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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5 Samuel N. Araya¹, S. Mercer Meding² and Asmeret Asefaw Berhe^{1,3}

6 [1]Environmental Systems Graduate Group, University of California, Merced

7 [2]Soil, Water, and Environmental Science Dept., University of Arizona, Tucson

8 [3]Life and Environmental Sciences Unit, University of California, Merced

9 Corresponding author: S. N. Araya (e-mail: saraya@ucmerced.edu)

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11 Abstract

12 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' 13 14 physical and chemical properties after they are exposed to a range of temperatures that are 15 expected during prescribed and wildland fires. For this study, we used topsoils collected from 16 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 17 18 significant differences in soil organic matter (SOM) concentration and mineralogy owing to the 19 effects of climate on soil development. Topsoils (0 - 5 cm depth) from the Sierra Nevada 20 climosequence were heated in a muffle furnace at six set temperatures that cover the range of 21 major fire intensity classes (150, 250, 350, 450, 550 and 650 °C). We determined the effects of 22 heating temperature on soil aggregate strength, aggregate size distribution, specific surface area 23 (SSA), mineralogy, pH, cation exchange capacity (CEC), and carbon (C) and nitrogen (N) 24 concentrations. With increasing temperature, we found significant reduction of total C, N and 25 CEC. Aggregate strength also decreased with further implications for loss of C protected inside aggregates. Soil pH and SSA increased with increase in temperature. Most of the statistically 26 27 significant changes (p <0.05) occurred between 350 and 450 °C. We observed relatively smaller

changes 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.

5 Keywords

6 Chemical properties, Climate change, Climosequence, Fire intensity, Physical properties, Soil7 heating

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9 1 Introduction

10 Fire is a common, widespread phenomenon in many ecosystems around the world (Bowman et 11 al., 2009). Vegetation fires burn an estimated 300 to 400 million hectares of land globally every 12 year (FAO, 2005). In the US alone, over 80,000 fires were reported in 2014-including about 13 63,000 wildland fires, and 17,000 prescribed burns that burned over 1.5 million and 970,000 ha 14 of land, respectively (National Interagency Fire Center, 2015). Climate and climatic variations 15 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 16 17 an increase in frequency of fires in the coming decades (Pechony and Shindell, 2010; Westerling 18 et al., 2006). However, our understanding of how climate change and changes in fire regimes 19 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. 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 aboveground 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).

14 The aim of this study is to investigate effects of heating temperatures on important soil 15 properties. Here we aim to determine how the same input of energy from fires affects topsoils 16 that vary significantly based on carbon content, mineralogy, and associated soil physical and 17 chemical properties. The inferences derived from this work are essential for determining how 18 changing climate regimes (and associated changes in vegetation dynamics and soil properties) 19 are likely to influence the response of topsoil to wild- and prescribed-fires. We use a laboratory 20 heating experiment on soils from a well-characterized climosequence in the western Sierra 21 Nevada mountain range to determine: (1) magnitudes of change in soil physico-chemical 22 properties associated with different fire heating temperatures; (2) identify critical thresholds for 23 major changes in soil-physico-chemical properties for soils that significantly vary based on 24 organic matter properties, texture, mineralogy, and other properties.; and (3) infer the 25 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 26 27 development of ability to predict the effect of different intensity fires on soil properties under 28 changing climate scenarios.

1 2 Materials and methods

2 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.,
11 1997; Rasmussen et al., 2007) (Table 1).

The lower elevation woodlands of Sierra Nevada experience less frequent fires than further upslope and the fires are often fast moving and lower severity (Skinner and Chang, 1996). At the middle-elevation zone of Sierran forest, the mixed conifer zones, frequent fires are low to moderate severity at lower altitudes but fire frequency generally increases with altitude towards the upper elevation of the mixed conifer forest (Caprio and Swetnam, 1995). Fires are infrequent and low severity within the high altitude, Subalpine, zone of Sierra (Skinner and Chang, 1996).

18 Soils from the lowest elevation site, Vista soils (210 m), fall within the oak woodland zone 19 (elevations < 1008 m). This is the only soil in our study that does not have an O-horizon, the 20 soil has dense annual grass cover, however, and the A-horizon SOM originates mainly from 21 root turnover. Musick soils (1384 m) lie within oak/mixed-conifer forest (1008-1580 m) and 22 mixed-conifer forest (1580-2626 m). These soils receive the highest biomass and litter fall. 23 Shaver and Sirretta soils (2317m) fall within the mixed-conifer forest range zone while Chiquito 24 soils (2865 m) lies within the subalpine mixed-conifer forest range (2626-3200 m). These 25 soils have lower biomass and litter fall compared to the lower elevation soils (van Wagtendonk 26 and Fites-Kaufman, 2006).

The western slope of central Sierra Nevada presents a remarkable climosequence of soils that developed under similar granitic parent material and are located in landscapes of similar age, relief, slope and aspect (Trumbore et al., 1996) with significant developmental differences

1 attributed to climate. The soils at mid-elevation range (1000 to 2000 m) tend to be highly 2 weathered while soils at high and low elevations are relatively less developed (Dahlgren et al., 3 1997; Harradine and Jenny, 1958; Huntington, 1954; Jenny et al., 1949). Among the most 4 important changes in soil properties along the climosequence include changes in soil organic carbon (SOC) concentration, base saturation, and mineral desilication and hydroxyl-Al 5 6 interlayering of 2:1 layer silicates. Soil pH generally decreases with elevation and the 7 concentrations of clay and secondary iron oxides show a step change at the elevation of present-8 day average effective winter snowline, i.e. 1600 m elevation (Tables 1 and 2) (California 9 Department of Water Resources, 1952-1962; Dahlgren et al., 1997).

10 **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).

18 Sub-samples from each soil were heated in muffle furnace to one of six selected maximum 19 temperatures (150, 250, 350, 450, 550 and 650 °C). To ensure uniform soil heating and reduce 20 formation of heating gradient inside, the soils were packed 1 cm high in a 7 cm diameter 21 porcelain flat capsule crucibles. Oxygen supply during the heating was only limited by the 22 availability of space, the furnace was not sealed and the volume of soil sample to volume air in 23 furnace was approximately 1:50. Furnace temperature was ramped a rate of 3 °C min⁻¹ and soils 24 were exposed to the maximum temperature for 30 minutes. Once cooled to touch, soils were 25 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 $^{\circ}$ C), medium intensity (350 and 450 $^{\circ}$ C),
- 29 and high intensity (550 and 650 °C). These fire intensity classes generally correspond with

1 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 2 3 preferred in laboratory fire simulation experiments (Giovannini et al., 1988; Terefe et al., 2008; 4 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 5 6 samples were exposed to the maximum set temperature for a period of 30 minutes. This length 7 of time ensures that the entire sample is uniformly heated at the set temperature and is in keeping 8 with wide majority of similar laboratory soil heating experiments (for example Badía and Martí, 9 2003a; Fernández et al., 2001; Giovannini, 1994; Varela et al., 2010; Zavala et al., 2010). The 10 duration of soil heating under vegetation fires is highly varied and not uniform across landscape 11 (Parsons et al., 2010). The same heating procedure was used for all the soils so that it would be 12 possible to compare how the soils from different climate regimes are likely to respond to the 13 fires.

14 **2.3 Laboratory analysis**

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

22 Water-stable aggregate percent was measured by wet-sieving methods of Nimmo and Perkins 23 (2002). For this procedure, a wet-sieving apparatus with 0.25mm mesh size sieve is used 24 (Eijkelkamp Agrisearch Equipment, Giesbeek, The Netherlands). Four grams of soil sample 25 was placed into a sieve and pre-wetted slowly from a moist tissue paper placed beneath the 26 sieve. The soil sample was then wet-sieved with an up-down motion at a rate of 35 cycles per 27 minute and a vertical distance of 1.3 cm. Mass of the soil passing through the sieve was recorded 28 (M_1) after evaporating the supernatant water in oven. The remaining soil sample was subjected 29 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 30

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:

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

6 Specific surface area was measured using automated N₂-BET analyzer (Micromeritics Tri-Star 7 3000, Micromeritics Instrument Corporation, Norcross, GA, USA). For this procedure, 8 approximately 1 g of soil was oven dried at 60 °C for 36 hours and out-gassed for another 30 9 minutes using a flow of N₂ gas with outgassing station mantle set to a temperature of 105 °C. 10 Measurement was done using ultra-high purity N₂ gas and the instrument was set to use seven-11 point N₂-BET adsorption isotherm measurement from which the Micromeritics software 12 calculated specific surface area.

13 Soil mineralogy was measured using X-ray diffraction analysis (XRD) using PANalytical Xpert 14 Pro diffractometer (PANalytical Inc., Westborough, MA, USA). We used the PANalytical Xpert Pro software for identification of mineral phases and Rietveld refinement for 15 16 quantification (Rietveld, 1969; Schulze and Dixon, 2002). Soil samples were ground to a fine 17 powder consistency using a ball-mill (8000M MiXer/Mill, with 65 ml stainless steel grinding 18 vial set, SPEX SamplePrep, LLC, Metuchen, NJ, USA) and oven dried at 60 °C for over 36 19 hours. Samples were scanned at generator setting of 45 mA by 40 kV. Scan start position was 20 set to $5^{\circ} 2\theta$ and end position was set to $120 2\theta$. Scan step time was set to 10 seconds with a step 21 interval size of 0.0170° 20. At least two replicate measurements were run for each sample and 22 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
CaCl₂ 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).

27 Cation exchange capacity (CEC) was measured by the barium exchange method. Barium was 28 used to quantitatively displace soil exchangeable cations, and excess barium was removed by 29 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).

4 Elemental concentrations of carbon (C) and nitrogen (N) were measured using an elemental 5 combustion system (Costech ECS 4010 CHNSO Analyzer, Costech Analytical Technologies, 6 Valencia, CA, USA) that is interfaced with a mass spectrometer (DELTA V Plus Isotope Ratio 7 Mass Spectrometer, Thermo Fisher Scientific, Inc, Waltham, MA, USA). For the analyses, the 8 soil samples were ground to a powder consistency using a ball-mill (8000M MiXer/Mill, with 9 a 55 ml tungsten Carbide Vial, SPEX SamplePrep, LLC, Metuchen, NJ, USA) and oven-dried 10 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 11 12 (Kaiser et al., 2015). The C and N concentration results were corrected for moisture by ovendrying subsamples at 105 °C overnight and to determine moisture content. The C and N 13 14 concentration results were corrected for moisture by and adjusting for moisture as: W_{adi} = 15 $W \times (100 - W_m)$. Where W_{adj} is the adjusted percent concentration, W is the concentration 16 before moisture adjustment and W_m is the percent moisture content. All concentration changes 17 resulting from moisture adjustment were a decrease of less than 1% of the value.

18 2.4 Statistical Analysis

19 All quantitative results are expressed as means of three replicates \pm standard error, unless otherwise indicated. Differences of means were tested by Analysis of Variance (ANOVA) and 20 21 pairwise comparison of treatments done using Tukey's HSD test at p<0.05 significance level. 22 All statistical analysis were performed using R statistical software (R Core Team, 2014). 23 ANOVA was implemented using aov() function and Tukey's HSD test subsequently 24 implemented on the aov fit by TukeyHSD() function. The ANOVA tables and Tukey's HSD 25 comparison of means tables are given in supplementary material. The ordinary linear regression 26 technique was used to examine relationships between soil properties.

1 3 Results

2 **3.1 Soil color**

3 We observed a marked soil color change, as inferred using the Munsell color system, with 4 increasing heating temperature (Figure 3). As the heating temperatures increased, the soils 5 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 6 7 became markedly lighter and became increasingly reddish in color (with hue changing from 8 10YR to 7.5YR at temperatures above 550 °C). Color change patterns were similar for both, 9 dry and moist soil measurements except for the marked color change occurring at 450 °C when 10 dry and at 550 °C when moist. Across the heating temperature range, Vista (210m) soils showed 11 the least pronounced increase in darkness at 350°C while Shaver (1737m) soils showed the most 12 pronounced darkening at that temperature range (from dry color of 10YR 5/2 unburned to 10YR 13 3/3 by 350°C). At higher temperatures, Musick soils (1384m) showed the largest change in dry 14 soil color going from 10YR 2/2 at 350 °C to 7.5YR 6/6 at 650 °C.

15 **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°C. In contrast, significant mass loss was observed for the two mid elevation soils of Musick (1384m) and Shaver (1737m) starting 250°C. There was no significant mass loss at temperatures above 450°C 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.

23 **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 1 these two temperatures. Statistically significant decrease in aggregate strength, compared to

- 2 unburned samples, was observed only at higher temperatures above 350 °C for Sirretta (2317m)
- 3 and Chiquito (2864m) soils, and above 450 °C for Musick (1384m), Vista (210m) and Shaver
- 4 (1737m) soils (Figure 4).

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.

10 **3.4** Specific surface area

11 For all soils, we observed a stepwise increase in specific surface area (SSA) for samples were 12 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 13 14 the high elevation soils Sirretta (2317 m) and Chiquito (2865 m). Sirretta soils showed a lot of variability and did not show any significant change in SSA throughout the temperature range 15 while the Chiquito soil showed statistically significant increase between low temperature 150 – 16 17 250 °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 18 19 heated at 250 °C, and highest SSA was observed at 350 °C (for Musick and Chiquito soils) or 20 450 °C (Vista and Shaver soils).

21 **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, 1 2 possibly due to dehydration and the removal -OH (Figure 6). Summary mineral composition 3 changes identified from XRD analysis using Rietveld method are presented in Figure 7. From 4 the minerals identified, the concentration of kaolinite was the most affected by heating. In all 5 soils, concentration of kaolinite showed a decrease when heated to 550°C and above. Gibbsite 6 was also not be detected in soils heated to > 450 °C. Furthermore, mica/illite, plagioclases and 7 amphibole mineral groups changed consistently with increasing temperature. The largest 8 change in soil mineralogy with heating was observed for the mid-elevation Music (1384m) and 9 Shaver (1737m) soils that are also among the most developed soils with highest proportions of 10 the 1:1 clay minerals (kaolinite).

11 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 m) soils which reached a pH of 10 at temperatures above 550 °C.

18 **3.7 Cation exchange capacity**

19 The CEC of our soils ranged from an average of 6 cmol_c/kg for Chiquito (2865 m) soils to 25 20 cmol_c/kg for Musick (1384 m) soils. With increase of heating temperature all soils showed 21 continued decrease of CEC. With the exception of Musick (1384m) soils, CEC eventually 22 dropped to below our detection limit (2 cmol_c/kg) at temperature above 550 °C (Figure 8). For 23 the poorly weathered Chiquito (2865 m) soils, CEC was below 2 cmol_c/kg at temperatures 250 24 °C and above. For the rest of the soils, statistically significant changes in CEC (p<0.05) occurred 25 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 26 slightly higher CEC than at 250 °C thus interrupting continuous pattern of CEC decrease with 27 28 increase in temperature.

3.8 Carbon and nitrogen concentration

2 The initial concentration of C ranged from 1.5% (Vista soil, 210 m) to 7.7 % (Musick soils, 3 1384 m). Soil C concentration decreased with increase in temperature (Figure 9) with the largest 4 decrease occurring between temperatures of 250 and 450 °C. At 450 °C, all soils had lost more 5 than 95% of their initial C. At temperatures above 450 °C, C concentration changes were small 6 and we did not find statistically significant changes at p<0.05. The C:N ratio ranged from 10 7 (Vista soils, 210 m) to 29 (Musick soils, 1384 m). The C:N ratio decreased with increase in 8 heating temperature in a similar pattern to the C concentration (Figure 9). 9 The loss of C and N from soils due to heating showed a similar response among all five soils.

10 After 250 °C, all the soils lost more than 25% of their initial C (except Shaver soils that lost 11 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. 12 13 At temperatures greater than 550 °C there was 5 to 15% of soil N still remaining. Consequently, 14 we observed a decrease of C:N ratio with increase in heating temperature. All soils continued 15 to lose about 15% soil N for every 100 °C increase and maintained more than 60% of their N at heating temperatures up to 350 °C. After heating at 450 °C, all soils lost more than 60% of the 16 17 initial soil N and 85% by 550 °C.

18 4 Discussion

19 The topsoil layer is most affected by extreme temperature during vegetation fires. Our results 20 show significant changes in soil properties as a result of temperature exposure. Our findings 21 demonstrate that alterations and loss of SOM in topsoil because of heating, rather than 22 alterations to soil minerals, was the most important driver for the observed changes in soil 23 physico-chemical properties. Our XRD analysis shows notable changes in soil mineralogy only 24 after the soils were heated to about 450 to 550 $^{\circ}$ C (Figure 7). In upland ecosystems, such as the 25 Sierra Nevada Mountains, the soils typically have low clay content and low concentration of 26 secondary minerals (Neary et al., 1999; Ubeda and Outeiro, 2009). In addition, these upland 27 temperate ecosystems also tend to have relatively high concentration of SOM, including a fairly 28 well developed O-horizon. Consequently, strong relationships are observed between SOM 29 concentrations and the soils' physical and chemical properties. Simple linear regression 30 analyses between C concentration changes and other soil physical and chemical changes for our

1 study soils shows that more than 80% of the variability in mass loss, aggregate strength, SSA,

- 2 pH, CEC and N concentrations are associated with changes in C concentration at the different
- 3 heating temperatures. Table 3 summarizes the correlation coefficients of soil property changes
- 4 with changes in C concentration.

5 The changes in soil color observed were consistent with the charring of SOM which leads to 6 darkening of the brownish color of the soils. At temperatures over 450 °C, the near complete 7 removal of SOM by combustion and the addition of ash products likely explains the observed 8 lighter color of soils. The increase in Munsell chromas and reddening of soils at these high 9 temperatures has been noted in previous works (e.g. Giovannini et al., 1988; Ketterings and 10 Bigham, 2000; Ulery and Graham, 1993) and is likely a result and the transformation of iron 11 oxides that occurs during heating.

12 The extent of mass loss in topsoil layers due to vegetation fires is strongly correlated to SOM 13 combustion (Rein et al., 2008). In all of our soils, statistically significant mass loss (p<0.05) 14 occurred within the temperature ranges of SOM combustion, between 250°C to 450°C. 15 Proportion of soil mass loss with temperature was proportional to initial C concentration of the 16 soils. For example, Musick (1384m) soils which had the highest initial C concentration (7.7%)17 had the steepest soil mass loss and lost 15% of mass at 550°C, while Vista (210m) which had 18 less than 2% C concentration showed the smallest mass loss losing less than 5% mass at even 19 at the highest temperature. Mass loss at temperatures below 250°C is likely driven by 20 dehydration processes rather than by loss of SOM. On the other hand, mass loss at temperatures 21 above which SOM combusts, >450°C, is likely driven by charring, ashing and volatilization 22 processes.

23 Most studies report significant soil aggregate stability reduction with fire heating (e.g. 24 Arcenegui et al. (2008); Zavala et al. (2010)), however, contrasting findings are also reported 25 in other studies. Mataix-Solera et al. (2011) explains that increase in aggregate strength with 26 heating is possible in clay-rich soils where the main cementing agents are inorganic minerals 27 such as calcium carbonates and metallic oxides which would fuse under fire heating increasing 28 aggregate stability. Another possibility is, soils which initially had weak hydrophobicity may 29 show increased aggregate stability due to increase in hydrophobicity, in such cases however 30 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 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. An 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 the aggregate stability test.

8 Soil specific surface area (SSA) is an important soil property that affects soil adsorption, ion 9 exchange capacity, reactivity, aggregation and porosity (Feller et al., 1992). SSA of soil is 10 largely dictated by clay-size particles and SOM (Carter et al., 1986). The increase in SSA with 11 heating that we observed in this work is most likely the result of physical disintegration and 12 charring of SOM, especially at temperatures below 500 °C. Changes in soil mineralogy are not 13 likely to be responsible for the changes in SSA we observed. XRD analysis showed notable 14 mineralogical changes only at temperatures above 450 °C where the kaolinite peak disappeared 15 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 16 17 particles are likely to have fragmented and reduced in size with an increase in heating 18 temperature leading to increase in bulk soil SSA. Furthermore, thermal alteration and the 19 removal of organic matter from mineral surfaces may have increased surface area by reducing 20 the size of particles. At higher temperatures (>450 °C), XRD spectra showed some collapse of 21 mineral complexes through dehydration and de-hydroxylation of clay minerals that may reduce 22 mineral particle size and increase surface area. These changes in mineralogy might have a 23 significant effect on SOM. A study by Rosa et al. (2013) found that soils released more organic 24 compounds during pyrolysis when the soils were treated with HF acid, suggesting that mineral 25 complexes play a role in protecting organic compounds from pyrolysis. The collapse of mineral 26 complexes we observed and the decrease in aggregate strength is likely to have enhanced 27 thermal oxidation of SOM. However mineralogical changes would play a more important role 28 affecting soil properties at these high temperatures since SOM has been almost completely 29 removed.

1 Soil pH generally increases with fire heating (Badía and Martí, 2003b; Chandler et al., 1983; 2 Ubeda and Outeiro, 2009). In a soil heating experiment similar to our study, Fernández et al. 3 (1997) observed a pH increase of 1.7 at 350 °C and 2.35 at 490 °C. Such increase in soil pH has 4 been attributed to the denaturation of organic acids, release of base cations from combustion 5 (K- and Na-hydroxides, Mg- and Ca-carbonates), deposition of ashes, and loss of hydroxyl 6 groups from clays (Badía and Martí, 2003b; Certini, 2005; Ulery et al., 1993). In our soils, the 7 higher elevation soils (Shaver, Sirretta and Chiquito) showed a statistically insignificant 8 decrease of 0.3 to 0.5 pH units (measured in water) at 250 °C. The change in pH in our high 9 elevation soils was consistent with previous results of Badía and Martí (2003b); Terefe et al. 10 (2008) that found similar initial decrease. Terefe et al. (2008) hypothesized that this may be due 11 to the combined effect of desiccation and heating effect which favor proton-reducing oxidation 12 reactions. And the fact that this initial increase occurs below the temperature for the start of 13 combustion of organic acids means contribution of SOM to this pH increase (organic acid 14 denaturation and ash liming effect) was absent at this temperature. In a similar heating 15 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 16 17 where we observed a decrease in pH when measured in water but not in CaCl₂ suggesting that 18 the decrease in pH might have to do with increase of soluble salts with heating up to 250 °C.

19 The capacity of soil to exchange positively charged ions between soil and soil solution (CEC) 20 decreased with increasing temperature. CEC of soils is a result of surface charges associated 21 with secondary clay minerals and SOM (Sparks, 2005), and in our study soils, Dahlgren et al. 22 (1997) had previously reported a strong relationship of CEC with soil organic carbon and clay 23 concentrations. Different authors have attributed loss of CEC during heating mainly with loss 24 of SOM (Fernández et al., 1997; Ubeda and Outeiro, 2009) partly because CEC loss starts to be 25 observed at temperatures above 200 °C with little or no decrease at lower temperatures where 26 SOM is not affected (Nishita and Haug, 1972; Soto and Diazfierros, 1993). The slight increase 27 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 28 29 compensated for the loss of SOM at that temperature. Furthermore, the contribution of surface 30 oxidation of char products has been shown to increase CEC per unit C (Liang et al., 2006) 31 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.

3 4.1 Importance of the 250 – 450 °C range

Taking maximum surface temperature as a metric of fire intensity, low intensity fires reach 4 5 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, 6 7 2008). In this study, the most significant changes of soil chemical properties occurred between 8 250 and 450 °C. Figure 10 illustrates the changes between unburned and 650 °C burned soils 9 and the amount of change that occurred within 250 to 450 °C heating temperature for a range 10 of the variables discussed above. In all cases, the change in the 250 - 450 °C range accounts for 11 most of the total change observed during our heating treatments. Among the variables we 12 investigated in this study, we observed changes along two general lines: (1) mass loss, SSA and 13 pH which showed a progressive increase with heating temperature, and (2) %C, %N, C:N ratio, 14 CEC, and wet aggregate stability that showed a progressive decrease with increase in 15 temperature (Figure 10), with the most significant changes in all cases being recorded in all soils between 250 - 450 °C. 16

17 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). 18 19 However, we found that temperatures below 200 °C have very little effect on quality or quantity 20 of SOM. This implies that lower intensity fires, such as typical prescribed fires, where soil 21 surface temperatures do not exceed below 250 °C temperature contribute little to soil C loss. 22 Similarly, temperatures above 500 °C do little to SOM, which already has been lost or 23 transformed into a pyrogenic product. The effect on soil inorganic particles starts at high 24 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. 25

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.

8 4.2 Climate Change Implications

9 Investigation of the response of climosequence soils to different heating temperature in this 10 study enables us to infer how changes in climate (and associated changes in soil properties) are 11 likely to alter the effect of fires on topsoil physical and chemical properties. The overall pattern 12 of soil property changes with increase in heating temperature were similar for all soils, however, 13 the magnitudes of change were variable. Hence, these findings lead us to conclude that climate 14 change is likely to alter the response of topsoil properties to different fire regimes. Along our 15 study climosequence, we observed critical differences in response of topsoils based mostly on 16 concentration OM in soil and soil development stages of each soil --both variables that are 17 expected to respond to changes in climate (Berhe et al., 2012). Consequently, changes in soil C 18 storage associated with climate change are expected to lead to different amounts of C loss due 19 to fires. This is evidenced by the observed highest total mass of C loss from the mid-elevation 20 Musick soil that had the highest carbon stock, compared to soils in either side of that elevation 21 range. Anticipated changes in climate in the Sierra Nevada mountain ranges are expected to 22 include upward movement of the rain-snow transition line exposing areas that now receive most 23 of their precipitation as snow to rainfall and associated runoff (Arnold et al., 2015, 2014; Stacy 24 et al., 2015). Moving of the rain-snow transition zone higher and promotion of more intense 25 weathering at higher elevation zones then is likely to render more C to loss during fires. As we 26 found in this study, more than 80% of the variability in mass loss, aggregate strength, SSA, pH, 27 CEC and N concentrations is associated with changes in C concentration at the different heating 28 temperatures (Table 3). Improving our understanding of how topsoil properties are likely to 29 respond to changes in climate becomes even more critical when we recognize that C 30 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).

6 The different responses of soil aggregation in our climosequence to the treatment temperatures 7 also suggest potential loss and transformation of the physically protected C pool in topsoil. 8 Degradation of aggregates during fire (Albalasmeh et al., 2013) is likely to render aggregate-9 protected C to potential loses through oxidative decomposition, leaching and erosion. 10 Moreover, in systems such as the Sierra Nevada where steep slopes and organic matter-rich 11 topsoils dominate, movement of the rain-snow transition zone upward is likely to increase 12 proportion of precipitation that occurs as rain. The kinetic energy of raindrops and observed 13 increase in hydrophobicity of soils post-fires (Johnson et al., 2007a; Johnson et al., 2004) can 14 lead to higher rates of erosional redistribution of especially the free light fraction or particulate 15 C that is not associated with soil minerals (Stacy et al., 2015). Moreover, the important 16 differences in changes in pH, mineralogy, CEC in response to heating at different temperatures 17 that we observed for soils along the climosequence suggest that changes in temperature are 18 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°C 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.

24 **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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- 22

1 Tables

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; <i>Quercus</i> 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	

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)

⁴ ^a Mean annual air temperature, calculated from regression equation of Harradine and Jenny (1958)

5 ^b Mean annual precipitation

6 ^c Dominant form of precipitation

7 ^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	Gravimet ric water	pH (CaCl ₂)	Carbon (%)	CEC (cmolc/kg)	SSA (m²/g)	Particle size distribution ^a (%)		
	(g/cm ³)	content (%)					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

4 ^a Particle size distribution of topsoil profile from Dahlgren et al. (1997): Vista (0 - 14 cm),

5 Musick (0 - 29 cm), Shaver (0 - 4 cm), Sirretta (0 - 6 cm) and Chiquito (0 - 6 cm)

6

7

	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			

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

1 Figures

2



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



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



1

2 Figure 3: Soil color change across heating temperatures. Colors produced from CIExyY

3 colorspace equivalents to Munsell colors (Munsell Color Science Laboratory). CIExyY colors

4 were converted to RGB system (Rossel et al., 2006; UC Davis Soil Resource Laboratory) and

5 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.



1

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

3 silt-clay (<0.053 mm) sizes.













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 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.</p>



5

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.



- 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.