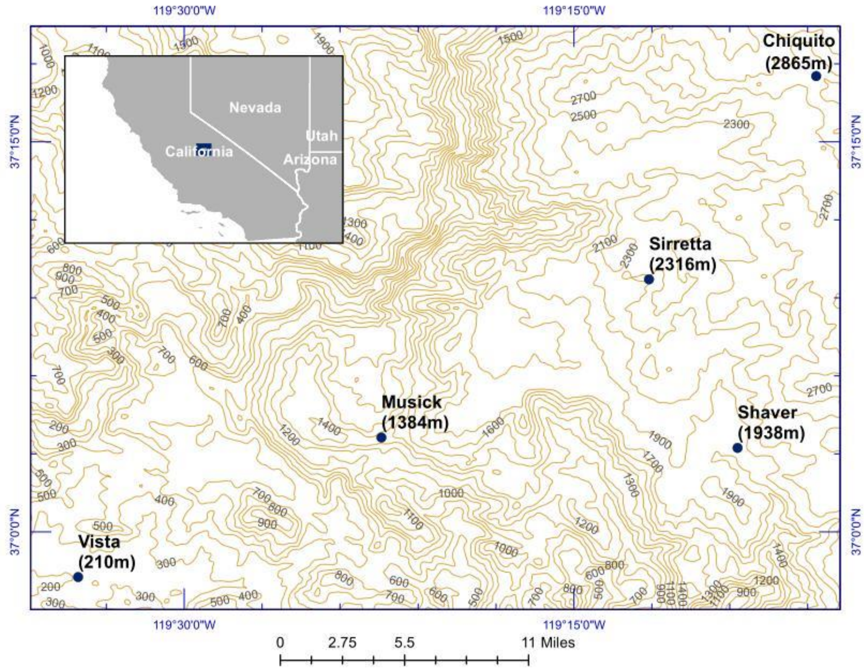

Figure 2: Map of the five sampling sites along elevational transect in the Western Sierra Nevada, California (Base map from U.S. Geological Survey, 2015)

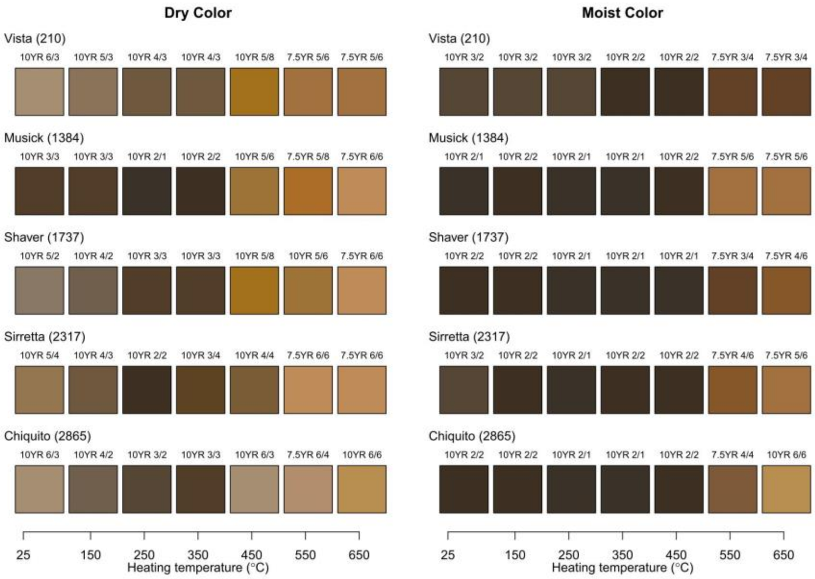

Figure 3: Soil color change across heating temperatures. Colors produced from CIExyY colorspace equivalents to Munsell colors (Munsell Color Science Laboratory). CIExyY colors were converted to RGB system (Rossel et al., 2006; UC Davis Soil Resource Laboratory) and visually compared with Munsell Soil Color book for plotting.

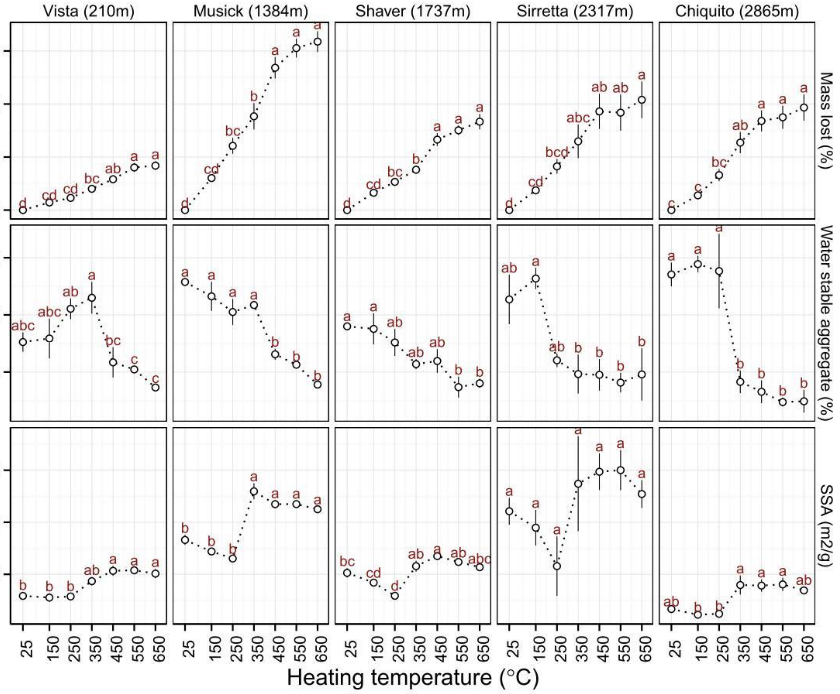

Figure 4: Percent mass lost, water-stable aggregate percent and specific surface area changes with increase in heating temperatures. Error bars represent standard error where n=3. Different letters represent significantly different means ($p<0.05$) at temperature after Tukey's HSD testing.

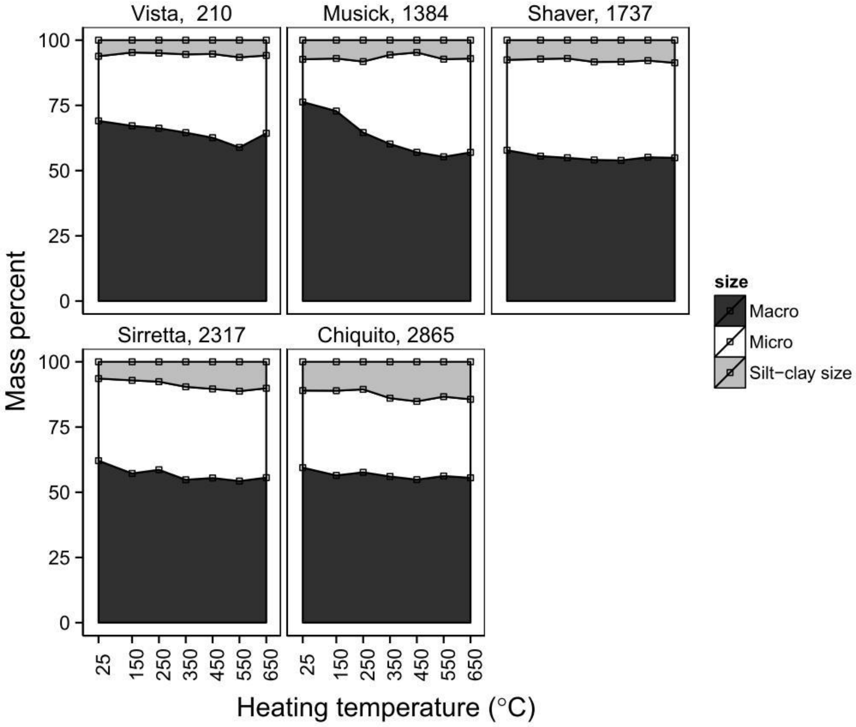

Figure 5: Weight fraction of aggregate sizes: macro (2-0.25 mm), micro (0.25-0.053 mm) and silt-clay (<0.053 mm) sizes.

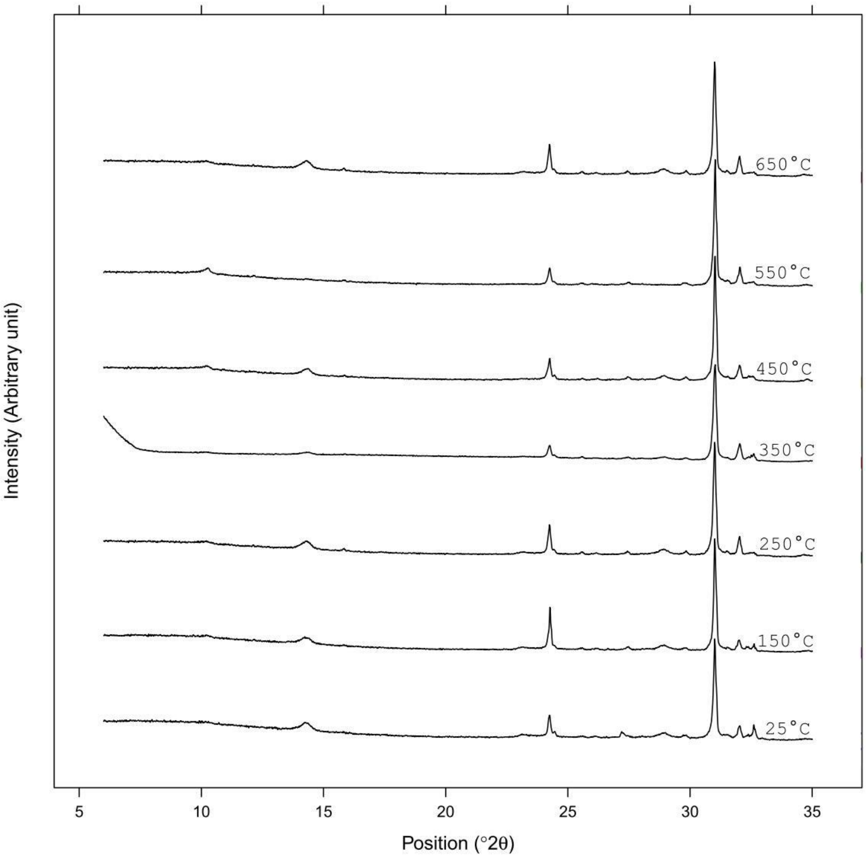

Figure 6: XRD diagram for Music series soils.

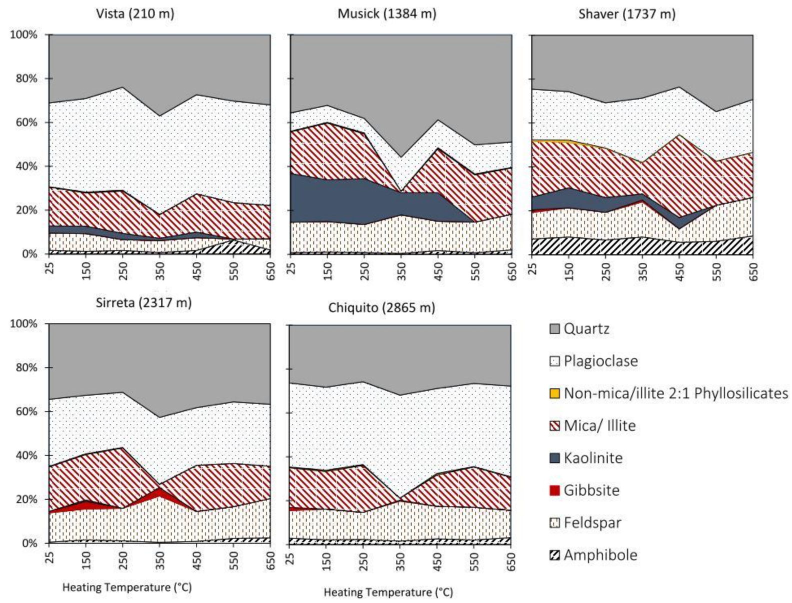

Figure 7: Relative amounts of minerals identified from powder XRD.

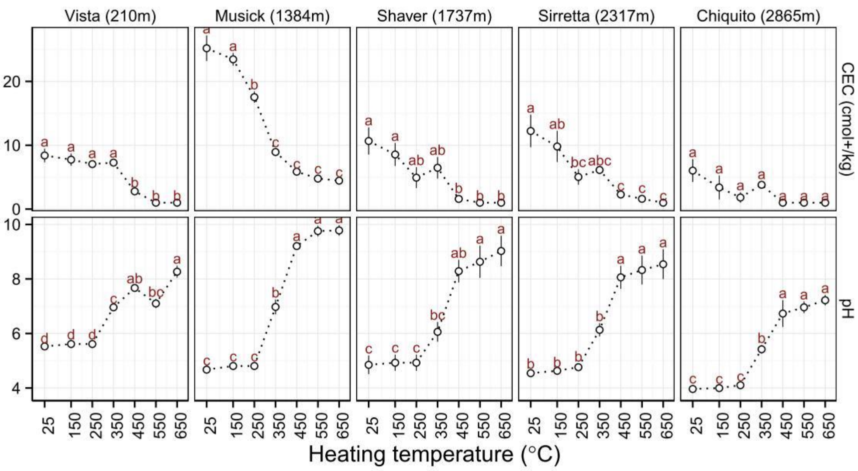

Figure 8: pH (geometric means) and cation exchange capacity (adjusted for mass loss) changes with increase in heating temperature. CEC values below the 2 cmol$_c$/kg are assigned a value of 1 for plotting. Error bars represent standard error where n=3. Different letters represent significantly different means (p<0.05) at each temperature after Tukey's HSD testing.

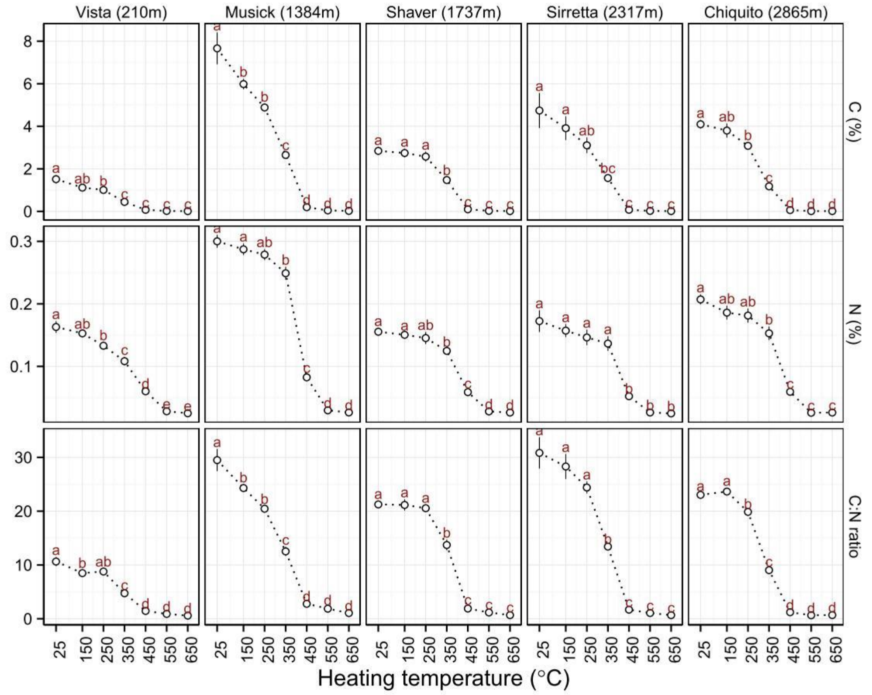

Figure 9: Carbon concentration, Nitrogen concentration and C:N atomic ratio changes with increase in heating temperature. Error bars represent standard error where n=3. Different letters represent significantly different means (p<0.05) at each temperature after Tukey's HSD testing.

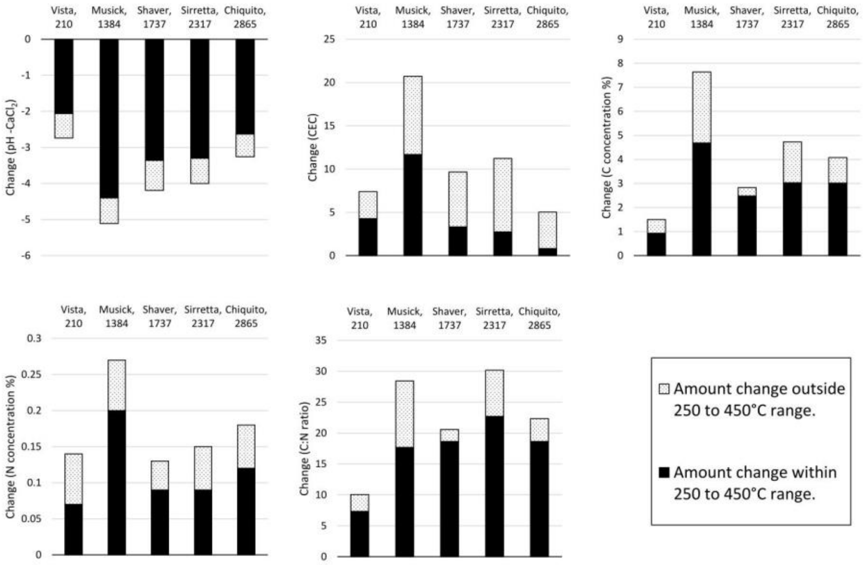

Figure 10: Total amount of change from unburned to 650 °C combusted soils showing amount of change within the 250 to 450 °C range.