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RESEARCH ARTICLE

These boots are made for burnin': Inferring the position of the corpse and the presence of leather footwears during cremation through isotope ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$) and infrared (FTIR) analyses of experimentally burnt skeletal remains



Kevin Salesse^{1,2,3,4*}, Elisavet Stamatakis^{1,3,4}, Ioannis Kontopoulos⁵, Georges Verly⁶, Rica Annaert^{3,7}, Mathieu Boudin⁸, Giacomo Capuzzo¹, Philippe Claeys⁴, Sarah Dalle^{3,9}, Marta Hlad^{1,3,4}, Guy de Mulder⁹, Charlotte Sabaux^{1,9}, Amanda Sengeløv^{1,9}, Barbara Veselka^{3,4}, Eugène Warmenbol¹⁰, Martine Vercauteren¹, Christophe Snoeck^{3,4,11}

1 Research Unit: Anthropology and Human Genetics, Department of Biology of Organisms and Ecology, Université Libre de Bruxelles, Brussels, Belgium, **2** UMR 5199: "PACEA—De la Préhistoire à l'Actuel: Culture, Environnement et Anthropologie", University of Bordeaux, Pessac Cedex, France, **3** Maritime Cultures Research Institute, Department of Art Sciences and Archaeology, Vrije Universiteit Brussel, Brussels, Belgium, **4** Research Unit: Analytical, Environmental and Geo-Chemistry, AMGC-WE-VUB, Vrije Universiteit Brussel, Brussels, Belgium, **5** GLOBE Institute, Section for GeoGenetics, University of Copenhagen, København, Denmark, **6** Faculté des Lettres, Sorbonne Université, Paris, France, **7** Flemish Heritage Agency, Brussels, Belgium, **8** Royal Institute for Cultural Heritage, Brussels, Belgium, **9** Department of Archaeology, Ghent University, Ghent, Belgium, **10** Centre de Recherches en Archéologie et Patrimoine, Department of History, Arts, and Archaeology, Université Libre de Bruxelles, Brussels, Belgium, **11** G-Time Laboratory, Université Libre de Bruxelles, Brussels, Belgium

* Kevin.Salesse@vub.be

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Abstract

Cremation is a complex mortuary practice, involving a number of activities of the living towards the dead before, during, and after the destruction of the bodily soft tissues by fire. The limiting information concerning these behavioral patterns obtained from the pyre remains and/or cremation deposits prevents the reconstruction of the handling of the corpse during the burning process. This pioneering study tries to determine the initial positioning of the corpse in the pyre and assess whether the deceased was wearing closed leather shoes during cremation through isotopic ($\delta^{13}\text{C}$, $\delta^{18}\text{O}$) and infrared (ATR-FTIR) analyses of experimentally burnt pig remains, used as a proxy for humans. The results obtained show that both the position of feet on or within the pyre and the presence of footwears may moderately-to-highly influence the oxygen isotope ratios of bone apatite carbonates and the cyanamide content of calcined bone in certain situations. By forming a protective layer, shoes appear to temporarily delay the burning of the underlying pig tissues and to increase the heat-shielding effect of the soft tissues protecting the bone mineral fraction. In such case, bioapatite bone carbonates exchange oxygen with a relatively more ^{18}O -depleted atmosphere (due to the influence of lignin-derived oxygen rather than cellulose-derived oxygen),

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resulting in more pronounced decrease in the $\delta^{18}\text{O}_{\text{carb}}$ values during burning of the shoed feet vs. unshoed feet. The shift observed here was as high as 2.5‰. A concomitant isotopic effect of the initial location of the feet in the pyres was also observed, resulting in a top-to-bottom decrease difference in the $\delta^{18}\text{O}_{\text{carb}}$ values of shoed feet of about 1.4‰ between each deposition level tested. Finally, the presence of cyanamide ($\text{CN/P} \geq 0.02$) seems to be indicative of closed footwear since the latter creates favorable conditions for its incorporation into bone apatite.

Introduction

Cremation is a complex process that has been performed in a wide range of geographic and cultural contexts from prehistory to present day [1–8], involving a chain of activities of the living towards the dead before, during, and after cremation [9, 10]. The investigation of the post-cremation history of burnt skeletal remains is particularly complicated due to their fragmentary and often incomplete nature [11–14], but reconstructing the handling of the corpse during the burning process probably constitutes an even more challenging venture. The ephemerality of the moment, the taphonomic vulnerability of the pyre locations [15–17], the choices made by the funerary ritual attendants, and the unpredictable contingencies inherent to the burning process (e.g. pyre collapse) are all variables that make each cremation a unique event [9, 18–20]. Yet, to better understand the cremation processes and, ultimately, the funerary ritual determining how the body of the deceased was managed during burning is vital (see [21] or [22] for archaeological examples; see [23] for ethnographical cases).

In open-air cremations, the manipulation of the corpse during the burning process is conditioned, mainly but not only, by its initial positioning in the pyre, and by the presence of clothing (shroud, garments, etc.), both of which can affect the efficiency of combustion in a variety of ways. Although it is commonly suggested that the deceased is placed on top of the pyre to be visible during the burning phase (e.g. [16]), other arrangements include placing the corpse under or in the heart of the pyre structure [24–26]. The deceased may have been previously clothed, placed in a shroud or left naked [24, 27]. When exposed to fire, clothing—typically made of vegetal fabrics or animal leathers [25]—is swiftly consumed, making its presence difficult to establish, unless metalwork, such as ornaments (e.g. belt buckles, brooches), reinforcements (e.g. hobnails, toe tips) or others accessories (e.g. pins, buttons) is recovered from the pyre remains [25, 26]. The handling of the corpse, its positioning and dressing, may differ from one individual and/or population to another depending on many factors, such as the biological and social status of the deceased [28–32], the religious beliefs and cultural practices of the participants in the funerary event [24, 32–34], and/or the expertise of the cremation operators [35]. Identifying the positioning and dressing of the corpse improves understanding of past societies that practiced cremation as a funeral ritual.

Recent work has highlighted the high potential of calcined skeletal remains to isotopically investigate cremation practices (see [36] and references therein). While all organic matter has been destroyed by the high temperatures reached during the burning process, the inorganic fraction (often called bone apatite or bioapatite) still contains a certain amount of carbon and oxygen under the form of carbonates. Lanting and colleagues [37, 38] showed that while it was possible to obtain reliable radiocarbon (^{14}C) ages from the carbonate fraction of calcined bones in specific archaeological contexts, the stable carbon isotope compositions ($\delta^{13}\text{C}$) of these bones had been significantly altered. It was proposed that three parallel mechanisms

could explain these alterations. First, carbon exchanges occur between the bone apatite carbonates and the surrounding cremation atmosphere. Second, carbon from soft tissues and bone collagen admixes to that of the bone apatite carbonates during heating. Third, most of the structural bone apatite carbonates are lost during the combustion process, and this loss is likely associated with time/temperature-dependent fractionation [39–50]. Experimental works have shown that fuel carbon replaces up to 95% of the biogenic bioapatite carbon as a result of these mechanisms [42, 47, 49].

Less consideration has been given to the stable oxygen isotope composition of calcined bone apatite carbonates ($\delta^{18}\text{O}_{\text{carb}}$). While a temperature-related change in the $\delta^{18}\text{O}_{\text{carb}}$ values of calcined bones was consistently recognized, the explanatory model behind this shift remains not yet fully understood [48, 50–53]. Indeed, in a cremation environment, many possible sources of oxygen can ultimately influence the bone $\delta^{18}\text{O}_{\text{carb}}$ signals (e.g. O_2 , CO , CO_2 , H_2O , NO_x from the ambient air, fuel or human/animal soft tissues and bone collagen). Oxygen isotope exchanges between bone apatite carbonates and CO_2 from the combustion atmosphere—and particularly from fuel—could have led to the observed ^{18}O depletion in calcined bones [50]. Temperature-induced isotopic fractionation associated with the loss of structural carbonates during the combustion process may also be at the origin of the decrease in the $\delta^{18}\text{O}_{\text{carb}}$ values of calcined bones [50, 53]. Even if the $\delta^{13}\text{C}_{\text{carb}}$ and $\delta^{18}\text{O}_{\text{carb}}$ proxies can no longer be used to infer life traits (i.e. diet and mobility) of the deceased in cremation contexts, they can be used as semi-independent lines of evidence to better characterize cremation conditions [50, 54, 55].

Fourier transform infrared (FTIR) indices offer the possibility to assess the extrinsic and/or intrinsic factors at the origin of the cremation-induced transformations [52, 56–60]. During cremation, bone is physically and chemically altered. These alterations occur in a generally orderly manner. As the temperature rises, the thermal shielding effect of the organic matter protecting the inorganic fraction diminishes and it is probably only after this loss that the carbonates are completely exposed to heat, and thus gradually lost [61–64]. The CO_2 emitted during the combustion of bone initially comes from the organic matter, and in a second stage from structural carbonates, with a transition period between the two [65]. As a consequence of heating, alterations in bone crystallinity occur, with larger crystals and higher degrees of crystallinity, making burnt bone, and in particular calcined bone, more resistant to post-burial alterations [57, 60]. Throughout its rearrangement, bone apatite can incorporate new chemical species such as cyanamide. Cyanamide molecules (H_2CN_2) form in the presence of ammonia and carbon, and CN_2^{2-} substitute for hydroxyl (OH^-) groups or type A carbonates in the apatite lattice [66–69]. The origin of ammonia is still uncertain; however, it could be a byproduct of the combustion of organic matter (e.g. body tissues) or fuel (e.g. coal) [45, 47]. A greater occurrence of cyanamide is expected to be found when combustion occurs under reducing conditions (i.e. low oxygen availability) [43, 50, 57, 70]. Therefore, the cyanamide content constitutes an useful indicator for identifying specific cremation settings.

This study aims to better understand past cremation practices and related funerary rituals by identifying the initial positioning of the corpse deposited on/in a pyre and assess whether the deceased was wearing closed leather shoes during cremation through isotopic ($\delta^{13}\text{C}_{\text{carb}}$ and $\delta^{18}\text{O}_{\text{carb}}$) and infrared (ATR-FTIR) analyses of experimentally burnt pig remains. Leather footwear is postulated to be the most fire-resistant type of clothing and foot bones encased in a closed type of leather shoe may experience heating conditions characteristic of confined-space cremation (low availability of oxygen, poor ventilation, etc.), which could lead to distinct bone apatite chemical and/or structural variations. The effects of the deposition levels of the feet (i.e. bottom, middle, or top of pyre) are also investigated to establish whether their initial position affects their chemical and structural characteristics due to different heating conditions.

Materials and methods

Setting up and running of experimental cremations

The experimental cremations were conducted in Wallonia (Belgium) during two different sessions (October 2018 and July 2019), the first in a clearing partially sheltered from the wind and the second in a disused quarry more exposed to the wind. Four fires were conducted per session.

European beech (*Fagus sylvatica*) wood was selected to build all eight experimental pyres (see Section 1 in [S1 Appendix](#) for the description of the pyre architecture). The firewood was cut from dried logs ($n \simeq 12$) originating from a small plot of land located in the French Ardennes (Houdain Lez Bavay, France). Vegetable tanned cow and goat leathers were purchased (Steffisburg, Switzerland) to make the experimental footwear. With a thickness of 3 mm and 1.5 mm respectively, these leathers were used to test the fire resistance of two different types of shoes. Seven shoes were created using the cowhide and only one with the goatskin (see Section 1 in [S1 Appendix](#) for details about the leather manufacturing process and the footwear making). Domestic pigs (*Sus scrofa domesticus*) were used as human substitutes since carcasses or body parts of such animals have long been involved in experimental cremations [71–74]. Feet of nine pigs raised in the same farm were bought from a local butcher (Anderlecht/Sint-Lievens-Houtem, Belgium).

In each experimental outdoor cremation, one unshoed foot and one shoed foot from a single animal were placed at the same level in the pyre structure and as far apart as possible from each other (ca. 40 cm). For each experimental session, pig feet were placed on top (x 1), in the middle (x 2) or at the bottom (x 1) of the pyres. Dry farmyard manure pellets ($\simeq 5$ kg) were added to one of the pyres (pyre 8) to create an ammonia-rich combustion atmosphere. The goatskin was used in only one case (pyre 3). Feet from the ninth pig were burned one by one in a muffle furnace (Thermo Heraeus M110) at 900°C for about 5h and allowed to cool down overnight.

On average, the pyres were reduced to ashes after 1–1.5h. All the pyres reached a temperature of 900°C and some went above 1000°C for a short period of time (S2.1 Table in [S2 Appendix](#)). There was no human intervention after the fire was lit ([Fig 1](#)) and no additional fuel was loaded into the fires during the cremation process. All structures, apart from pyre 7, collapsed on themselves, retaining the animal skeletal remains within. Pyre 7 toppled chaotically to the side, causing the shoed foot to fall out of the pyre. In all cases, the burning process lasted long enough to destroy the organic matter (leathers, pig soft tissues and bone collagen) and calcine most of the recovered foot bones. In the first session, the pyre remains were rapidly cooled down due to heavy rains during the night, while those burned in the second session were allowed to cool down naturally overnight. Bones were recovered on the top and within the ash piles on the day following the cremation experiments.

Pre-treatments and stable isotope analysis of materials used in the experiments

The metacarpals/metatarsals from unburnt pig feet were isolated by dissection, and the periosteum was removed with a scalpel. Bones were cleaned using a tungsten carbide drill bit to retain only the cortical part. Bones were then ground into powder using a countertop blender. Particles ranging between 0.3 and 0.7 mm were collected for collagen extraction, and those less than 0.3 mm were used for carbon and oxygen isotope analyses of bioapatite carbonates. Prior to the purification procedures, bone powder samples were defatted according to the protocol developed by Kates [75] and tested by Liden et al. [76], as presented in Salesse et al. [77]. Bone



Fig 1. Active burning of experimentally constructed pyres. Credits: Ahmad Alsaadi.

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collagen was extracted following the non-ultrafiltered procedure proposed by Brock et al. [78] while unburnt bone carbonate samples were prepared following a revised version of the protocol of Balasse et al. [79]. With regards to the calcined material, the outer layers of the bone fragments were scratched with a scalpel. The samples were prepared according to the procedure described by Snoeck et al. [80]. Between 4 and 7 different (calcined) bones per foot were sampled (amount: ca. 500 mg per sampled bone). Wood shavings, animal hide cuts and soft tissues of the pig (skin, muscle, and tendon) were sampled using a scalpel. They received no further treatment prior to isotopic analysis. All these pre-treatments were carried out at the AMGC Research Unit of the *Vrije Universiteit Brussel* (VUB, Brussels, Belgium). More information about the pre-treatments carried out is provided in Section 2 in [S1 Appendix](#).

Carbon and nitrogen abundances and isotope compositions for organic samples (amount: $\approx 500 \mu\text{g}$, in tin capsule) were measured in duplicate on different aliquots using a Eurovector Elemental Analyzer (EA) coupled with a Nu Perspective Isotope Ratio Mass Spectrometer (IRMS) at the AMGC Research Unit (S3.1, S3.2 and S4.1 Tables in [S3](#) and [S4](#) Appendices). The $\delta^{13}\text{C}_{\text{organic}}$ and $\delta^{15}\text{N}_{\text{organic}}$ values are reported as per mil (‰) difference relative to VPDB and AIR, respectively. Standards (IA-R041: $\delta^{13}\text{C} = -23.3\text{‰}$ and $\delta^{15}\text{N} = -5.6\text{‰}$; IAEA-C-6: $\delta^{13}\text{C} = -10.8\text{‰}$; IAEA-CH-7: $\delta^{13}\text{C} = -32.2\text{‰}$; IAEA-N-1: $\delta^{15}\text{N} = +0.4\text{‰}$; IAEA-N-2: $\delta^{15}\text{N} = +20.3\text{‰}$) were used for data correction. All samples and standards were analyzed randomly except IA-R041 that was used for instrumental drift correction. Analytical errors were better

than $\pm 0.2\%$ (1SD) for both $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$. Quality criteria for bone collagen–extraction yields (%Col), carbon and nitrogen contents (%C and %N), and atomic C:N ratios—are presented in S4.1 Table in [S4 Appendix](#).

Carbon and oxygen isotope compositions for unburnt and calcined bone samples (amount: ≈ 2 mg) were measured in duplicate on different aliquots via a NuCarb carbonate preparation device interfaced with a Nu Perspective IRMS (first batch) or via the NuCarb/IRMS system including a Nu GasPrep automatic gas sampler (second batch) at the AMGC Research Unit (S4.1 and S4.2 Tables in [S4 Appendix](#)). The results are reported as per mil (‰) deviation from VPDB reference standard scale. Two internal standards were used (ENF: $\delta^{13}\text{C} = -9.8\%$ and $\delta^{18}\text{O} = -5.4\%$; CBA: $\delta^{13}\text{C} = -14.8\%$ and $\delta^{18}\text{O} = -10.8\%$ —see de Winter et al. 2016), as well as Iso-Analytical IA-R022 ($\delta^{13}\text{C} = -28.6\%$ and $\delta^{18}\text{O} = -22.7\%$), and international standards IAEA-603 ($\delta^{13}\text{C} = 2.5\%$ and $\delta^{18}\text{O} = -2.3\%$), and IAEA-CO-8 ($\delta^{13}\text{C} = -5.8\%$ and $\delta^{18}\text{O} = -22.7\%$). Over the course of all analyses, the analytical precision was better than $\pm 0.25\%$ (1SD) for both $\delta^{13}\text{C}_{\text{carb}}$ and $\delta^{18}\text{O}_{\text{carb}}$ based on repeated measurements of CBA ($n = 26$). Δ is used to express isotopic differences between unshoed and shoed feet of the same pig (e.g. $\Delta^{13}\text{C}_{\text{shoed-unshoed}}$ or $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$).

Preparation and ATR-FTIR analysis of burnt bones

Only the pig bones from the second burning experiments were studied using infrared analyses. Sample preparation and infrared analysis of calcined bone were carried out according to Kontopoulos et al. [81] (see Section 2 in [S1 Appendix](#) for specifics). Bioapatite changes were assessed through FTIR spectroscopy using attenuated total reflection (ATR). Between 5 and 7 different bones per foot were investigated. ATR-FTIR measurements on bone samples (amount: 2 to 3 mg per sampled bone) were performed in triplicate under vacuum on different aliquots using a Bruker Vertex 70v FTIR spectrometer (range: $4000\text{--}400\text{ cm}^{-1}$; No. of scans: 64; resolution: 4 cm^{-1} ; mode: absorbance) at the AMGC Research Unit. A background measurement was run before each sample analysis to remove the background signal. After each measurement, the crystal plate and the anvil of the pressure applicator were cleaned using a dry Kimtech™ precision wipe. The Bruker OPUS software (v. 7.5) was used to determine the infrared indices (see Section 2 in [S1 Appendix](#) for the list of measured indices). Differences in infrared proxies between feet are expressed by using Δ (e.g. $\Delta\text{CN}/\text{P}_{\text{shoed-unshoed}}$; see S5.2 Table in [S5 Appendix](#)).

The Bruker Vertex 70v is a high performance spectrometer equipped with an evacuable optics bench, a RockSolid™ interferometer and DigiTect™ 24-bit ADC detectors. These features ensure improved sensitivity and spectral resolution as well as lower electronic noise and atmospheric interferences compared to conventional FTIR spectrometers. Because the detection threshold for cyanamide has been established on a conventional FTIR spectrometer operating at standard atmospheric pressure [57], an instrument intercomparison was performed for testing comparability of the CN/P values between studies. Samples initially measured by Snoeck et al. [57] using an Agilent Technologies Cary 640 (no vacuum) were rerun using the Bruker Vertex 70v (under vacuum) from the AMGC Research Unit.

Results and discussion

$\delta^{13}\text{C}_{\text{carb}}$ signals: The fuel effect

The $\delta^{13}\text{C}_{\text{carb}}$ values of pig foot bones shift drastically during the cremation process. While the unburnt bones have $\delta^{13}\text{C}_{\text{carb}}$ values ranging from -15.4 to -14.8% (S4.1 Table in [S4 Appendix](#)), the calcined bones (from all burning experiments) have $\delta^{13}\text{C}_{\text{carb}}$ values ranging from -29.8% to -17.8% (S4.2 Table in [S4 Appendix](#)). On average, the $\delta^{13}\text{C}_{\text{carb}}$ values of the unburnt bones

(mean = $-15.1 \pm 0.2\%$, 1SD) differ by -10.2% from the burnt bones (mean = $-25.3 \pm 1.9\%$, 1SD).

Heating experiments carried out in a fuel-free muffle furnace (pig 9) show that the $\delta^{13}\text{C}_{\text{carb}}$ values of calcined bones (mean_[unshoed] = $-20.6 \pm 0.5\%$, 1SD; mean_[shoed] = $-24.6 \pm 0.9\%$, 1SD; S4.2 Table in [S4 Appendix](#)) tend to decrease towards the $\delta^{13}\text{C}$ values of the pig skin, muscle, tendon and bone collagen (mean = $-23.1 \pm 0.8\%$, 1SD; S4.1 Table in [S4 Appendix](#)) and/or the leathers (mean = $-27.9 \pm 0.6\%$, 1SD; Section 3 in [S1 Appendix](#) and S3.1 Table in [S3 Appendix](#)) ([Fig 2](#)). This suggests that part of the carbon exchanges between bone apatite carbonates and combustion gases—especially CO₂—occurred during the burning of organic matter, including leathers. However, these bones exhibit less negative $\delta^{13}\text{C}_{\text{carb}}$ values than those cremated in outdoor experiments (pigs 1 to 8; mean = $-25.4 \pm 1.8\%$, 1SD; S4.2 Table in [S4 Appendix](#)).

When cremated on pyres, the unshoed feet yield $\delta^{13}\text{C}_{\text{carb}}$ values ranging from -28.8 to -22.5% (mean = $-25.5 \pm 1.5\%$, 1SD) while the shoed feet have $\delta^{13}\text{C}_{\text{carb}}$ values ranging from -29.8 to -17.8% (mean = $-25.4 \pm 2.1\%$, 1SD) ([Fig 2](#); [Table 1](#) and S4.2 Table in [S4 Appendix](#)). There is no significant difference in $\delta^{13}\text{C}_{\text{carb}}$ values between these two groups of feet (Mann-Whitney (MW) test, $p = 0.74$; S6.2 Table in [S6 Appendix](#)). This absence of difference is also observed within each pyre and between pyres (MW tests, $p \geq 0.06$; S6.3 Table in [S6 Appendix](#)) ([Fig 2](#)). Still, the larger variability in $\delta^{13}\text{C}_{\text{carb}}$ values seen in shoed feet ($\pm 2.1\%$, 1SD) compared to unshoed feet ($\pm 1.5\%$, 1SD) might be linked to the presence of more carbon sources and/or more variable burning conditions for the shoed feet because of the presence of footwear. The $\delta^{13}\text{C}_{\text{carb}}$ values of bones recovered above or below the ashes are similar (MW test, $p = 0.81$; S6.4 Table in [S6 Appendix](#)) ([Fig 2](#)). Therefore, neither the presence of footwear nor the initial position of the feet in the pyres nor the final location of the bones in the ashes seem to visibly affect the $\delta^{13}\text{C}_{\text{carb}}$ values of calcined bones.

This is most likely because wood is present in much larger amounts and, when burned, releases much larger quantities of CO₂ compared to any other material cremated simultaneously. Wood-derived carbon thus not only participates predominantly in the various chemical exchanges that occur in the cremation environment, but also largely influences the isotopic signature of the CO₂ present in the cremation atmosphere. Still, in none of the experiments carried out did the $\delta^{13}\text{C}_{\text{carb}}$ values of calcined bones perfectly match the $\delta^{13}\text{C}_{\text{org}}$ values of the pig soft tissues, the pig bone collagen, the leathers, or the firewood. This observation is consistent with the results of previous work showing that most but not all the carbon in the carbonate fraction of calcined bones is replaced by carbon from the fuel [[42](#), [47](#), [49](#)].

$\delta^{18}\text{O}_{\text{carb}}$ signals: The local heating conditions

During the burning process, the $\delta^{18}\text{O}_{\text{carb}}$ values of pig foot bones drop similarly to the $\delta^{13}\text{C}_{\text{carb}}$ values ([Fig 3](#)). While the raw bones have $\delta^{18}\text{O}_{\text{carb}}$ values ranging from -10.8 to -10.2% , (S4.1 Table in [S4 Appendix](#)), the calcined bones (from all burning experiments) have $\delta^{18}\text{O}_{\text{carb}}$ values ranging from -19.6% to -12.1% (S4.2 Table in [S4 Appendix](#)). On average, the $\delta^{18}\text{O}_{\text{carb}}$ values of the unburnt bones (mean = $-10.4 \pm 0.2\%$, 1SD) differ by -5.6% from the burnt bones (mean = $-16 \pm 1.6\%$, 1SD).

The unshoed and shoed feet from the laboratory burning experiments have $\delta^{18}\text{O}_{\text{carb}}$ values that differ by 1.3% on average. Such a pattern is also found in most of the outdoor burnings (Tables [1](#) and S3.2 Table in [S3 Appendix](#)). When cremated on pyres, the unshoed and shoed feet have $\delta^{18}\text{O}_{\text{carb}}$ values ranging from -18.6 to -12.1% (mean = $-15.6 \pm 1.3\%$, 1SD) and from -19.6 to -13% (mean = $-16.6 \pm 1.7\%$, 1SD), respectively ([Fig 3](#); Tables [1](#) and S3.2 Table in [S3 Appendix](#)). The difference in $\delta^{18}\text{O}_{\text{carb}}$ values between the two groups of feet is statistically significant (MW test, $p = 0.01$; S6.5 Table in [S6 Appendix](#)) ([Fig 3](#)). If this difference is examined

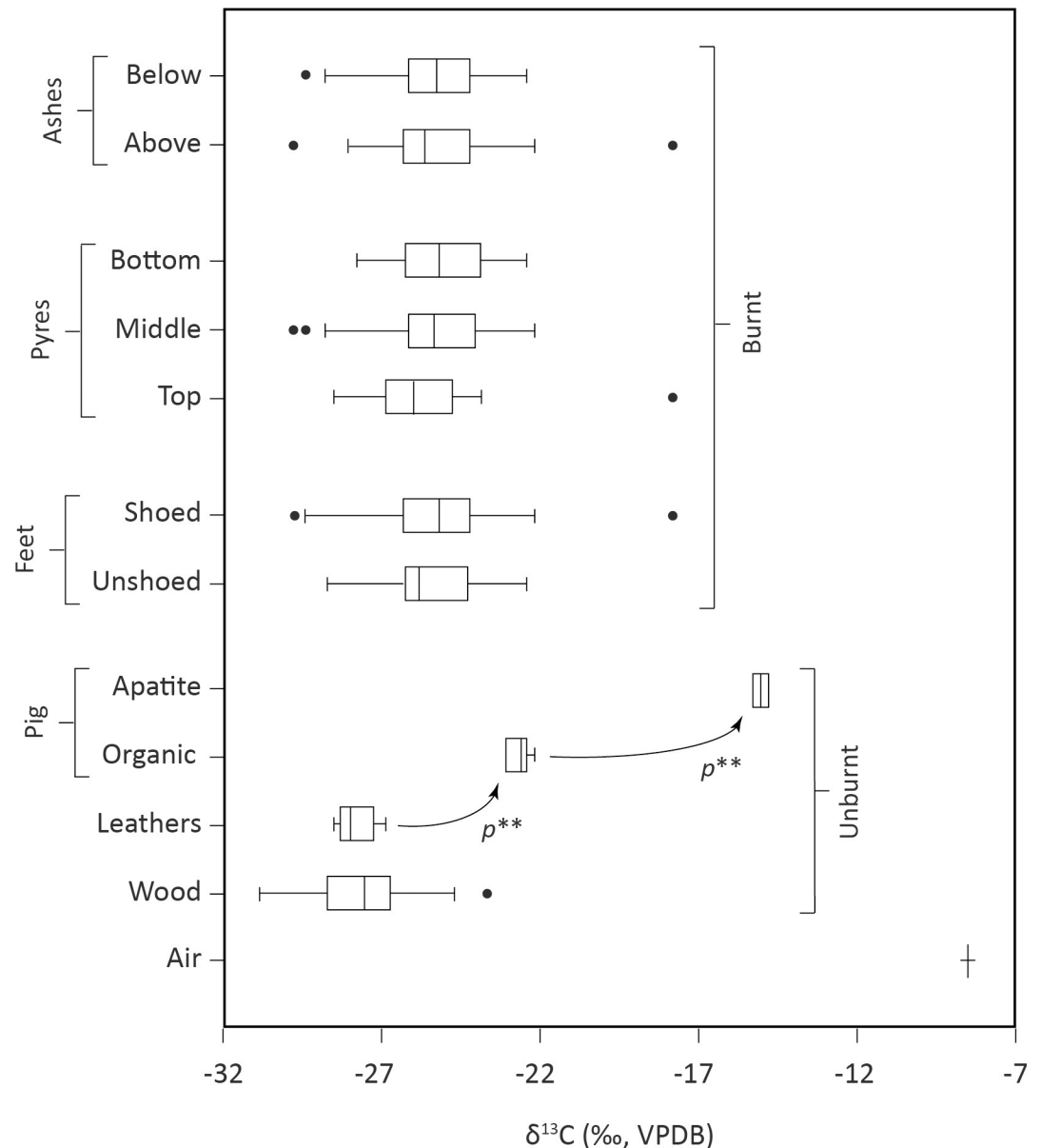


Fig 2. Boxplots presenting the distribution of the $\delta^{13}\text{C}$ values measured on the wood, leathers and pig tissues. p^{**} represents statistically significant differences (based on Mann-Whitney U tests).

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per pyre, the outcome is less straightforward. There is no difference in $\delta^{18}\text{O}_{\text{carb}}$ values between the feet placed on top or in the middle of the pyres (MW tests, $p \geq 0.18$; S6.6 Table in S6 Appendix) (Fig 3). In contrast, when deposited at the bottom of the structure, the $\delta^{18}\text{O}_{\text{carb}}$ values of the unshoed feet statistically significantly differ from the shoed feet by 2.5‰ (MW test, $p < 0.01$; S6.6 Table in S6 Appendix) (Fig 3). Overall, the burnt shoed feet are more depleted in ^{18}O than the burnt unshoed feet, demonstrating a certain isotopic effect of wearing closed leather footwear during cremation.

By forming a temporary protective layer around the foot, the shoe creates conditions that prolong the pyrolysis of the underlying pig tissues, while delaying the ignition and subsequent burning processes of the latter. Inside the shoe, pyrolysis-derived volatiles, gases and residues

Table 1. Mean $\delta^{13}\text{C}_{\text{carb}}$ and $\delta^{18}\text{O}_{\text{carb}}$ values and discrepancies between feet of experimentally cremated pigs.

Sample	Position	SHOED (‰ -VPDB)					UNSHOED (‰ -VPDB)					DIFFERENCES (‰ -VPDB)			
		n	$\delta^{13}\text{C}_{\text{carb}}$	SD	$\delta^{18}\text{O}_{\text{carb}}$	SD	n	$\delta^{13}\text{C}_{\text{carb}}$	SD	$\delta^{18}\text{O}_{\text{carb}}$	SD	$\Delta^{13}\text{C}_{\text{shoed-unshoed}}$	SD	$\Delta^{18}\text{O}_{\text{shoed-unshoed}}$	SD
Pig 1 / Pyre 1	Top	4	-26.6	1.9	-14.8	1.5	5	-26.0	1.5	-15.2	1.8	-0.6	0.9	0.4	0.8
Pig 2 / Pyre 2	Middle	5	-26.3	3.3	-16.5	1.4	5	-26.6	0.8	-15.1	0.5	0.3	0.9	-1.4	0.6
Pig 3 / Pyre 3*	Middle	4	-24.9	2.0	-15.3	0.6	5	-25.3	1.9	-14.3	1.4	0.4	0.9	-1.0	0.7
Pig 4 / Pyre 4	Bottom	5	-26.5	1.2	-18.3	1.7	4	-24.0	2.5	-15.9	1.7	-2.5	0.9	-2.4	0.7
Pig 5 / Pyre 5	Bottom	5	-25.5	0.9	-18.8	0.4	5	-24.8	1.1	-16.3	0.6	-0.7	0.6	-2.5	0.5
Pig 6 / Pyre 6	Middle	6	-24.7	1.1	-17.1	1.0	6	-25.8	0.7	-16.7	1.2	1.1	0.5	-0.4	0.6
Pig 7 / Pyre 7	Top	7	-24.7	3.3	-16.2	1.1	6	-25.9	0.8	-15.2	0.8	1.2	0.8	-1.0	0.5
Pig 8 / Pyre 8†	Middle	6	-24.5	0.9	-15.4	1.0	5	-25.1	2.2	-15.8	1.2	0.6	0.8	0.4	0.6
Pig 9 / Furnace	900°C / 5h	2	-24.6	0.9	-14.4	0.3	2	-20.6	0.5	-15.7	0.3	-4.0	0.8	1.3	0.5

Full data available in S4.2 Table in S4 Appendix.

*Pyre where the pig foot was placed in goatskin instead of cow leather.

†Dry farmyard manure pellets were added to this pyre.

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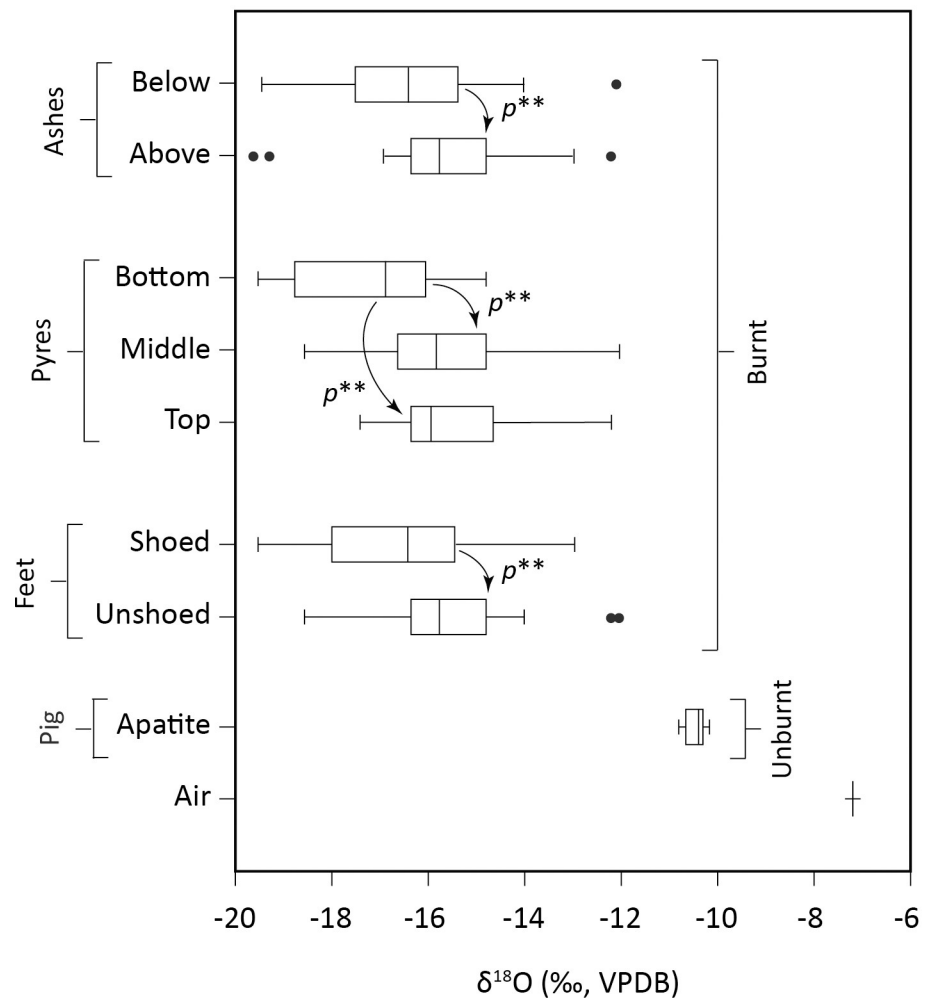


Fig 3. Boxplots presenting the distribution of the $\delta^{18}\text{O}$ values measured on the wood, leathers and pig tissues. p^{**} represents statistically significant differences (based on Mann-Whitney U tests).

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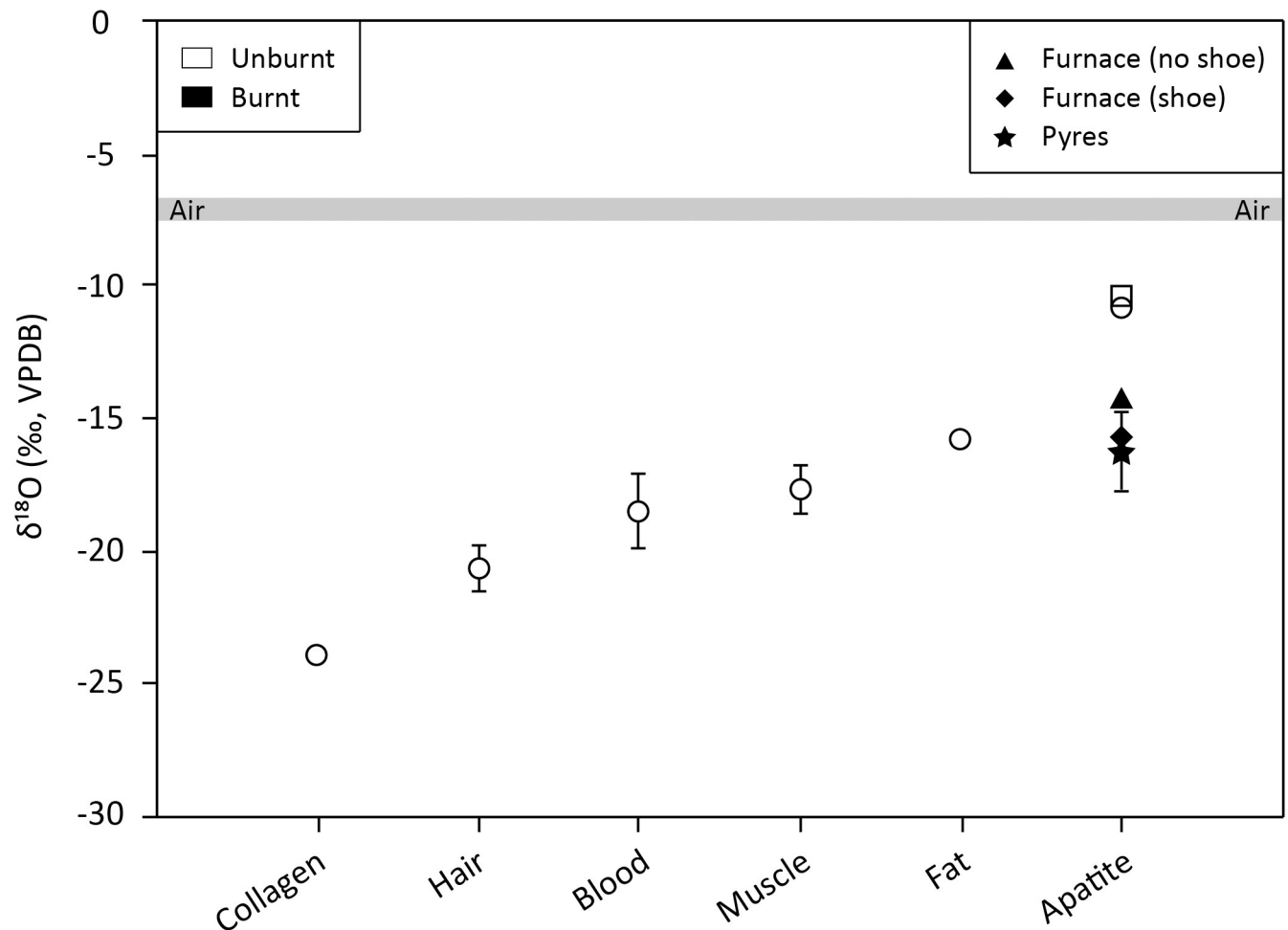


Fig 4. $\delta^{18}\text{O}$ values of pig tissues from this study (square, triangle, diamond, star) compared to the dataset from Tuross et al. [82] and Warinner and Tuross [83] (circle).

<https://doi.org/10.1371/journal.pone.0257199.g004>

would accumulate, setting up a specific local atmosphere, with $\delta^{18}\text{O}$ values distinct from those on the outside. The organic $\delta^{18}\text{O}$ values (hides/skins used for footwear) are expected to be lower than mineral $\delta^{18}\text{O}$ values. For pigs with similar $\delta^{18}\text{O}_{\text{carb}}$ values to those obtained in this study (-10/-11‰), Tuross et al. [82] as well as Warinner and Tuross [83] found that bone collagen (-23.9 ± 0.2‰), hair (-20.7 ± 0.9‰), blood (-18.5 ± 1.4‰), muscle (-17.7 ± 1‰) and fat (-15.8 ± 0.5‰), were depleted in ^{18}O compared to unburnt bone apatite (-10.9 ± 0.4‰) (Fig 4). Such differences between tissues are also recorded for other domesticated herbivores (e.g. [84, 85]). Based on the environment in which the cattle and goats used for leather production were reared, the hides/skins are expected to have $\delta^{18}\text{O}$ values in the range of those of the pig organic tissues.

However, oxygen exchanges between the inner shoe atmosphere and the bone apatite carbonates are thought to be minimal. If structural rearrangements or chemical changes in apatite have been reported at low temperatures, this generally concerns synthetic carbonated hydroxyapatites (e.g. AB-CO₃-Aps) or skeletal tissues containing a small quantity of organic matter (e.g. tooth enamel) [86, 87]. In bones, soft tissues and collagen matrix shield the inorganic fraction, delaying carbonates exposure to heat and consequently restricting the oxygen exchanges with the surrounding atmosphere [61–64]. Meanwhile in the pyre, wood is consumed under

the action of fire which is conditioned by the thermal degradation properties of the wood compounds: i.e. holocellulose and lignin. Holocellulose starts to burn first, followed by lignin. Initially largely influenced by holocellulose, the combustion-derived oxygen pool is then mainly influenced by lignin as burning progresses [88]. These compounds contain both very distinct oxygen isotopic compositions, with (holo-)cellulose being significantly enriched in ^{18}O compared to lignin (up to 11‰) [89–92]. The bones from the shoed foot are apparently exposed to fire at a more advanced stage of burning and exchange oxygen with a relatively more ^{18}O -depleted atmosphere than bones from the unshoed foot; the leather shoe acting as an additional protective organic layer to the inorganic fraction of the bones.

Regardless of footwear, feet put on top or in the middle of the pyres present $\delta^{18}\text{O}_{\text{carb}}$ values that statistically significantly differ from those of feet laid out at the bottom of the structure ($\Delta^{18}\text{O}_{\text{top/middle-bottom}} = 1.7\text{‰}$; MW test, $p < 0.01$; S6.7 Table in S6 Appendix). Feet deposited at the bottom of the pyres exhibit the lowest mean $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$ values (below -2.4‰ ; pyres 4 and 5), while those placed on top present the highest mean $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$ value ($+0.4\text{‰}$; pyre 1) (Fig 5; Table 1). The observed decrease in $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$ values from the top to the bottom of the pyres suggests that local variations in heating conditions occur. The fires of

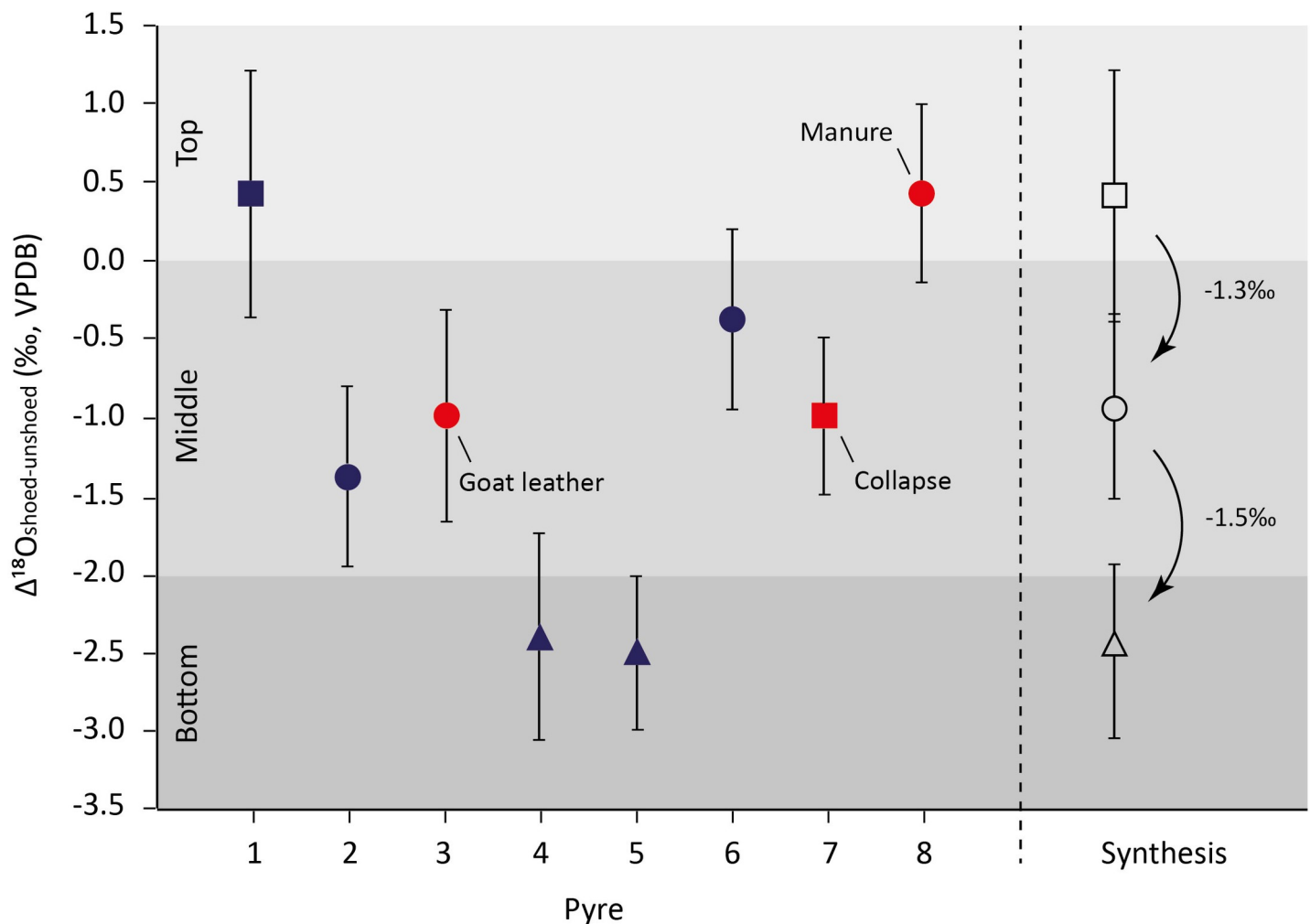


Fig 5. $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$ values (\pm 1SD) of the pigs burned in the experimental cremations. Squares refer to top-position pyres, circles for middle-position pyres, and triangles for bottom-position pyres. The synthesis excludes pyre 7 and 8.

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open-air cremations burn heterogeneously and samples are exposed to varying temperatures and changing atmospheres [93]. Thermal variations could have been larger and exposure times longer at the bottom than on top of the pyres, resulting in distinct exchanges and/or isotope fractionations of oxygen. In addition, the oxygen availability probably changed drastically within the wooden structures. The oxygen supply, the accumulation and removal of combustion by-products, and the interaction with the various gases were probably different in the heart or deep down in the pyres than closer to the surface. Moreover, the diffusion of oxygen could have been more complex or slower inside the pyre than closer to the surface, resulting in different isotopic fractionations and/or exchanges.

Furthermore, the shoes appear to have been subjected to different physical constraints depending on their position in the pyres. The shoes within the pyres stayed closed longer, probably because of the pressure from the firewood, while the shoe placed on top of pyre 1 opened quickly, exposing the foot to the same burning regime than the unshoed foot. This would explain the small but positive difference observed between shoed and unshoed feet recorded for pyre 1 ($\Delta^{18}\text{O}_{\text{shoed-unshoed}} = +0.4\text{‰}$). It can also be observed that the mean $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$ values decrease by -1.4‰ in average between each position, from top to bottom (Fig 5). This clearly evidences a concomitant influence of the initial position of the feet in the pyres and the wearing of footwear. It also highlights that, regardless the type of leather used, a comparable isotopic effect is noted for the shoed feet placed in the middle of the pyres.

Comparing the $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$ results obtained in each burning session, they are strikingly similar, especially for the bones burned at the bottom of the pyre (pyres 4 and 5 –Fig 3; Table 1) suggesting that, at the bottom of a pyre, conditions are quite similar regardless of the location of the pyre within the landscape (enclosed area in session 1 vs open hill in session 2). The bones burned in the middle position also show similar $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$ values but the slight difference between pyre 2 (cow leather) and 3 (goat leather) could be explained by the difference in thickness between the two leathers, the latter being thinner and thus burning away faster. The difference between pyres 2 and 6, where pyre 6 has a slightly less negative mean $\Delta^{18}\text{O}_{\text{shoed-unshoed}}$ value (-1.4‰ and -0.4‰ respectively) could be the results of a difference in openness of the cremation area where an open area could create a more homogeneous combustion atmosphere in the middle area of the pyre.

The results of pyres 7 and 8 further show the impact of fuel and position on the pyre on the oxygen isotope results. While the unshoed foot from pyre 7 has an identical mean $\delta^{18}\text{O}_{\text{carb}}$ value to that of pyre 1 (top), the shoed foot from pyre 7 presents a similar mean $\delta^{18}\text{O}_{\text{carb}}$ value than those of the shoed feet from pyres 2, 3 and 6 (middle). This is most likely related to the fact that pyre 7 toppled chaotically, subjecting the shoed foot to a different burning regime. When the pyre collapsed, a few pieces of burning wood covered the shoed foot, recreating to some extent an atmosphere comparable to that experienced by the feet placed in the middle of the pyres.

With regards to pyre 8, it can be observed that the shoed foot has a slightly higher mean $\delta^{18}\text{O}_{\text{carb}}$ value than the unshoed foot. Manure has a similar decomposition pattern to lignin [94], and, in general, fertilizers appear to have relatively high $\delta^{18}\text{O}$ values [95]. Assuming that the ^{18}O -enriched manure could mitigate the influence of the ^{18}O -depleted lignin and create a more homogenous combustion atmosphere throughout the cremation process in comparison to the other pyres where cellulose is expected to burn away faster than lignin (i.e. creating a change in combustion atmosphere through time), the shoed and unshoed feet from pyre 8 could have been burned in a more comparable atmosphere.

Whether they were initially encased in shoes or not, the $\delta^{18}\text{O}_{\text{carb}}$ values of bones (from all pyres) recovered below the ashes differ from those of bones retrieved from above the ashes by -0.9‰ (MW test, $p = 0.01$; S6.8 Table in S6 Appendix) (Fig 3). This indicates that the presence

of an ash layer covering foot elements have likely restricted the oxygen exchanges between bone apatite carbonates and the combustion atmosphere during and after the collapse of the pyre, or conversely, that the absence of such a layer have likely promoted oxygen exchanges between bone apatite carbonates and the atmospheric CO₂ at the end of the cremation process. Charcoal generally presents lower $\delta^{18}\text{O}$ values than the wood from which it originates (e.g. by about -9‰ for beech according to Schumacher et al. [94]). This suggests that below the ashes, a severely ¹⁸O-depleted atmosphere prevails, and that carbon and oxygen exchanges can still occur, which is not totally unsurprising as some of the ash layers were still hot (> 400°C) the next morning. The location below the ashes would therefore be a constraining condition that may promote the influence of the char-derived oxygen to the $\delta^{18}\text{O}_{\text{carb}}$ signal. Interestingly, the final position of bones in ashes in addition to the presence of shoes and the initial position of the feet in the pyre seem to have a cumulative effect on the $\delta^{18}\text{O}_{\text{carb}}$ values, with the greater difference in $\delta^{18}\text{O}_{\text{carb}}$ values found between 1) the group of bones recovered above ashes but deriving from unshoed feet placed on top of the pyres, and 2) the group of bones recovered below ashes but deriving from shoed feet initially deposited at the bottom of the pyres (i.e. 3.7‰). Especially, this latter group contains the most negative $\delta^{18}\text{O}_{\text{carb}}$ values from the whole dataset.

The cyanamide content: The initial position in the pyre and the presence of footwear

The CN/P values derived from Agilent Technologies Cary 640 and Bruker Vertex 70v spectrometers are shown to be well correlated ($y = 0.0944x - 0.0025$; $R^2 = 0.88$) (S7.1 Table in [S7 Appendix](#)). Due to its ability to work under vacuum, the Bruker Vertex 70v almost eliminates atmospheric disturbances, resulting in a decrease of the signal-to-noise ratio and thus in the measured cyanamide contents according to a ratio of about 13:1. A significance threshold of 0.02 for detecting the presence of cyanamide is proposed based on the instrument intercomparison carried out in this study and the work of Snoeck et al. [57].

The amount of cyanamide in calcined bone is relatively higher in shoed feet (mean = 0.042 ± 0.024 , 1SD) than in unshoed ones (mean = 0.010 ± 0.010 , 1SD) at the bottom of the pyre (pig 5) (MW test, $p = 0.03$; S6.9 Table in [S6 Appendix](#)), while a reverse pattern is observed between the shoed (mean = 0.010 ± 0.007 , 1SD) and unshoed feet (mean = 0.044 ± 0.032 , 1SD) located on top (pig 7) (MW test, $p = 0.02$; S6.9 Table in [S6 Appendix](#)) (S5.1 Table in [S5 Appendix](#) and see the other FTIR results for these feet in Section 4 in [S1 Appendix](#)). Overall, a slight top-to-bottom decrease in the mean $\Delta\text{CN/P}_{\text{shoed-unshoed}}$ values is observed (S5.2 Table in [S5 Appendix](#)). A joint effect of the initial position of the feet in the pyre and the presence of closed leather footwear seems to influence the CN/P values of the pig feet.

It is possible that leather contains low to moderate ammonia concentrations. Indeed, in leather processing, dehairing, deliming and/or softening are generally carried out with ammonium salts or urine derivatives, generating large amounts of ammonia [95–97]. The more reducing conditions surrounding the shoed feet, in conjunction with a higher presence of ammonia released during the combustion of the shoe, have likely promoted the incorporation of cyanamide into the apatite structure. Since the unshoed feet placed on the same deposition levels do not have cyanamide or have relatively low levels of cyanamide, it can be hypothesized that either the presence of ammonia in the combustion atmosphere was localized or reducing conditions were not present around the unshoed feet; one not excluding the other.

The OH/P and CN/P values of pigs 5 to 8 display a weak inverse correlation ($R^2 = 0.23$) (Fig 6; S5.1 Table in [S5 Appendix](#)). This is caused by the shoed foot located on top of pyre 7. It

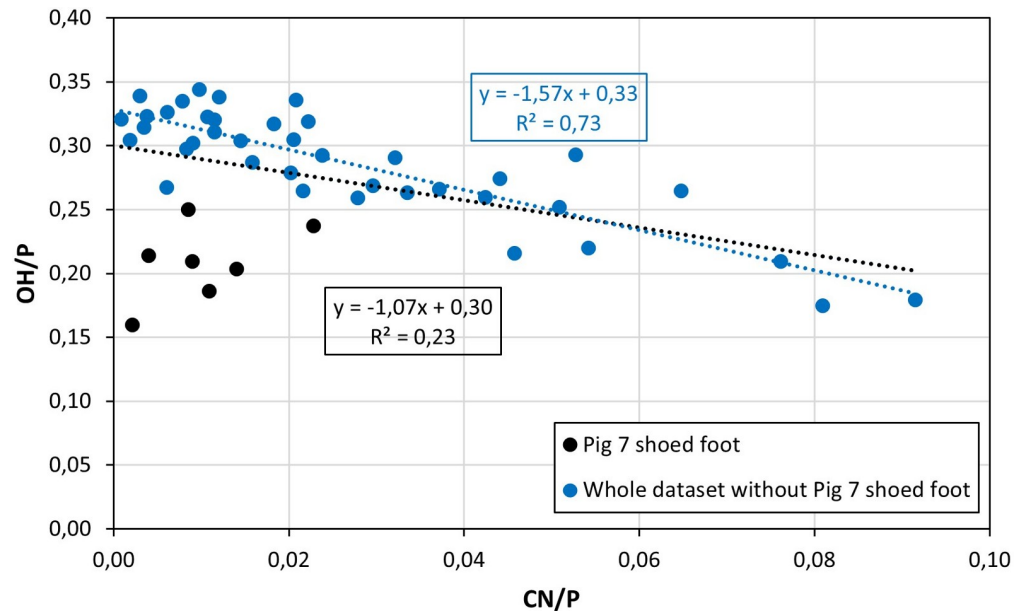


Fig 6. Biplot presenting the CN/P values against the OH/P values for pigs 5 to 8.

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presents the lowest mean OH/P value of the corpus (0.21), which likely is related to the collapse of pyre 7 and to the fall of the shoed foot out of the pyre. When this foot is excluded, the inverse correlation improves ($R^2 = 0.74$) (Fig 6). Such a strong relationship confirms that cyanamide substitutes for hydroxyl groups within the bone matrices [57, 66]. However, there is no plausible explanation for the presence of cyanamide in the unshoed foot placed on top of pyre 7, although it is certainly related to the collapse of the structure and/or could be linked to the other inconsistent infrared indices obtained for this foot (see Section 4 in S1 Appendix and S5.1 Table in S5 Appendix). Furthermore, there is no statistically significant difference between the CN/P values of the feet deposited in the middle of pyres 6 and 8, with or without shoes (MW tests, $p \geq 0.11$; S6.10 Table in S6 Appendix). Therefore, either the manure used in pyre 8 did not induce the expected ammonium-rich atmosphere or failed to produce cyanamide and its incorporation into the heated bones. Another explanation could be that the potentially reductive conditions necessary for the incorporation of cyanamide were not met at the time the manure was burned. Carrying out this experiment again but in bottom position should refine this interpretation. Finally, the position of bones in ashes does not influence the CN/P values (MW test, $p = 0.99$; S6.11 Table in S6 Appendix).

Implications for archaeological cremations and future research

This study is a step forward to a more thorough comprehension of the cremation practices and rituals. The results obtained show that the initial positioning of feet on or within the pyre and the wearing of closed leather footwears may moderately-to-highly influence the oxygen isotope ratios of bone apatite carbonates and the cyanamide content of calcined bone. However, these effects should not be viewed as definitive or strict offsets, but rather as relative trends or orders of magnitude that can be expected when foot bones are cremated under similar conditions.

Because a multitude of cremation scenarios is technically possible, the number of parameters—controlled or not—coming into play is almost infinite. This is what makes the study of cremations challenging. However, previous work and ongoing research have shown that the oxygen isotope ratios of apatite carbonates in bone and the cyanamide content of calcined

bone can vary locally (intra-site), regionally (inter-site), and chronologically (within and across time periods) [50, 54, 98]. The present study demonstrates that the initial positioning of feet, and thereby of the individual, as well as the wearing of closed leather shoes, and by extrapolation of clothing items, are plausible parameters that can explain these geographical and chronological variations. However, many other factors, that are not yet well circumscribed or understood, may also affect the oxygen isotope ratios of bone apatite carbonates and the cyanamide content of calcined bone. Among the extrinsic factors are all the actions of the living towards the body and the funeral pyre such as dressing and shoeing (with open or closed footwear) the deceased, tending and stirring the fire, reloading the fire with wood, placing offerings and grave goods. These extrinsic factors are influenced by practices and beliefs of the living and but also by the skills and know-how of the cremation operators (e.g. [35, 99]). Intrinsic factors include the type of materials used to build the pyre, the heating duration, the way fire is quenched, the body size of the deceased, the pyre settings, etc. All these parameters can theoretically lead to isotopic, structural and chemical changes similar to those observed. However, we believe that by sufficiently refining the sampling strategy and selecting the most relevant bones to answer specific archaeological questions, biomolecular archaeology can be of great interest and help. An important aspect of this work is that a single isotopic or infrared measurement per bone is likely to bias the interpretations that can be drawn from it. Although time consuming, only repetitive measurements per bone and per anatomical region studied can provide a reliable signal that can be exploited for archaeological purposes.

The replication of this study on forensic anthropological material is envisaged to definitively corroborate the results obtained on modern animals. It is very likely that the two feet of the same individual burned on an open-air pyre were treated in an identical manner, i.e. either shoed or unshoed. It can be hypothesized that foot bones enclosed in a closed leather shoe will have different isotopic and infrared values than leg bones from the same individual. Even if clothed, the leg will certainly not be in a garment as fire resistant as the leather of a shoe. The shield effect of any clothing relative to the leg will therefore be less than for a foot and this should be reflected in the results obtained. In addition, complementary data from well-defined archaeological contexts e.g. Pompeian necropolises [20]) could help us better understand the observed variations and their potential meanings. Overall, this work and its multi-proxy approach / multi-sampling strategy represents a milestone with the potential to lead to a major breakthrough in cremation archaeology, and more broadly in archaeological science.

Conclusion

This study demonstrates that from calcined foot bones it is possible to detect whether a deceased was wearing closed leather shoes during cremation and to identify the initial positioning of the feet, and thereby of the individual, on/in a pyre. By forming an additional protective layer around the foot, the footwear appears to temporarily delay the burning of the underlying pig tissues and, simultaneously, increase the heat-shielding effects of organic matter protecting the mineral fraction of bone. Bone apatite seems to be then exposed to fire at a more advanced stage of combustion of the pyre, during which carbonates exchange oxygen with a relatively more ^{18}O -depleted atmosphere (due to the influence of lignin-derived oxygen). This results in more pronounced—and negative—shifts in the $\delta^{18}\text{O}_{\text{carb}}$ values of shoed feet than feet burned bare (up to 2.5‰). Such an isotopic pattern is not recognized when the feet are placed on top of the pyre, probably due to the lack of physical constraint (pressure/weight of the logs above) to maintain the shoes closed, although weaknesses in the shoe patterns cannot be ruled out. Further research and experimental burnings need to be conducted to better understand the processes that occur on top of the pyres.

In addition, a concomitant isotopic effect of the initial location of the feet in the pyres is observed, resulting in a decrease in the $\delta^{18}\text{O}_{\text{carb}}$ values of shoed feet by about 1.4‰ between each deposition level from the top to the bottom of the pyre. Another isotopic effect of approximately 0.9‰ is identified based on the relative position of the bones in relation to the ash heaps at the end of cremation. Bones recovered within the piles of ashes display lower values of $\delta^{18}\text{O}_{\text{carb}}$ than bones recovered above the pile, probably due either to a decrease in oxygen intake into the bone apatite or to privileged oxygen exchanges with charcoal that are depleted in ^{18}O . Finally, the presence of cyanamide ($\text{CN/P} \geq 0.02$) is likely indicative of the wearing of unventilated footwear since the latter creates favorable conditions for its incorporation into the bones (reducing atmosphere and a greater amount of organic matter).

From an archaeological perspective, ritual sequences related to cremation practices are usually well identified, but generally poorly understood with regard to the organization of the burning act. This study opens new avenues to obtain additional information about the treatment of the deceased during cremation and the cremation operator's range of skills. However, it is only when confronted with archaeological sources and field data that this approach yields its full potential. In the case of the presence of shoe nails in cremation-related deposits, it becomes possible to discuss if closed shoes were worn by the deceased or only placed next to the body on the pyre, which are different funerary gestures with possible different cultural or ritual meanings. Based on a multi-sampling strategy of the skeleton, and considering anthropological and anthracological data, it could be inferred if the pyre was left unattended during cremation or managed by operators. These are just a few examples of the promising prospects offered by isotopic and infrared evidence obtained from calcined bones.

Supporting information

S1 Appendix. Supporting text and figures for materials, methods, results and discussion sections.

(DOCX)

S2 Appendix. Heating temperatures in pyres.

(XLSX)

S3 Appendix. Isotopic results ($\delta^{13}\text{C}_{\text{org}}$ and $\delta^{15}\text{N}_{\text{org}}$) for unburnt leather and wood.

(XLSX)

S4 Appendix. Isotopic results ($\delta^{13}\text{C}_{\text{org}}$, $\delta^{15}\text{N}_{\text{org}}$, $\delta^{13}\text{C}_{\text{carb}}$ and $\delta^{18}\text{O}_{\text{carb}}$) for unburnt and burnt pig tissues (bone, muscle, skin and tendon).

(XLSX)

S5 Appendix. Infrared results for burnt pig bones (shoed and unshoed).

(XLSX)

S6 Appendix. Results from Mann-Whitney U tests.

(XLSX)

S7 Appendix. CN/P results from the instrument intercomparison.

(XLSX)

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Author Contributions

Conceptualization: Kevin Salesse, Christophe Snoeck.

Data curation: Kevin Salesse, Christophe Snoeck.

Formal analysis: Kevin Salesse, Elisavet Stamataki, Ioannis Kontopoulos, Christophe Snoeck.

Funding acquisition: Mathieu Boudin, Guy de Mulder, Martine Vercauteren, Christophe Snoeck.

Investigation: Kevin Salesse, Christophe Snoeck.

Methodology: Kevin Salesse, Christophe Snoeck.

Project administration: Martine Vercauteren, Christophe Snoeck.

Resources: Kevin Salesse.

Supervision: Martine Vercauteren.

Validation: Kevin Salesse, Christophe Snoeck.

Visualization: Kevin Salesse.

Writing – original draft: Kevin Salesse, Christophe Snoeck.

Writing – review & editing: Kevin Salesse, Elisavet Stamataki, Ioannis Kontopoulos, Georges Verly, Rica Annaert, Mathieu Boudin, Giacomo Capuzzo, Philippe Claeys, Sarah Dalle, Marta Hlad, Guy de Mulder, Charlotte Sabaux, Amanda Sengeløv, Barbara Veselka, Eugène Warmenbol, Martine Vercauteren, Christophe Snoeck.

References

1. Bowler JM, Jones R, Allen H, Thorne AG. Pleistocene human remains from Australia: A living site and human cremation from Lake Mungo, western New South Wales. *World Archaeology*. 1970; 2(1):39–60. <https://doi.org/10.1080/00438243.1970.9979463> PMID: 16468208
2. Walthall JA. Mortuary Behavior and Early Holocene Land Use in the North American Midcontinent. *North American Archaeologist*. 1999; 20(1):1–30. <https://doi.org/10.2190/MDEP-VM2K-YB2Q-L1D0>
3. Fengming L. Cremation process: China. In: Davies D, editor. *Encyclopaedia of cremation*. London: Ashgate; 2005. p. 132–5.
4. Scott RM, Buckley HR, Spriggs M, Valentin F, Bedford S. Identification of the first reported Lapita cremation in the Pacific Islands using archaeological, forensic and contemporary burning evidence. *Journal of Archaeological Science*. 2010; 37(5):901–9. <https://doi.org/10.1016/j.jas.2009.11.020>.
5. Potter BA, Irish JD, Reuther JD, Gelvin-Reymiller C, Holliday VT. A Terminal Pleistocene Child Cremation and Residential Structure from Eastern Beringia. *Science*. 2011; 331(6020):1058. <https://doi.org/10.1126/science.1201581> PMID: 21350175
6. Gil-Drozdz A. The Origins of Cremation in Europe. *Analecta Archaeologica Ressoiviensia*. 2011; 5:9–94.
7. Gray Jones A. Cremation and the Use of Fire in Mesolithic Mortuary Practices in North-West Europe. In: Cerezo-Román J, Wessman A, Williams H, editors. *Cremation and the Archaeology of Death*. Oxford: Oxford University Press; 2017. p. 27–51.
8. Capuzzo G, Snoeck C, Boudin M, Dalle S, Annaert R, Hlad M, et al. Cremation vs. inhumation: modeling cultural changes in funerary practices from the Mesolithic to the Middle Ages in Belgium using Kernel Density Analysis on ^{14}C data. *Radiocarbon*. 2020; 62(6):1809–32. <https://doi.org/10.1017/RDC.2020.88>

9. Williams H. Death Warmed up: The Agency of Bodies and Bones in Early Anglo-Saxon Cremation Rites. *Journal of Material Culture*. 2004; 9(3):263–91. <https://doi.org/10.1177/1359183504046894>
10. Oestigaard T. Cremations in Culture and Cosmology In: Tarlow S, Nilsson Stutz L, editors. *The Oxford Handbook of the Archaeology of Death and Burial*. Oxford: Oxford University Press; 2013. p. 497–510.
11. Gonçalves D, Pires AE. Cremation under fire: a review of bioarchaeological approaches from 1995 to 2015. *Archaeological and Anthropological Sciences*. 2017; 9(8):1677–88. <https://doi.org/10.1007/s12520-016-0333-0>.
12. Rebay-Salisbury K. Cremations: fragmented bodies in the Bronze and Iron Ages. In: Rebay-Salisbury K, Stig Sørensen ML, Hughes J, editors. *Body parts and bodies whole: changing relations and meanings*. Oxford: Oxbow; 2010. p. 64–71.
13. McKinley JI. Bone Fragment Size in British Cremation Burials and its Implications for Pyre Technology and Ritual. *Journal of Archaeological Science*. 1994; 21(3):339–42. <https://doi.org/10.1006/jasc.1994.1033>.
14. McKinley JI. Bone fragment size and weights of bone from modern British cremations and the implications for the interpretation of archaeological cremations. *International Journal of Osteoarchaeology*. 1993; 3(4):283–7. <https://doi.org/10.1002/oa.1390030406>
15. Williams H. Ephemeral Monuments and Social Memory in Early Roman Britain. *Theoretical Roman Archaeology Journal* (2013). 2004:51–61.
16. McKinley JI. Cremations: excavation, analysis, and interpretation of material from cremation-related contexts. In: Tarlow S, Nilsson Stutz L, editors. *The Oxford Handbook of the Archaeology of Death and Burial*. Oxford: Oxford University Press; 2013. p. 147–72.
17. Fülöp K. Why is it so rare and random to find pyre sites? Two cremation experiments to understand the characteristics of pyre sites and their investigational possibilities. *Papers from the Institute of Archaeological Sciences*. 2018; 3(6):287–311.
18. Cerezo-Román JI. Pathways to Personhood Cremation as a Social Practice Among the Tucson Basin Hohokam. In: Kuijt I, Quinn CP, Cooney G, editors. *Transformation by Fire. The Archaeology of Cremation in Cultural Context*. Tucson: University of Arizona Press; 2014. p. 148–67. <https://doi.org/10.1016/j.forsciint.2013.12.044> PMID: 24485301
19. Pearce J. From Death to Deposition: The Sequence of Ritual in Cremation Burials of the Roman Period. *Theoretical Roman Archaeology Journal* (1997). 1998:99–111.
20. Van Andringa W, Duday H, Lepetz S, Joly D, Lind T. Mourir à Pompéi. Fouille d'un quartier funéraire de la nécropole romaine de Porta Nocera (2003–2007). Rome: École Française de Rome; 2013. 1014 p.
21. McKinley JI. Complexities of the Ancient Mortuary Rite of Cremation: An Osteoarchaeological Conundrum. In: Grupe G, McGlynn GC, editors. *Isotopic Landscapes in Bioarchaeology*. Berlin, Heidelberg: Springer Berlin Heidelberg; 2016. p. 17–41.
22. Duday H, Van Andringa W. Archaeology of Memory: About the Forms and the Time of Memory in a Necropolis of Pompeii. *Memoirs of the American Academy in Rome Supplementary Volumes*. 2017; 13:73–85.
23. Parry JP. *Death in Banaras*. Cambridge: Cambridge University Press; 1994.
24. Wild JP. The textile archaeology of Roman burials: eyes wide shut. In: Carroll M, Wild JP, editors. *Dressing the Dead in Classical Antiquity*. Gloucestershire: Amberley; 2012. p. 17–25.
25. McKinley JI. Archaeology of Britain. In: Davies DJ, Mates LH, editors. *Encyclopedia of Cremation*. Aldershot: Ashgate; 2005. p. 9–15.
26. McKinley JI. Cremation. . . the cheap option? In: Gowland R, Knüsel C, editors. *The Social Archaeology of Funerary Remains*. Oxford: Oxbow Books; 2006. p. 81–8.
27. Williams H. Ethnographies for early Anglo-Saxon cremation. In: Riddler I, Keys L, Soulat J, editors. *Le témoignage de la culture matérielle: mélanges offerts au Professeur Vera Evison*. Autun: Éditions Mergoil; 2016. p. 139–54.
28. Goodenough WH. Rethinking 'Status' and 'Role': Toward a General Model of the Cultural Organization of Social Relationships. In: Banton M, editor. *The Relevance of Models in Social Anthropology*. London: Tavistock Publications; 1965. p. 1–24.
29. Duday H. *The archaeology of the dead: lectures in archaeoethanatology*. Oxford: Oxbow Books; 2009. 230 p.
30. Mohamad Z. I Am What I Wear: The Dialectics Of Dress And Individuality. *Ideology*. 2018; 3(1):109–13.
31. Rose GM, Shoham A, Kahle LR, Batra R. Social Values, Conformity, and Dress. *Journal of Applied Social Psychology*. 1994; 24(17):1501–19. <https://doi.org/10.1111/j.1559-1816.1994.tb01560.x>.
32. Roach-Higgins ME, Eicher JB. Dress and Identity. *Clothing and Textiles Research Journal*. 1992; 10(4):1–8. <https://doi.org/10.1177/0887302x9201000401>

33. Interlandi P. [A]dressing death: Fashioning garments for the grave. Melbourne: RMIT University; 2013.
34. Newholm T, Hopkinson GC. I just tend to wear what I like: contemporary consumption and the paradoxical construction of individuality. *Marketing Theory*. 2009; 9(4):439–62. <https://doi.org/10.1177/1470593109346896>
35. Oestigaard T. The deceased's lifecycle rituals in Nepal. Present Cremation burials for the interpretations of the past. Oxford: British Archaeological Reports; 2000. 76 p.
36. Wolska B. Applying isotope analyses of cremated human bones in archaeological research—a review. *Analecta Archaeologica Ressoiviensia*. 2020; 15:7–16.
37. Lanting JN, Brindley AL. Dating Cremated Bone: The Dawn of a New Era. *The Journal of Irish Archaeology*. 1998; 9:1–7.
38. Lanting JN, Aerts-Bijma AT, van der Plicht J. Dating of cremated bones. *Radiocarbon*. 2001; 43(2A):249–54. WOS:000174444000018.
39. Olsen J, Heinemeier J, Bennike P, Krause C, Margrethe Hornstrup K, Thrane H. Characterisation and blind testing of radiocarbon dating of cremated bone. *Journal of Archaeological Science*. 2008; 35(3):791–800. <https://doi.org/10.1016/j.jas.2007.06.011>.
40. Olsen J, Heinemeier J, Hornstrup KM, Bennike P, Thrane H. 'Old wood' effect in radiocarbon dating of prehistoric cremated bones? *Journal of Archaeological Science*. 2013; 40(1):30–4. <https://doi.org/10.1016/j.jas.2012.05.034>.
41. Zazzo A, Saliège JF, Person A, Boucher H. Radiocarbon dating of calcined bones: where does the carbon come from? *Radiocarbon*. 2009; 51(2):601–11. WOS:000270530800019.
42. Zazzo A, Saliège J-F, Lebon M, Lepetz S, Moreau C. Radiocarbon dating of calcined bones: insights from combustion experiments under natural conditions. *Radiocarbon*. 2012; 54(3–4):855–66.
43. Zazzo A, Lebon M, Chiotti L, Comby C, Delqué-Količ E, Nespolet R, et al. Can we Use Calcined Bones for 14C Dating the Paleolithic? *Radiocarbon*. 2013; 55(3):1409–21. Epub 02/09. <https://doi.org/10.1017/S0033822200048347>
44. van Strydonck M, Boudin M, Hoefkens M, De Mulder G. 14C-dating of cremated bones, why does it work? *Lunula*. 2005; 13:3–10.
45. van Strydonck MV, Boudin M, Mulder GD. The Carbon Origin of Structural Carbonate in Bone Apatite of Cremated Bones. *Radiocarbon*. 2010; 52(2):578–86. Epub 07/18. <https://doi.org/10.1017/S0033822200045616>
46. van Strydonck M, Decq L, den Brande TV, Boudin M, Ramis D, Borms H, et al. The Protohistoric 'Quicklime Burials' from the Balearic Islands: Cremation or Inhumation. *International Journal of Osteoarchaeology*. 2015; 25(4):392–400. <https://doi.org/10.1002/oa.2307>
47. Hüls CM, Erlenkeuser H, Nadeau MJ, Grootes PM, Andersen N. Experimental study on the origin of cremated bone apatite carbon. *Radiocarbon*. 2010; 52(2):587–99. WOS:000285437800042.
48. Harbeck M, Schleuder R, Schneider J, Wiechmann I, Schmahl WW, Grupe G. Research potential and limitations of trace analyses of cremated remains. *Forensic Science International*. 2011; 204(1–3):191–200. <https://doi.org/10.1016/j.forsciint.2010.06.004> PMID: 20609539
49. Snoeck C, Brock F, Schulting R. Carbon Exchanges between Bone Apatite and Fuels during Cremation: Impact on Radiocarbon Dates. *Radiocarbon*. 2014; 56(2):591–602.
50. Snoeck C, Schulting RJ, Lee-Thorp JA, Lebon M, Zazzo A. Impact of heating conditions on the carbon and oxygen isotope composition of calcined bone. *Journal of Archaeological Science*. 2016; 65:32–43. <http://dx.doi.org/10.1016/j.jas.2015.10.013>.
51. Lindars ES, Grimes ST, Matthey DP, Collinson ME, Hooker JJ, Jones TP. Phosphate $\delta^{18}\text{O}$ determination of modern rodent teeth by direct laser fluorination: an appraisal of methodology and potential application to palaeoclimate reconstruction. *Geochimica et Cosmochimica Acta*. 2001; 65(15):2535–48. [https://doi.org/10.1016/S0016-7037\(01\)00606-8](https://doi.org/10.1016/S0016-7037(01)00606-8).
52. Munro LE, Longstaffe FJ, White CD. Burning and boiling of modern deer bone: Effects on crystallinity and oxygen isotope composition of bioapatite phosphate. *Palaeogeography, Palaeoclimatology, Palaeoecology*. 2007; 249(1–2):90–102.
53. Munro LE, Longstaffe FJ, White CD. Effects of heating on the carbon and oxygen-isotope compositions of structural carbonate in bioapatite from modern deer bone. *Palaeogeography, Palaeoclimatology, Palaeoecology*. 2008; 266(3–4):142–50.
54. Snoeck C, Pouncett J, Claey's P, Goderis S, Mattielli N, Parker Pearson M, et al. Strontium isotope analysis on cremated human remains from Stonehenge support links with west Wales. *Scientific Reports*. 2018; 8(1):10790. <https://doi.org/10.1038/s41598-018-28969-8> PMID: 30072719
55. Snoeck C, Jones C, Pouncett J, Goderis S, Claey's P, Mattielli N, et al. Isotopic evidence for changing mobility and landscape use patterns between the Neolithic and Early Bronze Age in western Ireland.

- Journal of Archaeological Science: Reports. 2020; 30:102214. <https://doi.org/10.1016/j.jasrep.2020.102214>.
56. Thompson TJU. Heat-induced Dimensional Changes in Bone and their Consequences for Forensic Anthropology. *Journal of Forensic Sciences*. 2005; 50(5):JFS2004297–8. <https://doi.org/10.1520/JFS2004297> PMID: 16225204
 57. Snoeck C, Lee-Thorp JA, Schulting RJ. From bone to ash: Compositional and structural changes in burned modern and archaeological bone. *Palaeogeography, Palaeoclimatology, Palaeoecology*. 2014; 416:55–68. <https://doi.org/10.1016/j.palaeo.2014.08.002>.
 58. Greiner M, Rodríguez-Navarro A, Heinig MF, Mayer K, Kocsis B, Göhring A, et al. Bone incineration: An experimental study on mineral structure, colour and crystalline state. *Journal of Archaeological Science: Reports*. 2019; 25:507–18. <https://doi.org/10.1016/j.jasrep.2019.05.009>.
 59. Lebon M, Reiche I, Fröhlich F, Bahain JJ, Falguères C. Characterization of archaeological burnt bones: contribution of a new analytical protocol based on derivative FTIR spectroscopy and curve fitting of the ν_{13} PO₄ domain. *Analytical and Bioanalytical Chemistry*. 2008; 392(7):1479–88. <https://doi.org/10.1007/s00216-008-2469-y> PMID: 18972105
 60. Thompson TJU. The Analysis of Heat-Induced Crystallinity Change in Bone. In: Schmidt CW, Symes SA, editors. *The Analysis of Burned Human Remains (Second Edition)*. San Diego: Academic Press; 2015. p. 323–37.
 61. Etok SE, Valsami-Jones E, Wess TJ, Hiller JC, Maxwell CA, Rogers KD, et al. Structural and chemical changes of thermally treated bone apatite. *J Mater Sci*. 2007; 42(23):9807–16. <https://doi.org/10.1007/s10853-007-1993-z>
 62. Lebon M, Reiche I, Bahain JJ, Chadeffaux C, Moigne AM, Fröhlich F, et al. New parameters for the characterization of diagenetic alterations and heat-induced changes of fossil bone mineral using Fourier transform infrared spectrometry. *Journal of Archaeological Science*. 2010; 37(9):2265–76. <https://doi.org/10.1016/j.jas.2010.03.024>.
 63. Person A, Bocherens H, Mariotti A, Renard M. Diagenetic evolution and experimental heating of bone phosphate. *Palaeogeography, Palaeoclimatology, Palaeoecology*. 1996; 126(1):135–49. [https://doi.org/10.1016/S0031-0182\(97\)88906-7](https://doi.org/10.1016/S0031-0182(97)88906-7).
 64. Thompson TJU, Islam M, Bonniere M. A new statistical approach for determining the crystallinity of heat-altered bone mineral from FTIR spectra. *Journal of Archaeological Science*. 2013; 40(1):416–22. <https://doi.org/10.1016/j.jas.2012.07.008>.
 65. Mamede AP, Gonçalves D, Marques MPM, Batista de Carvalho LAE. Burned bones tell their own stories: A review of methodological approaches to assess heat-induced diagenesis. *Applied Spectroscopy Reviews*. 2018; 53(8):603–35. <https://doi.org/10.1080/05704928.2017.1400442>
 66. Habelitz S, Pascual L, Durán A. Transformation of tricalcium phosphate into apatite by ammonia treatment. *J Mater Sci*. 2001; 36(17):4131–5. <https://doi.org/10.1023/A:1017996217452>
 67. Habelitz S, Pascual L, Durán A. Nitrogen-containing apatite. *Journal of the European Ceramic Society*. 1999; 19(15):2685–94. [https://doi.org/10.1016/S0955-2219\(99\)00048-5](https://doi.org/10.1016/S0955-2219(99)00048-5).
 68. Vignoles M, Bonel G, Young RA. Occurrence of nitrogenous species in precipitated B-type carbonated hydroxyapatites. *Calcified Tissue International*. 1987; 40(2):64–70. <https://doi.org/10.1007/BF02555707> PMID: 3032379
 69. Dowker SEP, Elliott JC. Infrared absorption bands from NCO⁻ and NCN²⁻ in heated carbonate-containing apatites prepared in the presence of NH₄⁺ ions. *Calcified Tissue International*. 1979; 29(1):177–8. <https://doi.org/10.1007/BF02408075> PMID: 116758
 70. Marques MPM, Gonçalves D, Mamede AP, Coutinho T, Cunha E, Kockelmann W, et al. Profiling of human burned bones: oxidising versus reducing conditions. *Scientific Reports*. 2021; 11(1):1361. <https://doi.org/10.1038/s41598-020-80462-3> PMID: 33446708
 71. DeHaan JD, Campbell SJ, Nurbakhsh S. Combustion of animal fat and its implications for the consumption of human bodies in fires. *Science & Justice*. 1999; 39(1):27–38. [https://doi.org/10.1016/S1355-0306\(99\)72011-3](https://doi.org/10.1016/S1355-0306(99)72011-3).
 72. Moritz AR, Henriques FC. Studies of Thermal Injury: II. The Relative Importance of Time and Surface Temperature in the Causation of Cutaneous Burns. *The American journal of pathology*. 1947; 23(5):695–720. PMID: 19970955.
 73. Yermán L, Wall H, Carrascal J, Browning A, Chandraratne D, Nguyen C, et al. Experimental study on the fuel requirements for the thermal degradation of bodies by means of open pyre cremation. *Fire Safety Journal*. 2018; 98:63–73. <https://doi.org/10.1016/j.firesaf.2018.04.007>.
 74. Jonuks T, Konsa M. The Revival of Prehistoric Burial Practices: Three Archaeological Experiments. *Folklore*. 2007; 37:91–110.

75. Kates M. Techniques of lipidology: analysis and identification of lipids. Amsterdam: Elsevier; 1986. 464 p.
76. Liden K, Takahashi C, Nelson DE. The effects of lipids in stable carbon isotope analysis and the effects of NaOH treatment on the composition of extracted bone collagen. *Journal of Archaeological Science*. 1995; 22(2):321–6. <http://dx.doi.org/10.1006/jasc.1995.0034>.
77. Salesse K, Kaupová S, Brůžek J, Kuželka V, Velemínský P. An isotopic case study of individuals with syphilis from the pathological-anatomical reference collection of the national museum in Prague (Czech Republic, 19th century A.D.). *International Journal of Paleopathology*. 2019; 25:46–55. <https://doi.org/10.1016/j.ijpp.2019.04.001> PMID: 31051405
78. Brock F, Geoghegan V, Thomas B, Jurkschat K, Higham TFG. Analysis of Bone “Collagen” Extraction Products for Radiocarbon Dating. *Radiocarbon*. 2013; 55(2):445–63. <https://doi.org/10.1017/S0033822200057581>
79. Balasse M, Ambrose SH, Smith AB, Price TD. The seasonal mobility model for prehistoric herders in the south-western Cape of South Africa assessed by isotopic analysis of sheep tooth enamel. *Journal of Archaeological Science*. 2002; 29(9):917–32.
80. Snoeck C, Lee-Thorp J, Schulting R, de Jong J, Debouge W, Mattielli N. Calcined bone provides a reliable substrate for strontium isotope ratios as shown by an enrichment experiment. *Rapid Communications in Mass Spectrometry*. 2015; 29(1):107–14. <https://doi.org/10.1002/rcm.7078> PMID: 25462370
81. Kontopoulos I, Presslee S, Penkman K, Collins MJ. Preparation of bone powder for FTIR-ATR analysis: The particle size effect. *Vibrational Spectroscopy*. 2018; 99:167–77. <https://doi.org/10.1016/j.vibspec.2018.09.004>.
82. Tuross N, Warinner C, Kirsanow K, Kester C. Organic oxygen and hydrogen isotopes in a porcine controlled dietary study. *Rapid communications in mass spectrometry*. 2008; 22(11):1741–5. <https://doi.org/10.1002/rcm.3556> PMID: 18446769
83. Warinner C, Tuross N. Alkaline cooking and stable isotope tissue-diet spacing in swine: archaeological implications. *Journal of Archaeological Science*. 2009; 36(8):1690–7.
84. von Holstein ICC, Hamilton J, Craig OE, Newton J, Collins MJ. Comparison of isotopic variability in proteinaceous tissues of a domesticated herbivore: a baseline for zooarchaeological investigation. *Rapid Communications in Mass Spectrometry*. 2013; 27(23):2601–15. <https://doi.org/10.1002/rcm.6725> PMID: 24591021
85. Kirsanow K, Makarewicz C, Tuross N. Stable oxygen ($\delta^{18}\text{O}$) and hydrogen (δD) isotopes in ovicaprid dental collagen record seasonal variation. *Journal of Archaeological Science*. 2008; 35(12):3159–67.
86. Shi J, Klocke A, Zhang M, Bismayer U. Thermally-induced structural modification of dental enamel apatite: Decomposition and transformation of carbonate groups. *European Journal of Mineralogy*. 2005; 17(5):769–76.
87. Tõnsuaadu K, Gross KA, Plūduma L, Veiderma M. A review on the thermal stability of calcium apatites. *Journal of Thermal Analysis and Calorimetry*. 2012; 110(2):647–59. <https://doi.org/10.1007/s10973-011-1877-y>
88. Bartlett AI, Hadden RM, Bisby LA. A Review of Factors Affecting the Burning Behaviour of Wood for Application to Tall Timber Construction. *Fire Technology*. 2019; 55(1):1–49. <https://doi.org/10.1007/s10694-018-0787-y>
89. Barbour MM, Andrews TJ, Farquhar GD. Correlations between oxygen isotope ratios of wood constituents of *Quercus* and *Pinus* samples from around the world. *Functional Plant Biology*. 2001; 28(5):335–48. <https://doi.org/10.1071/PP00083>.
90. Gray J, Thompson P. Climatic information from $^{18}\text{O}/^{16}\text{O}$ analysis of cellulose, lignin and whole wood from tree rings. *Nature*. 1977; 270(5639):708–9. <https://doi.org/10.1038/270708a0>
91. Battipaglia G, Jäggi M, Saurer M, Siegwolf RTW, Cotrufo MF. Climatic sensitivity of $\delta^{18}\text{O}$ in the wood and cellulose of tree rings: Results from a mixed stand of *Acer pseudoplatanus* L. and *Fagus sylvatica* L. *Palaeogeography, Palaeoclimatology, Palaeoecology*. 2008; 261(1):193–202. <https://doi.org/10.1016/j.palaeo.2008.01.020>.
92. Ferrio JP, Voltas J. Carbon and oxygen isotope ratios in wood constituents of *Pinus halepensis* as indicators of precipitation, temperature and vapour pressure deficit. *Tellus B: Chemical and Physical Meteorology*. 2005; 57(2):164–73. <https://doi.org/10.3402/tellusb.v57i2.16780>
93. DeHaan JD. Fire and Bodies. In: Schmidt A, Symes S, editors. *The Analysis of Burned Human Remains*. Amsterdam: Academic Press; 2008. p. 1–13.
94. Schumacher M, Werner RA, Meijer HAJ, Jansen HG, Brand WA, Geilmann H, et al. Oxygen isotopic signature of CO_2 from combustion processes. *Atmos Chem Phys*. 2011; 11(4):1473–90. <https://doi.org/10.5194/acp-11-1473-2011>

95. Sivakumar V, Ponnusawmy C, Sudalaimani K, Rangasamy T, Muralidharan C, Mandal AB. Ammonia free delimiting process in leather industry based on eco-benign products. *Journal of Scientific & Industrial Research*. 2015; 74(9):518–21.
96. Hashem A, Islam A, Paul S, Nasrin S. Generation of ammonia in delimiting operation from Tannery and its environmental effect: Bangladesh perspective. *International Journal of Renewable Energy and Environmental Engineering*. 2014; 2(4):266–70.
97. Dercy B. *Le travail des peaux et du cuir dans le monde grec antique. Tentative d'une archéologie du disparu appliquée au cuir*. Naples: Centre Jean Bérard; 2015.
98. Stamataki E, Kontopoulos I, Salesse K, McMillan R, Veselka B, Sabaux C, et al. Is it hot enough? A multi-proxy approach shows variations in cremation conditions during the Metal Ages in Belgium. *Journal of Archaeological Science*. Accepted.
99. Goldhahn J, Oestigaard T. Smith and death. Cremations in furnaces in Bronze and Iron Age Scandinavia. In: Prescott C, Chilidis K, Lund J, editors. *Facets of Archaeology. Essays in Honour of Lotte Hedegaard on her 60th Birthday*. Oslo: Oslo University; 2008. p. 215–241.