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Estimation of temperatures beneath archaeological campfires using carbon stable isotope composition of soil organic matter

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Abstract

An Inceptisol and an Alfisol from Western Maryland were sampled by horizon and exposed to the elevated temperatures associated with heat transfer measured beneath campfires. Substantial loss in organic carbon and change in carbon stable isotope composition was observed, even after exposure to relatively low temperatures (200–400 °C). Analyses showed that a comparison of carbon isotope enrichment in organic matter beneath a suspected campfire site to that of unaltered soil would give quantitative information about the minimum effective campfire temperature. Application of these results may lead to particular insight for archaeological sites where soil surfaces have been eroded or otherwise truncated.

Keywords: Campfire; Soil carbon; Temperature estimate

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1. Introduction

The identification of anthropogenic fire (e.g., campfires) is a major factor in defining and describing the past human habitation of an archaeological site. The characterization of past anthropogenic fires is becoming an increasingly sophisticated practice, involving both archaeologists and physical scientists (e.g. [1]). Fires have been set by past humans in order to facilitate a wide range of activities, including food preparation, religious ceremonies, disease control and warfare (reviewed within [28]). Classical methods of assessing campfire temperature include the examination of obsidian (e.g. [30]) and the evaluation of the morphology of various charred substances (e.g. [31]) and stone tool residues (e.g. [38]). Each of these approaches is limited to qualitative inference about fire presence or absence. With this work we sought to establish a measurable chemical threshold that would imply a minimum fire temperature within the range of common combustion. Towards this, we have produced a thorough description of the carbon stable isotope and organic mass changes within the genetic horizons of mineral soils after exposures to temperatures seen during fires.

Soil organic matter is a ubiquitous and potentially quantitative indicator of fire intensity. The mineral horizons of common soils (the A and B horizons) may contain up to 20% C_{org} by mass; this carbon has the potential to be released through heat-transport processes. Carbon isotope patterns within soils also show a characteristic enrichment with depth [4] and have the potential for alteration during the selective removal of selected compounds (e.g., cellulose) at elevated temperatures. Because the physical chemistry of the basic components of soil organic matter are well known (Table 1), it should be possible to create a quantitative model of temperature and soil carbon loss after exposure to the elevated temperatures commonly produced by campfires.

2. Selection of field sites: pedogenic end-members

Two soils were selected for study; we identify them as effective pedogenic end-members, which means that they span a wide range of soil properties (Table 2 and Fig. 1) and effectively encompass all soil attributes we consider important to

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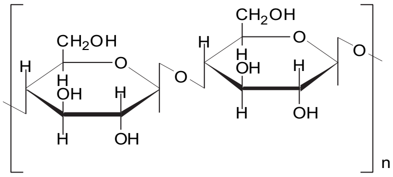
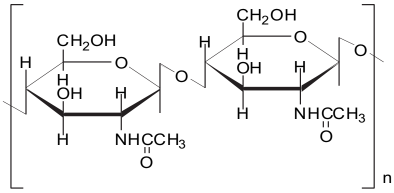
Table 1
Combustion and related properties for some compounds

Compound	($\delta^{13}C_{\mathrm{Compound}}$− $\delta^{13}C_{\mathrm{bulk\ tissue}}$) (‰)	Combustion temperature (°C)	Empirical equation for the predication of HG[i] (kcal/mol)	HG (kcal/mol) calculated; measured [j]
Cellulose (poly-*n*-glucose)	(+1.0 to +3.5)[a]	(310 to 370)[b]	n.d.	n.d.
				
Lignin (poly-*n*-phenylpropane)	(−1.0 to −3.0)[c]	(360 to 400)[d]	n.d.	n.d.
Chitin (poly-*n*-*N*-acetylglucosamine)	−0.5[e]	(270 to 285)[f]	n.d.	n.d.
				
Lipids, waxes, cutin and miscellaneous fatty acids				
Fatty acids: saturated and monosaturated molecules	(−1.0 to −10.0)[g]	n.d.[h]	see note below	see note below
14:0 Fatty acid methyl ester	n.d.	n.d.	$25.11 + 25.80N$	2244; 2254
18:0 Fatty acid methyl ester	n.d.	n.d.	$25.11 + 25.80N$	2863; 2859
20:0 Fatty acid methyl ester	n.d.	n.d.	$25.11 + 25.80N$	3172; 3170
14:1 Fatty acid methyl ester	n.d.	n.d.	$27.36 + 26.02N$	2213; 2208
18:1 Fatty acid methyl ester	n.d.	n.d.	$27.36 + 26.02N$	2838; 2828
18:2 Fatty acid methyl ester	n.d.	n.d.	$27.36 + 26.02N$	2785; 2794
18:3 Fatty acid methyl ester	n.d.	n.d.	$27.36 + 26.02N$	2733; 2750

Note: due to large values of *n* (>1000) inherent to the structures of cellulose and chitin, the relatively low HG values determined for fatty acids within SOM are consistent with even lower temperatures of combustion than those reported for cellulose, lignin and chitin.

[a] [36,32,29].

[b] [33,8,29,9,10,19,25].

[c] [36,29].

[d] [33,19,13].

[e] [37,35].

[f] [26,21,5].

[g] [13].

[h] (n.d., Not determined); combustion temperatures have not been determined for the fatty acid molecules of interest.

[i] Empirical equations are available to predict gross heats of combustion (HG) [23] using the number of valence electrons (*N*).

[j] Empirical equations were used to calculate these quantities [23], and compared to those measured [14].

this study. The soils are classified as an Alfisol (a soil known to be mature in its properties) and an Inceptisol (a soil known for its lack of development). The soils indeed exhibit striking differences: the Alfisol shows genetic horizons to almost twice the depth of the Inceptisol; the Alfisol also contains twice as much organic carbon in the upper horizons, twice as much clay at depth and several times more silt-sized particles, relative to the Inceptisol.

Soils were collected from Camp Singewald, a research field site owned by Johns Hopkins University, is located approximately 3 miles northwest of Clear Spring, MD in Washington County. In March 2003, two soils were sampled from locations bordering Lane Run, a stream that bisects the Camp. Due to a shift in streambed location, a lower terrace has been cut along the stream bank in one location allowing a younger soil, an Inceptisol to develop just a few meters from the older forest soil, an Alfisol. The Alfisol (CS-1) sample location is located approximately 15 m east of the Inceptisol (CS-2). Because the soils are a short distance apart and all other soil-forming factors remain constant, the soils represent a chronosequence with time of development the only change in soil-forming factor between the two sites. The selection of a chronosequence for this study allows for the direct comparison of the effects of elevated temperatures on two soils with identical biologic and mineral source material but with different contributions from each within pedogenic horizons.

The climate of Western Maryland is temperate and moderately humid with an average temperature of 12 °C and an annual precipitation average of 96.04 cm. The site lies in a flat

Table 2
Pedon descriptions for soils studied

Horizon	Depth (cm)	Color	Organic C (g kg^{-1} bulk)	Sand (%bulk)	Silt (%bulk)	Clay (%bulk)	$\delta^{13}C_{org}$ (‰)
Alfisol: fine loamy mixed mesic Ultic Hapludalf [a]							
Oa$_1$	0–6	10YR 3/1	218.40	n.m.[b]	n.m.	n.m.	−28.1
A$_1$	6–16	10YR 5/3	96.80	45.3	44.6	10.1	−27.0
A$_2$	16–23	2.5Y 5/4	4.82	30.5	57.5	12	−25.4
BA$_1$	23–34	2.5Y 6/6	2.87	34.8	50.3	14.9	−24.8
Bw$_2$	34–45	10YR 6/6	2.88	54.9	28.2	16.9	−24.8
C$_1$	45–56	10YR 5/6	1.94	63.6	14	22.4	−23.5
C$_2$	56+	10YR 5/8	3.09	58	25.2	16.8	−24.4
Inceptisol: loamy mixed mesic Typic Dystrochrept [a]							
Oa$_1$	0–3	10YR 3/2	90.30	n.m.	n.m.	n.m.	−27.3
A$_1$	3–12	10YR 4/3	16.14	79.3	11.6	9.1	−26.4
A$_2$	12–19	10YR 3/6	7.22	78.7	13.3	8.0	−25.7
Bw$_1$	19–26	7.5YR 4/4	6.22	79.1	13.1	7.8	−25.7
B$_2$	26–37	10YR 4/6	4.39	84.6	5.1	10.3	−25.6
C$_1$	37+	10YR 3/6	2.71	79.1	7.8	13.1	−25.2

Note: samples are stored in the collections of Johns Hopkins University Department of Earth and Planetary Sciences, Baltimore, MD 21218.

[a] Soil Survey Staff and Soil Conservation Service (1999) Keys to Soil Taxonomy.

[b] No mineral component present.

valley in the Ridge and Valley Province of the Appalachian Mountains and is dominated by a mixed hardwood deciduous forest. Some of the more common trees seen onsite were American beech, yellow poplar, witch hazel and white oak. Select soil properties are included in Table 2. The valley is underlain by meta-dolostone; sandstone and shale from nearby outcrops can be found within the soil profile and stream area and can be considered to be the parent material.

3. Observations of below-ground temperatures during campfires

Soil temperatures ranging from less than 100 °C to over 700 °C have been recorded underneath surface fires [7,39]. In this study, we quantified both isotopic change and C_{org} loss from soil horizons exposed to a large range of temperatures, including the relatively low temperatures that would be present within a thermally attenuating soil profile. In order to verify published temperature ranges, we created several small ($\sim 5\ m^2$) fires directly over both soils of the site; only fallen wood and leaf litter was used as fuel. Temperature in the soil was monitored at 1 cm intervals to a depth of 4 cm using K-type thermocouples within ceramic sleeves; we cleaned a small vertical profile and inserted the thermocouples into undisturbed soil. Approximately 3 cm of litter layer was present above the soil and was comprised of deciduous leaf material and a few small branches. Fig. 2 shows the temperatures recorded during an average fire event. During the 2-h event, temperatures in the soil rose rapidly to ~95 °C and persisted for the short time required to evacuate any soil water ($\leq$15 min) [7]. The uppermost 1 cm of soil reached nearly 700 °C, the second and third cm of organic soil rose to temperatures of 400–500 °C and 300–400 °C, respectively. The uppermost mineral horizon saw a maximum temperature of 280 °C for several minutes. All depths remained above 150 °C for at least 1 h.

4. Methods

There are several reasons why it was not possible to accurately measure the change in carbon isotope signature or the change in soil carbon amount within soil horizons directly resulting from the burning events described above. During any fire event, ash and debris from above-ground burning filters down into the upper horizons of the soil. Burned material of the soil organic horizons is mixed between layers, and distinct boundaries are no longer visible. Because of this, we relied on the laboratory methods described below.

Approximately 1 kg of soil from each horizon listed in Table 2 was transported back to the laboratory where %sand, %silt and %clay values were determined gravimetrically. In addition, each mineral horizon (genetic horizons A, B and C) was sieved using a 2 mm screen in order to remove rocks and root material. Organic horizons (genetic horizon O) were not sieved, but rock and larger roots ($\geq$1 mm) were identified visually and hand-picked. Ten grams of each resulting sample were air-dried and weighed into porcelain crucibles. The samples were then oven dried at 100 °C for 24 h and reweighed to determine air-dry water content and to determine pre-fire ignition weight.

Each crucible was placed in an Isotemp Programmable Muffle Furnace and subjected to an elevated temperature held constant for 6 h. Several studies have shown that nearly all mass lost from plant material occurs within several minutes of fire [34,20,6]. However, it has also been shown in this study (Fig. 2) and previous works [7,22,11] that elevated temperatures can persist in soils for several hours after fire. We chose to expose our samples to 6 h of elevated temperature in order to approximate a normal soil-heating regime. The muffle furnace exhaust material was liberated into a chemical hood; the furnace itself is subject to considerable air flow during operation; oxygen and other gases flow freely into the furnace during incineration. We used elevated temperatures ranging from 150 °C to 500 °C in 50 °C increments. This range represented the range of temperatures documented within the top 5 cm of soil during a fire event [11]. After exposure to 6 h of a given elevated temperature, crucibles were removed from the muffle furnace and reweighed in order to estimate total mass lost during incineration. After exposure to temperatures higher than 500 °C, all samples retained $< 0.06\%$ C.

All samples (i.e., soils before and after exposure to elevated temperatures from each horizon of the two profiles) were prepared for analysis in keeping with the methods described by Boutton [2]. An Eurovector Elemental Analyzer (EA) was used in conjunction with a Micromass Isoprime IRMS to determine the %carbon (by mass) and the $\delta^{13}C$ value of each sample. Organic carbon stable isotope ratios ($^{13}C/^{12}C$) are reported according to standard permil [‰] convention, relative to Pee Dee belemnite.

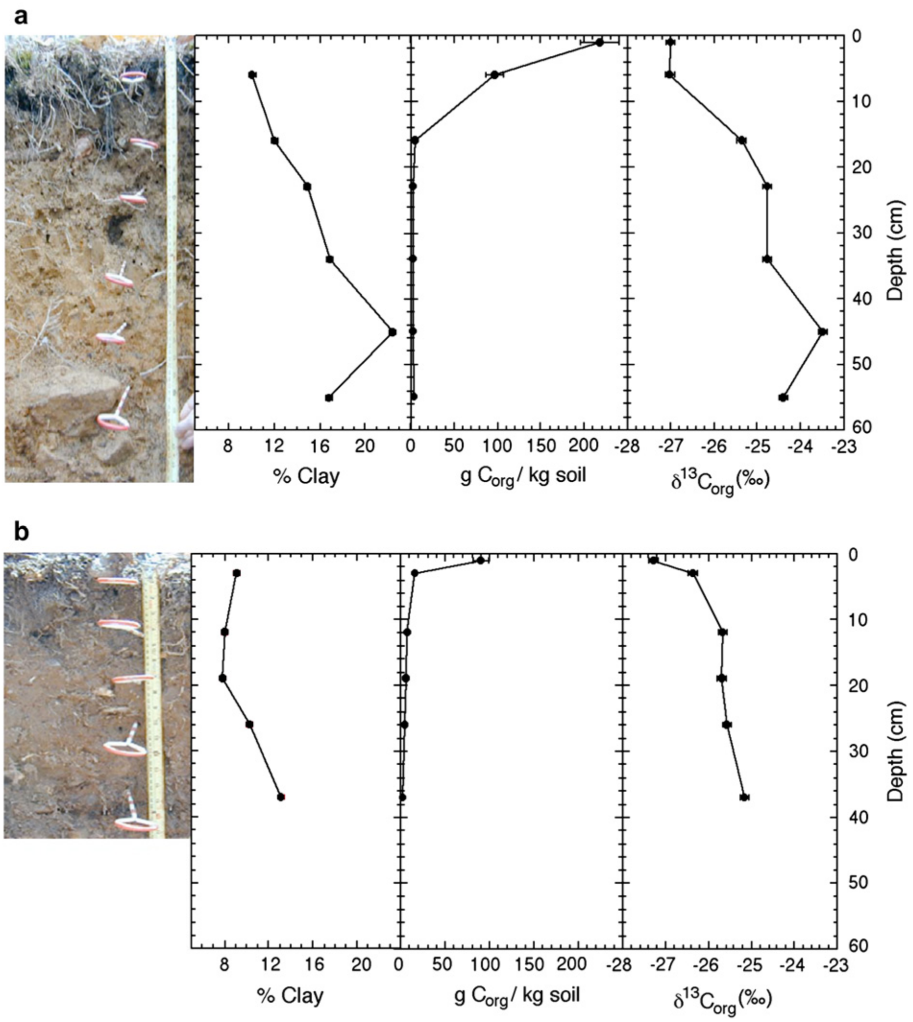


Fig. 1. This figure depicts the original levels of clay, C_{org} content and $\delta^{13}C$ values vs. depth in both the Alfisol (a) and the Inceptisol (b). The $\delta^{13}C$ values reported for the unburned soil horizons in this study (-28 to -23‰; Table 1) are within the range of values seen in C-3 plant dominated ecosystems [15,16] and the soils that form beneath them [3], which is consistent with our vegetation surveys.

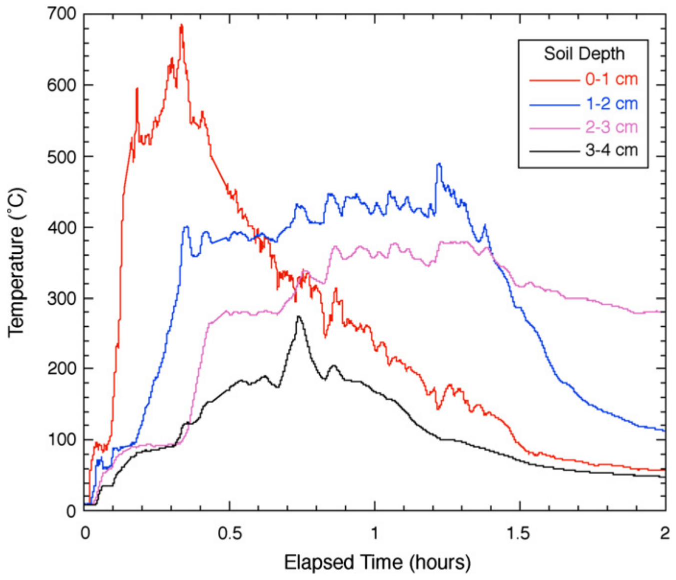


Fig. 2. The soil temperatures beneath an average experimental burn at the field site. The temperature rise was delayed at 95 °C until the soil water was evacuated, then there was a rapid increase to temperatures that remained high for over 1 h. Temperature uncertainty = 0.1% of measurement.

5. Change in soil $\delta^{13}C_{org}$ beneath campfires

We defined net change in $\delta^{13}C$ value for all samples:

$$\Delta\delta^{13}C_{org} = \delta^{13}C_I - \delta^{13}C_U \tag{1}$$

where $\delta^{13}C_U$ and $\delta^{13}C_I$ refer to the stable isotope value of the unheated and incinerated soil, respectively. Net isotopic change ($\Delta\delta^{13}C$) generally increased after exposure to 250–400 °C (Fig. 3 top left and right). At 400 °C, all horizons show $\Delta\delta^{13}C$ values ranging from 2.3 to 3.5‰. The largest differences in $\Delta\delta^{13}C$ between temperature increments tended to occur between 250 and 300 °C, ranging from 0.5 to 3.0‰. After exposures between 150 and 250 °C, all O and A soil horizons showed increasing $\Delta\delta^{13}C$ values, but the lower horizons (B and C) showed decreasing $\Delta\delta^{13}C$ values, pointing to an intriguing relationship between carbon content and isotopic fractionation during burning (further discussed below). In most cases (i.e., below exposures = 300 °C), the greater the depth of the horizon within the soil profile, the lower the $\Delta\delta^{13}C$ value at each temperature exposure.

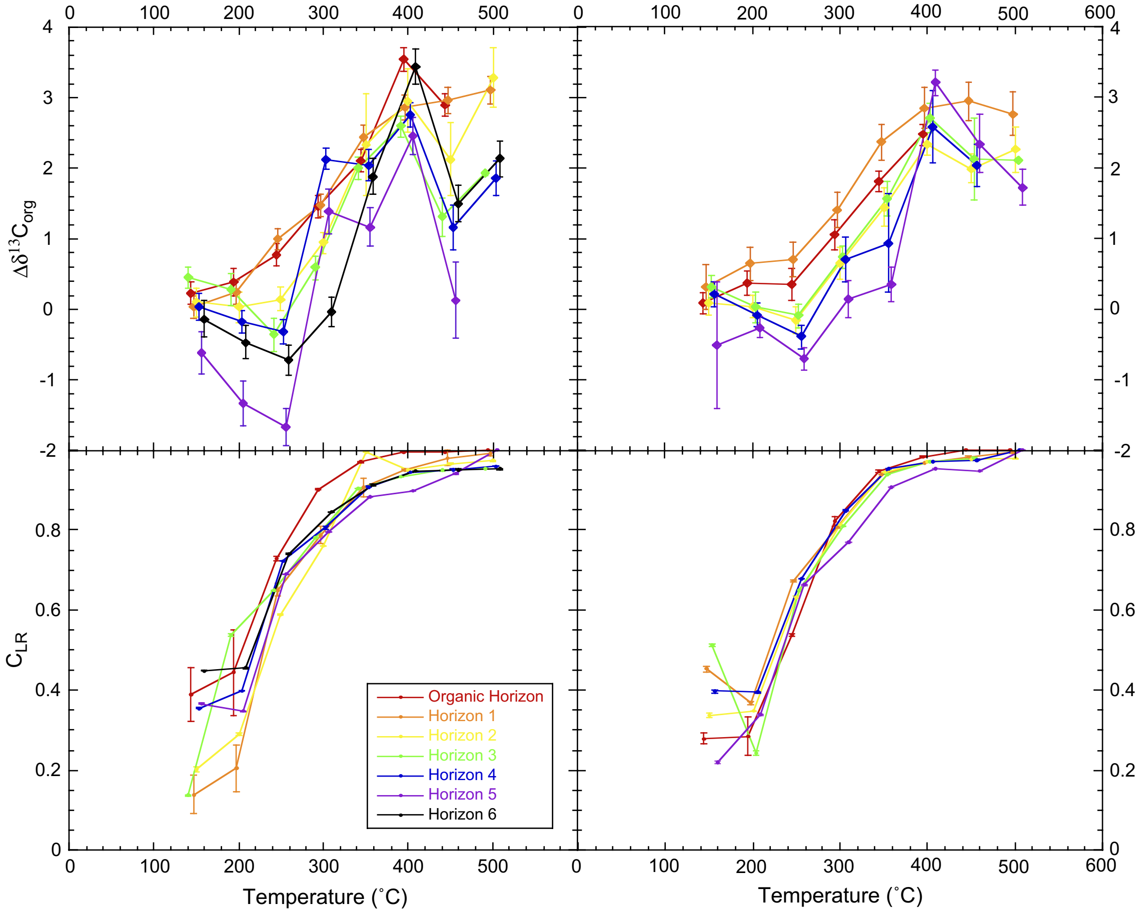


Fig. 3. The effects of elevated temperature on $\delta^{13}C$ values for both the Alfisol (top left) and Inceptisol (top right); the effects of elevated temperature on carbon loss ratio for both the Alfisol (bottom left) and Inceptisol (bottom right). Uncertainties: %clay carries a standard error of 5% based on the variability seen across five replicates; %carbon uncertainty is reported as the standard deviation seen across three samples; analytical precision of $\delta^{13}C_{org} = \pm 0.1‰$. The temperature uncertainty of the furnace was ±5 °C.

We observed a strong overall trend for soil organic carbon to become isotopically enriched during exposure to elevated temperature (Fig. 3 top left and right); our experiments revealed enrichments of up to 3.5‰ relative to unburned soil. This enrichment is presumably caused by the preferential departure of isotopically light carbon compounds from the soil organic carbon pool at each elevated temperature. Table 1 illustrates the relatively low temperatures at which ^{13}C-depleted SOM compounds such as lignin, chitin and various fatty acids are thought to combust; such a departure would lead to the trends we see in Fig. 3 top left and right. We note that that horizons from greater depth in the profiles do not show a consistent $\delta^{13}C$ enrichment with elevating temperature, but instead become depleted by >1‰ after exposure to temperatures of 150 °C through 250 °C. Fernandez et al. [18] demonstrated that recently-incorporated (i.e., less decomposed) SOM is less thermally resistant than more decomposed SOM. Applying this principle in a generalized fashion, lower soil horizons contain SOM in an advanced state of decomposition relative to horizons closer to the surface; it is therefore likely that lower-profile SOM better resisted thermal alteration after exposure to the lowest temperatures of the experiments.

6. Loss of soil carbon from beneath campfires

We determined the amount of organic carbon lost after exposure to elevated temperature as a carbon loss ratio (C_{LR}):

$$C_{LR} = \frac{(M_U C_U \times 0.01) - (M_I C_I \times 0.01)}{(M_U C_U \times 0.01)} \qquad (2)$$

where M_U and M_I refer to the mass [g] of the unheated and incinerated soil, respectively; C_U and C_I refer to the %C (by mass) in the unheated and incinerated soil, respectively, as determined by the EA. This results in C_{LR}, a unitless measure of organic carbon lost from each sample relative to its original content; a value = 1 signifies that all carbon has been lost as a result of the temperature elevation process.

We observed C_{LR} to increase dramatically between 200 and 350 °C with average $C_{LR} = 0.36$ at 200 °C and average $C_{LR} = 0.96$ at 400 °C (Fig. 3 bottom left and right). After exposure to temperatures above 200 °C, horizons within each soil differed in C_{LR} by less than 0.12 (94% were within 0.07), indicating that the highly variable soil properties inherent to each horizon caused no systematic or significant difference in C_{LR}. However, for both the Alfisol and the Inceptisol, the organic horizons (O_a) showed a complete loss of carbon (i.e., $C_{LR} \approx 1$) at lower temperatures than did any other horizon. Every horizon studied achieved maximum C_{LR} value at approximately 400 °C.

Loss of SOM C_{org} after exposure to highly elevated temperatures has been hypothesized in numerous publications (e.g. [12,24]); carbon emissions from various above-ground fuel types are well-established [34,33]. Ninety percent or more of the C_{org} within our soil samples was liberated after exposure to 400 °C (Fig. 3 bottom left and right); this is similar to reports describing the incineration of live vegetation and litter material [8,27]. The striking result of our study is that in the absence of flame, soil horizon average C_{LR} increased from 0.36 up to 0.81 between 200 and 300 °C (C_{LR} values averaged across all horizons). These temperatures are far lower than those required for the flaming combustion of very common organic materials; our data imply that as heat is transferred downward through soil, substantial C_{org} loss may occur. This claim is consistent with previous qualitative observations: Fernandez et al. [17] noted a complete disappearance of SOM after a natural fire within a Mediterranean forest.

7. Correlation between soil properties and isotopic change and/or carbon loss

In order to quantify the extent to which C_{LR} and $\Delta\delta^{13}C$ values obtained from the burn treatments depended upon the original amount of C_{org} and/or clay present we performed the statistical analyses illustrated by Table 2 and Fig. 1. In each case, a linear regression through the data returns an R^2 value near zero and $P > 0.05$ (Fig. 4). Considering the large range of both C_{org} content and %clay in these samples, we conclude from the statistical analyses that neither of these important soil characteristics governed organic carbon loss or isotopic fractionation during exposure to elevated temperatures in a straightforward (e.g., linear) fashion.

8. Change in soil organic matter morphology beneath campfires

Diagnosis of archaeological fires has relied on macro- and microscopic changes in the organic matter contained in the soil profile at or near sites of habitation. To further explore

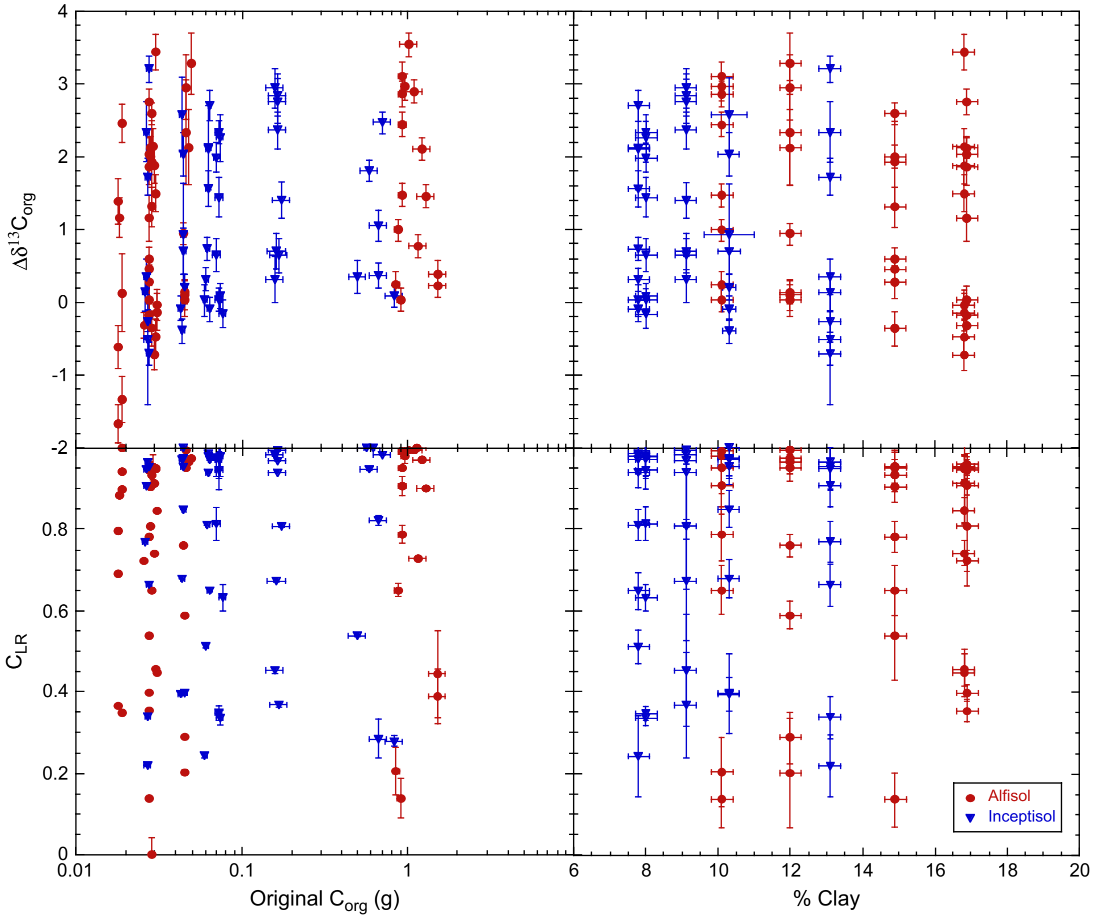


Fig. 4. Carbon loss ratio for all soil horizons plotted against the corresponding original C_{org} content and percent clay; $\Delta\delta^{13}C_{org}$ values plotted against the corresponding original C_{org} content and percent clay. Errors are propagated from those specified in main text; no significant correlation was found for any of these relationships.

this practice, we have analyzed these incinerated and non-incinerated soils treated in the muffle furnace under Scanning Electron Microscopy (SEM) to examine the morphological changes which occurred in the soil as the temperature increased. Fig. 5 shows evidence of plant structures at temperatures of 300 °C showing that complete combustion at this temperature has not occurred even though the horizons for our soils have an average C_{LR} of 0.80 at that temperature. It is also interesting to note that the structure of fungal spores appears resistant to higher temperatures, even at 400 °C. For these reasons we believe that changes in the carbon isotopic composition of soil organic matter may be a more reliable indicator of campfire intensity than the microfossil content of soil organic profiles.

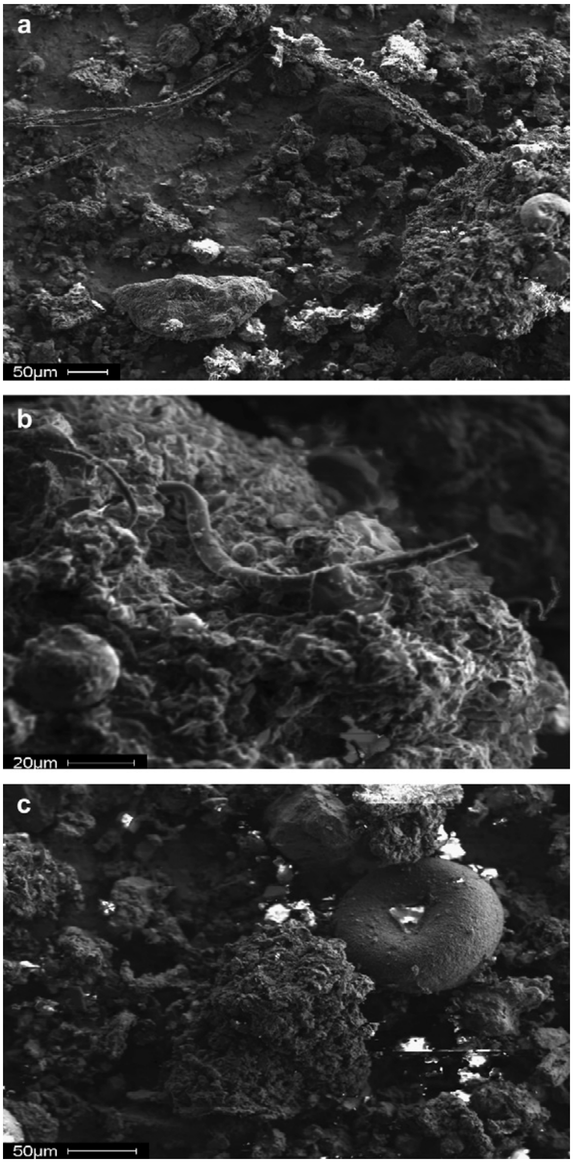


Fig. 5. These scanning electron micrographs of select soil horizons treated in the laboratory reveal that at temperatures as high as 300 °C (a and b), there is considerable evidence of plant remains indicating that combustion is not complete at this temperature. Evidence of the structural remains of fungal spores (c) can also be seen at temperatures as high as 400 °C.

9. Prediction of temperature beneath campfires based on the carbon isotope composition of archaeological soils

As described above, we determined the changes in stable carbon isotope ($\delta^{13}C_{org}$) value caused by exposure to elevated temperatures. SOM includes a myriad of compounds that are of extremely variable molecular weight, however, certain groups of compounds are thought to constitute the most important components (Table 1). Because these compounds exhibit characteristically light or heavy carbon isotope composition relative to bulk SOM, change in $\delta^{13}C_{org}$ value shed light on changes in SOM composition resulting from exposure to elevated temperatures.

One prediction that emerges unequivocally from Fig. 3 top left and right is that an observed ^{13}C enrichment in SOM of $+1$ to $+3‰$ relative to unburned adjacent SOM implies exposure to temperatures between 200 and 400 °C. These mid-range fire temperatures are a useful interpretation of either fire intensity or temperature at depth. Because the analytical error associated with $\delta^{13}C_{org}$ measurements is commonly $\leq 0.1‰$, such enrichment can be quantified without uncertainty. The interpretation of mid-range temperatures might be particularly useful for the reconstruction of human activities: 200–400 °C is more than sufficient for the boiling of water, or the reducing of fluids, however, these temperatures are not high enough for the flaming combustion of wood, more useful for warmth than specific cooking practices. We look forward to applying these results to archaeological sites undergoing active excavation. Our results might be of particular importance to sites where the surficial features of ancient campfires have previously eroded or the soil profile has otherwise been truncated. A description of the implementation of the temperature-estimation technique that we propose is as follows: two sets of soil samples are taken, one set below what archaeologists suspect to have been a fire of some type, the other of the same age soil, at some unburned location. The carbon content, both the amount of organic carbon and the carbon isotope value of the organic carbon, should be compared between soils. The larger the difference in the two sets of properties, the higher the temperature that can be postulated. Specific quantitative claims could follow a comparison to patterns put forward in Fig. 3.

10. Conclusions

It is notable that, given the widely varied characteristics of the soil horizons included in the study, essentially no difference in the C responses was revealed between any of the epipedon or subsurface horizons. The amount of C_{org} present in the original samples and sediment size of the soil appear to play no major role in defining the loss of C_{org} or fractionation of ^{13}C with increasing temperature. Temperature is shown to independently control the changes we saw in these soils, with the possible exception of the importance SOM depth at very low temperature exposure. Our work shows that considerable change in $\delta^{13}C_{org}$

value as well as carbon loss from soils at temperatures well below what are commonly assumed to be required for widespread ecosystem burning, and are more germane to the physical parameters of a campfire. Because of the variation we have captured in the characteristics of these two soils, the results here can appropriately be generalized to the majority of soils that exhibit intermediate properties. As such, this study presents a new method for the estimation of campfire minimum temperature, based on the comparison of $\delta^{13}C_{org}$ values within SOM within and outside of the burned site.

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