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1 **Responses of soil physico-chemical properties to**

2 **combustion: a space for time substitution study to infer**

3 **how changes in climate are likely to affect response of**

4 **topsoil to fires**

5

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11

12 **Abstract**

13 Fire is a common ecosystem perturbation that affects many soil properties. As global fire

14 regimes continue to change with climate change, we investigated the effect of fire heating

15 temperatures on the physical and chemical properties of soils across a climosequence transect

16 along the Western slope of the Sierra Nevada that spans from 210 to 2865 m.a.s.l. All the soils

17 we studied were formed on a granitic parent material and have significant differences in soil

18 organic matter (SOM) concentration and mineralogy owing to the effects of climate on soil

19 development. The dominant vegetation from lowest to highest elevation across the transect

20 range from oak woodland, oak/mixed-conifer forest, mixed-conifer forest and subalpine mixed-

21 conifer forest. Topsoils (0 - 5 cm depth) from the Sierra Nevada climosequence were heated in

22 a muffle furnace at six set temperatures that cover the range of major fire intensity classes (150,

23 250, 350, 450, 550 and 650 °C). We determined the effects of fire heating temperature on soil

24 aggregate strength, aggregate size distribution, specific surface area (SSA), mineralogy, pH,

25 cation exchange capacity (CEC), and carbon (C) and nitrogen (N) concentrations. With increase

26 of temperature, we found significant reduction of total C, N and CEC. Aggregate strength also

27 decreased with further implications for loss of C protected inside aggregates. Soil pH and SSA

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1   increased with increase in temperature. Most of the statistically significant changes ($p < 0.05$)

2   occurred at temperature ranges of 350 to 450 °C. We observed relatively smaller changes at

3   typical temperature ranges of prescribed fires (i.e. less than 250 °C). This study identifies

4   critical combustion temperature thresholds for significant physico-chemical changes in soils

5   that developed under different climate regimes, allowing inferences for how soils are likely to

6   respond to different fire intensities under anticipated climate change scenarios.

7   **Keywords**

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

9   heating

10

11 **1   Introduction**

12 Fire is a common, widespread phenomenon in many ecosystems around the world (Bowman et

13 al., 2009). Vegetation fires burn an estimated 300 to 400 million hectares of land globally every

14 year (FAO, 2005). In the US alone, over 80,000 fires were reported in 2014–including about

15 63,000 wild-land fires, and 17,000 prescribed burns that burned over 1.5 million and 970,000

16 ha of land, respectively (National Interagency Fire Center, 2015). Climate and climatic

17 variations exert strong control on distribution, frequency, and severity of fires (Harrison et al.,

18 2010). Significant changes on global fire regimes are anticipated because of climate change

19 including an increase in frequency of fires in the coming decades (Westerling et al.,

20 2006a;Pechony and Shindell, 2010). However, as of yet, we only have limited understanding of

21 how changes in fire regimes and climate change are likely to interact to influence response to

22 topsoils in fire affected ecosystems to wild- or prescribed-fires.

23 Even though humans are responsible for causing a substantial proportion of vegetation fires

24 (Caldararo, 2002), vegetation fires are also natural phenomena with an important role in

25 maintaining the health of many ecosystems around the world. Many ecosystems in the US and

26 across the world depend on fires to maintain ecosystem health and productivity. In the Sierra

27 Nevada, vegetation fires have a major influence on the landscapes (McKelvey et al., 1996).

28 Lightning fires were historically common in the dry season in the upland forests of the Sierra

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1 Nevada Mountains and also fires were used by some of the Native American tribes to modify

2 the environment for their needs (Parsons and van Wagtendonk, 1996).

3 In addition to alteration of the overland vegetation, fire also significantly affects the physical,

4 chemical and biological properties of soils. The degree of alteration caused by fires depends on

5 fire intensity and duration, which in turn depend on factors such as amount and type of fuels,

6 properties of above ground biomass, air temperature and humidity, wind, topography, and soil

7 properties of moisture content, texture and SOM content (DeBano et al., 1998). The first-order

8 effects of fire on soil are caused by the input of heat causing extreme soil temperatures in topsoil

9 (Badía and Martí, 2003;Neary et al., 1999) resulting in loss and transformation of SOM, changes

10 in soil hydrophobicity, changes in soil aggregation, loss of soil mass, and addition of charred

11 material and other combustion products (Albalasmeh et al., 2013;Rein et al., 2008;Mataix-

12 Solera et al., 2011). Soil temperature thresholds for some important soil transformations are

13 illustrated in Figure 1.

14 Fires also impact soil by altering and removing above-ground vegetation and topsoil biomass,

15 and increasing soil erodibility (Carroll et al., 2007;DeBano, 1991), subsequently leading to a

16 shift in plant and microbial populations (Janzen and Tobin-Janzen, 2008). Fires with longer

17 durations are typically expected to have more impact on soil physical and chemical properties,

18 and loss of SOM than fast-moving, high temperature fires (González-Pérez et al., 2004).

19 The aim of this study is to investigate effects of combustion temperatures on important soil

20 properties. Here we aim to determine how the same input of energy from fires affects topsoils

21 that vary significantly based on carbon content, mineralogy, and associated soil physical and

22 chemical properties respond to combustion. The inferences derived from this work are essential

23 for determining how changing climate regimes (and associated changes in vegetation dynamics

24 and soil properties) are likely to influence the response of topsoil to wild- and prescribed-fires.

25 We use a laboratory heating experiment on soils from a well-characterized climosequence in

26 the western Sierra Nevada mountain range in our study to determine: (1) magnitudes of change

27 in soil physico-chemical properties associated with different fire heating temperatures; (2)

28 identify critical thresholds for major changes in soil-physico-chemical properties for soils that

29 significantly vary based on organic matter properties, texture, mineralogy, and other properties.;

30 and (3) infer the implications of changing climate on topsoil physico-chemical properties that

1 might experience changing fire regime. This study aims to contribute to the systematic

2 evaluation and development of ability to predict the effect of different intensity fires on soil

3 properties under changing climate scenarios.

4 **2   Materials and methods**

5 **2.1   Study site and soil description**

6 The Sierra Nevada ecosystems are fire-adapted systems. Fires are common perturbations that

7 maintain ecosystem health and plant productivity in the region. However, over the last couple

8 of decades, the frequency and severity of fires has been increasing due to changes in climate

9 (Westerling et al., 2006b). For this study, we collected soils from five sites across elevation

10 transect along the western slope of central Sierra Nevada (Figure 2); the sites were previously

11 characterized by Dahlgren et al. (1997). We selected four forested sites that are likely to

12 experience forest fires and a fifth lower elevation grassland site for comparison.

13 All the sites have a Mediterranean climate characterized by warm to hot dry summers and cool

14 to cold wet winters. Mean annual air temperature ranges from 16.7 °C at the lowest site located

15 at 210 m to 3.9 °C at the highest elevation site which is at an elevation of 2865 m. Annual

16 precipitation ranges from 33 cm at the lowest site to 127 cm at the highest site (Dahlgren et al.,

17 1997;Rasmussen et al., 2007) (Table 1).

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, however, and the A-horizon SOM originates mainly from root

21 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).

27 The western slope of central Sierra Nevada presents a remarkable climosequence of soils that

28 developed under similar parent material and are located in landscapes of similar age, relief,

29 slope and aspect (Trumbore et al., 1996) formed on a granitic parent material with significant

1 developmental differences attributed to climate. The soils at mid-elevation range (1000 to 2000

2 m) tend to be highly weathered while soils at high and low elevations are relatively less

3 developed (Jenny et al., 1949;Huntington, 1954;Harradine and Jenny, 1958;Dahlgren et al.,

4 1997). Among the most important changes in soil properties along the climosequence include

5 changes in soil organic carbon (SOC) concentration, base saturation, and mineral desilication

6 and hydroxyl-Al interlayering of 2:1 layer silicates. Soil pH and the concentrations of clay and

7 secondary iron oxides show a step change at the elevation of present-day average effective

8 winter snowline, i.e. 1600 m elevation (Tables 1 and 2) (California Department of Water

9 Resources, 1952-1962;Dahlgren et al., 1997).

10 **2.2 Experimental design and sample collection**

11 To investigate the effect of combustion temperature on physico-chemical properties of soils

12 with significantly different carbon contents, mineralogy, and overall development, we collected

13 top soils (0 to 5 cm depth) from five sites. Triplicate samples, approximately 10 m apart, were

14 collected from each site. The soils were air-dried at room temperature and passed through 2 mm

15 sieve. Prior to furnace heating, the soils were oven dried at 60 °C overnight. Soil bulk density

16 and field soil moisture were determined from separate undisturbed core samples collected from

17 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. Furnace temperature was ramped a rate of 3 °C min^{-1} and soils

22 were exposed to the maximum temperature for 30 minutes. Once cooled to touch, soils were

23 stored in in air-tight polyethylene bags prior to analysis.

24 The six heating temperatures were selected to correspond with fire intensity categories that are

25 based on maximum surface temperature (Janzen and Tobin-Janzen, 2008;DeBano et al.,

26 1977;Neary et al., 1999), that is, low intensity (150 and 250 °C), medium intensity (350 and

27 450 °C), and high intensity (550 and 650 °C). These fire intensity classes generally correspond

28 with thresholds for important thermal reactions in soils observed by differential thermal

29 analyses (Giovannini et al., 1988;Varela et al., 2010;Soto et al., 1991) The relatively slow

1   heating rate of 3 °C min⁻¹ is recommended for laboratory based fire simulation experiments

2   (Giovannini et al., 1988;Terefe et al., 2008;Varela et al., 2010) to prevent sudden combustion

3   when soil's ignition temperature is reached at about 220 °C (Fernández et al., 1997, 2001;Varela

4   et al., 2010). The identical treatment of all soils, then allows for comparison of how the soils

5   from the different climate regimes are likely to respond to fires. Furthermore, once the set

6   temperature is reached, samples were exposed to that temperatures for a period of 30 minutes.

7   This is approximately equivalent to the time it takes to burn off small dry logs (Stoof et al.,

8   2010;Chandler et al., 1983) and 30 to 40 minutes has become the standard in laboratory soil

9   heating experiments (for example Varela et al., 2010;Giovannini, 1994;Fernández et al.,

10   2001;Zavala et al., 2010).

11   **2.3   Laboratory analysis**

12   Soil color was measured using the Munsell Color Charts. Dry color was measured from air-

13   dried samples and moisture was added to same sample for moist color measurement. Dry-

14   aggregate size distribution was measured by sieving. Samples were dry sieved into three

15   aggregate size classes: 2–0.25 mm (macro-aggregates), 0.25–0.053 mm (micro-aggregates) and

16   <0.053 mm (silt and clay sized particles). These aggregate size classes were selected to enable

17   comparison with other studies that investigated the effect of different natural and anthropogenic

18   properties on soil aggregate dynamics and aggregate protected organic matter (Six et al 2000).

19   Water-stable aggregate percent was measured by wet-sieving methods of Nimmo and Perkins

20   (2002) using a wet-sieving apparatus (Eijkelkamp Agrisearch Equipment, Giesbeek, The

21   Netherlands) with 0.25mm mesh size. Four grams of soil is weighed into sieve and slowly pre-

22   wetted by capillary rise. Sample was then wet-sieved with an up-down motion with a vertical

23   distance of 1.3 cm and a rate of 35 cycles per minute. Soil passing through sieves was collected

24   in the cans and weighed after evaporating the supernatant water in oven (M_1). The samples

25   remaining in the sieve were then subjected to a second round of wet-sieving using another set

26   of cans filled with dispersing solution (2 gL⁻¹ of sodium hexametaphosphate for the soils with

27   pH >7 and 2 gL⁻¹ NaOH for the soils with pH <7). Samples were wet-sieved until all particles

28   smaller than the screen opening pass. Mass of soil collected in the second set of cans (M_2) was

29   determined by evaporating supernatant solution in oven and subtracting the weight of the

30   dispersing-agent. The water-stable aggregate fraction was calculated as:

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

2   Specific surface area was measured using automated N_2-BET analyzer (Micromeritics Tri-Star

3   3000, Micromeritics Instrument Corporation, Norcross, GA, USA). For this procedure,

4   approximately 1 g of soil samples were oven dried at 60 °C for 36 and out-gassed for another

5   30 minutes using a flow of N_2 gas with outgassing station mantle set to a temperature of 105

6   °C. Measurement was done using ultra-high purity N_2 gas and the instrument was set to seven

7   point measurement. The isotherm is analyzed using Micromeritics software.

8   Soil mineralogy was measured from X-ray diffraction analysis (XRD) using PANalytical Xpert

9   Pro diffractometer (PANalytical Inc., Westborough, MA, USA). We used the PANalytical

10   Xpert Pro software for identification of mineral phases and Rietveld refinement for

11   quantification (Schulze and Dixon, 2002;Rietveld, 1969). Soil samples were ground to fine

12   powder consistency using ball-mill (8000M MiXer/Mill, with 65 ml stainless steel grinding vial

13   set, SPEX SamplePrep, LLC, Metuchen, NJ, USA) and oven dried at 60 °C for over 36 hours.

14   Samples were scanned at generator setting of 45 mA by 40 kV. Scan start position was set to 5°

15   2θ and end position was set to 120 2θ. Scan step time was set to 10 seconds at step interval size

16   of 0.0170° 2θ. Two or three replicate measurements were run for each sample and samples were

17   measured in random order.

18   Soil pH was measured 1:2 solid:solution ratio mixtures in a deionized water and 0.01 M $CaCl_2$

19   solution. Five grams of soil was mixed by shaking with 10 ml of solution and allowed to stand

20   for 30 minutes with stirring every 10 minutes. The pH reading was taken by placing electrodes

21   directly in the sediment slurry immediately after stirring (Thomas, 1996).

22   Cation exchange capacity (CEC) was measured by the barium exchange method. Barium was

23   used to quantitatively displace soil exchangeable cations, and excess barium was removed by

24   four deionized water rinses. A known quantity of calcium is then exchanged for barium and

25   excess solution calcium is measured in order to determine CEC by the difference in the quantity

26   of the calcium added and the amount left in the resulting solution. The method has a detection

27   limit of 2.0 $cmol_c$/kg (Rible and Quick, 1960).

28   Elemental concentrations of carbon (C) and nitrogen (N) were measured using an elemental

29   combustion system (Costech ECS 4010 CHNSO Analyzer, Costech Analytical Technologies,

1   Valencia, CA, USA) that is interfaced with a mass spectrometer (DELTA V Plus Isotope Ratio

2   Mass Spectrometer, Thermo Fisher Scientific, Inc, Waltham, MA, USA). For the analyses, air-

3   dried < 2 mm soil samples were ground to powder consistency on a ball-mill (8000M

4   MiXer/Mill, with a 55 ml tungsten Carbide Vial, SPEX SamplePrep, LLC, Metuchen, NJ, USA)

5   and oven dried at 60 °C for over 36 hours. The values for C and N concentration were corrected

6   for oven dried weights by oven-drying subsamples at 105 °C.

7   *Statistical Analysis*

8   All quantitative results are expressed as means of three replicates ± standard error, unless

9   otherwise indicated. Differences of means were tested by Analysis of Variance (ANOVA) and

10 pairwise comparison of treatments done using Tukey's HSD test at $p<0.05$ significance level.

11 The ordinary linear regression technique was used to examine relationships between soil

12 properties. All statistical analysis were performed using R statistical software (r-project.org).

13 **3   Results**

14 **3.1   Soil color**

15 We observed a marked soil color change, as inferred using the Munsell color system, with

16 increasing heating temperature (Figure 3). With increase in heating temperature, all the soils

17 exhibited a similar trend in color change. As the heating temperatures increased, the soils

18 initially got darker with, reaching their darkest color in mid temperatures (250 - 350 °C when

19 dry and 250 - 450 °C when moist). At higher temperatures, the soils became markedly lighter

20 and became increasingly reddish in color (with hue changing from 10YR to 7.5YR at

21 temperatures above 550 °C). Color change patterns were similar for dry and for moist soils

22 except for the marked color change occurring at 450 °C in dry soils and at 550 °C in moist soils.

23 Across the heating temperature range, Vista (210m) soils showed the least pronounced increase

24 in darkness at 350°C while Shaver (1737m) soils showed the most pronounced darkening at that

25 temperature range (from dry color of 10YR 5/2 unburned to 10YR 3/3 by 350°C). At higher

26 temperatures, Musick soils (1384m) showed the largest change in dry soil color going from

27 10YR 2/2 at 350 °C to 7.5YR 6/6 at 650 °C.

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.

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).

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.

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

9

1   variability and did not show any significant change in SSA throughout the temperature range

2   while the Chiquito soil showed statistically significant increase between low temperature 150 –

3   250 °C and higher temperature 350 – 550 °C range. The pattern of change in SSA with

4   temperature was similar for all soils. The lowest SSA was recorded for all soils when soils were

5   heated at 250 °C, and highest SSA was observed at 350 °C (for Musick and Chiquito soils) or

6   450 °C (Vista and Shaver soils).

7   ## 3.5   Soil mineralogy

8   The bulk soil XRD results of changes in soil mineralogy in response to heating are presented

9   for basic mineral groups as: feldspar (microcline and orthoclase); plagioclase (albite and

10 oligoclase); amphibole; mica/illite (biotite); kaolinite; gibbsite; and expandable phyllosilicate

11 (montmorillonite and vermiculite). We identified vermiculite with low confidence, since we did

12 not correct with oriented clay treatments, hence it is not certain if the identified peaks are indeed

13 representative of vermiculite, chlorite, or both. The XRD diagrams showed some significant

14 transformations in soil mineralogy with heating, with shifted peaks at higher temperatures

15 suggesting transformation of clay minerals. Layer silicates appeared to collapse structurally,

16 possibly due to dehydration and the removal –OH (Figure 6). Summary mineral composition

17 changes identified from XRD analysis using Rietveld method are presented in Figure 7. Across

18 all the soils, the largest mineral composition change with increased heating temperatures were

19 observed for kaolinite that experiences loss at temperatures above 550ºC. Gibbsite was not

20 found in soils that it was originally present after 450ºC. Furthermore, mica/illite, plagioclases

21 and amphibole mineral groups changed consistently with increasing temperature. The largest

22 change in soil mineralogy with heating was observed for the mid-elevation Music (1384m) and

23 Shaver (1737m) soils that are also among the most developed soils with highest proportions of

24 the 1:1 clay minerals (kaolinite).

25 ## 3.6   Soil pH

26 With increase in temperature, all soils showed a similar pattern of increase in pH (Figure 8).

27 For all soils the largest increase in pH (2.5 – 5 units) occurred between 250 and 450 °C. All the

28 soils started out with slightly to moderately acidic pH and with the exception of Chiquito (2865)

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

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

2   °C.

3   **3.7   Cation exchange capacity**

4   The CEC of our soils ranged from an average of 6 cmol$_c$/kg for Chiquito (2865 m) soils to 25

5   cmol$_c$/kg for Musick (1384 m) soils. With increase of heating temperature all soils showed

6   continued decrease of CEC. With the exception of Musick (1384m) soils, CEC eventually

7   dropped to below our detection limit (2 cmol$_c$/kg) at temperature above 550 °C (Figure 8). For

8   the poorly weathered Chiquito (2865 m) soils, CEC was below 2 cmol$_c$/kg at temperatures 250

9   °C and above. For the rest of the soils, statistically significant changes in CEC ($p < 0.05$) occurred

10   at 450 °C with the exception of Musick (1384 m) soils which showed statistically significant

11   drop at 250 °C and again 350 °C. At 350 °C, all the soils except Musick (1384 m) showed a

12   slightly higher CEC than at 250 °C thus interrupting continuous pattern of CEC decrease with

13   increase in temperature.

14   **3.8   Carbon and nitrogen concentration**

15   The initial concentration of C range from less than 2% (for the Vista soil, 210 m) to over 7 %

16   (for the Musick soils, 1384 m). Soil C concentration decreased with increase in temperature

17   (Figure 9) with the largest decrease occurring between temperatures of 250 and 450 °C. At 450

18   °C, all soils lost more than 95% of their initial C and changes at higher temperatures were small

19   and statistically insignificant ($p < 0.05$). Soil's C:N ratio ranged from 10 (Vista soils, 210 m) to

20   29 (Musick soils, 1384 m). For all soils C:N ratio decreased with increase in heating temperature

21   in a similar pattern as what we observed for the changes in C concentration (Figure 9).

22   The loss of C and N from soils due to heating showed a similar response among all five soils.

23   After 250 °C, all the soils lost more than 25% of their initial C (except Shaver soils that lost

24   only about 10%). At 350 °C all soils lost 50 to 70% of C. Combustion at 450 °C led to loss of

25   more than 95% of their initial C for all soils in this study. Loss of N was lower than that of C.

26   At temperatures greater than 550 °C there was 5 to 15% of soil N still remaining. Consequently,

27   we observed a decrease of C:N ratio with increase in heating temperature. All soils continued

28   to lose about 15% soil N for every 100 °C increase and maintained more than 60% of their N at

1   heating temperatures up to 350 ºC. After heating at 450 °C, all soils lost more than 60% of the

2   initial soil N and 85% by 550 °C.

3   **4   Discussion**

4   Topsoil layer is most affected by extreme temperature during vegetation fires. Our results show

5   significant changes in soil properties as a result of temperature exposure. Our findings

6   demonstrate that alterations and loss of SOM in topsoil, rather than alterations to soil minerals,

7   was the most important driver for the observed changes in soil physico-chemical properties after

8   combustion. Our XRD analysis shows notable changes in soil mineralogy only after the soils

9   were heated to about 450 to 550 °C (Figure 7). In upland ecosystems, such as the Sierra Nevada

10   Mountains, the soils typically have low clay content and low concentration of secondary

11   minerals (Neary et al., 1999;Ubeda and Outeiro, 2009). In addition, these upland temperate

12   ecosystems also tend to have relatively high concentration of SOM, including a fairly well

13   developed O-horizon. Consequently, strong relationships are observed between SOM

14   concentrations and the soils' physical and chemical properties. Simple linear regression

15   analyses between C concentration changes and other soil physical and chemical changes for our

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

17   pH, CEC and N concentrations are associated with changes in C concentration at the different

18   heating temperatures. Table 3 summarizes the correlation coefficients of soil property changes

19   with changes in C concentration.

20   The changes in soil color observed were consistent with the charring of SOM which leads to

21   darkening of the brownish color of the soils. At temperatures over 450 °C, the near complete

22   removal of SOM by combustion and the addition of ash products likely explains the observed

23   lighter color of soils. The increase in Munsell chromas and reddening of soils at these high

24   temperatures has been noted in previous works (e.g. (Giovannini et al., 1988;Ulery and Graham,

25   1993;Ketterings and Bigham, 2000) and is likely a result to oxidation and transformation of

26   iron oxides in a manner analogous to aging of soils and transformations of mineral soil after

27   intense weathering.

28   The extent of mass loss in top soil layers due to vegetation fires is strongly correlated to SOM

29   combustion (Rein et al., 2008). In all of our soils, statistically significant mass loss (p<0.05)

30   occurred within the temperature ranges of SOM combustion. Proportion of soil mass loss with

1   temperature was proportional to initial C concentration of the soils, Musick (1384m) soils,

2   which had the highest initial C concentration (7%) had the steepest soil mass loss and lost 15%

3   of mass at 550ºC while Vista (210m) which had less than 2% C concentration showed the

4   smallest mass loss losing less than 5% mass at even at the highest temperature. The influence

5   of other mass loss mechanisms likely account for mass loss outside of the temperature range

6   where SOM plays a dominant role, dehydration processes are most likely responsible for mass

7   loss at temperatures below 250ºC where the loss and transformation of SOM is minimal. Mass

8   loss at higher temperatures where most of SOM has been combusted (i.e. >450°C) is likely

9   dominated by charring, ashing and volatilization processes.

10   Most studies report significant soil aggregate stability reduction with fire heating (e.g. Zavala

11   et al. (2010);Arcenegui et al. (2008)), however, contrasting findings are also reported in other

12   studies. Mataix-Solera et al. (2011) explains that increase in aggregate strength with heating is

13   possible in clay-rich soils where main cementing agents are inorganic minerals such as calcium

14   carbonates and metallic oxides which would fuse under fire heating increasing aggregate

15   stability. Another possibility is, soils which initially had weak hydrophobicity may show

16   increased aggregate stability due to increase in hydrophobicity, in such cases however aggregate

17   strength would decline at higher severity fires as hydrophobicity is destroyed.

18   Aggregate stability in all our soils generally decreased with increase in heating temperature.

19   Although we did not find it to be statistically significant difference, it is worth noting, that the

20   lowest elevation soil (Vista, 210 m) showed a trend of aggregate stability increase up to 350 °C

21   while the high elevation soils, Sirretta (2317 m) and Chiquito (2865 m), showed a slight increase

22   in aggregate stability at 150 °C. Increase in hydrophobicity at these temperatures is the most

23   likely cause, substantial hydrophobicity was apparent with Chiquito soils (2865 m) heated at

24   250 °C where resistance to slaking was remarkably evident during aggregate stability test.

25   Soil specific surface area (SSA) is an important soil property that affects soil adsorption, ion

26   exchange capacity, reactivity, aggregation and porosity (Feller et al., 1992). SSA of soil is

27   largely dictated by clay-size particles and SOM (Carter et al., 1986). The increase in SSA with

28   heating that we observed in this work is most likely the result of physical disintegration and

29   charring of SOM, especially at temperatures below 500 °C. Changes in soil mineralogy are not

30   likely to be responsible for the changes in SSA we observed. XRD analysis showed notable

1   mineralogical changes only at temperatures above 450 °C where the kaolinite peak disappeared

2   at 550 °C and the phyllosilicate peaks were diminished or disappeared at 450 – 550 °C (e.g.

3   Figure 6). Because of combustion and heat-induced dehydration, larger organic matter particles

4   are likely to have fragmented and reduced in size with increase in heating temperature leading

5   to increase in bulk soil SSA. Furthermore, removal of organic matter from mineral surfaces due

6   to combustion may also increase surface area by reducing overall size of particles. At higher

7   temperatures, XRD spectra showed some collapse of mineral complexes through dehydration

8   and de-hydroxylation of clay minerals that may reduce mineral particle size and increase surface

9   area. These changes in mineralogy might have a significant effect on SOM. A study by Rosa et

10   al. (2013) found that soils released more organic compounds during pyrolysis when the soils

11   were treated with HF acid, suggesting that mineral complexes play a role in protecting organic

12   compounds from combustion. The collapse of mineral complexes we observed and the decrease

13   in aggregate strength is likely to have enhanced thermal oxidation of SOM. However

14   mineralogical changes would play a more important role affecting soil properties at these high

15   temperatures since SOM has been almost completely removed.

16   Soil pH generally increases with fire heating (Ubeda and Outeiro, 2009;Badía and Martí,

17   2003;Chandler et al., 1983). In a similar heating experiment as we followed in our study,

18   Fernández et al. (1997) observed a pH increase of 1.7 at 350 °C and 2.35 at 490 °C, where the

19   authors attributed the change in pH to denaturation of organic acids, the release of base cations

20   from combustion (K- and Na-hydroxides, Mg- and Ca-carbonates), the deposition of ashes and

21   loss of hydroxyl groups from clays (Certini, 2005;Badía and Martí, 2003). In our soils, the

22   higher elevation soils (Shaver, Sirretta and Chiquito) showed a statistically insignificant

23   decrease of 0.3 to 0.5 pH units (measured in water) at 250 °C. The change in pH in our high

24   elevation soils was consistent to previous results of Badía and Martí (2003);Terefe et al. (2008)

25   that found similar initial decrease. Terefe et al. (2008) hypothesized that this may be due to the

26   combined effect of desiccation and heating effect which favor proton-reducing oxidation

27   reactions. And the fact that this initial increase occurs below the temperature for start of

28   combustion of organic acids means contribution of SOM to pH increase (organic acid

29   denaturation and ash liming effect) was absent at this temperature. In a similar heating

30   experiment Badía and Martí (2003) found an increase in electric conductivity and soluble Ca

31   along with decrease in pH at 250 °C. Such increase in soluble cations might explain our findings

1   where we observed a decrease in pH when measured in water but not in CaCl$_2$ suggesting that

2   the decrease in pH might have to do with increase of soluble salts with heating up to 250 °C.

3   The capacity of soil to exchange positively charged ions between soil and soil solution (CEC)

4   decreased with increasing temperature. CEC of soils is a result of surface charges associated

5   with secondary clay minerals and SOM (Sparks, 2005), and in our study soils, Dahlgren et al.

6   (1997) had previously reported a strong relationship of CEC with soil organic carbon and clay

7   concentrations. Different authors have attributed loss of CEC during heating mainly with loss

8   of SOM (Ubeda and Outeiro, 2009;Fernández et al., 1997) partly because CEC loss starts to be

9   observed at temperatures above 200 °C with little or no decrease at lower temperatures where

10   SOM is not affected (Soto and Diazfierros, 1993;Nishita and Haug, 1972). The slight increase

11   of CEC we observed at 350 °C may be due to the steep increase of specific surface area at that

12   temperature (Figure 4). The additional surface for cation adsorption might have to an extent

13   compensated for the loss of SOM at that temperature. Furthermore, the contribution of surface

14   oxidation of char products has been shown to increase CEC per unit C (Liang et al., 2006)

15   because of the almost complete loss of C at temperatures above 450 °C and very little loss at

16   temperatures below 250 °C. The soils most likely had highest concentration of charred SOM at

17   350 °C temperature.

4.1   Importance of the 250 – 450 °C range

19   Based on maximum surface temperature, fires are often classified as low, medium or high

20   intensity. Low intensity fires reach surface temperatures of up to 250 °C, medium intensity fires

21   reach surface temperatures of 400 °C, and high intensity fires reach surface temperatures above

22   675 °C (Janzen and Tobin-Janzen, 2008). In this study, the most significant changes of soil

23   chemical properties occurred at the transition between low and medium severity fires, between

24   250 and 450 °C. Figure 10 illustrates the changes between unburned and 650 °C burned soils

25   and the amount of change that occurred within 250 to 450 °C heating temperature for a range

26   of the variables discussed above. In all cases, the change in the 250 – 450 °C range accounts for

27   most of the total change observed during our combustion treatments. Among the variables we

28   investigated in this study, we observed changes along two general lines: (1) mass loss, SSA and

29   pH which showed a progressive increase with heating temperature, and (2) %C, %N, C:N ratio,

30   CEC, and wet aggregate stability that showed a progressive decrease with combustion

1   temperature (Figure 10), with the most significant changes in all cases being recorded in all

2   soils between 250 - 450ºC.

3   Temperatures below 250 °C are very critical for many processes, water is lost at 95 °C and this

4   has a significant effect on soil heat conduction and soil biota (Janzen and Tobin-Janzen, 2008).

5   However, temperatures below 200 °C have very little effect on quality or quantity of SOM. This

6   means low intensity fires, such as typical prescribed fires, contribute little to soil C loss.

7   Similarly, temperatures above 500 °C do little change to SOM, which already has been lost or

8   transformed into a pyrogenic product. The effect on soil inorganic particles starts at high

9   temperature but the significance of change on minerals is not as large (Figure 1). Hence, we

10   found that the most important soil changes occur the 250 – 450 °C range.

11   Important modifications of fire conditions that still allow for comparison of responses of

12   different types of soils have to be adopted to conduct the type of heating experiments that we

13   undertook in this work. A heating rate of 3 °C min^{-1} is common in laboratory soil heating

14   studies, often because of technical consideration and because such slow rate prevents sudden

15   combustion which otherwise would happen as soil's ignition temperature is reached at about

16   220 °C (Fernández et al., 1997, 2001;Varela et al., 2010). However, it is important to recognize

17   that during vegetation fires the rate of temperature increase experienced by the topmost layer

18   of soil that is exposed to fire can be significantly higher. The rate of heating alone might have

19   additional significant effects on soil properties beyond what we observe here. For example,

20   Albalasmeh et al. (2013) found that slow rate of heating underestimates soil aggregate

21   destruction of moist soils due to a slower buildup of pore-pressure.

22   **4.2   Climate Change Implications**

23   Investigation of the response of climosequence soils to different heating temperature in this

24   study enables us to infer how changes in climate (and associated changes in soil properties) are

25   likely to alter the effect of fires on topsoil physical and chemical properties. Even though the

26   general pattern of change in soil physical and chemical conditions with increasing combustion

27   temperature were mostly consistent for all soils along our study climosequence, the actual

28   magnitude of change in the investigated variables was not. Hence, these finds lead us to

29   conclude that climate change is likely to alter the response of topsoil properties to different fire

1   regimes. Along our study climosequence, we observed critical differences in response of

2   topsoils based mostly on concentration OM in soil and soil development stages of each soil --

3   both variables that are expected to respond to changes in climate (Berhe et al., 2012).

4   Consequently, changes in soil C storage associated with climate change are expected to lead to

5   different amounts of C loss due to fires. This is evidenced by the observed highest total mass of

6   C loss from the mid-elevation Musick soil that had the highest carbon stock, compared to soils

7   in either side of that elevation range. Anticipated changes in climate in the Sierra Nevada

8   mountain ranges are expected to include upward movement of the rain-snow transition line

9   exposing areas that now receive most of their precipitation as snow to rainfall and associated

10 runoff. Moving of the rain-snow transition zone higher and promotion of more intense

11 weathering at higher elevation zones then is likely to render more C to loss during fires. As we

12 found in this study, more than 80% of the variability in mass loss, aggregate strength, SSA, pH,

13 CEC and N concentrations is associated with changes in C concentration at the different

14 combustion temperatures (Table 3). Improving our understanding of how topsoil properties are

15 likely to respond to changes in climate becomes even more critical when we recognize that C

16 concentration in soil is likely to respond quickly to changes in climate, compared to other soil

17 physical and chemical properties (Berhe et al., 2012). Furthermore, the long-term fate of soil

18 carbon in fire-affected ecosystems is also likely to be accompanied by changes in microbial

19 community composition and OM decomposition kinetics (Holden et al., 2015;Tas et al., 2014)

20 which are likely to have further implications for nutrient availability post-fire (Johnson et al.,

21 2007b;Johnson et al., 1997).

22 The different responses of soil aggregation in our climosequence to the treatment temperatures

23 also suggest potential loss and transformation of the physically protected C pool in topsoil.

24 Degradation of aggregates during fire (Albalasmeh et al., 2013) is likely to render aggregate-

25 protected C to potential loses through oxidative decomposition, leaching and erosion.

26 Moreover, in systems such as the Sierra Nevada where steep slopes and organic matter-rich

27 topsoils dominate, movement of the rain-snow transition zone upward is likely to increase

28 proportion of precipitation that occurs as rain. The kinetic energy of raindrops and observed

29 increase in hydrophobicity of soils post-fires (Johnson et al., 2007a;Johnson et al., 2004) can

30 lead to higher rates of erosional redistribution of especially the free light fraction or particulate

31 C that is not associated with soil minerals (Stacy et al., 2015). Moreover, the important

1 differences in changes in pH, mineralogy, CEC in response to heating at different temperatures

2 that we observed for soils along the climosequence suggest that changes in temperature are

3 likely to lead to different effects on soil chemical properties in soils after fires.

4 Finally, with changes in climate it is anticipated that fires will increase in severity (Westerling

5 et al., 2006b). Our findings of important changes in soil physical and chemical properties

6 occurring between 250-450ºC are important for recognizing that critical transformations of

7 topsoil physical and chemical properties are likely to occur when, as a result of climate change,

8 systems that are adapted to low severity fires experience medium to high severity fires.

9 **5   Conclusion**

10 Findings of this study showed that changes in soil properties during heating are closely related

11 to changes in C concentrations in soil. The temperatures most critical to C loss and alteration

12 were found to be 250 °C, where charring of organic matter starts and 450 °C where most of the

13 SOM is combusted. Most soil properties exhibited a steep change in this temperature range.

14 Soil aggregate stability, CEC, and C and N concentrations significantly decreased with

15 increased combustion temperature while soil pH and SSA significantly increased. The most

16 important effect of combustion on soil mineralogy as observed by XRD analysis was the

17 collapse of kaolinite, which was undetectable at temperatures above 500 °C.

18 This study presented the effects of heat input on topsoil properties. The study is necessary to

19 understand the changes that occur under fires that result in heating of soil without additional

20 variables such as the addition of charred plant material and ash, and the influence of soil

21 moisture. Findings from this study will contribute towards estimating the amount and rate of

22 change in carbon and nitrogen loss, and other essential soil properties that can be expected from

23 topsoil exposure to different intensity fires under anticipated climate change scenarios.

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12

1 **Tables**

2 Table 1 Soil classification and site description for the five sites along elevational transect in the western slopes of
3 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

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

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1

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

Soil series and elevation (m)	Bulk density (g/cm³)	Gravimetric water content (%)	pH (CaCl₂)	Carbon (%)	CEC (cmol_c/kg)	SSA (m²/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

4   [a] Particle size distribution of top soil 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

1

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

Soil	Correlation coefficient (r^2) values					
	Mass loss	SSA	Aggregate Stability	pH (CaCl$_2$)	CEC	N concentration
Vista	0.74	0.73	0.21	0.77	0.78	0.89
Musick	0.89	0.58	0.77	0.89	0.96	0.83
Shaver	0.82	0.58	0.68	0.74	0.78	0.93
Sirretta	0.60	0.34	0.47	0.67	0.87	0.86
Chiquito	0.82	0.62	0.78	0.88	0.44	0.87

3

1   **Figures**

2
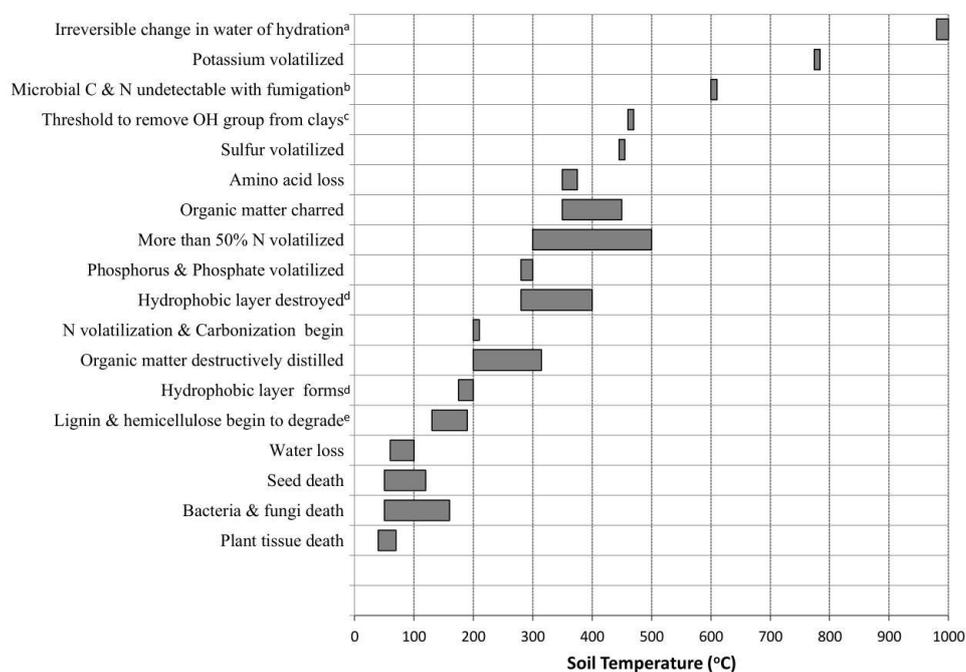
3

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))
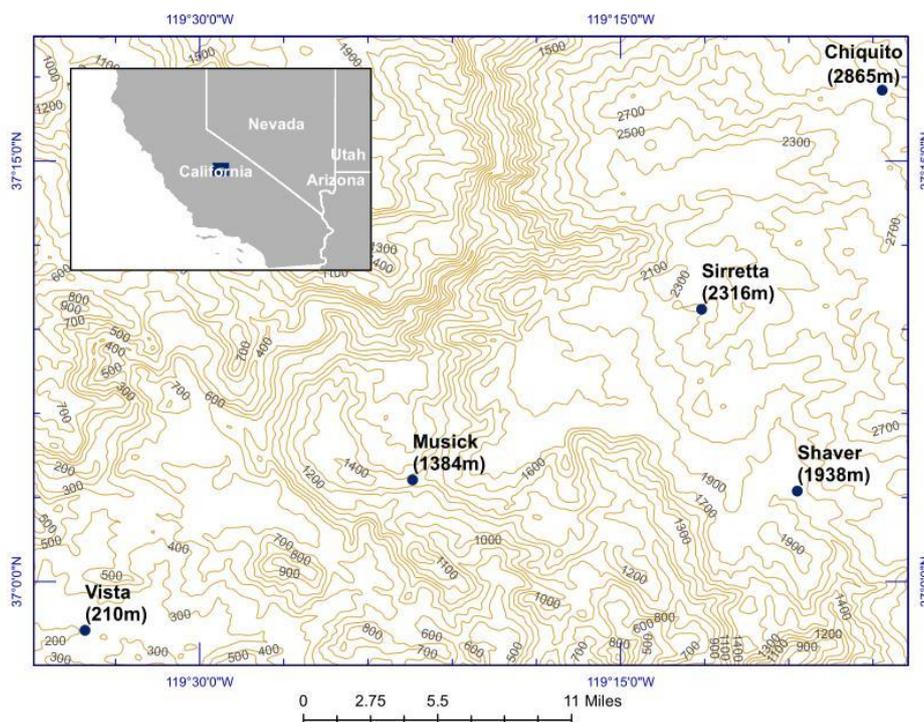
1

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)
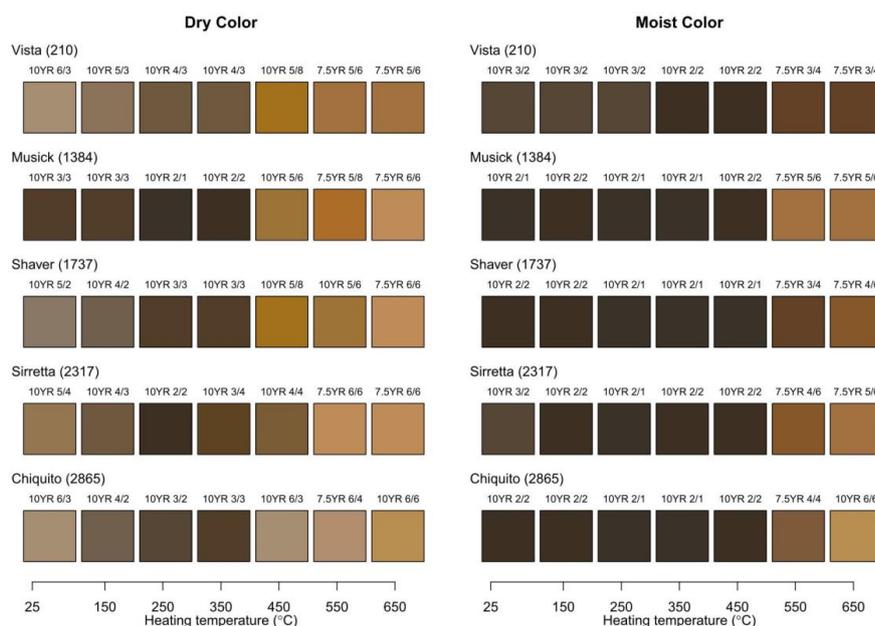
1

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.

1
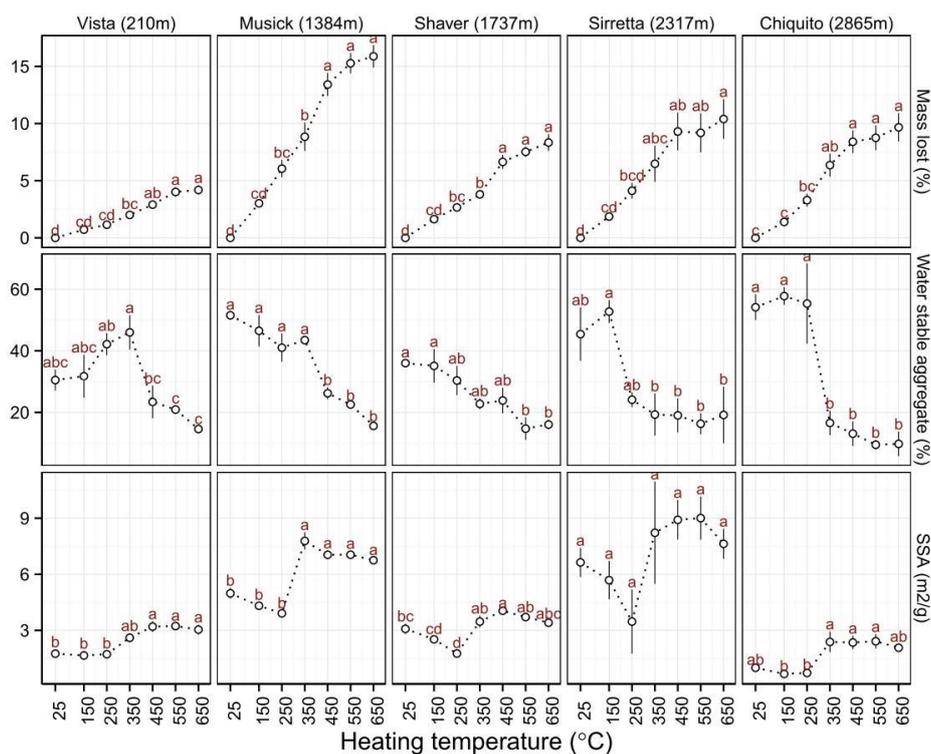
Figure 4: Percent mass lost, water-stable aggregate percent and specific surface area changes
with increase in heating temperatures. Error bars represent standard error where n=3. Different
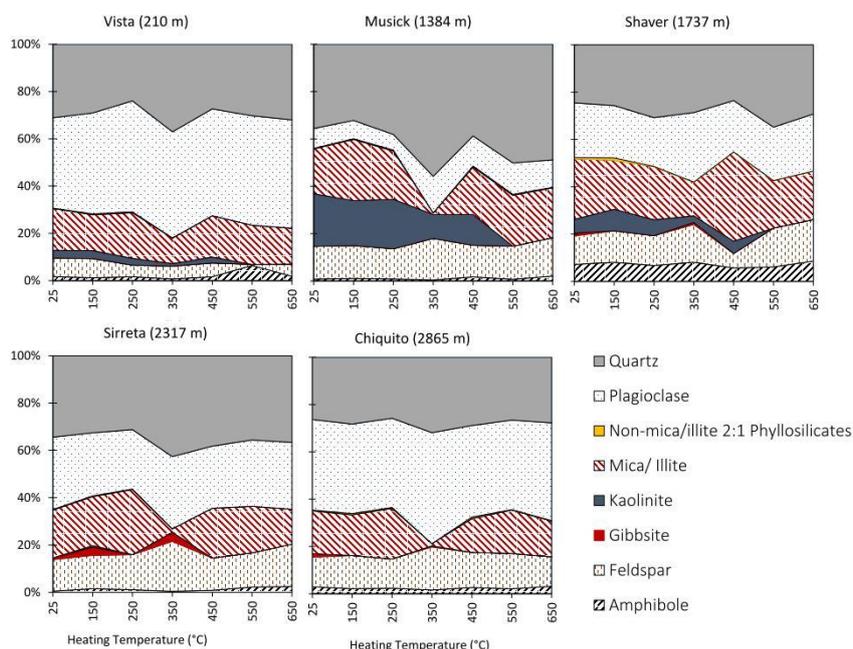letters represent significantly different means ($p<0.05$) at temperature after Tukey's HSD
testing.
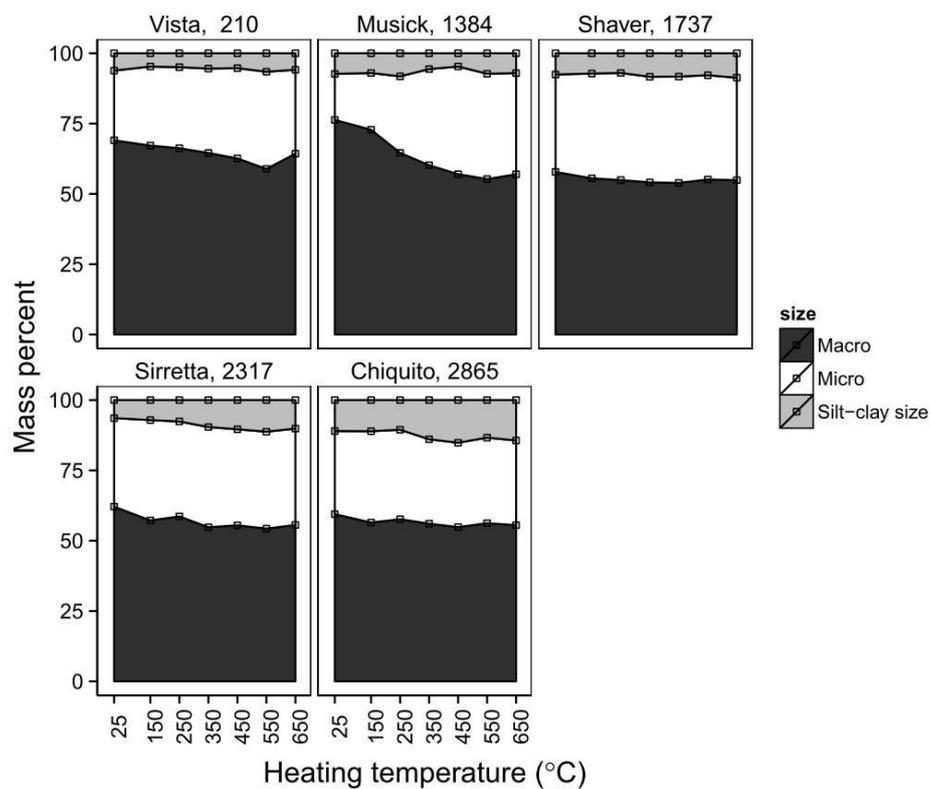
1

2   Figure 5: Weight fraction of aggregate sizes: macro (2-0.25 mm), micro (0.25-0.053 mm) and
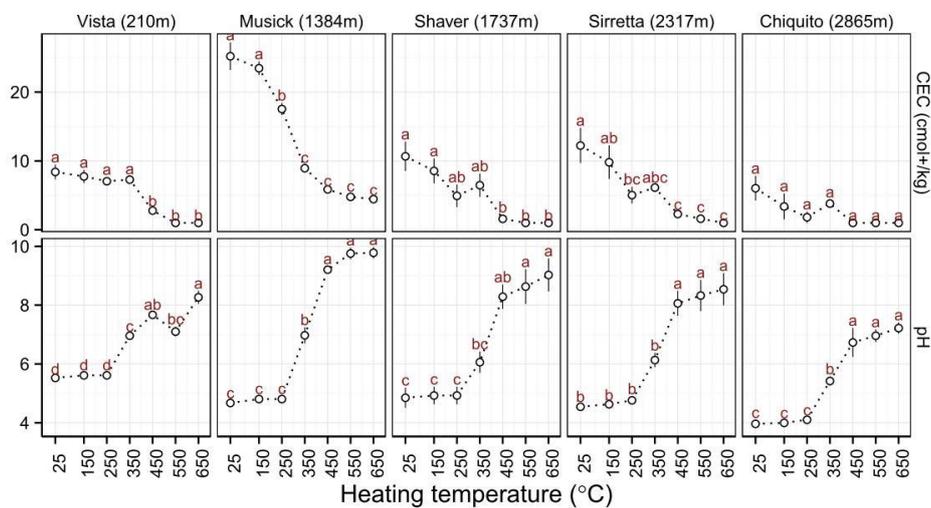
3   silt-clay (<0.053 mm) sizes.
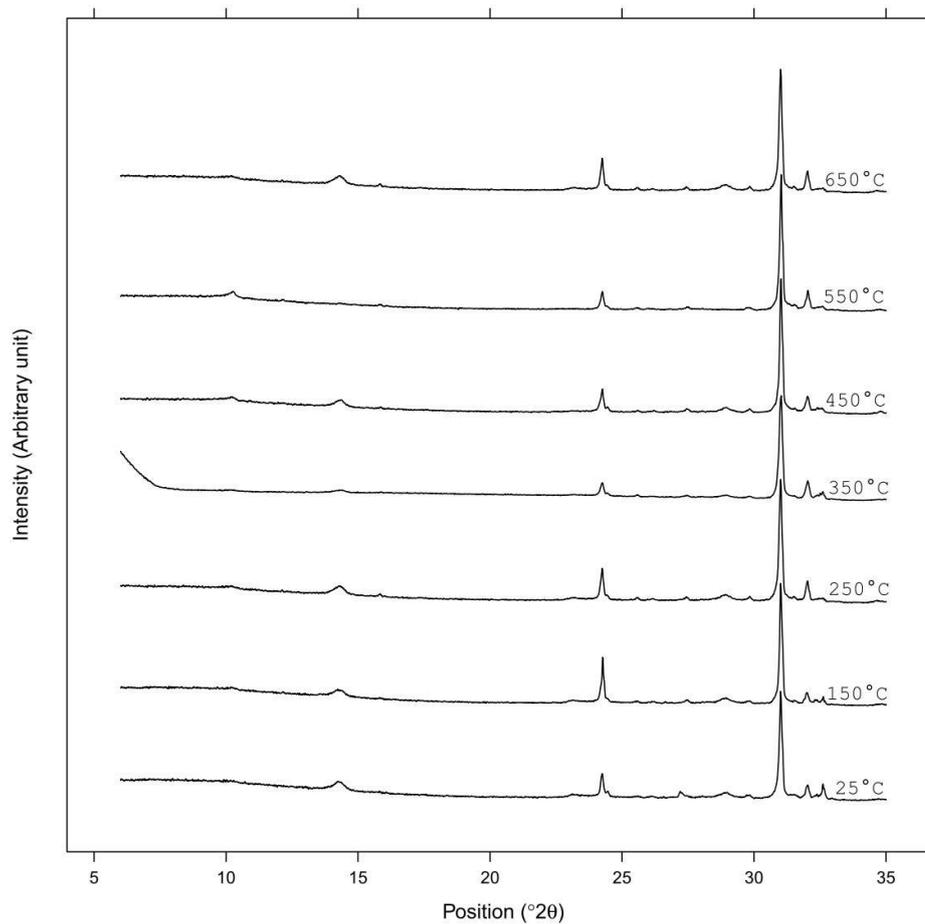
1

2   Figure 6: XRD diagram for Music series soils.

1

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

3

4

SOIL
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1 Figure 8: pH (geometric means) and cation exchange capacity (adjusted for mass loss) changes

2 with increase in heating temperature. CEC values below the 2 $cmol_c$/kg are assigned a value of

3 1 for plotting. Error bars represent standard error where n=3. Different letters represent

4 significantly different means ($p < 0.05$) at each temperature after Tukey's HSD testing.
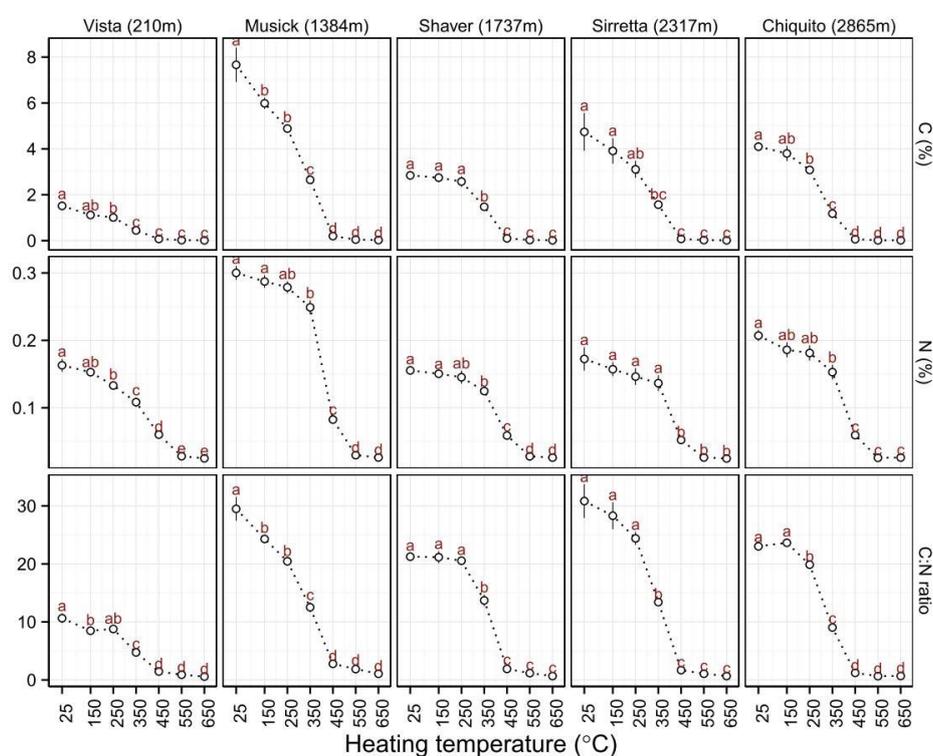
5

6 Figure 9: Carbon concentration, Nitrogen concentration and C:N atomic ratio changes with

7 increase in heating temperature. Error bars represent standard error where n=3. Different letters

8 represent significantly different means ($p < 0.05$) at each temperature after Tukey's HSD testing.
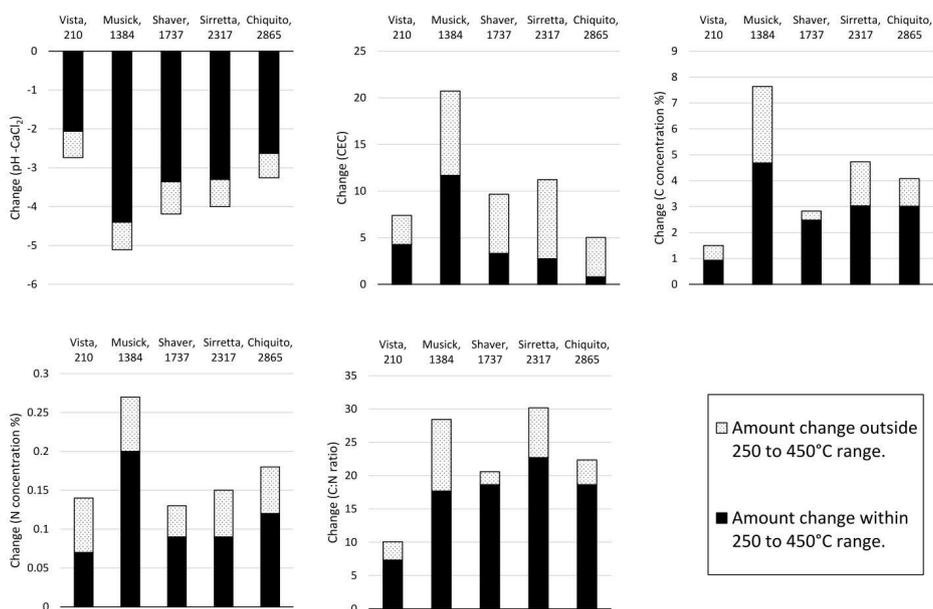
1

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.