

A modern analogue matching approach to characterize fire temperatures and plant species from charcoal

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ABSTRACT

Charcoal identification and the quantification of its abundance in sedimentary archives is commonly used to reconstruct fire frequency and the amounts of biomass burning. There are, however, limited metrics to measure past fire temperature and fuel type (i.e. the types of plants that comprise the fuel load), which are important for fully understanding the impact of past fire regimes. Here, we expand the modern reference dataset of charcoal spectra derived from micro-Fourier Transformed Infrared Spectroscopy (FTIR) and apply an analogue matching model to estimate the maximum pyrolysis temperature and the type of plant material burned. We generated laboratory-created reference charcoal from nine plant species that were heated to six temperature categories (100 °C increments between 200 °C–700 °C). The analogue matching approach used on the FTIR spectra of charcoal estimated the maximum pyrolysis temperatures with an accuracy of 57%, which improved to 93% when accuracy was considered ± 100 °C. Model accuracy for the type of plant material burned was 38% at the species level, which increased to 67% when species were grouped into trait-based categories. Our results show that analogue matching is an effective approach for estimating pyrolysis temperature and the type of plant material burned, and we suggest that it can also be applied to charcoal found in palaeoecological records, improving our understanding of past fire regimes and fuel dynamics.

1. Introduction

Modern synergies between direct human activity and the indirect influence of climate change are altering fire regimes through positive feedbacks that increase fire susceptibility, fuel loads, and fire intensity (IPCC, 2014; McLauchlan et al., 2020; Pyne, 2001). Fire regimes can be defined as the general characteristics of recurrent fires through time (size, extent, frequency, intensity) (Gill, 1975; Keeley, 2009), and the magnitude of the ecological effects of fire (severity) (organic matter loss sensu Keeley, 2009; including impact on vegetation sensu McLauchlan et al., 2020) (Table 1). Fire regimes can shape species populations (Bradstock and Myerscough, 1988), biological functioning (Bigalke and Willan, 1984), community/assembly structure and composition (Foster et al., 1990), and ecosystem function (Kruger, 1983; Díaz Baradas et al., 1999). Understanding how fire regimes have changed through time (e.g. decadal to millennial scales) can improve our

understanding of fire-ecosystem linkages (Marlon, 2020) and inform future climate-human-fire model projections (Le Page et al., 2017).

Changes in fire severity (sensu Keeley, 2009), and frequency (for definitions of fire characteristics, see Table 1) can be inferred from observational data, such as maps of area burned, fire occurrence records, and satellite imagery (Abedi Gheshlaghi et al., 2020; Giglio et al., 2016; Morgan et al., 2014; Roy et al., 2006; Weng, 2005; White et al., 1996). Beyond the observational record, subfossil charcoal fragments (Birks and Birks, 1980; Blackford, 2000) extracted from sediments have been used extensively to reconstruct various components of past fire regimes (Clark, 1988; Clark and Patterson, 1997; Clark and Uhl, 1987; Conedera et al., 2009; Duffin et al., 2008; Hudspith and Belcher, 2017; Iglesias et al., 2015; Mooney and Tinner, 2011; Patterson et al., 1987; Swain, 1978; Whitlock et al., 2010; Whitlock and Millspaugh, 1996). Measurements of charcoal abundance (i.e. particle counts, area or volume measurements, chemical extraction of charcoal) preserved in lake,

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Table 1

Terminology and definitions associated of the characteristics comprising fire regimes and our interpretation of whether, or how, they can be parameterised using subfossil charcoal base on previously published work (palaeofire triangle, see Fig. 1).

Terminology and definition(s)	Examples of method by which aspects of fire regimes can be parameterized through the analysis of subfossil charcoal
<p>Fire size/extent: The geographic area over which a fire occurs and spreads.</p> <p>Fire severity: The impact of a fire event on vegetation. Parameterised as:</p> <ol style="list-style-type: none"> The loss of, or change in, above or below ground biomass (sensu Keeley, 2009) <p>The magnitude of the effect of the fire event on the vegetation, including injury and mortality (sensu McLauchlan et al., 2020).</p>	<p>None, but see Fire severity sensu Keeley (2009).</p> <ol style="list-style-type: none"> Abundance of subfossil charcoal contained within the sedimentary record (Whitlock and Larsen, 2001). Comparison of charcoal morphology of different plant types to identify material burnt (Umbanhowar Jr. and McGrath, 1998). Quantification of optical reflectance in reference fire type; canopy vs. surface fire (Roos and Scott, 2018). Abundance of polycyclic aromatic hydrocarbons within the sedimentary record to detect burning of particular vegetation types; steppe and tundra vegetation (Vachula et al., 2020), and softwood taxa (McWethy et al., 2020). The relationship between the abundance of subfossil charcoal contained within the sedimentary record and the vegetation record (either derived from the charcoals themselves, or from other proxies; (Grimm, 1983).
<p>Fire frequency: Number of fire events per unit of time.</p>	<p>Time series analysis of sedimentary charcoal records, such as: (i) wavelet or cross-wavelet analyses are used to compare the frequency of fire events within and between records (Brown et al., 2005; Mariani and Fletcher, 2017) and (ii) decomposition of signal-to-noise index in the influx rate of charcoal into the sedimentary record (Clark and Royall, 1996; Higuera et al., 2011; Kelly et al., 2011).</p>
<p>Fire intensity: The energy release by a fire event per unit time in W/m^2 (Keeley, 2009). Parameterised as:</p> <ol style="list-style-type: none"> Temperature Reaction intensity Fireline intensity Residence time Radiant energy 	<ol style="list-style-type: none"> Comparison of FTIR spectra derived from subfossil charcoals with FTIR spectra obtained from reference charcoal heated to a known range of temperatures; 200–700 °C (Gosling et al., 2019). Abundance of polycyclic aromatic hydrocarbons contained within the sedimentary records; maximum production of 3–4 rings linked to temperatures of 400–500 °C (Argiriadis et al., 2018), and quantification of dehydration products formed below 350 °C (Dietze et al., 2019^a). Comparison of FTIR spectra derived from subfossil charcoals with FTIR spectra obtained from reference charcoal heated to a known temperature for a known amount of time; c. 300–700 °C for 5300–9000 s (Constantine et al., 2021). None. None. Quantification of the optical reflectance of charcoals that have been produced for a known amount of time and released a known amount of heat; duration 200–1100 s, 11–111 MJ/kg heat release (Belcher and Hudspith, 2016^b; Hudspith and Belcher, 2017).

^a Dietze et al. (2019) also noted the potential for this approach to be used to identify plant type (aspect of fire severity).

^b Belcher and Hudspith (2016) also noted the importance of these parameters for understanding past fire severity.

swamp, bog, marine sediments, and ice cores (Conedera et al., 2009; Gill, 1979; Koeppen, 1972; Komarek, 1973; Lewis, 1982; Osmont et al., 2019; Weng, 2005; Winkler and March, 1985) can be used to estimate changes the amount of biomass consumed by past fires (i.e. greater/lower abundance of charcoal in a sample indicates relatively more/less biomass burned, reflecting changes in fire severity) (Brown and Power, 2013; Whitlock and Larsen, 2002) (Fig. 1A). Changes in charcoal abundances can also be used to parameterize fire frequency and the timings of significant biomass burning events (Higuera et al., 2011; Kelly et al., 2011) (Fig. 1B).

Fire intensity (strictly defined as the energy released by a fire per unit time in Wm^{-2} ; Keeley, 2009; Fig. 1C, Table 1) is, however, more challenging to parameterise in the modern fires because “no single metric captures all of the relevant aspects of fire energy” (Keeley, 2009, p. 117). This also holds true for parameterising past fire intensity. Metrics of modern fire intensity include temperature, reaction intensity, fireline intensity (the rate of heat transfer per unit length of the fireline (kWm^{-1}) (Byram, 1959), residence time (heating duration), radiant energy, and others are useful for different purposes (Keeley, 2009). Of these accepted metrics for fire intensity, temperature is linked of human fire use. Fires used to maintain lands for slash and burn cultivation typically burn at lower temperatures (e.g. <500 °C) (Thomaz, 2017), while canopy fires that cause mass mortality and deforest the landscape often reach >500 °C (Kennard et al., 2002; Kennard and Gholz, 2001).

The chemical composition of charcoal is determined by the temperature to which charcoal is heated, with a key transition in thermochemical stability occurring between 400 and 500 °C (Antal and Grønli, 2003). The morphological and optical reflectance characteristics of charcoal are correlated with the amount of light reflected and the temperature at which the charcoal was formed (Belcher and Hudspith, 2016; Bustin and Guo, 1999; Glasspool and Scott, 2005; Guo and Bustin, 1998; Hudspith et al., 2015; Hudspith and Belcher, 2017; Jones et al., 1991; Roos and Scott, 2018; Scott, 2000). The chemical composition (reflectance) of modern charcoal fragments burned at temperatures above and below 400–500 °C are distinguishable using methods such as

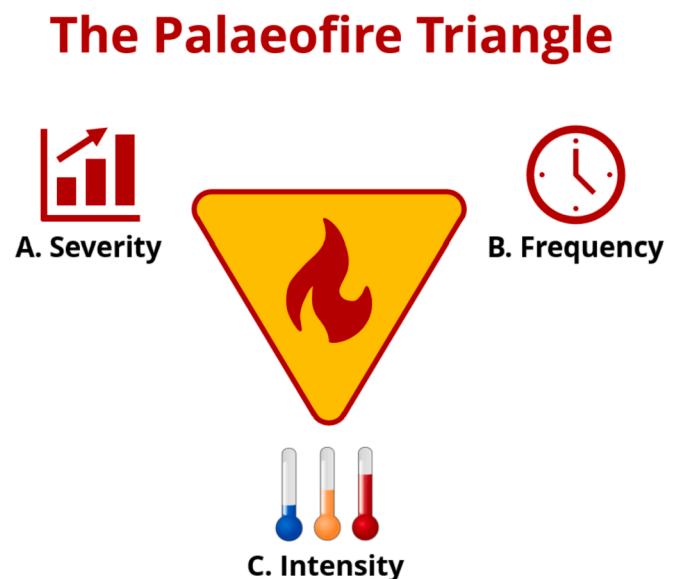


Fig. 1. The Palaeofire Triangle presents the three main components that can be parameterized from subfossil charcoal records to reconstruct past fire regimes: (A) severity (biomass consumed), (B) frequency (number of events per unit time), (C) Intensity (inferred from temperature). For color versions of the figure please see online version.

infrared spectroscopy (Belcher and Hudspith, 2016; Hudspith and Belcher, 2017). Subfossil charcoal (at least those occurring in the last few thousands of years) remain chemically stable, and thus the spectroscopy techniques can also be used to reconstruct past fire temperatures from subfossil charcoal (Belcher et al., 2018; Bezerra et al., 2015; Costa et al., 2018; Davrieux et al., 2010; De la Rosa et al., 2019; Gosling et al., 2019; Guo and Bustin, 1998; Labbé et al., 2006; Monnier, 2018; Monteiro et al., 2010; Pyle et al., 2015; Ramalho et al., 2017; Tintner et al., 2020).

Previous studies implementing optical reflectance data have argued that laboratory produced charcoal is not a suitable substitute for charcoal produced in natural environments, such as wildfires, because ovens are not able to replicate the transient nature of a wild fire nor capture the full range of combustion (Belcher et al., 2018; Belcher and Hudspith, 2016; Cohen-Ofri et al., 2006; Hudspith et al., 2018; Hudspith and Belcher, 2017; Roos and Scott, 2018). However, recent studies linking the vibrational spectra derived from laboratory created and subfossil charcoal suggest that these differences are likely overstated (Constantine et al., 2021; Gosling et al., 2019; Theurer et al., 2021).

Insights into palaeofire severity (vegetation impact sensu McLachlan et al., 2020) can be gauged from correlations of subfossil charcoal abundance records with vegetation proxies such as pollen, phytoliths, or plant macrofossils. Additional information on the type plant material consumed by fires can be obtained when the structure of the plant material is preserved (Wheeler, 2011). Charcoal morphotypes have been used to distinguish between woody and grass taxa (Aleman et al., 2013; Crawford and Belcher, 2014; Enache and Cumming, 2006; Jensen et al., 2007; Leys et al., 2015; Maezumi et al., 2015; Tweiten et al., 2009; Umbanhowar Jr. and Mcgrath, 1998), and to identify species selected by people for burning (Bodin et al., 2020). However, taxonomic identification of plant species from charcoal is generally restricted to woody/grass components based on length-to-width ratios on smaller fragments (<250 µm) or on larger fragments (>4 mm) where key plant structures are preserved (Scheel-Ybert, 2004; Wheeler, 2011). FTIR spectra also characterize chemical properties that can be used to distinguish the types of plants being burned (Bezerra et al., 2015; Costa et al., 2018; Davrieux et al., 2010; Gosling et al., 2019; Guo and Bustin, 1998; Marchant et al., 2009; Ramalho et al., 2017) and may consequently offer a promising insights into fuels consumed in past fires (Fig. 1D).

Thus, while our understanding of past fire regimes from the subfossil charcoal records allows for some insight into their frequency, our understanding remains incomplete due to a paucity of information related to aspects of: (i) intensity (e.g. past fire temperature), and (ii) severity (e.g. the nature of plant material consumed) (Fig. 1; Table 1). Here, we build upon the reference database and approach used by Gosling et al. (2019) that uses the FTIR spectra of charcoal fragments to infer the burn temperature ranges and the types of plants that were being burned. Specifically, we generate an expanded modern charcoal FTIR spectra reference dataset and employ modern analogue matching approaches. We assess the performance of the modern analogue matching approach by comparing its results with previously published methodologies (Gosling et al., 2019).

2. Materials and methods

2.1. Modern charcoal material

The modern charcoal reference dataset was designed to examine the influence of maximum pyrolysis temperatures and plant species on its chemical composition (Fig. 2). The modern charcoal reference dataset of Gosling et al. (2019) contained only one tree (*Alnus glutinosa*) and one grass (*Panicum capillare*) species burned at array of temperatures ranging from 200 °C–700 °C in 100 °C increments. To extend the representation of the plant types within the modern charcoal reference collection we added: (i) tree and shrub taxa, including *Peltophorum africanum* (Fabaceae), *Combretum woodii* (Combretaceae), *Diospyros whyteana*

<i>Alnus glutinosa</i> (Gosling et al. 2019)	700 °C	n=27	<i>Combretum woodii</i>	700 °C	n=25	<i>Peltophorum africanum</i>	700 °C	n=24
	600 °C	n=27		600 °C	n=30		600 °C	n=30
	500 °C	n=27		500 °C	n=30		500 °C	n=30
	400 °C	n=27		400 °C	n=30		400 °C	n=30
	300 °C	n=27		300 °C	n=30		300 °C	n=30
	200 °C	n=27		200 °C	n=20		200 °C	n=29
<i>Elegia tectrotum</i>	700 °C	NA	<i>Diospyros whyteana</i>	700 °C	n=30	<i>Protea cynaroides</i>	700 °C	n=15
	600 °C	n=15		600 °C	n=30		600 °C	n=15
	500 °C	n=15		500 °C	n=30		500 °C	n=15
	400 °C	n=15		400 °C	n=30		400 °C	n=15
	300 °C	n=15		300 °C	n=30		300 °C	n=15
	200 °C	n=15		200 °C	n=25		200 °C	n=15
<i>Cannomois virgata</i>	700 °C	n=15	<i>Grewia occidentalis</i>	700 °C	n=20	<i>Panicum capillare</i> (Gosling et al. 2019)	700 °C	n=27
	600 °C	n=15		600 °C	n=30		600 °C	n=27
	500 °C	n=15		500 °C	n=30		500 °C	n=27
	400 °C	n=15		400 °C	n=30		400 °C	n=27
	300 °C	n=15		300 °C	n=30		300 °C	n=27
	200 °C	n=13		200 °C	n=30		200 °C	n=27

Fig. 2. Experimental Design: Charcoal was generated for nine plant species, at six temperatures (200 °C to 700 °C at 100 °C intervals). The number of replicates (n) vary based on sample availability and preservation during pyrolysis. Information for two taxa (*Panicum capillare* and *Alnus glutinosa*) was previously published (Gosling et al., 2019). For color versions of the figure please see online version.

(Ebenaceae), *Grewia occidentalis* (Tiliaceae), (ii) Fynbos taxon *Protea cynaroides* (Proteaceae), and (iii) reed taxa *Elegia tectrotum* and *Cannomois virgata* (both Restionaceae) (Fig. 2). These samples were collected from living, mainstem material from specimens provided by the Hortus Botanicus Amsterdam. The six different maximum pyrolysis temperature groups and nine vegetation groups defined 54 distinct treatment groups, each with a number of replicates that varied based on sample availability (i.e. 13–30 replicates per group; $n = 1260$ samples in total; Fig. 2).

The plant material was pyrolyzed to either 200 °C, 300 °C, 400 °C, 500 °C, 600 °C or 700 °C ($n = 6$ temperatures, Fig. 2) following standard methods (Orvis et al., 2005) to induce varying amounts of carbonization. One gram of plant material per sample was wrapped in standard laboratory aluminium foil and surrounded by 250–500 µm sand. Samples were transferred into a preheated oven and pyrolyzed at the target temperature for 10 min to achieve full charcoalification following the protocol for the creation of voucher reference charcoal (Orvis et al., 2005). Samples were allowed to cool in the oven between 25 (200 °C) and 90 (700 °C) minutes and were then manually ground into a fine powder (< 45 µm) using a ceramic pestle and mortar to homogenize within sample variability.

2.2. Characterisation of charcoal using FTIR spectra

The FTIR spectra obtained from the charcoal characterizes its chemical composition (molecular composition and functional groups) following the methodology established by Guo and Bustin (1998). To obtain FTIR spectra from laboratory processed charcoal the samples were first homogenized and then spread evenly onto zinc selenium slides for analysis. A nitrogen gas purge system was used to suppress changes in the composition of atmospheric air surrounding samples. A

background scan was performed before each analytical scan to subtract the spectral signatures of the zinc selenium slides and the nitrogen air. An FTIR spectrum was obtained for each sample using a Nicolet iN10 MX Infrared Imaging Microscope and Omnic software. Samples were analysed using a liquid nitrogen cooled MCT detector with the following settings: (i) transmission mode, (ii) 2 cm^{-1} spectral resolution, (iii) 128 scans, and (iv) a spatial range consisting of wavenumbers from 950 to 3500 cm^{-1} . An aperture size of $>150\text{ }\mu\text{m}$ was used for all samples. Scattering may have impacted FTIR spectra but the impact of this was assumed to be consistent between samples because sample preparation and analysis followed the same protocol. The raw FTIR spectra were subjected to a Beer-Norton filter to improve the signal-to-noise ratio, and the standard linear baseline correction was applied using the “auto baseline” function in the commercially available IR spectra analysis software package Omnic (Thermo Nicolet Analytical Instruments, Madison WI, Lakiza 2008) to enable the integration with previously published data following Gosling et al. (2019).

Spectra measured at wavenumbers below 950 and above 3500 were found to have very high standard deviation values and were therefore removed before further analysis (Gosling et al., 2019; Varmuza and Filzmoser, 2009). After removing these wavenumbers, spectra were centered and scaled using z-score standardisation to eliminate differences in the mean of each measurement (Gosling et al., 2019).

2.3. Analogue matching

Analogue matching (AM) is used in palaeoecological studies to identify samples in a modern multivariate dataset that are closest matches for those in the fossil assemblages (Chevalier et al., 2020; Flower et al., 1997; Overpeck et al., 1985; Simpson, 2012). AM uses (dis) similarity matrices to compare modern multivariate assemblages (the training dataset, which has known and measured environmental characteristics) with fossil assemblages in a sedimentary sequence (the testing dataset) to discriminate similar and dissimilar sites. Here we determine whether AM can be used to accurately match charcoal fragments with known pyrolysis temperatures and species composition using FTIR spectra (Fig. 2). To define a threshold for identifying analogues for a given samples, we selected the value corresponding to the 2.5% lower tail of the distribution of randomly selected pairwise Euclidean distances between samples in the reference dataset using a Monte-Carlo approach (Simpson, 2012). With this criteria, reference (training) samples that fell within this threshold value at 2.5% similarity were considered analogues for the test fragment. Additionally, to ensure robustness of the approach, we only estimate the maximum pyrolysis temperature and type of plant material burned for test fragments that had at least five identified analogues within the reference dataset.

To assess whether AM can accurately identify the pyrolysis temperature and species of plant burned, we took the reference dataset of FTIR spectra ($N = 1260$, Fig. 2), and separated into a training and testing partition. We then performed AM, using Euclidean distance as the dissimilarity metric, on the testing and training partitions to identify analogues for each sample of the testing dataset. Two statistics based on the identified analogues for each sample were used to infer pyrolysis temperature and the species of the burned plant: i) the mode value (i.e. the most frequent maximum pyrolysis temperature or species found within the identified analogues), and ii) with the lowest mean Euclidean distance between groups (i.e. the analogues are grouped by temperature or species categories, and the group with the lowest mean distance is the solution) of identified analogues for maximum pyrolysis temperature categories (e.g. 200–700 °C) or the species of plant burned.

We used 10-fold cross-validation (CV) to split the data into the training and testing partitions to assess the accuracy of the AM model in identifying the pyrolysis temperature and species of the FTIR spectra. This process first split reference dataset into 10 groups (folds) of equal size ($n = 126$). Nine of the ten folds were assigned as the training dataset ($n = 1134$), and the tenth was assigned as the testing dataset ($n = 126$).

The AM model was then run and assessed using the two statistics described above. The process was repeated until each of the folds were used as the testing dataset. The results were pooled to estimate model accuracy.

Model accuracy was defined as the percentage of accurate predictions for each category (i.e. pyrolysis temperature category and species). For temperature we also derived a secondary measure of accuracy of how many samples were accurately reconstructed at $\pm 100\text{ }^\circ\text{C}$ intervals (e.g. for all the samples burnt at $300\text{ }^\circ\text{C}$, how many were classified in the $200\text{ }^\circ\text{C}$, $300\text{ }^\circ\text{C}$ or $400\text{ }^\circ\text{C}$ categories). For plant species we also derived a secondary measure of accuracy for plant species grouped into trees/shrubs and reed/grass (e.g. for all the tree/shrub samples burnt, how many classified in the tree/shrub category).

The ‘ChemoSpec’ (Hanson, 2020, version 5.2.12), ‘Nnet’ (Venables and Ripley, 2002), and ‘analogue’ (Simpson and Oksanen, 2014) packages for R (version 3.4.2; (R Foundation for Statistical Computing, 2020) were used for data pre-processing and statistical analyses.

3. Results

3.1. Characteristics of modern charcoal material

Charcoalification of the samples was not complete at lower temperatures. Samples at $200\text{ }^\circ\text{C}$ were baked (not carbonized), samples at $300\text{ }^\circ\text{C}$ were partially charcoalified (i.e. some of the sample was partially carbonized), and samples at $>400\text{ }^\circ\text{C}$ samples were fully charcoalified (i.e. all of the sample was pyrolysed). At $700\text{ }^\circ\text{C}$ ca. 10% ashing was observed in *P. capillare* samples; ash was removed manually before grinding and FTIR analysis. No samples of *E. tectrotum* were preserved at $700\text{ }^\circ\text{C}$ as the fine needle-like structure completely ashed at this temperature and thus were not measured.

The raw spectral data ($n = 1260$) and the magnitude and direction of the standard deviations of the wavenumbers showed visible differences between the samples heated to low ($200\text{ }^\circ\text{C}$, $300\text{ }^\circ\text{C}$), mid ($400\text{ }^\circ\text{C}$, $500\text{ }^\circ\text{C}$), and high ($600\text{ }^\circ\text{C}$ or $700\text{ }^\circ\text{C}$) temperature categories (Fig. 3A); notably relatively higher absorbance between wavenumber 2000–2700 at high temperature, and over 3000 at low temperatures. Spectra also show some visible differences when plotted by plant species although no clear overall pattern is dominant (Fig. 3B).

3.2. Analogue matching

Pairwise Euclidean distances between samples within the reference dataset of FTIR spectra ranged from 0.55 to 7.94. The upper value of 7.94 was selected as the threshold for the lowest 2.5% of pairwise Euclidean distances of the reference dataset that was then used to identify analogues for a given sample. Using this threshold, 1202 of the 1260 fragments matched to statistically similar analogues within the dataset, with an average of 96 analogues for temperature and for species per sample. There were a total of 58 samples in the dataset that had no significant matches (no-analogues, NAs) identified for neither temperature nor species (Fig. 4B). The distribution of NA values across temperatures and between species is very even (maximum number of temperature NAs = 26 for $700\text{ }^\circ\text{C}$ and minimum = 3 for $600\text{ }^\circ\text{C}$; maximum number of species NAs = 17 for *Panicum capillare* and minimum = 1 for *Grewia occidentalis*). Using a higher distance threshold reduced the number of unmatched samples, but it decreased the average quality and distribution of the identified analogues (SOM Fig. 1).

The results of the model iterations and cross-validation resulted in a total of 10 model runs on the 1260 samples. The total number of analogues identified across categories using the iterative cross-validation of the AM model ranged from 6139 to 39,206 for temperature, and from 5413 to 21,506 for species. More analogues were found for samples burned at $500\text{ }^\circ\text{C}$ and $600\text{ }^\circ\text{C}$ than at other temperatures (Fig. 4A). The distributions of correctly identified analogues varied between temperature categories, with 34% correctly identified across all model runs at

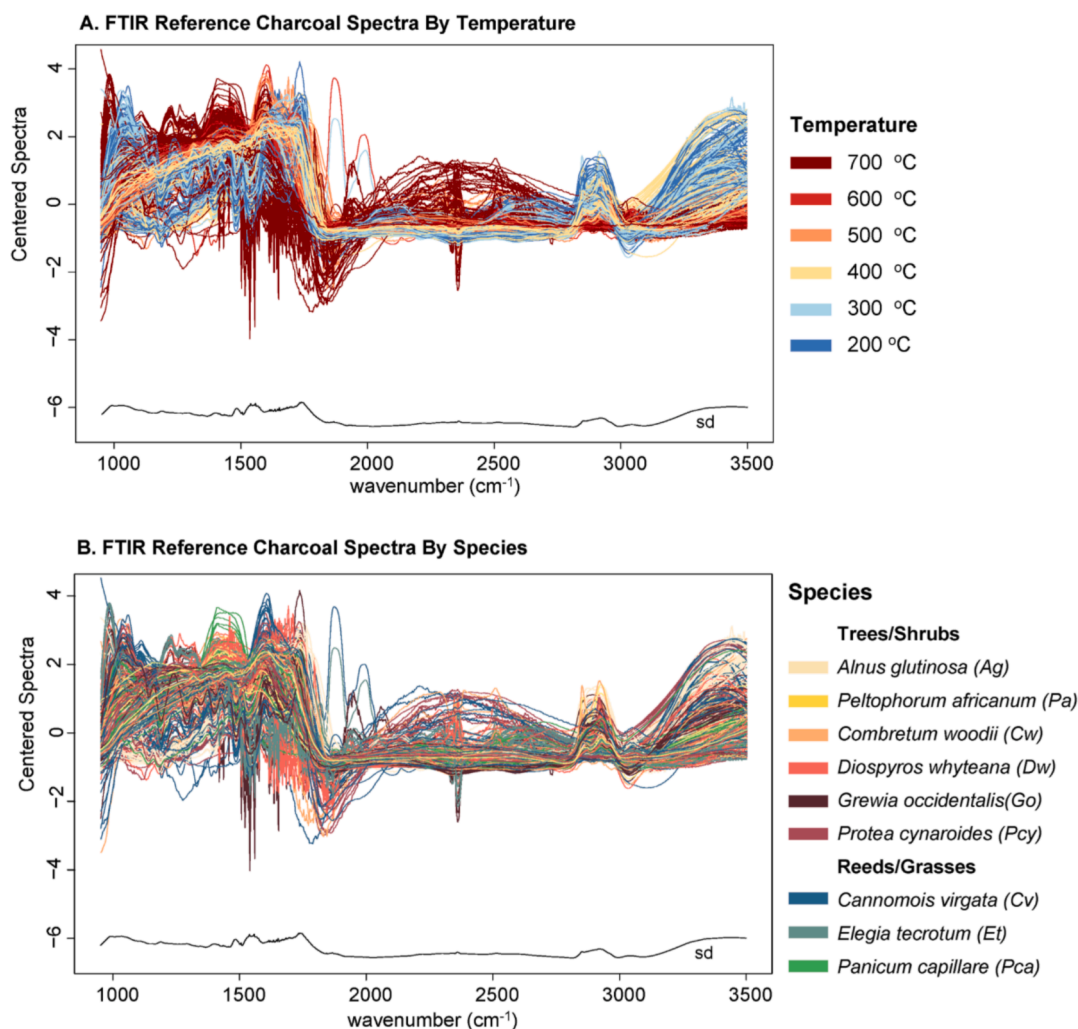


Fig. 3. Fourier Transformed Infrared Spectroscopy (FTIR) absorbance spectra for modern reference charcoal material: (A) Samples color-coded by temperature category, and (B) Samples color-coded by plant species. sd indicates standard deviations of FTIR absorbance spectra grouped into trees/shrubs and reeds/grasses (not at scale). For color versions of the figure please see online version.

200 °C (2088 analogues), 40% at 300 °C (3336 analogues), 37% at 400 °C (6870 analogues), 47% at 500 °C (18,402 analogues), 45% at 600 °C (16,680 analogues) and 39% at 700 °C (4258 analogues) (Fig. 4B; Table 2). Including the ± 100 °C intervals for each temperature category increased the correct identification of analogues to 74% at 200 °C (4569 analogues), 89% at 300 °C (7515 analogues), 79% at 400 °C (14,806 analogues), 94% at 500 °C (36,868 analogues), 92% at 600 °C (34,258 analogues), and 88% at 700 °C (9608 analogues). The percentages of no-analogue matches ranged from 1 to 6% across the temperature categories, except in the 700 °C category where it reached 14%.

The iterative AM model runs and cross-validation also resulted in uneven distributions of the total number of analogues identified for each plant species burned. *A. glutinosa*, *P. africanum*, *C. woodii*, and *D. whyteana* had more analogues matches than the other species in the dataset (Fig. 4A). The percentage of correctly identified analogues resulting from these model runs varied between species, with 17% correctly identified for *A. glutinosa* (1700), 20% for *P. africanum* (4076), 18% for *C. woodii* (3046), 20% for *D. whyteana* (4242), 21% for *G. occidentalis* (4262), 12% for *P. cynaroides* (1038), 10% for *C. virgata* (732), 13% for *E. tectrotum* (680), and 19% for *P. capillare* (1936) (Fig. 4D). When grouped into broad categories of trees/shrubs and reeds/grasses, the number of correctly identified analogues increased to 83% and 28%, respectively (Fig. 4C, B, Table 3). The distribution of no-analogue matches was varied across species categories, ranging from

0.5–11% (Fig. 4D).

For determining the maximum pyrolysis temperature of a charcoal fragment, the metrics of mode and mean Euclidean distance of all identified analogues provided similar results (Fig. 5A, B). Accuracy (percentage correctly classified) of the AM model using the modal and lowest mean Euclidean distance, respectively, was 41/53% for samples burned at 200 °C, 56/51% at 300 °C, 56/74% at 400 °C, 78/78% at 500 °C, 63/61% at 600 °C, and 48/69% at 700 °C (Fig. 5A, B). When a prediction of the AM was considered accurate if it fell within 100 °C of the actual pyrolysis temperature, accuracy for the modal and mean lowest Euclidean distance metrics increased to 87/84% for samples burned at 200 °C, 93/97% at 300 °C, 94/90% at 400 °C (205 analogues), 98/96% at 500 °C (214 analogues), 98/95% at 600 °C (215 analogues), and 85/86% at 700 °C (155 analogues).

Accuracy assessments of the AM model were also similar between the modal or lowest mean Euclidean distance metrics when predicting the species of plant material that was burned. When using the modal and mean lowest Euclidean distance, respectively, 52/54% of fragments were correctly classified for for *A. glutinosa*, 52/46% for *P. africanum*, 32/58% for *C. woodii*, 34/30% for *D. whyteana*, 48/45% for *G. occidentalis*, 27/41% for *P. cynaroides*, 26/61% for *C. virgata*, 28/68% for *E. tectrotum*, 41/45% for *P. capillare* (Fig. 5C, D, Table 3). The taxonomic grouping of growth forms yielded 91/83% accuracy for trees/shrubs and 43/66% for reeds/grasses.

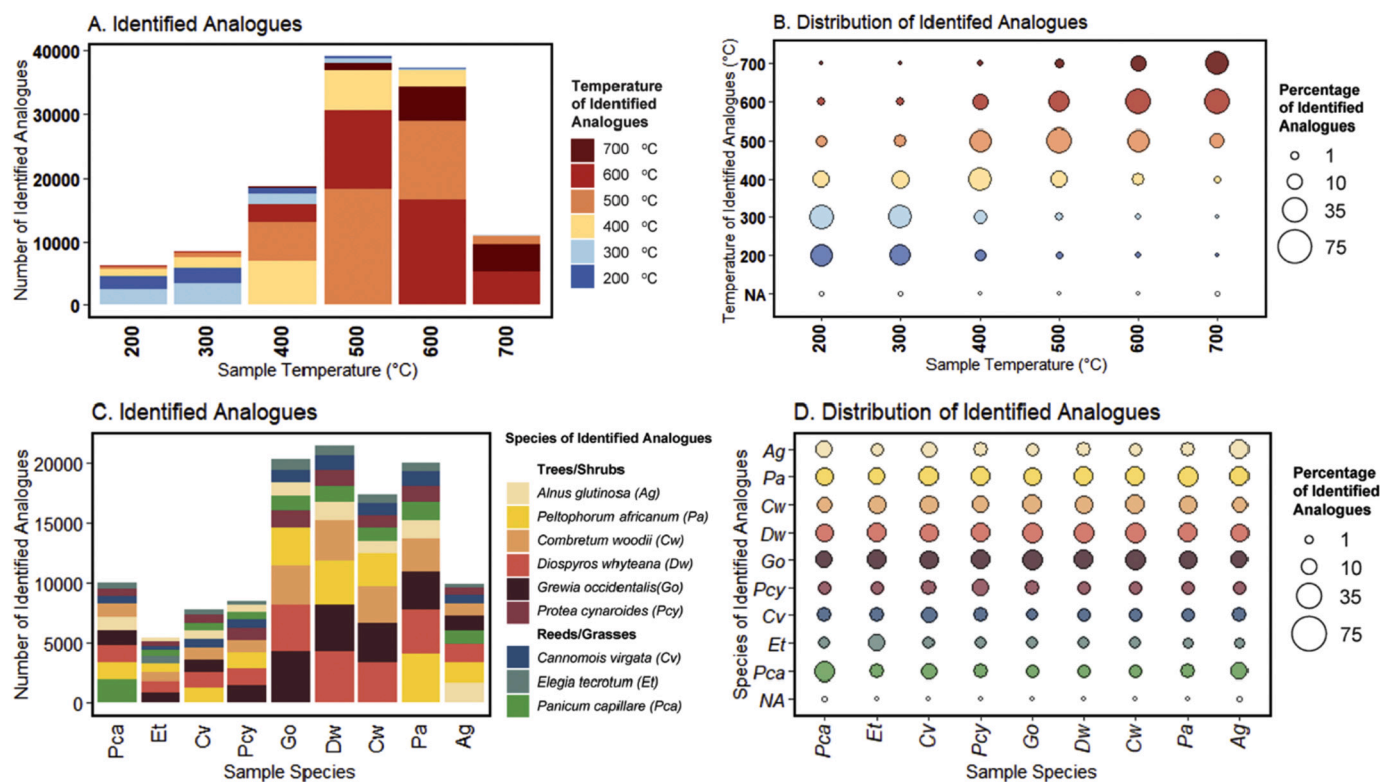


Fig. 4. Results of Analogue Matching to characterize the temperature and species of plant burned: (A) Number of temperature analogues by category under the 2.5% dissimilarity threshold. (B) Percentage of temperature analogues under 2.5% dissimilarity threshold. (C) Total number of identified analogues for the 1260 charcoal fragments in the reference dataset FTIR spectra of the species shown in Fig. 2, and (D) Percentage of all analogues identified for each of the species contained within the FTIR reference dataset.

Table 2

Accuracy (% correctly identified) of Analogue Matching for each temperature category. Percentages are shown for all analogues under the 2.5 percentile, and using the modal value or lowest mean Euclidean distance. Percentages of all statistics are also shown for the secondary metrics of accuracy at the range of ± 100 °C.

Temperature category (°C)	All <2.5 analogues	Mode	Lowest mean	<All 2.5 analogues (± 100 °C)	Mode (± 100 °C)	Lowest mean (± 100 °C)
200	34	41	53	74	87	84
300	40	56	51	89	93	97
400	37	58	74	79	94	90
500	47	78	78	94	98	96
600	45	63	61	92	98	95
700	39	48	69	87	85	86
Model Performance	40	57	64	86	93	91

4. Discussion

4.1. Inferring pyrolysis temperature and the type of plant material burned based on FTIR spectra

Model-based clustering has been previously used to extract information from FTIR spectra derived from charcoal fragments, and had excellent success in classifying maximum pyrolysis temperature (67–93% accuracy dependent on temperature class) (Gosling et al., 2019). Model-based clustering generates static groups determined by the spectra of the reference charcoal fragments. The modern analogue approach, however, does not depend on the reference charcoal fragments being divided into pre-defined groups. Instead, it quantifies the known characteristics of the reference fragments that are statistically similar to the fragment being analysed.

Model-based clustering also assigns all test charcoal fragments to a cluster even if the spectra fell outside the bounds of the training dataset (reference dataset). The modern analogue approach does not force a test sample to a group, but instead simply reports that there are no reference samples that fall within a given threshold of multivariate similarity (NA

values). The forced classification of model-based clustering likely increases the potential for error in inferring the characteristics of charcoal and determining whether the sample material is actually charcoal. In many soils and sediments where charcoal is analysed to reconstruct fire histories, other types of black material appear in samples and can be misidentified as charcoal (Earle et al., 1996; Whitlock and Anderson, 2003). The lack of forced classification with the analogue matching approach can even be used as a tool to identify whether material is actually charcoal. Overall, the analogue matching (AM) approach retains a high accuracy of classification (Tables 2 and 3), can be used to infer multiple characteristics of charcoal, and does not force classify charcoal fragments. For these reasons, we suggest that analogue matching is a preferred approach to model-based clustering for inferring the pyrolysis temperature and type of plant material burned.

Two different statistical metrics for inferring charcoal characteristics (pyrolysis temperature and type of plant material burned) of unknown (test) spectra were used to assess the robustness of the analogue matching approach: (i) the mode value of all identified analogues (reference samples falling within the 2.5% similarity threshold), and (iii) the lowest mean Euclidean distance of all identified analogues (Fig. 5A-

Table 3

Model performance comparison for plant species reconstructions shown in percentages for all identified analogues, the mode of the species analogues, the lowest mean Euclidean distance, and the model performance across all species categories. Plant species are organized based on growth form (i.e. tree/shrub, reed/grass).

Species	Growth form	All <2.5 analogues	Mode	Lowest mean
<i>Alnus glutinosa</i>	Tree/Shrub	17	52	54
<i>Peltophorum africanum</i>	Tree/Shrub	20	51	46
<i>Combretum woodii</i>	Tree/Shrub	18	32	58
<i>Diospyros whyteana</i>	Tree/Shrub	20	34	30
<i>Grewia occidentalis</i>	Shrub	21	48	45
<i>Protea cynaroides</i>	Shrub	12	27	41
Grouped Tree/shrub		83	91	83
<i>Cannomois virgata</i>	Reed	10	26	61
<i>Elegia tectorum</i>	Reed	13	28	68
<i>Panicum capillare</i>	Grass	19	41	45
Grouped Reed/Grass		28	43	66
Average model accuracy		17	38	50
Average group accuracy		56	67	75

D). Both metrics had a good overall model accuracy (57% and 64% respectively), which increased with the inclusion of adjacent temperature categories ± 100 °C (93% and 91% respectively; Table 2). Model accuracy was lower for the taxonomic identification of plant species compared with pyrolysis temperature using both metrics (38% and 50%), but increased markedly once species were grouped by growth form; trees and shrubs (91% and 83%) and herbs and grasses (43%, 66%) (Table 3).

The number of analogues identified under the 2.5% threshold was higher for samples burned at 500 °C and 600 °C compared with other

temperature categories (Fig. 4A). This likely reflects the shift in thermochemical stability of the charcoal that occurs between 400 °C and 500 °C during formation (Antal and Grønli, 2003). Our interpretation is consistent with existing charcoal-pyrolysis temperature studies that demonstrate that as temperatures increase, organic material becomes dominated by stable, condensed polyaromatics (Pyle et al., 2015; Tintner et al., 2018; Zhao et al., 2017). Furthermore, it is interesting to note that even when duration of heating is considered it is still between 400 °C and 500 °C that the largest shifts in the FTIR spectral data are observed (Constantine et al., 2021; Table 1).

The lowest accuracy of analogue matches in our dataset were in the lower temperature samples at 200 °C (41% and 53%) and 300 °C (56% and 51%) (Table 2). There was also a lower number of analogues identified for samples burned at these temperatures during the cross-validation runs of the model (Fig. 4). Lowered accuracy of these groups is likely attributed to the incomplete charring of the plant material at lower pyrolysis temperatures as charring only begins at 200 °C, below which dehydration is the main reaction (Pyle et al., 2015). Samples at 300 °C were likely only partially charred. As a result of the incomplete charring at the pyrolysis temperatures, other factors (e.g. unique species chemical signatures), likely had a greater influence on charcoal chemistry than temperature and decreased the accuracy of the temperature analogues. In the highest temperature sample (700 °C), the number of analogues under the 2.5% threshold again declined (Fig. 4A), which is likely the result of the loss of chemical information associated with ashing at and above this temperature (Gosling et al., 2019; Gur-Arieh et al., 2014; Gur-Arieh et al., 2013). Our interpretation is supported by ca. 10% ashing observed in *P. capillare* samples at 700 °C (Gosling et al., 2019). These findings suggest that at lower temperatures (200 °C and 300 °C) more chemical information related to plant type is likely to be preserved, while at high temperatures insufficient chemical information may be available to make meaningful reconstructions of

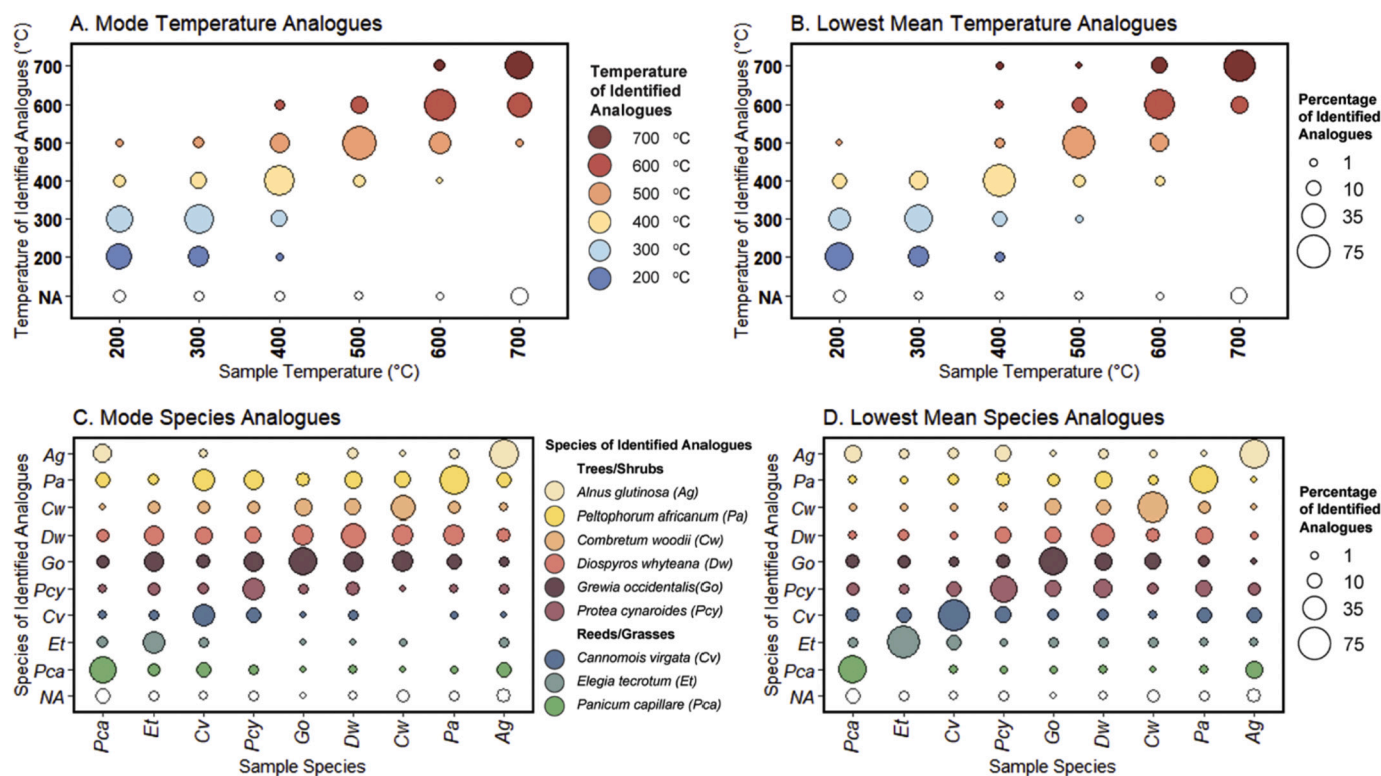


Fig. 5. The percentage of correctly identified analogues within the reference dataset for each temperature and species category. (A) the inferred temperature of charcoal fragments burned using the mode value, and (B) the inferred temperature of charcoal fragments using the lowest mean Euclidean distance of all identified analogues. (C) the mode of analogues by plant species category and (D) the lowest mean Euclidean distance by plant species category. For color versions of the figure please see online version.

either temperature or plant type.

The differences in the number of identified analogues when assessing the species or type of plant material burned likely relates to intrinsic differential susceptibility to fire due to the structural and chemical composition of the plants. Notably, three of the four plants that produce the lowest number of analogues lack woody components, a grass (*Panicum capillare*) and two reeds (*Elegia tectorum* and *Cannomois virgata*), while the other is genetically distant from the other woody species being a protea shrub (*Protea cynaroides*). Despite the fewer number of analogues identified, there does not seem to be a relationship between the number of analogues per species and the classification success rate, e.g. *Diospyros whyteana* has the highest number of analogues (Fig. 4), but the lowest classification success rate (Table 3).

The limited number of species currently available in our reference dataset led us to group the plant species into broad groups on the basis of their growth forms (i.e. trees/shrubs, vs. reeds/grasses). Importantly for charcoal formation these two groups differ significantly in terms of their lignin content (trees/shrubs typically 25%, (Novaes et al., 2010); reeds/grasses typically 9–19%, (Juneja et al., 2011; Kou et al., 2017; Novaes et al., 2010; Tutt and Olt, 2011; Wöhler-Geske et al., 2016)). By assigning the FTIR spectra into these groups we significantly increased the model performance for the two modern analogue matching models (i.e. from 38% and 50% to 67% and 75% respectively; Table 3). Further work is required to test the link between the plant charcoal chemistry and the growth form and/or genetic relatedness of species; however, indications from this small subset of the plant kingdom indicate that expanding the chemical characterisation in this way could provide a new tool for identifying the fuel consumed by past fires.

4.2. Application to the subfossil charcoal record

Current reconstructions of past fire regimes using subfossil charcoal found in sedimentary archives lack components of the palaeofire triangle, particularly estimates of fire intensity (temperature) (Fig. 1). While fire intensity includes multiple parameters, temperature is a main component (Table 1). Thus, pyrolysis temperature is likely a proxy for fire intensity. Every fire, however, burns at a variety of temperatures, with temperature gradients measurable across landscapes (Veraverbeke et al., 2018) and within individual plants (Wesolowski et al., 2014). Therefore, it is important to consider what reconstructing the temperature of an individual subfossil charcoal fragment might mean for reconstructing the temperature of a past fire event. Firstly, it should be recognized that the variation in the FTIR spectra of the charcoal produced in the laboratory under controlled conditions is consistent on broad temperature scales, e.g. see increased classification success when temperature classes are grouped (Table 2). This supports the findings of Gosling et al. (2019) that within broad temperature groups (± 100 °C), the temperature under which that charcoal was formed can be successfully characterised using FTIR spectra. Given the variability of temperatures known to occur within a single fire event, it is likely that trying to assign a smaller temperature range to a single charcoal fragment would lead to an overinterpretation of the data.

Secondly, it should be recognized that within many palaeofire records, multiple charcoal fragments are present within samples. Therefore, within a sample it is recommended to develop a profile of the temperature ranges under which the various charcoals have been formed. For example, assume a sample contained 100 charcoal fragments of which 5 matched to 200 °C, 10 matched to 300 °C, 10 matched to 400 °C, 40 matched to 500 °C, 30 matched to 600 °C, and 5 matched to 700 °C. Using the analogue matching approach, which obtained ca. 90% accuracy at classifying pyrolysis temperatures at ± 100 °C, these data would suggest that the majority (70%) of fire(s) occurring in that time interval burned around 500 °C–600 °C. It would therefore be reasonable to infer that the sample reflects predominantly high temperature fire(s). We believe that further inference obtained by calculating metrics, such as mean temperature of the fire from these data

would not be meaningful and would be an overinterpretation of the dataset. We also recommend against assuming that multiple subfossil charcoal fragments recovered from within a given sample were created during a single fire event. It should always be considered that the profile of reconstructed temperatures from a sample may reflect multiple fire events, and that the likelihood is dependent on the rate of sedimentation or soil accumulation. Even with these considerations, the temperature profiles generated for a sample provide invaluable information related to fire intensity and filling in the missing components of the palaeofire triangle (Fig. 1).

The analogue matching approach applied to our dataset suggests that classifying plant types in the subfossil charcoal record on the basis of their FTIR spectral properties has high potential, particularly when taxa are assigned to broader groups, e.g. trees/shrubs and reeds/grasses (Table 3). We, however, recommend against making inferences at the species level when assessing subfossil charcoal fragments. Our reference database currently has a limited number of representative species ($N = 9$), and even if that number were multiplied tenfold, it would likely not reflect the diversity of species found within an ecosystem. Our reference database also contains predominantly species from African ecosystems (Fig. 2). If we were to compare this reference database to subfossil charcoal fragments from North or South American sites, it would be meaningless to make species-level inferences. However, making inferences regarding the broader plant groups would be reasonable, keeping in mind the representation of plant groups within the reference dataset and within the ecosystem where the subfossil charcoal fragments were derived. Optimally, reference datasets should be generated so that the representation of plant types within the reference dataset reflects the ecosystem being studied. Morphometric approaches such as charcoal length-to-width ratio (Aleman et al., 2013; Umbanhowar Jr. and Mcgrath, 1998) and anthracological studies (Bodin et al., 2020) are commonly used to identify charcoal fragments to genus or species level. This approach can be time-consuming because a given palaeofire record may contain hundreds of samples, with hundreds of charcoal fragments within each sample. We suggest the FTIR analysis of charcoal fragments provides a relatively fast and efficient way to broadly characterize the type of plant material that was burned, and combining our approach with visual based studies of morphometrics and anthracology can more fully develop our understanding of past fire regimes.

5. Conclusions

The development of a modern analogue matching approach for extracting information from FTIR spectra derived from subfossil charcoal has been demonstrated to: (i) allow multiple types of information to be extracted independently from the same data (i.e. temperature and taxonomic information), and (ii) to avoid the potential for incorrect ‘forced’ classification. The classification success rates for assigning temperature classes are not as high as previously published statistical approaches when only one temperature category is considered, however, they are exceeded when adjacent categories are included (± 100 °C). This suggests that the reconstruction of past fire temperatures from this approach should be restricted to broad classifications, such as: predominantly high temperatures (> 500 °C charcoals) potentially indicative of canopy fires. Through reconstructing past fire temperatures in this way, a new insight into a constituent component of fire intensity, and how it changed through time, can be gained. The ability to identify species representing a variety of different growth forms (trees/shrubs and reeds/grasses) from their charcoal offers an interesting new opportunity to identify the nature of the fuel-temperature relationships from past fires. The characterisation of maximum pyrolysis temperature, and fuel type, extracted from subfossil charcoal data provide new insights into the influence of land use and crop cultivation on past fire regimes.

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Authors' contributions

Conceptualization: WDG, CNHM, SYM; Data curation: HC, TH; Formal analysis: SYM, JK, MC, CHNM; Funding acquisition: SYM, WDG, CHNM; Investigation: SYM, WDG, JK, MC, CHNM; Methodology: WDG, SYM, CHNM; Project administration: SYM, WDG, CHNM; Resources: WDG, CHNM, SYM; Supervision: WDG, CHNM; Validation: SYM, CHNM, MC; Visualization: SYM with input from WDG, CHNM; Writing - original draft: SYM, WDG, CHNM, MC; Writing - review & editing: SYM, WDG, JK, MC, HC, TH, CHNM.

Data availability

The FTIR reference data and R Source code used in this analysis are available on the online data repository Zenodo (<https://doi.org/10.5281/zenodo.5156746>).

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.palaeo.2021.110580>.

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