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Responses of soil physico-chemical properties to combustion: a space for time substitution study to infer how changes in climate are likely to affect response of topsoil to 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 the effect of fire heating temperatures on the physical and chemical properties of soils across 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 have significant differences in soil organic matter (SOM) concentration and mineralogy owing to the effects of climate on soil development. The dominant vegetation from lowest to highest elevation across the transect range from oak woodland, oak/mixed-conifer forest, mixed-conifer forest and subalpine mixed-conifer forest. 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 fire 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 increase of 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

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increased with increase in temperature. Most of the statistically significant changes ($p < 0.05$) occurred at temperature ranges of 350 to 450 °C. We observed relatively smaller changes at typical temperature ranges of prescribed fires (i.e. less than 250 °C). This study identifies critical combustion temperature thresholds for significant physico-chemical changes in soils that developed under different climate regimes, allowing 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 ""wildland" more normal 63,000 wild-land 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 on global fire regimes are anticipated because of climate change including an increase in frequency of fires in the coming decades (Westerling et al., 2006a;Pechony and Shindell, 2010). However, as of yet, we only have limited understanding of how changes in fire regimes and climate change are likely to interact to influence the response of to topsoils in fire affected ecosystems to wild- or prescribed-fires. "wild or prescribed fires"

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. Essentially repeats previous sentence. REF needed ~~Many ecosystems in the US and across the world depend on fires to maintain ecosystem health and productivity.~~ In the Sierra Nevada, vegetation fires have a major influence on the landscapes (McKelvey et al., 1996). Lightning fires were historically common in the dry season in the upland forests of the Sierra

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Can you be more specific?

Nevada Mountains and also fires were used by some of the Native American tribes to modify the environment for their needs (Parsons and van Wagtendonk, 1996).

In addition to alteration of ~~the overland~~ vegetation, fire also significantly affects the physical, chemical and biological properties of soils. The degree of alteration caused by fires depends on 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 of moisture content, texture and 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 (Badia and Marti, 2003;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;Rein et al., 2008;Mataix-Solera et al., 2011). Soil temperature thresholds for some important soil transformations are illustrated in Figure 1.

Need to define abbreviation at first use

Spaces needed where multiple refs listed - correct formatting throughout

Fires also impacts 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). Fires with longer durations are typically expected to have more impact on soil physical and chemical properties, and loss of SOM than fast-moving, high temperature fires (González-Pérez et al., 2004).

Slightly conflating temperature and RoS here, latter probably most important

The aim of this study is to investigate effects of combustion 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 ~~respond to combustion~~. 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 ~~in our study~~ 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

I like what the study is trying to do but at this point am rather skeptical of the climate angle. It seems to be more of a study of the effects of variation in simulated fire severity (~duration of heating of soil). Fire severity may change with a changing climate but also varies hugely between fires - the inference might be how varying fire severity (~ fire weather and fuel structure) would effect soils but it's something of a leap to go from this to climate change as there are likely to be complex interacting processes involved. To infer a climate effect you're at least two steps removed from the process you're studying (climate ~ fire weather ~ fire severity/behavior ~ soil chemico/physico/biological changes

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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

This repeats information presented earlier, can be cut.

~~The Sierra Nevada ecosystems are fire adapted systems. Fires are common perturbations that maintain ecosystem health and plant productivity in the region. However, over the last couple of decades, the frequency and severity of fires has been increasing due to changes in climate (Westerling et al., 2006b).~~ 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).

Soils from the lowest elevation site, Vista soils (210 m), 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 m) lie within oak/mixed-conifer forest (1008—1580 m) and mixed-conifer forest (1580—2626 m). These soils receive the highest biomass and litter fall. Shaver and Sirretta soils (2317m) fall within the mixed-conifer forest range zone while Chiquito soils (2865 m) lies within the subalpine mixed-conifer forest range (2626—3200 m). 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 parent material and are located in landscapes of similar age, relief, slope and aspect (Trumbore et al., 1996) formed on a granitic parent material with significant

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developmental differences attributed to climate. The soils at mid-elevation range (1000 to 2000 m) tend to be highly weathered while soils at high and low elevations are relatively less developed (Jenny et al., 1949;Huntington, 1954;Harradine and Jenny, 1958;Dahlgren et al., 1997). 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 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).

in what direction? step up or step down - please clarify

2.2 Experimental design and sample collection

To investigate the effect of combustion 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).

So soils were seived and dried prior to heating. To what extent does this effectively simulate what happens during a real fire? Seems to be it would have been better to examine changes over a range of moisture contents

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. 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 (Janzen and Tobin-Janzen, 2008;DeBano et al., 1977;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;Varela et al., 2010;Soto et al., 1991) The relatively slow

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Compare to heating rates seen in real fires?

heating rate of 3 °C min^{-1} is recommended for laboratory based fire simulation experiments (Giovannini et al., 1988;Terefe et al., 2008;Varela et al., 2010) to prevent sudden combustion when soil's ignition temperature is reached at about 220 °C (Fernández et al., 1997, 2001;Varela et al., 2010). The identical treatment of all soils, then allows for comparison of how the soils from the different climate regimes are likely to respond to fires. Furthermore, once the set temperature is reached, samples were exposed to that temperatures for a period of 30 minutes. This is approximately equivalent to the time it takes to burn off small dry logs (Stoof et al., 2010;Chandler et al., 1983) and 30 to 40 minutes has become the standard in laboratory soil heating experiments (for example Varela et al., 2010;Giovannini, 1994;Fernández et al., 2001;Zavala et al., 2010).

Presumably this occurs as material is oven dry, begs the question why use oven dry material?

Again, I would have preferred to see some study of the effects of heating duration. 30-40 minutes is a long time for a fire to be resident at a site. I warrant that it might approximate conditions under a smouldering log but then to what extent are you actually simulating changes more generally associated with a fire - logs occupy a small proportion of the soil surface.

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) using a wet-sieving apparatus (Eijkelkamp Agrisearch Equipment, Giesbeek, The Netherlands) with 0.25mm mesh size. Four grams of soil is weighed into sieve and slowly pre-wetted by capillary rise. Sample was then wet-sieved with an up-down motion with a vertical distance of 1.3 cm and a rate of 35 cycles per minute. Soil passing through sieves was collected in the cans and weighed after evaporating the supernatant water in oven (M_1). The samples remaining in the sieve were then subjected to a second round of wet-sieving using another set of cans filled with 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). Samples were wet-sieved until all particles smaller than the screen opening pass. Mass of soil collected in the second set of cans (M_2) was determined by evaporating supernatant solution in oven and subtracting the weight of the dispersing-agent. The water-stable aggregate fraction was calculated as:

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

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 ~~samples~~ was ~~were~~ oven dried at 60 °C for 36 minutes/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 seven point measurement. The isotherm is analyzed using Micromeritics software.

Soil mineralogy was measured from 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 (Schulze and Dixon, 2002;Rietveld, 1969). Soil samples were ground to fine powder consistency using 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 at a step interval size of 0.0170° 2θ. Two or three replicate measurements were run for each sample and samples were measured in random order. Why not consistent?

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 with 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,

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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 < 2 mm 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. The values for C and N concentration were corrected for oven dried weights by oven-drying subsamples at 105 °C.

Statistical Analysis

There's no need to engage in "sacrificial pseudoreplication" and average values for the cores. Why not fit a mixed effects model with core as a random effect?

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 ordinary linear regression technique was used to examine relationships between soil properties. All statistical analysis were performed using R statistical software (r-project.org).

Not the correct citation for R. Need to state what functions were used for the analyses

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). With increase in heating temperature, all the soils exhibited a similar trend in color change. As the heating temperatures increased, the soils initially got darker ~~with~~, reaching their darkest color in mid temperatures (250 - 350 °C when dry and 250 - 450 °C when 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 dry and for moist soils except for the marked color change occurring at 450 °C in dry soils and at 550 °C in moist soils. Across the heating temperature range, Vista (210m) soils showed the least pronounced increase in darkness at 350ºC while Shaver (1737m) soils showed the most pronounced darkening at that temperature range (from dry color of 10YR 5/2 unburned to 10YR 3/3 by 350ºC). At higher temperatures, Musick soils (1384m) showed the largest change in dry soil color going from 10YR 2/2 at 350 ºC to 7.5YR 6/6 at 650 ºC.

Slight change in language needed here to remind us that colours were measured when soils were dry and moist not treated when dry and moist

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

Results of the ANOVA (or mixed effects model need to be reported in full (i.e. d.f., F and P values)

3.3 Aggregate stability and size distribution

Aggregate stability generally decreased with temperature for all soils. While aggregate stability seemed to decrease in an almost uniform manner with increase in temperature for the lower to mid elevation soils, the higher elevation Sirretta (2317m) and Chiquito (2865m) soils showed a stepwise decrease in aggregate stability at 250 °C and 350 °C respectively. At higher 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 (2317m) and Chiquito (2864m) soils, and above 450 °C for Musick (1384m), Vista (210m) and Shaver (1737m) soils (Figure 4).

Please report results of stats in full

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 (1384m) soils showed a statistically significant decrease in macro-aggregate fraction between 150 and 350 °C temperatures.

Please report all stats in full

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 m) and Chiquito (2865 m). Sirretta soils showed a lot of

Please report stats in full

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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).

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. Across all the soils, the largest mineral composition change with increased heating temperatures were observed for kaolinite that experiences loss at temperatures above 550ºC. Gibbsite was not found in soils that it was originally present after 450ºC. 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 (1384m) and Shaver (1737m) soils that are also among the most developed soils with highest proportions of the 1:1 clay minerals (kaolinite).

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 all soils became alkaline at temperatures above 450 °C. The largest increase in pH was

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observed for the Musick (1384 m) soils which reached a pH of 10 at temperatures above 550 °C.

3.7 Cation exchange capacity

The CEC of our soils ranged from an average of 6 $cmol_c$/kg for Chiquito (2865 m) soils to 25 $cmol_c$/kg for Musick (1384 m) soils. With increase of heating temperature all soils showed continued decrease of CEC. With the exception of Musick (1384m) 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 m) 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 m) soils which showed statistically significant drop at 250 °C and again 350 °C. At 350 °C, all the soils except Musick (1384 m) showed a slightly higher CEC than at 250 °C thus interrupting continuous pattern of CEC decrease with increase in temperature.

3.8 Carbon and nitrogen concentration

The initial concentration of C range d from less than 2% (for the Vista soil, 210 m) to over 7 % (for the Musick soils, 1384 m). 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 lost more than 95% of their initial C and changes at higher temperatures were small and statistically insignificant ($p<0.05$). Soil's C:N ratio ranged from 10 (Vista soils, 210 m) to 29 (Musick soils, 1384 m). For all soils C:N ratio decreased with increase in heating temperature in a similar pattern as what we observed for the changes in C concentration (Figure 9).

Report stats in full please

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. Combustion at 450 °C led to 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

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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.

4 Discussion

The Topsoil layer is most affected by extreme temperatures 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, rather than alterations to soil minerals, was the most important driver for the observed changes in soil physico-chemical properties after combustion. 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.

Don't introduce new statistical analyses in the Discussion. All results and analyses should be presented in the Results. The results of the regressions need to be reported in full. I'm not sure you need to do an ANOVA and regression - what does the extra analysis tell you? Correlation analysis was not mentioned in the Methods or Results

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;Ulery and Graham, 1993;Ketterings and Bigham, 2000) and is likely a result to oxidation and transformation of iron oxides in a manner analogous to aging of soils and transformations of mineral soil after intense weathering.

The extent of mass loss in top soil 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. Proportion of soil mass loss with temperature was proportional to initial C concentration of the soils, Musick (1384m) soils, which had the highest initial C concentration (7%) had the steepest soil mass loss and lost 15% of mass at 550ºC while Vista (210m) which had less than 2% C concentration showed the smallest mass loss losing less than 5% mass at even at the highest temperature. The influence of other mass loss mechanisms likely account for mass loss outside of the temperature range where SOM plays a dominant role, dehydration processes are most likely responsible for mass loss at temperatures below 250ºC where the loss and transformation of SOM is minimal. Mass loss at higher temperatures where most of SOM has been combusted (i.e. >450°C) is likely dominated by charring, ashing and volatilization processes.

Most studies report significant soil aggregate stability reduction with fire heating (e.g. Zavala et al. (2010);Arcenegui et al. (2008)), 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 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 ~~it to be s~~ a statistically significant difference, it is worth noting, that the lowest elevation soil (Vista, 210 m) showed a trend of aggregate stability increase up to 350 °C while the high elevation soils, Sirretta (2317 m) and Chiquito (2865 m), showed a slight increase in aggregate stability at 150 °C. Increase in hydrophobicity at these temperatures is the most likely cause, substantial hydrophobicity was apparent with Chiquito soils (2865 m) heated at 250 °C where resistance to slaking was remarkably evident during aggregate stability test.

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

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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 and heat-induced dehydration, larger organic matter particles are likely to have fragmented and reduced in size with increase in heating temperature leading to increase in bulk soil SSA. Furthermore, removal of organic matter from mineral surfaces due to combustion may also increase surface area by reducing overall size of particles. At higher temperatures, 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 combustion. 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 (Ubeda and Outeiro, 2009;Badía and Martí, 2003;Chandler et al., 1983). In a similar heating experiment as we followed in our study, Fernández et al. (1997) observed a pH increase of 1.7 at 350 °C and 2.35 at 490 °C, where the authors attributed the change in pH to denaturation of organic acids, the release of base cations from combustion (K- and Na-hydroxides, Mg- and Ca-carbonates), the deposition of ashes and loss of hydroxyl groups from clays (Certini, 2005;Badía and Martí, 2003). 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 to previous results of Badía and Martí (2003);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 start of combustion of organic acids means contribution of SOM to pH increase (organic acid denaturation and ash liming effect) was absent at this temperature. In a similar heating experiment Badía and Martí (2003) 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

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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 (Ubeda and Outeiro, 2009;Fernández et al., 1997) 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 (Soto and Diazfierros, 1993;Nishita and Haug, 1972). 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

Based on maximum surface temperature, fires are often classified as low, medium or high 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 at the transition between low and medium severity fires, 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 combustion 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 combustion

If you're talking temperatures at the soil surface I would refer to differences in severity. Intensity is generally taken to have a specific meaning in fire science - the rate of heat output per m of fireline (kW/m)

What processes occur in this range that would explain this observation?

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temperature (Figure 10), with the most significant changes in all cases being recorded in all soils between 250 - 450ºC.

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, temperatures below 200 °C have very little effect on quality or quantity of SOM. This means low intensity fires, such as typical prescribed fires, contribute little to soil C loss. Similarly, temperatures above 500 °C do little ~~change~~ 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.

I know what you mean but perhaps rephrase this as temperatures above 500 are also temperatures above 250

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 consideration and because such 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.

This is important and should be discussed earlier on and the methodological issues acknowledged in the Methods section. Duration of heating, the fact that the soils were dry and ground may also be important.

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. Even though the general pattern of change in soil physical and chemical conditions with increasing combustion temperature were mostly consistent for all soils along our study climosequence, the actual magnitude of change in the investigated variables was not. Hence, these finds 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. 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 combustion 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).

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

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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.

I think this is the strongest link you have to argue climate change implications. I'm not convinced about the climate series idea - soils are likely to change only slowly to climate change with very significant lag. The soils represent the results of underlying geological conditions and millenia of differing biological activity - surely that won't be erased overnight by climate change alone. What might happen is that alteration to disturbance regimes will alter vegetation and microbial communities and, in the process, alter soil properties and soil forming processes

Finally, with changes in climate it is anticipated that fires will increase in severity (Westerling et al., 2006b). Our findings of important changes in soil physical and chemical properties occurring between 250-450ºC 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 increased combustion temperature while soil pH and SSA significantly increased. The most important effect of combustion on soil mineralogy as observed by XRD analysis was the collapse 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 the changes that occur under fires that result in heating of soil without additional variables such as the addition of charred plant material and ash, and the influence of soil moisture. 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 top soil profile from Dahlgren et al. (1997): Vista (0 – 14 cm), Musick (0 – 29 cm), Shaver (0 – 4 cm), Sirretta (0 – 6 cm) and Chiquito (0 – 6 cm)

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

	Correlation coefficient (r^2) values					
Soil	**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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Figures

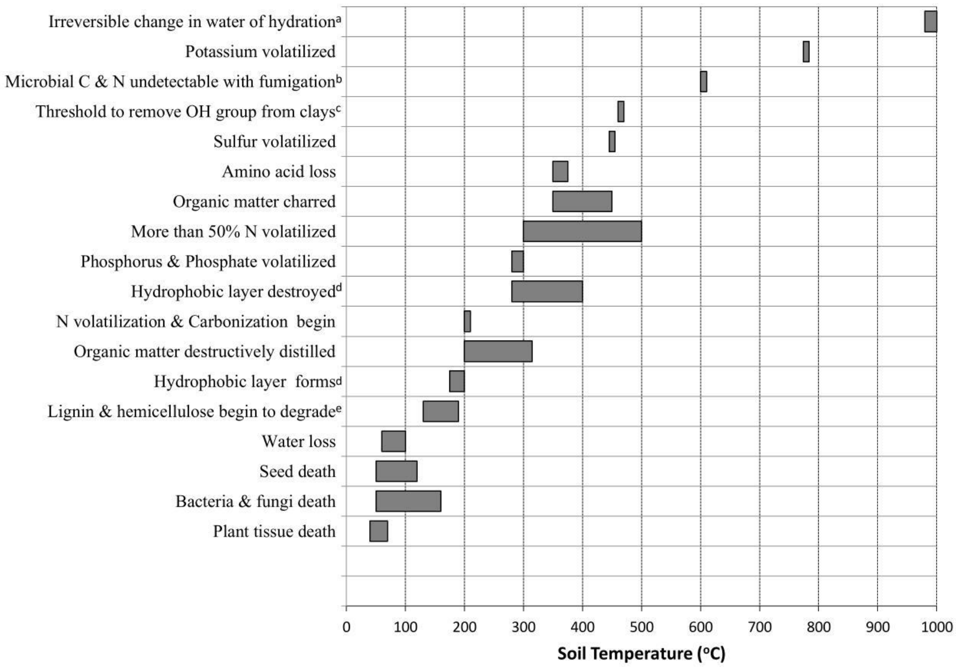

Figure 1: Temperature thresholds and ranges associated with fire heating. Figure adopted and expanded from Massman et al. (2010). ([a] DeBano et al. (1977), [b] Diaz-Ravina et al. (1992), [c] Giovannini et al. (1988), [d] DeBano (2000), and [e] Knicker (2007))

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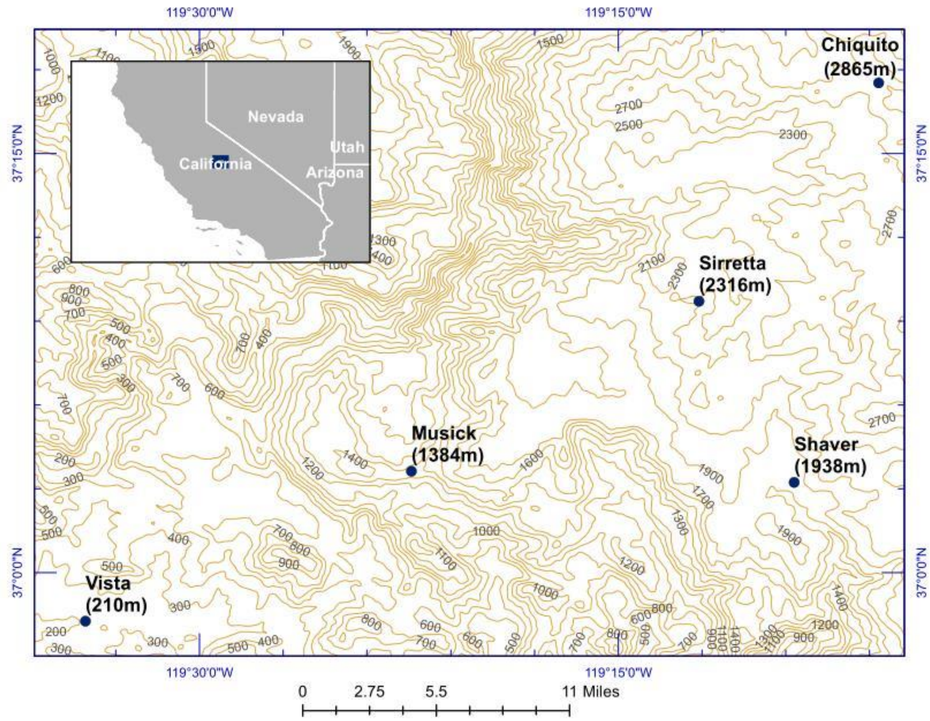

Figure 2: Map of the five sampling sites along elevational transect in the Western Sierra Nevada, California (Base map from U.S. Geological Survey, 2015)

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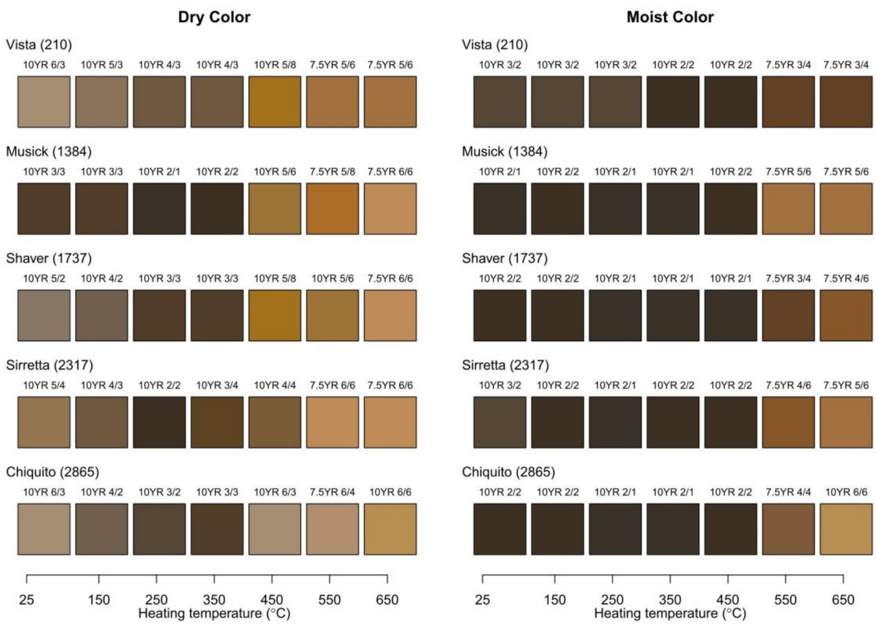

Figure 3: Soil color change across heating temperatures. Colors produced from CIExyY colorspace equivalents to Munsell colors (Munsell Color Science Laboratory). CIExyY colors were converted to RGB system (Rossel et al., 2006;UC Davis Soil Resource Laboratory) and visually compared with Munsell Soil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

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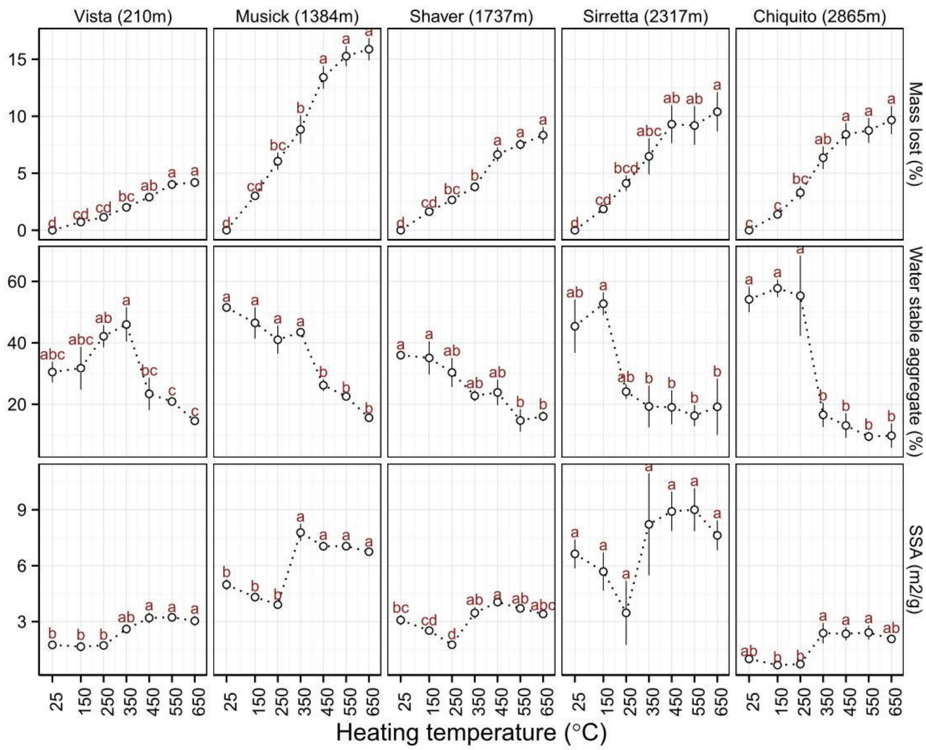

Figure 4: Percent mass lost, water-stable aggregate percent and specific surface area changes with increase in heating temperatures. Error bars represent standard error where n=3. Different letters represent significantly different means (p<0.05) at temperature after Tukey's HSD testing.

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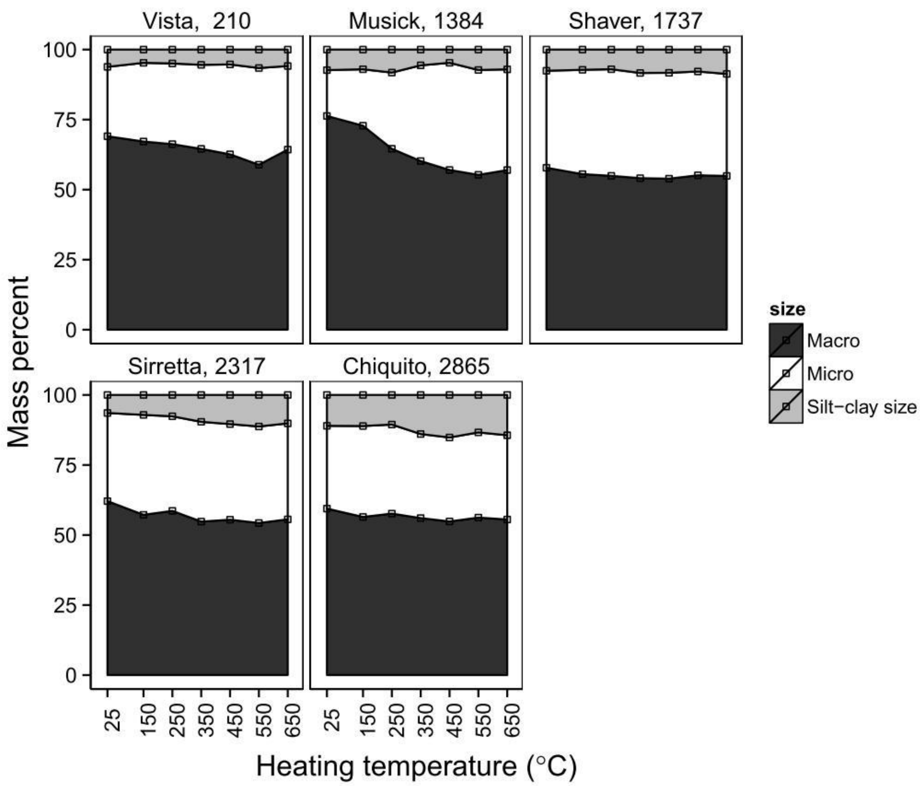

Figure 5: Weight fraction of aggregate sizes: macro (2-0.25 mm), micro (0.25-0.053 mm) and silt-clay (<0.053 mm) sizes.

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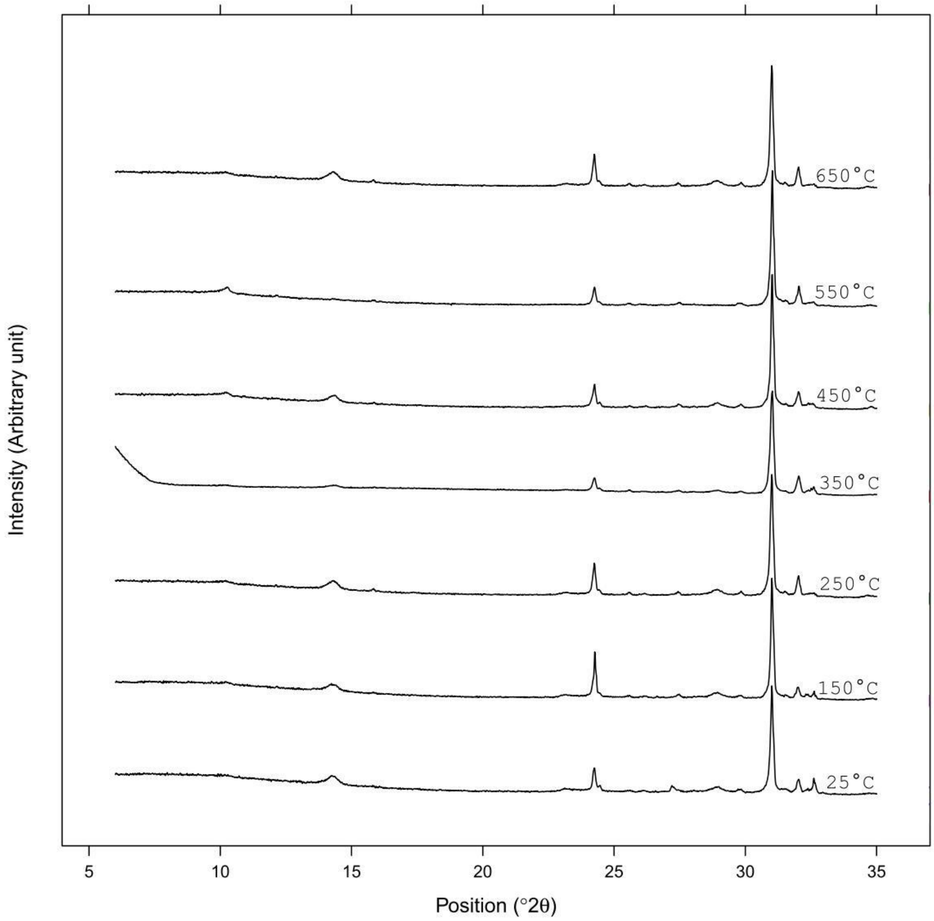

Perhaps highlight areas of the figure where you believe you're seeing important/interesting changes in peaks?

1

2 Figure 6: XRD diagram for Music series soils.

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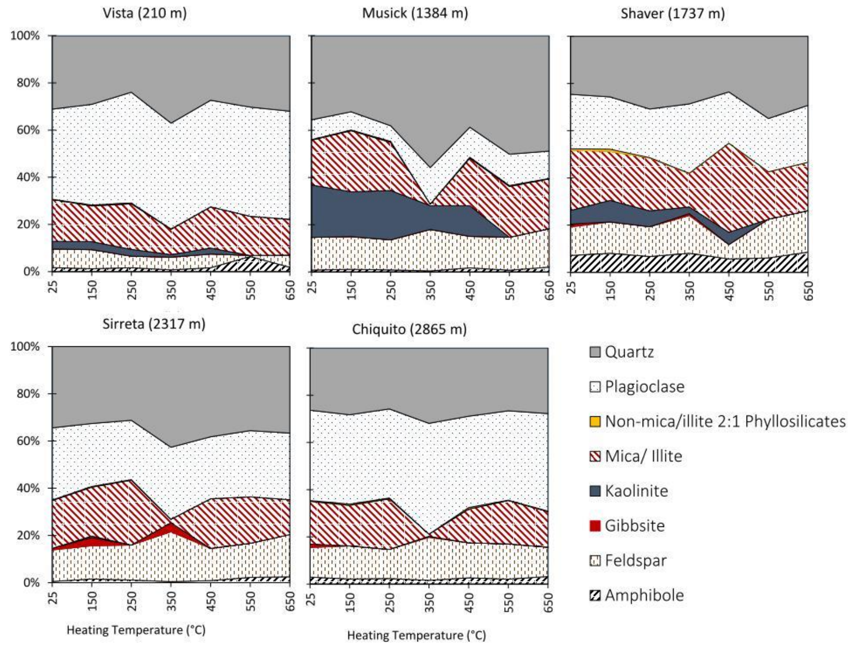


Figure 7: Relative amounts of minerals identified from powder XRD.

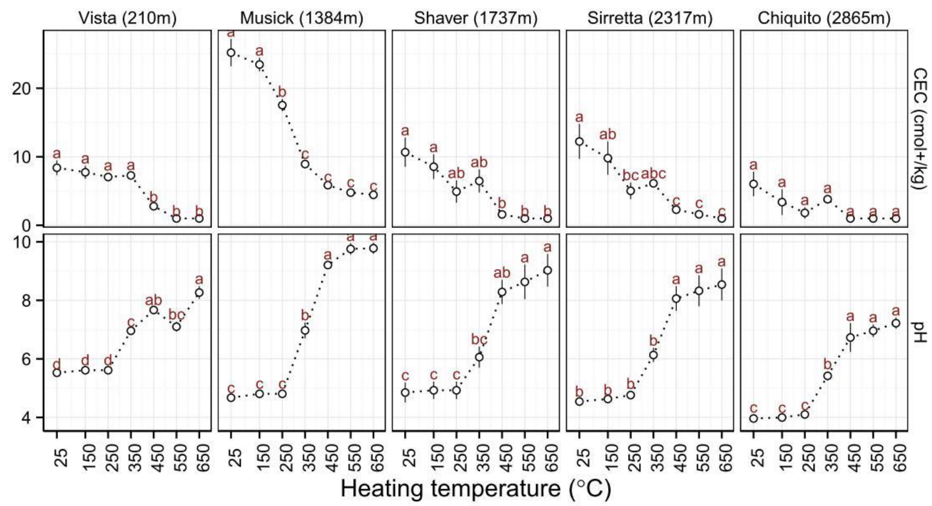


Figure 8: pH (geometric means) and cation exchange capacity (adjusted for mass loss) changes with increase in heating temperature. CEC values below the 2 $cmol_c/kg$ are assigned a value of 1 for plotting. Error bars represent standard error where n=3. Different letters represent significantly different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

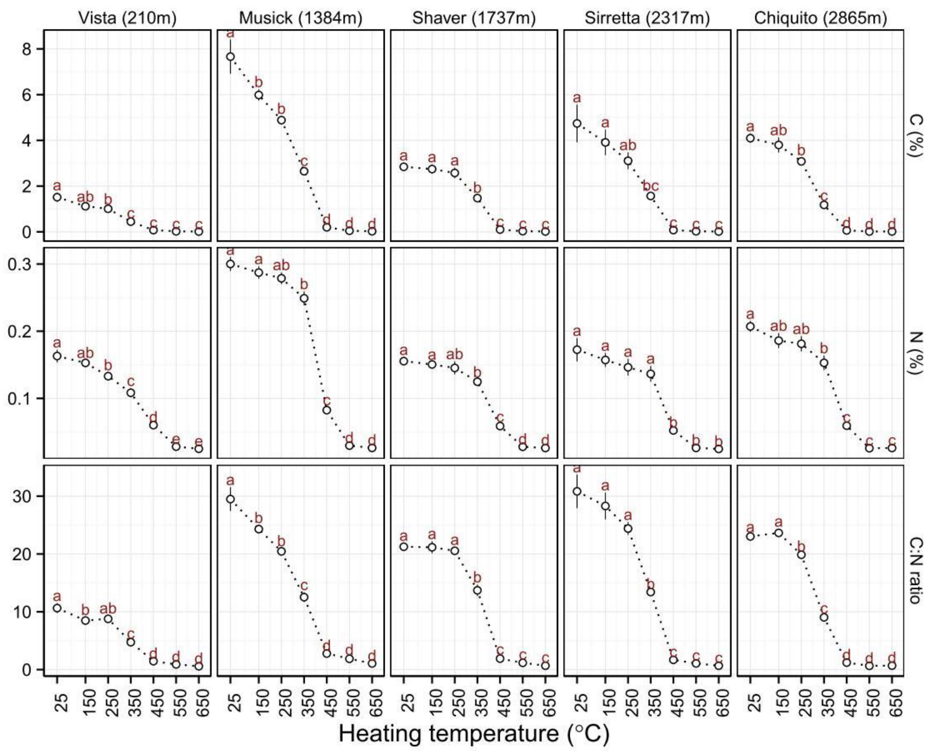
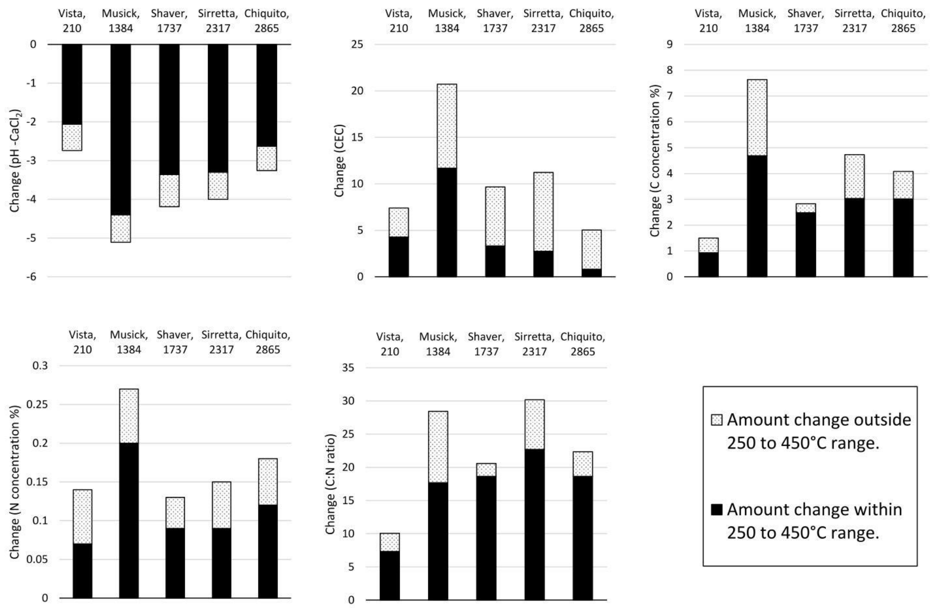


Figure 9: Carbon concentration, Nitrogen concentration and C:N atomic ratio changes with increase in heating temperature. Error bars represent standard error where n=3. Different letters represent significantly different means ($p<0.05$) at each temperature after Tukey's HSD testing.



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