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Fire and heat, from hearth to charcoal: An experimental approach to temperature in the context of Palaeolithic hearths^{\ddagger}

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ABSTRACT

Whether one is interested in palaeoeconomics or technical aspects of fire use, or in taphonomy, the concept of temperature plays a central role in charcoal analysis. What does the temperature reveal about the function of the hearths? Can prehistoric hearth temperatures be accurately measured a posteriori, and what information could be gleaned from such measurements? Changing scale, what are the effects of fire temperatures on the residues themselves in terms of taphonomy and what are the consequences for the preservation of isotopic and molecular signatures?

To address these questions, we conducted over two hundred experimental standardised combustions under laboratory conditions. Our results, supported by mathematical data processing, provide insight into the properties of wood combustion, including fragmentation processes. We also explored the challenges of measuring temperatures in both the combustion structures and the charcoal itself.

Our results show that temperatures in the open-air fireplaces are highly labile, with average temperatures always within the same range regardless of the taxa. We also provide information on the effect of temperature on fragmentation processes but also on the isotopic and molecular signature.

1. Introduction

"Temperature" is a central but often ambiguous concept in Anthracology that can either refer to the temperature of the fire/hearth, the temperature at which the charcoal assemblage was formed, the temperature reached by the remains themselves, and the temperature finally recorded by the archaeological charcoal after post-depositional processes. Not only are they not the *same* temperature, but there may be little or no correlation between them. Temperatures vary not only during the successive drying, pyrolysis and oxidation phases of the combustion, in the flames and at the base of the fire, but also in the wood, where the temperature reached by the log depends on its morphological, phenological and physiological state, and decreases from the bark to the heart. It is therefore important to always specify what temperature we are talking about. Depending on the scale, "temperature" can document either some technical aspects related to the use of fire (anthropological scale), taphonomy or representativeness (anthracological scale); it also influences the results of some isotopic and chemical analyses applied to charcoal (charcoal scale) (Fig. 1). In this regard, the notion meets both the palaeoecological and palaeoenvironmental perspectives of Anthracology. This is true of the Palaeolithic sites that are the subject of this paper, but it is also true of more recent sites.

Paradoxically, perhaps because it does not meet the epistemological challenges of the discipline, this issue has rarely or only indirectly been addressed in Anthracology. Charcoal remains are in-situ residues of anthropological activities carried out in a palaeoenvironmental context that one sought to define as a priority. The first methodological developments aimed to define the composition, diversity, representativeness and capacity of charcoal to describe the context of human evolution, their activities and the environment in which societies developed (Badal-Garcia, 1992; Chabal, 1997, Kabukcu and Chabal, 2021). The temperature of the hearths, whether the anthracological deposit was formed at one temperature or another, the temperature reached by the residual charcoal were at first considered of little

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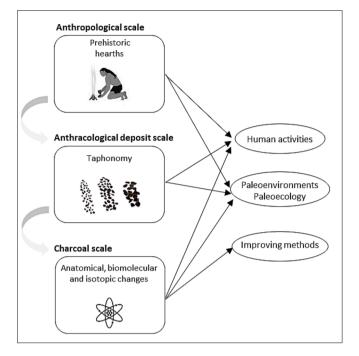


Fig. 1. Main concerns about "temperatures" in Anthracology.

interest, since it seemed obvious that nothing distinguished one fire from another, except the open hearths and the closed structures (eg., kiln) in which the laws of physics governed the flow of heat: the former was on average "less hot" than the latter. But also because of the methodological obstacles to be overcome: measuring the "temperature" a posteriori is a challenge, as there is no real tool to deal with this issue. Anthracology has subsequently focused on anthropological issues related to the use of fire by studying what was strictly within its remit: the identification of wood as a fuel and its properties, on the assumption that the notion of heat transfer mode and the management of the phenological and seasonal state of wood were better able to document the operation of fireplaces than temperature itself (Vidal-Matutano et al., 2015, 2020; Allué et al., 2022; Théry-Parisot, 2002). At the same time, ecological approaches have evolved, enriched by new methods for studying palaeoenvironments. So, what would be the point of revisiting the concept of temperature for Anthracology?

1.1. About the Palaeolithic hearths

Firstly, the notion of temperature brings together Anthracology and the study of Palaeolithic hearths, contributing to the debate on both the origin and the control of fire. Given the biological, social, technical and symbolic implications of fire, it holds a unique status in the history of research in Prehistory (Perlès 1977; Binford, 1998; Wadley and Jacobs, 2006; Vitezović, 2013). Identifying the oldest evidence through the recognition of structures, soils or artefacts that show thermal alteration was the first target (Sandgathe et al., 2011, 2017; Karkanas et al., 2007; Sorensen et al, 2014; Hérisson et al., 2020; Kumar Jha et al., 2021; Pietraszek et al. 2022, Allué et al., 2022). The question of the oldest evidence remains a topic of ongoing debate, but the advent of new techniques that now allow the measurement of the most transient traces of thermal alteration in sediments or in the burnt artefacts themselves will make it possible to reconsidered this issue (Mallol et al., 2013; Lacanette, 2017; Leierer, 2020; Allue et al., 2022). In addition to cognitive capacity of the oldest « users », next came the question of the domestication of fire, which is distinct from those of its origin and control. Fire became a central issue perceived as a means of accessing a range of crucial information about societies and their environment from (i) social aspects: organisation, housing, mobility and seasonality; (ii)

technical aspects, including all fire-related activities, from lighting to the thermal treatment of raw materials; (iv) symbolic systems, which are the most difficult to highlight; to (v) the environment, in terms of resources and climate (Théry-Parisot, 2002; Mallol et al., 2013). In that context, the notion of fire temperature has entered the debate, with the postulate that (*e.g.*, Courty et al., 2012; March et al., 2014) it would document the function and/or functioning of hearths and make it possible to identify activities such as cooking food, thermal treatments or lighting afterwards. But is temperature the best approach to dealing with these issues? What are the temperatures of the prehistoric hearths? What is the level of control and technicality of the cultural entities, and what information can we glean about the evolution of past human societies or social structures?

1.2. On the formation of the anthracological record

By shifting the scale, the temperature of combustion is directly related to the formation of the anthracological assemblage. During combustion, thermal degradation has a significant impact on the properties of wood, leading to mass loss and other mechanical and physical changes; the higher the heat, the more properties are altered. It seems obvious that the temperature of the hearth, which must be distinguished from the temperatures reached by the wood itself, potentially has an impact on the charcoal assemblages in terms of composition, mass of residues, occurrence and representation of taxa. But the correlation between fragmentation and temperature could it be evidenced?

From the 1970s onwards, experiments were carried out to understand the processes of fragmentation and mass reduction of burnt wood For logical methodological reasons related to the representativeness of the assemblages, most of the work has focused on charcoal fragmentation, addressing the potential bias due to species-dependent behaviour (correlation between fragmentation and species) independent of temperature itself (Mas et al., 2020; Chrzavzez et al., 2014; Deforce, 2013; Dussol et al., 2017; Frejaville et al, 2013; Hudspith et al., 2018; Kabukcu and Chabal, 2021; Thery-Parisot et al., 2010; 2014). The results vary greatly from one author to another, and generally the intrinsic and extrinsic variables of combustion are opposed as factors determining the residue rate. According to some authors, the residue rate depends on the physical, chemical and mechanical properties of the species (Rossen and Olson, 1985; Smart and Hoffman, 1988; Loreau 1994; Braadbaart and Poole, 2008), while others defend the predominant influence of extrinsic variables (type of structure, temperatures, oxygenation) (Scott and Jones, 1991; Belcher et al., 2005; Vaughan and Nichols 1995). According to Rossen and Olson (1985), less dense woods produce fewer charcoal than hard woods, while according to Loreau (1994) the opposite is true. According to the same author, high wood moisture content significantly reduces the residue rate, whereas our results tend to minimise the effect of this parameter (Théry-Parisot and Chabal, 2010). For some others, it is the size of the log that determines the residue rate (Smart and Hoffman, 1988). Lingens suggests that the differences in residue rate are more related to the chemical composition of the wood than to its density (Lingens et al., 2005), while the work of Belcher et al. (2005) and Scott and Jones (1991) see residue rate as a concomitant effect of combustion temperature and oxygenation. Finally, according to Vaughan and Nichols (1995), the temperature reached in the furnace determines the size, density and morphology of the residues, while Chrazvzez (2014) suggests that it results from a combination of anatomical features and mechanical properties. However, the effect of temperature on fragmentation is less studied or indirectly as a discret variable, during combustion and under post-depositional processes (Lancelotti et al., 2010; Chrzavzez et al., 2014). In a previous work, we highlight the complexity of the combustion process on the residue rate (Théry-Parisot and Chabal, 2010). We observed high intra-specific variability and differences between taxa, not explained by intrinsic variables (density, anatomy), nor by batch conformation. However, the effect of combustion characteristics on the residue rate was difficult to

determine: firstly, how could a temperature curve be mathematically correlated with a residue rate; secondly, even if the conformation of the batches was standardised, how could differences in volume and their effect on the combustion process be managed. "To address these difficulties, we conducted new experiments and rethought our approach to interpreting the results, taking greater account of the effect of temperature".

1.3. On residual charcoal temperatures

Finally, identifying the temperature at which charcoal was formed is a new challenge, either as an element in understanding the archaeological context itself (natural fire/anthropogenic fires), the type of combustion structure, firewood management and taphonomy (e.g., anatomical signatures), or for methodological applications such as geometric morphometrics, AI-based identification methods. Recent challenges in the field of molecular approaches and isotopy require a detailed understanding of the chemical degradation process induced by combustion and the consequences for the preservation of isotopic and molecular signatures. The molecular changes undergone during combustion have a direct impact on the isotopic signals of charcoal, including a progressive ¹³C depletion up to 500–600 °C, corresponding to the increasing evolution of the [(hemicellulose + cellulose) / lignin] ratio (Czimczik et al., 2002; Turney et al., 2006), with significant implications for paleoclimatic interpretations (Ferrio et al., 2020; Fiorentino et al., 2015; Hall et al., 2008). The similar impact of charring in a broad spectrum of 400-700 °C on the physico-chemical signals of charcoal has, in particular, helped to consolidate observations made on the isotopic signals (δ^{13} C) in anthracological remains and their climatic and environmental interpretations from the Holocene (Aguilera et al., 2008, 2012; Arranz-Otaegui et al. 2017; Baton et al., 2017; Deckers, 2016; Ferrio et al. 2006; Fiorentino et al., 2012; Hall et al., 2008) to the Palaeolithic (Audiard et al., 2019, 2021, 2024, submitted; Belli et al., 2024; Caracuta et al., 2021; Masi et al., 2013). This explains the development of methods for measuring the temperature of charcoal itself. The main paleothermometers include: (i) Hd/Hg ratio analysis by micro-Raman spectroscopy (Deldicque et al., 2016; Deldicque and Rouzaud, 2020; Mouraux et al., 2022; Rouzaud et al., 2015), (ii) charcoal reflectance measurement (Ascough et al., 2010; McParland et al., 2009), (iii) the use of atomic content (%C or H/C) (Aguilera et al., 2012; Audiard et al., 2018; du Boisgueheneuc et al., 2023; Ferrio et al., 2006), or (iv) FTIR measurements (Gosling et al., 2019; Maezumi et al., 2021; Mouraux et al., 2022). These approaches are based on a sound knowledge of the physico-chemical evolution of wood into charcoal. As a result of incomplete combustion (in an O2-free/restricted environment), this transformation can be summarised by a degradation of organic material and its restructuring by counting: (i) an elimination of volatiles $(>300 \degree C)$, (ii) an elimination of hemicellulose then cellulose ($<450 \degree C$) and (iii) the progressive formation of aromatic elements and their organisation into stable condensed compounds (Bird and Ascough, 2012; Braadbaart and Poole, 2008; Czimczik et al., 2002; Tintner et al., 2018; Wiedemeier et al., 2015). Above 400 °C, aromatic elements are mainly structured in large planar aromatic sheets, the size of which increases with temperature (Braadbaart and Poole, 2008; Pyle et al., 2015; Wiedemeier et al., 2015). While experimental calibrations highlight the advantages of these approaches, they vary according to (i) the formation context and combustion temperatures of the charcoal and/or (ii) the impact of post-dispositional processes on the quality and validity of the data obtained. In these contexts, the combination of several approaches may make it possible to consolidate some of the temperatures obtained. The use of Raman spectrometry provides solid results for combustion temperatures above 500–600 °C, but does not take into account heating conditions below 400 °C (absence of the Hd band; Deldicque et al., 2016). Conversely, the interpretation of infrared spectra allows to account for molecular evolution at low heating temperatures (evolution of the holocellulose peak, <400 °C; appearance of aromatic elements,

visible from 400 °C; Gosling et al., 2019; Tintner et al., 2018). An approach combining Raman and FTIR analysis can then cover the entire temperature spectrum (Mouraux et al., 2022). Similarly, the determination of aromaticity and degree of aromatic condensation by NMR provides information on high heating temperatures (McBeath et al., 2011). However, for FTIR analysis, the inclusion of non-removable mineral components in archaeological charcoal can complicate the reading of their absorption spectra (e.g., influence on the intensity and position of infrared bands of organic compounds, juxtaposition of O-SI-O absorption bands; Smidt et al., 2020). These sediment inclusion problems can also be observed in other methods, such as the use of %C, which can underestimate the heating temperature of charcoal fragments (Smidt et al., 2020; Audiard et al., 2024). Some authors therefore suggest coupling the use of %C with FTIR control (Audiard et al., 2021, 2024, submitted), which allows to verify (i) the presence of residual cellulose, (ii) sedimentary inclusions and (iii) the significant presence of oxidised elements (absence of significant carboxylic acid peaks; Vaiglova et al., 2014).

More generally, several studies have highlighted the sensitivity of archaeological charcoal to oxidation phenomena (Ascough et al., 2010; 2011; Braadbaart et al., 2009; Cohen-Ofri et al., 2006). These phenomena can be classified into two groups. Oxidation, which occurs at low heating temperatures (<400 °C), affects volatiles or residual cellulose components (Ascough et al., 2011). Thermometers used on fragments heated to these temperatures and sensitive to oxidation rates (e.g., %C) may then be biased (Mouraux et al., 2022). However, in an archaeological context, these residual oxidised elements could be eliminated by leaching (alkaline environment favoured by the presence of ash; Ascough et al., 2011; Audiard et al., 2024). Above 400 °C, the structural elements of charcoal are considered "inert" and more resistant to oxidation (Bird and Gröcke, 1997; Braadbaart et al., 2009; Retallack, 1998; Skjemstad et al., 2002). However, in certain contexts (e.g., highly alkaline environment, manure, fertile agricultural/forest soils; Ascough et al., 2011; Cheng et al., 2006; Cohen-Ofri et al., 2006; Wiedner et al., 2015), oxidation can occur as a 'self-humification' process affecting the graphitic component and probably also the unorganised phase (Cohen-Ofri et al., 2006). The structure of high-temperature-burned charcoal is then affected, with assessable effects on Raman data, reflectance or FTIR measurements, among others (Alon et al., 2002; Braadbaart et al., 2009; Cohen-Ofri et al., 2006). Furthermore, a recent study shows that charcoal burned above 400 °C appears to be subjected to an organic coating by rapid decomposition simulation, which affects Raman spectra and probably other thermometric approaches (Delarue et al, 2024). The question of the temperature reached and recorded by the charcoal is therefore an ongoing debate on which there is no clear consensus.

The primary objective of this article is to reconsider the concept of temperature through the lens of three successive scales: (i) the hearth scale, (ii) the anthracological assemblage scale and (ii) the charcoal scale. Does measuring the temperatures of prehistoric hearths document their function, their functioning or any other biological, cultural and technical answer? Do temperatures have an effect on the composition of the anthracological assemblage? What is at stake and can we measure the formation temperature of the charcoal itself? Based on complementary experiments in open fires and new experiments in muffle furnaces, we present unpublished results supported by mathematical data processing, providing insight into the combustion structures, and the formation of the anthracological record, through the prism of "temperature". Additionally, to provide a broad overview of the concept of temperature, we have explored the challenges of measuring temperature of the charcoal itself, based on a review of the literature.

2. Material and methods

A first series of experiments was conducted in an open combustion structure in a laboratory setting. The room, equipped and dedicated to the experiments allows for the limitation of the extrinsic parameters (wind, atmospheric humidity) that would be difficult to control outdoors (Théry-Parisot et al, 2020). However, even with stric control of the intrinsic and extrinsic factors, this type of structure introduces variability that acts as background noise, making it difficult to disentangle species behaviour from experimental hazards. In particular, (i) it is difficult to achieve accurate batch calibration, and (ii), temperatures, even when recorded, could not be controlled. For this reason, a second set of experiments was carried out in a muffle furnace, where sample size and the temperatures could be precisely controlled. Measurements taken at the end of the carbonisation process allow the results of the two structures to be compared and modelled as accurately as possible.

2.1. Openfire experiments (Fig. 2) (supplementary file 1)

The batches conformation varies according to three variables: taxa (10), calibre (7 to 12 cm) and number of logs (4,6,8,12). Ten taxa have been selected because of their common occurrence in anthracological assemblages: deciduous *Quercus; Betula pubescens; Olea europea; Corylus avellana; Carpinus betulus; Ostrya carpinifolia; Pinus pinaster; Pinus halepensis; Pinus sylvestris; Populus cf. alba.* In order to limit intraspecific variability, batches of wood for each taxon come from two geographically separate stations. The logs are healthy, unsplit and oven-dried to a standardized moister content of 12 to 14 %. Each log is measured before combustion: calibre, length, mass before drying, mass after drying, density, hygrometry, are scrupulously recorded. Combustions are composed either of 4,6,8 or 12 logs with systematic replications (6 to 10) of each modality to record all the variability. Fire was lit using a blowtorch to avoid adding twigs. One hundred and sixteen experimental combustions were carried out.

For a combination of reasons, temperature is a difficult parameter to measure in an open structure. It is highly variable and the recording depends mainly on the position of the sensors in the fire. As a result, punctual measurements of temperature are unable to describe combustion correctly (Fig. 3). That's why temperatures were recorded simultaneously by 12 sensors evenly distributed throughout the fireplace. The sensors are placed at the base of the fire so that the recording relates to the 'foyer' and not the flames.

One mean temperature curve was then modelled for each combustion (mean of the 12 sensors/combustion) and equated (Fig. 4). As a result, the modelling reduced the total of 1392 temperature curves to 1 mean curve per experiment (116) and 1 mean curve per taxa (10) (Fig. 4). On this basis, several factors can be used as variables to describe

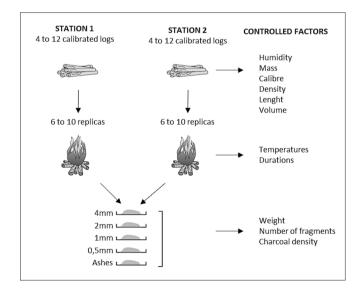


Fig. 2. Experimental protocol in open fire structure (. adapted from Théry-Parisot et al., 2010)

- the combustions, which is not possible with conventional curves. The «variables» are:
- D: Total duration of combustion in the range between 100 $^\circ C$ up and 100 $^\circ$ down
- F: The duration of combustion with flames
- C: The time during which the average temperature curve exceeds 500°.
- Tmean: Mean temperature modelled from 12 sensors over the period D (total duration of combustion)
- Tmax: Maximum temperature reached modelled from 12 sensors
- $-\,$ a: Equation for the slope of the temperature rise from ignition
- b: Equation for the slope of the temperature drop between 500° and 100°
- area (A) of the curve above 500 $^\circ$ C

Thanks to the model, the relationship between batch conformation, combustion processes and the production of remains, taxon by taxon can be better understood.

After each combustion, the residues were sieved with four meshes in order to separate five fractions (>4 mm, 2 to 4 mm, 1 to 2 mm, 0,5 to 1 mm and < 0,5mm), which were weighed and counted (for the > 4 and 2 to 4 mm classes). Charcoal specific gravity were measured with a pycnometer after waterproofing (because of its porosity).

The results we present are based on 116 combustions, representing 808 logs of 30 cm long, 26 kg of charcoal and 352,707 charcoal pieces. All the results are compiled in a data base (see sup. data).

2.2. Muffle furnace experiments (Fig. 5; supplementary file 2)

Logs of the same species *Betula pubescens*, *Corylys avellana*, *Carpinus betulus*, *Populus* sp., *Olea europaea*, *Quercus deciduous*, *Pinus halepensis* and *Pinus sylvestris* were cut into seasoned cubes of 4x4x 4 cm to obtain a homogeneous moisture content of 14 %. Each cube was weighed and measured to determine its mass and specific weight. The cubes were wrapped in aluminium foil to limit oxygen during combustion. The samples were heated at 400 °C, 500 °C, 600 °C, 750 °C and 900 °C for 30 min. They were placed in the furnace when the set temperatures were reached. They were then left to carbonise for half an hour before the furnace was switched off and the contents removed. A total of 240 samples were treated (8 taxa × 5 temperatures × 6 replicates). Individual numbers of fragments, % of residual mass, specific gravity were recorded after heat treatment (Table 1).

2.3. Statistical methods

The following statistical tests were used to analyse the data collected: – Spearman correlation test to evaluate the linear relationship between the quantitative variables; Kruskal-Wallis test with a significance value of 0.05 to evaluate the differences between the data variance of different groups (the normality hypothesis is not met on our data), together with the Dunn post-hoc test to identify the data groups with similar variance. Temperature curves were modelled using R and Excel Stat software.

3. Results

3.1. What are the combustion temperatures in a lab open fireplace?

Here we studied the effect of 3 variables (volume, number of logs and species) on the mean temperatures (Tmean in the model) and the maximum temperatures (Tmax in the model). The results relating to the effects of volume and number of logs are based on the 116 combustions, while those relating to the effects of the taxa are limited to combustions carried out with 6 logs in order not to introduce additional variability.

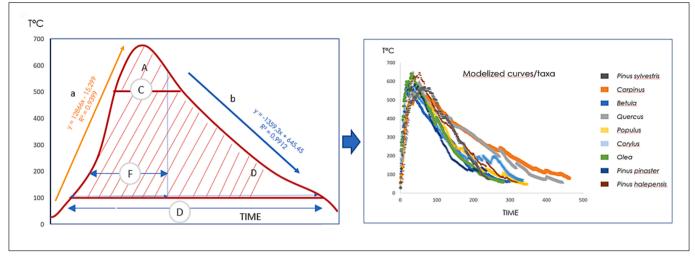


Fig. 3. Example of the variability of temperatures recorded by 12 sensors in the same experimental fire.

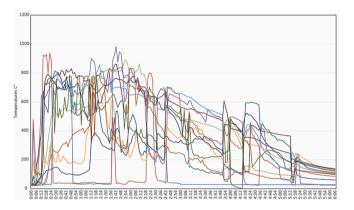


Fig. 4. Mathematical modelling of the temperature.

3.1.1. Mean combustion temperature in an open fireplace (Fig. 6)

The projection of the mean temperatures as a function of wood volume clearly shows the significant variability. For wood volumes ranging from 5000 to 20000 cm³, the mean temperature recorded (Tmean) varied between 277° and 506°. However, even when the values were grouped according to the number of logs, the correlation test showed low correlation coefficients between volume and mean temperature (R between -0.46 and 0.47).

As shown in Fig. 7, even when the amount of wood was tripled from 4 to 12 logs, the mean temperatures ranged from 250 °C for the lowest to 550 °C for the highest (Fig. 7), with no significant differences between the groups considered (number of logs) (Kruskal-Wallis test; p-value = 0.7194). In our experiments, the number of logs doesn't influence the mean temperature.

When analysing the taxa effect, we first notice a high variability of mean temperatures within replica, even if we only consider the combustion with 6 logs (Fig. 8). For example, the mean temperatures of *Pinus sylvestris* ranged from 275 °C to 480 °C, while those of *Populus* sp. varied

MUFFLE FURNACE



240 samples 8 taxa 2 provenances 5 temperatures 6 repetitions/modalities Weight Specific gravity

Standardized cubes





9

Fig. 5. Experimental protocol in a muffle furnace.

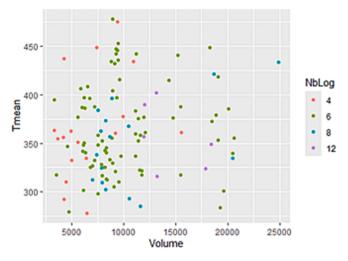


Fig. 6. Mean combustion temperature as a function of volume (Tmean).

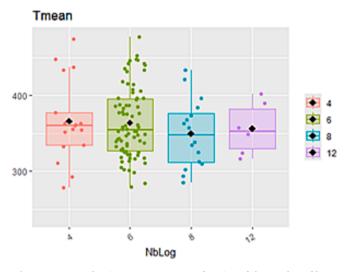


Fig. 7. Mean combustion temperature as a function of the number of logs.

between 300 °C and 380 °C. The Kruskal-Wallis test showed significant differences between the groups (p-value = 0.0008975), however, the Dunn post-hoc test doesn't allow a unique discrimination of species by considering the mean temperature variable.

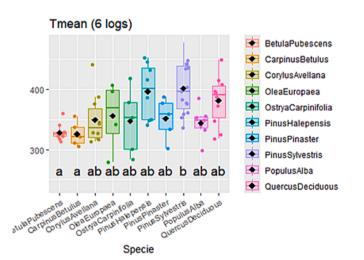


Fig. 8. Mean combustion temperature as a function of number of taxa.

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3.1.2. Maximum mean temperature of combustion (Tmax)

With wood volumes ranging from 5000 to 20000 cm³, the mean maximum temperature (Tmax) ranged from 460 °C to 813 °C. Higher temperatures can be reached locally with a single sensor, 996 °C being the higher temperature reached during the combustion of 6 logs of *Quercus*. However, even when grouping the values by number of logs, the statistical test showed no correlation between the volume and the maximum temperatures (R between –0.097 and 0.301) (Fig. 9).

The absence of correlation is confirmed when analysing the effect of increasing logs. The mean maximum temperatures range between 650 and 700 °C with a substantial variability within combustions with the same number of logs. The Kruskal-Wallis test showed non-significant differences between the considered groups (p-value = 0.3088) (Fig. 10).

Considering the specie effect on mean maximum temperature, we first notice high variability within combustions of the same taxa (eg., 600° to 800° for *Olea*; 520° to 720° C for *Betula*). The Kruskal-Wallis test showed significant differences between the considered groups (p-value = 0.03759) but the Dunn post-hoc test concluded that all groups have similar variance, marked by the letter "a" in the plot. This is justified by the fact that the Kruskal-Wallis p-value result is very close to the significance level = 0.05. The results don't allow a unique discrimination of the species by considering the mean maximum temperature variable (Fig. 11).

To sum up, in an open fire structure such as the one we experimented with, the average temperatures recorded ranged from 250 °C to 550 °C and the average maximum temperatures from 460 °C to 800 °C, with no correlation with the variable tested (volume, number of logs, species). In particular, we observed the high variability of the results within replicates of the same experience. As these are average temperatures, we can't rule out higher local temperatures. We will discuss their significance for the study of prehistoric hearths below.

3.2. What is the effect of temperature on the charcoal assemblages?

Next, we examine the effect of temperature on the charcoal assemblage and its potential influence on the representation of certain species. Specifically, we investigate how temperature impacts the post-combustion number of fragments under two complementary experimental conditions: open fire and muffle furnace.

3.2.1. Temperature and fragmentation in muffle furnace experiments

First, we tested the effect of the temperature on the total number of charcoal (Nb frag.). Fig. 12 shows that temperature has a significant influence on the number of fragments: the positive correlation is almost perfect (R = 0.9798934) between the Temperature (400 °C, 500 °C, 600 °C, 750 °C, 900 °C) and the mean values of Nbfrag for each

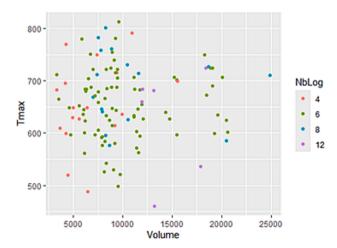


Fig. 9. Maximum combustion temperature as a function of volume (Tmax).

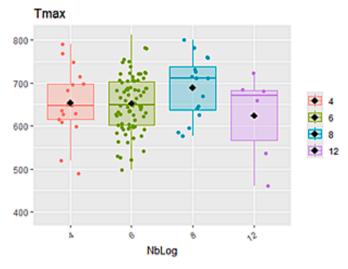


Fig. 10. Maximum combustion temperature as a function of number of logs.

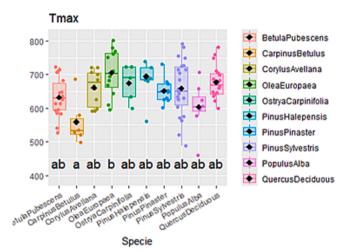


Fig. 11. Maximum combustion temperature as a function of taxa.

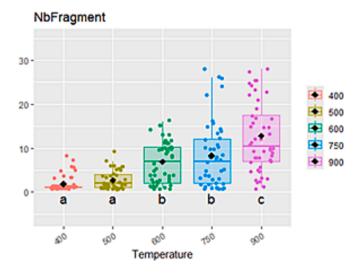


Fig. 12. Number of fragments versus temperature.

temperature (respectively 1,8; 2.6, 6,8; 8,2; 12,8). We also observe a greater variability at 600 $^{\circ}$ C and above. The Kruskal-Wallis test distinguish 3 stages in the fragmentation process with significant differences

between the considered groups (p-value < 2.2e-16): low fragmentation below 600 °C with no difference between 400 and 500 °C, a second group up to 750 °C with no difference between 600 °C and 750° and, a final group at 900 °C with a significantly higher number of fragments and greater variability.

Some of the observed variability may be attributed to differential fragmentation resulting from taxon-specific effects. Fig. 13 provides a clear illustration of both intraspecific and interspecific variability. The Kruskal-Wallis test showed significant differences between the considered groups (p-value = 1.781e-12) and groups with similar variance: groupe a: *Betula, Carpinus,* Corylus, *Olea,* Pin halepensis, *Pinus pinaster, Quercus;* groupe b: *Betula, Olea,* Pin halepensis, *Populus, Quercus;* groupe c: *Pinus sylvestris, Populus (*Dunn post-hoc.) However, the results don't allow a unique discrimination of species by considering the number of fragments, which explains the presence of the same taxon in 2 groups (eg. *Olea is* present in both groups a and b).

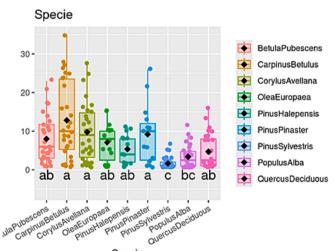
When we combine temperature and taxa, we observe that fragmentation is homogeneous for all taxa at 400 °C and 500 °C whereas a noticeable differentiation between the taxa occurs up to 500 °C (Fig. 14). Whatever the temperature, the mean number of fragments of *Pinus sylvestris* are consistently lower, while those recorded by *Carpinus* and *Corylus* are consistently higher. The behaviour of the intermediate group, composed of *Betula*, *Olea*, *P. halepensis*, *P. pinaster* and *Quercus* is more erratic.

Intuitively, one might assume that density plays a role in the fragmentation process. Or, the density is not correlated with the number of fragments (R = 0.26, pvalue < 2.2e-16). *Carpinus*, more fragmented on average, is one of the densest taxa while *Pinus sylvestris*, which is less fragmented, is one of the least dense taxa. Even if we admit some counterintuitive behaviour (the densest wood would fragment a lot, while less dense wood would fragment little), Corylus, which density is almost identical to that of *Pinus sylvestris* has a mean number of fragments among the highest (close to *Carpinus*). In contrast, *Quercus*, which has a higher density stand with the less fragmented taxa.

3.2.2. Temperature and fragmentation in open fire experiments

These results are based on more than 352,707 pieces of charcoal weighed and counted by size class. For each taxon, we modelled fragmentation, which allows to present the results in a standardized form, accounting for variations in volume and size since it was not feasible to obtain perfectly homogeneous wood batches.

Overall, charcoal represents less than 2 % of the weight of the wood burnt. Overall, charcoal represents less than 2 % of the weight of the burnt wood. This finding is consistent with the results of other experiments we have carried out on open-air fires (Théry-Parisot et al., 2018).



Specie

Fig. 13. Number of fragments as a function of specie.

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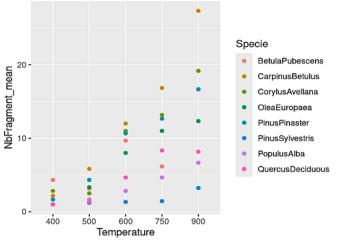


Fig. 14. Number of fragments as a function of temperature and taxa.

The first measure is the number of fragments > 2 mm/species for an identical volume of modelled wood. Significant differences between species were observed (Kruskal-Wallis test, p-value = 2.887e-12), and groups of similar variances emerged (Dunn's post-hoc test): group a: Betula, Carpinus, Ostrya, Corylus, which produced more fragments on average; group b: an intermediate group composed of Olea, Pinus halepensis, Pinus sylvestris, Populus and Quercus; a last group c) composed of Quercus, Populus, Pinus halepensis, which on average produce fewer fragments than the other taxa. The test does not allow a single discrimination of species, which is explained by the high variability with the replica of a same modality and the overlap between the groups (Fig. 15). This means that even if we cannot distinguish each taxon from another, we can distinguish a group that produces more residues on average (Betula, Carpinus, Corylus and Ostrya) from another that produces fewer residues on average (P. halepensis., P. sylvestris, Olea, Populus, Quercus).

According to the correlation matrix, the differences between groups of species can be explained neither by batch conformation nor by combustion parameters (Spearman correlation test, R between -0.14 and 0.44) (Fig. 16). The total duration of the combustion has for instance no effect on the number of fragments > 2 mm, nor the maximum and mean temperatures reached. Conversely, the conformation of the batches (volume and calibre) had a significant effect on the duration of

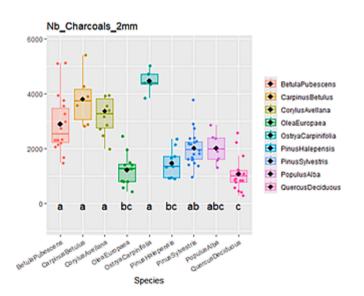


Fig. 15. Number of fragments > 2 mm for a same volume of wood.

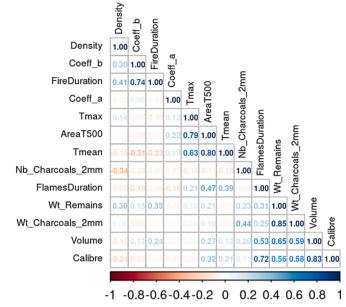


Fig. 16. Correlation between all factors.

the flames (R = 0.72), and the total weight of charcoal < 2 mm (R = 0.56).

Among the intrinsic parameters of the taxa, density seems to have little or no effect on the number of fragments (R between -0.34 and 0.40 for the considered variables). It is also noteworthy that none of the combustion parameters (duration, maximum and average temperatures, coefficient a for ignition, b for depletion, etc.) had an effect on the number of fragments > 2 mm.

In summary, the muffle furnace experiments clearly show the increasing number of fragments above 500 $^{\circ}$ C for each of the taxa studied. Carpinus and Corylus are on average more fragmented and Pinus sylvestris less, while other taxa show a more erratic behaviour. In open fire, 2 groups of taxa are distinguished, but the differences are not explained by the combustion characteristics. The number of fragments never correlates with the density, neither in the muffle furnace nor in the open fire.

4. Discussion

4.1. Fire and fictions: What are the combustion temperatures in an open fireplace?

Laboratory experiment makes significant contribution to defining temperatures recorded in open fireplaces. Firstly, we highlighted a significant variability in temperatures even under standardised conditions, which foreshadows the difficulty of producing a posteriori intelligible measurements of archaeological hearth temperatures. In our experiments, the average temperature of a fire composed of 6 logs is 365 °C (353 °C considering all combustion), while the maximum temperature is 662 °C (656 °C considering all combustion), with local variations that can occasionally reach 980 °C depending on the position of the sensor in the fire. However, these temperatures are probably overestimates compared to an open-air fire where atmospheric humidity contributes to lower temperatures. We showed that even when the hearth size is quadrupled, the variability does not depend on taxon, volume or log size. It is conceivable that a tenth larger fire would result in higher temperatures, but this has yet to be demonstrated. For example, experiments carried out in semi-closed conditions (rock shelter) with 30 kg of dry wood never exceed 600C° (Lacanette et al., 2017). Parameters we have not tested may of course have an effect on the temperatures. Kils or Polynesian structures can reach around 1000 °C in the oven (Chabal,

2001). However, this type of structure has not been found in the Palaeolithic period. It is also well known that moisture content has a significant effect on combustion temperature: the greener the wood, the lower the temperature (Trabaud, 1992). At least, the experiments carried out with other fuels (lignite and bone) have also highlighted the variability of temperatures, which are generally in the same ranges as wood (Pérez et al., 2017; Karkanas and Kyparissi-Apostolika, 2024; Théry-Parisot and Costamagno, 2005).

On this basis, we can compare experimental data with measurements made a posteriori on archaeological contexts. Measurements are based on physical, chemical and biomolecular measurements of either the sediment or the artefacts (Allue et al. 2022). According to the authors, these temperatures range from 250 °C to 600 °C (Berna et al., 2007; Brodard et al., 2016; Leierer et al., 2020). The difference may be due to the heterogeneity of the archaeological contexts themselves: the nature of the structures and artefacts measured, but also the post-depositional changes in the sediment that contribute to blind the original signal. It also probably reflects the variability observed in the experimental conditions. Most likely it's a localised temperature and/or a higher temperature reached without clear significance It can reflect either the average temperature reached, the maximum temperature, a local temperature or none of these. For these reasons, temperature measurements from archaeological hearths should be regarded with caution. We can also look at the issue from another angle. Most palaeolithic activities related to fire required temperatures of 400 °C, even for technically complex tasks like making adhesives from birch bark (Rageot et al., 2018). Temperatures of 250 °C are sufficient for cooking, roasting or smoking food, while lighting and heating depend primarily on the size of the flames, regardless of temperature. It can be concluded that this may not be the most reliable parameter for describing the behaviour of an open fireplace, whereas modes of heat transfer (conduction, radiation, convection) better describe the thermal characteristics of a hearth even if it is also difficult to highlight (Théry-Parisot et al., 2018; Vidal-Matutano et al, 2017). However, the control and raising of temperature became really important with the introduction of handicrafts (eg, ceramics, glass, metallurgy) which explain the diversification of fire structures from the Neolithic onwards.

4.2. Charcoal to ashes: What is the effect of temperature on the charcoal assemblages?

The experiments also provide insight into the effect of temperature on charcoal assemblage. We first demonstrated the significant intraspecific and interspecific variability observed under both highly standardised conditions in the muffle furnace and in open hearth conditions. In the open hearth, the number of fragments > 2 mm has no correlation with any combustion variable or with the characteristics of the wood batches. This is probably due to the consistency of the average temperatures across all experiments. Conversely, temperature has a significant effect on total fragmentation above 500 °C in the muffle furnace experiments. However, our experiments have shown that this cannot be transferred to open fire, as temperatures rarely exceed 500 °C. Therefore, only closed structures with temperatures above 500 °C could theoretically cause differential fragmentation of the wood. This remains to be proven, however, as the lack of oxygen in closed combustion structures is likely to alter the behaviour of the wood completely.

A comparison of the results of the two experiments (open fire/muffle furnace) reveals certain trends in the species behaviour. It is worth noting that taxa such as *Carpinus* and Corylus consistently produce more remains, while *Populus* and *Pinus sylvestris* produces fewer. In contrast, other taxa show a more erratic behaviour. It should be noted that these differences are only valid under equivalent conditions, *i.e.* in standardised batches of wood. However, this does not reflect real combustion conditions where logs of different sizes, calibres, phenological and physiological states are mixed. It is therefore reasonable to conclude that the results would be significantly different if a mixture of taxa, varying sizes and unequal proportions were used. Another limitation of this result could be the origin of the wood used for both experimental conditions which came from the same lots. We cannot exclude that the differential fragmentation is the result of the "station effect".

When we consider the additional effect of post-deposition processes, it seems even more difficult to highlight the general characteristics of the fragmentation process. Temperature has a real impact on the mechanical properties of the wood, which are probably responsible for the greater fragmentation due to combustion above 750°. However, experiments aimed at assessing the impact of taphonomic processes on charcoal preservation have not been able to illustrate the effect of temperature on fragmentation. (Lancelotti et al. 2010; Chravzvez et al., 2014). Therefore, even if we were to consider the hypothesis of different temperatures depending on the function of the hearth, which we have not demonstrated, this temperature would not be able to condition fragmentation and/or bias the representation of wood species.

4.3. Matter and memory: What are temperatures reached by the residues?

Changing scale, since we are proposing a global approach to the notion of temperature in Anthracology, we must include considerations of the temperatures reached by the charcoal itself as a factor of potential limitation of some new methodological approaches from a literature review. Several physico-chemical studies have attempted to define a method for estimating the formation temperature of charcoal fragments as accurately as possible. While there is still some debate about the degree of carbonisation of archaeological charcoal, there is general agreement that charcoal burnt in a broad range of temperatures around 400-700 °C is preferentially preserved in burial contexts. It should be noted that, for the sake of simplicity, we convert the state of the charcoal as a function of the burning temperature, but that it would be more accurate to speak of the "degree of carbonisation" (integrating temperature and heating time, as well as the state of the ignited material). Anthracological remains preserved in archaeological deposits are the result of several factors, starting with the physiological and phenological state of the fuel and the intensity of carbonisation, followed by postdepositional processes that depend on the archaeological and sedimentary context (Chrzavzez et al., 2014; Braadbaart et al., 2009; Retallack, 1998; Théry-Parisot and Henry, 2012; Henry and Théry-Parisot, 2014; Vidal-Matutano et al., 2017).

Considering that temperatures within the heat zone are heterogeneous and depend on fuel exposure (e.g., branch position), charcoal fragments cover the entire temperature spectrum. Charcoal found in natural hearths at this stage has generally been heated to low temperatures, with pyrolysis stopping early during slow, flameless combustion, thus avoiding complete ignition from ember to ash ("smoldering" process, typically < 600 °C; Braadbaart and Poole, 2008; Jones and Chaloner, 1991; Li et al., 2022; Ohlemiller, 1985; Wang et al., 2021). However, by limiting the combustion air or even drastically stopping thermodynamic combustion, charcoal can be preserved at higher heating temperatures.

The chemical and mechanical sensitivities of the charcoal obtained will be crucial for archaeological preservation. In this respect, different observations depending on methods and contexts still fuel the debate on the subject. For example, Raman studies on charcoal obtained after the fire of Notre Dame show carbonisation temperatures ranging from 500 to 1300 °C for the general spectrum (Deledique and Rouzaud, 2020) and around 500–700 °C for the internal carbonised zones in the preserved burnt logs (Rocha et al., 2024). Raman studies in archaeological contexts confirm these observations, for example the preferential preservation of charcoal around 700 °C at the Neolithic site of Lac de Chalain (Baton et al., 2017). On the contrary, most published measurements on preserved archaeological charcoal place these temperatures at 400–500 °C, based on several indicators: (i) the need for metallisation prior to SEM analysis of many anthracological assemblages, implying production at temperatures below 600 °C (Beall et al., 1974); (ii) reflectance analyses

(vitrified or not), with a critical look at the preservation of charcoal in alkaline contexts (Ascough et al, 2010; Braadbaart et al., 2009; McParland et al., 2010); (iii) FTIR data (Audiard et al., 2024; Ascough et al., 2011); or (iv) the experimental and archaeological study of %C (Aguilera et al., 2012; Audiard et al., 2024; Ferrio et al., 2006; Turney et al., 2006). This preferential preservation of archaeological charcoal is due to its resistance to post-depositional chemical and mechanical processes (Chravzvez et al., 2014; Cohen-ofri et al., 2006; Lancelotti et al., 2010). Indeed, at these temperatures (<380-400 °C), unburned or slightly burned fragments are biodegradable and may disappear after deposition. Conversely, burnt fragments above 600 °C become mechanically brittle, increasing their fragmentation and shifting from macro to micro charcoal (not extractable by flotation or sieving). This loss is in addition to that observed during combustion, with charcoal at high temperatures turning to ash. We can therefore hypothesise that only charcoal carbonised in the 400-600 °C range, in open-fire structures, is well preserved in archaeological sites, and even more so in ancient sites where post-depositional processes can have a significant effect.

5. Conclusions

Our experiments show that the average temperature of an open hearth, like those found at Palaeolithic sites, is approximately 400 °C, regardless of the hearth's size or composition. This is therefore a poor proxy for studying the functions and functioning of hearths. Consequently, it is preferable to favour multi-proxy approaches, combining (i) all cultural information, traces of fire-related activities such as food preparation, thermal treatments; (ii) detailed study of combustion structures, including micromorphology and biomolecular analyses; (iii) fuels, including all anatomical signatures and calibres, but also searching for other fuels such as bones and dung ((Lancelotti and Madella, 2012; Karkanas et al. 2024); (iv) then environmental proxies, dating and isotopic analyses. Biochemical analyses using lipidomics and proteomics (Connolly et al., 2019), together with ongoing research on Benzene Polycarboxylic Acid (Scheneider et al., 2010), and isotopic analyses are providing promising insights into the nature of fuels and the frequency of combustion events. Changing scale, if the effect of temperature on the fragmentation is effective, it seems to act with the same intensity for all taxa. Even if there are some differences, the study of the behaviour of species with regard to combustion does not reveal any major or significant differences in the representation of the different taxa. Moreover, these differences are difficult to explain and probably involve mechanical, chemical and environmental parameters. It should be noted that the intraspecific variability observed under standardized combustion conditions is even more marked under real conditions, when parameters such as fire oxygenation, stem size or the shape of the combustion structure vary. We are touching on the limitations of experimenting with biological organisms. Their variability, their diversity and the multitude of parameters involved in species characteristics, combustion and postdepositional processes completely obscure the signal. It's fair to say that even with maximum standardization of experiments and infinite replications, I doubt that it's possible to define a mathematical law that shows statistical differences between the fragmentation of taxa. Although these results do not allow us to infer the behaviour of all the taxa not tested in our experiments, they do support the validity of anthracological studies for the study of practices and palaeoenvironments. They partly explain the ecological coherence of the assemblages, the similarity of data from contemporary sites and the convergence of the results of anthracological studies at different spatio-temporal scales.

Finally, measuring the temperature at which charcoal is formed is emerging as a new challenge for Anthracology, linked to the development of isotopic and molecular approaches. However, there are still differences between the methods, probably due to the specificity of the measurement methods themselves. Pooling measurements carried out on the same samples but using different methods will probably make it

possible to refine the tools. The development of new physico-chemical approaches requires to pursue our studies (i) in different contexts, as well as (ii) on key transformation processes such as vitrification phenomena or post-dispositional oxidation (Delarue et al., 2024), or (iii) on instrumental and analytical development. In another area, studies of the physico-chemical evolution of vitrified charcoal, which is common in archaeological remains, are sorely lacking. For example, while studies of vitrification by casi-instantaneous "flash-pyrolysis" combustion above 800 °C agree with the proportions given by Raman (Courty et al., 2020), measurements for charcoal vitrified at lower temperatures (Henry, 2011; McParland et al., 2010) need to be investigated at elemental, molecular and isotopic levels. While these methods appear to be objectively applicable to experimentally produced samples, the accuracy of measurement in archaeology requires further development. Furthermore, measuring the temperature of charcoal alone is not enough to determine the combustion temperatures of fireplaces in an archaeological context. The two measurements are not correlated because of the combustion process itself: the temperature of the fire is not the temperature reached by the wood itself but also due to the effect of post-depositional process.

Finally, temperature, an a priori simple and unambiguous measure, appears as a compliant concept combining several distinctive temperatures from the hearth to the charcoal residues. This study shows either the complexity of the processes themselves, the difficulty of obtaining robust measures even in experimental contexts, the lack of consensus on measurement methods and the high degree of interaction between ante-, sin- and post-combustion factors, paving the way for further research.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work the author(s) used DeepL Write in order to correct and improve the English language. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

CRediT authorship contribution statement

Isabelle Théry-Parisot: Writing – original draft, Conceptualization, Data curation, Funding acquisition, Supervision, Validation, Writing – review & editing. Benjamin Audiard: Writing – original draft. Alain Carre: Investigation. Vanna-Lisa Coli: Data curation, Formal analysis, Investigation, Validation. Pauline Garberi: Data curation, Formal analysis, Software, Validation. Anne Lavalette: Data curation, Formal analysis, Investigation, Methodology, Validation.

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.jasrep.2025.104977.

Data availability

Data will be made available on request.

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