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A multi-proxy approach shows variations in cremation conditions during the Metal Ages in Belgium

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Abstract

Studies of funerary practices provide information about many aspects of death in past societies. However, only limited archaeological evidence documents the circumstances under which cremations occurred and the person(s) who were performing the funerary rituals. Lying at the border between Atlantic and Continental cultural traditions, the Scheldt and Meuse basins of Belgium represent a unique location to investigate variations in ancient pyre technology and body management as well as the transfer of knowledge related to cremation techniques during the Metal Ages (ca. 2100-52 BCE). The combined use of Fourier Transform Infrared spectroscopy and carbon and oxygen isotope analysis of different skeletal elements of cremation deposits from four archaeological sites clearly shows differences between the Meuse and Scheldt basins which could be linked to different wood availability or selection and variations in the skills and/or experience of the cremation operator. These observed differences are likely linked to ways in which cremation was performed in the two basins, indicating that during the Metal Ages burning processes were not homogeneous in the Belgian region. Instead, cremation practices appear to align with the different cultural influences also observed in ceramics and bronze artifacts from the same time period. These observed differences in funerary practices between the two basins in Belgium show the immense potential of combining infrared and carbon and oxygen isotope analyses to investigate cremation rituals in any period and region around the world.

Keywords: Cremated bones; FTIR-ATR; Carbon & Oxygen isotope analysis; Late Bronze Age; Early Iron Age

Introduction

The diversity of ancient funerary practices relates to different attitudes of communities towards death and may be visible in the differences in how the deceased were treated. The spatial organisation of cemeteries, the diversity of treatments of the corpse after death (e.g. inhumation, cremation), the orientation and position of the body in a grave, the place where the deceased was cremated, where the cremated bones were deposited, and the presence or absence of grave goods have been used to distinguish ancient societies with different beliefs and customs (Oestigaard 2000; Pearson 2001; Oestigaard and Goldhahn 2006; Gligor 2014). While the study of funerary practices provides information about many aspects of death in past societies, only limited archaeological evidence documents the circumstances under which cremations occurred (e.g., temperature, duration, fuel, pyre size, pyre location, position of the body on the pyre) as well as the person(s) who were performing the funerary rituals. This person (i.e. the cremator or cremation operator) could have been a ritual specialist, a skilled worker (e.g. a smith or a potter) (Goldhahn & Oestigaard 2008; McKinley 2015; 2016; Carroll & Squires 2020), or perhaps a family member (Oestigaard 2000). The transformation of the dead body from flesh to burnt bones and ash requires technical knowledges regarding the management of funeral pyre, which is linked to the skills and specialisation of the cremator (Cenzon-Salvayre 2014). In addition to the experience and knowledge of the cremator, other factors can affect the cremation settings, including the cremation environment, the biological profile of the cremated individual (e.g. sex, age), their social status, as well as the cultural, moral, ethical, and religious beliefs of the society within which the cremation took place.

During the Metal Ages in Belgium (ca. 2100-52 BCE), the Scheldt and Meuse basins were the main axes of transportation and communication (De Mulder 2013; 2017) (Figure 1). Studies of the recovered ceramics and bronze artifacts indicate that Belgium was located at the crossroads between the so-called Atlantic (Channel-North Sea) and Continental (Eastern France, the Rhine area, and Western Switzerland) cultural traditions, while influences by the Nordic Bronze Age culture are also visible but to a lesser extent (Mariën 1951; Warmenbol 1988a; Lehoërff et al. 2012; Leclercq 2013; De Mulder 2013; 2017). The Meuse basin was mostly oriented towards the Central European cultural context, presenting strong connections with the Rhin-Suisse-France orientale (RSFO) group during the period ca. 1100-900 BCE (Ha A2-B1; see Sabaux et al. 2021 for more details). The Scheldt basin appears to primarily be influenced by the Atlantic cultural area and to a lesser extent by the RSFO group and northern Germany (Mariën 1951; Warmenbol 1988a; De Mulder et al. 2008; Leclercq 2013; Leclercq and Warmenbol 2017).

Figure 1. The Scheldt and Meuse basins in Belgium, France and the Netherlands with the location of the studied sites.

Archaeological evidence from ancient Belgian cemeteries indicates that inhumation and cremation practices coexisted from the Mesolithic to the Early Medieval period. In the Late Bronze Age (LBA) (ca. 1200-800 BCE), cremation seems to be the predominant funeral rite, but it was already used on a large scale from the Middle Bronze Age (MBA) (ca. 1800-1200 BCE) onwards (De Mulder 2010; Capuzzo et al. 2020). Both the Late Bronze Age and Early Iron Age (EIA) are characterised by urnfield cemeteries and the adoption of cremation as a pan-European phenomenon (Barceló et al. 2014; Capuzzo & Barceló 2015). Nevertheless, a large variation in the type of cremation burials is observed in Belgian archaeological contexts (see De Mulder et al. 2009 for more details). Even with the large amount of information that can be extracted from the study of cremation burials, the questions about the variation in cremation settings between the different cultural traditions and their link to the skills and specialisation of those performing and managing the cremation funerary rituals in the Metal Ages remain unanswered. Further, the lack of written sources from the Metal Ages in Belgium makes addressing questions related to the burning conditions and specialisation of funerary rituals more difficult and creates a gap in our understanding of funerary practices from a period when cremation was the dominant funerary rite, as is the case in Belgium from the LBA to the EIA (ca. 1200-500/450 BCE).

Until the end of the 20th century, researchers thought that most biological, chronological, and environmental information recorded in bone was destroyed during the burning process. As a result, the chemical and structural analyses of cremated skeletal remains received little attention for many years (Shipman et al. 1984; Grupe & Hummel 1991; Taylor et al. 1995; Shahack-Gross et al. 1997). This changed when researchers demonstrated that obtaining reliable radiocarbon dates (^{14}C) from fully calcined bone was possible (Lanting et al. 1998; 2001; Van Strydonck et al. 2010; Zazzo et al. 2012). Although these new developments also raised questions about the origin of the carbon used for radiochronology (Surovell 2000; Van Strydonck et al. 2005; Munro et al. 2008; Olsen et al. 2008; Zazzo et al. 2009; Hüls et al. 2010; Van Strydonck et al. 2010; Harbeck et al. 2011; Zazzo et al. 2012; Olsen et al. 2013; Zazzo et al. 2013; Snoeck et al. 2014a; Van Strydonck et al. 2015), they opened new avenues for the study of burned bone. Studies have shown that during the combustion process, up to 95% of biogenic carbon in bone can be replaced by carbon from the fuel, although the extent of this replacement is highly variable (Surovell 2000; Van Strydonck et al. 2005; Munro et al. 2008; Olsen et al. 2008; Zazzo et al. 2009; Hüls et al. 2010; Van Strydonck et al. 2010; Harbeck et al.

2011; Zazzo et al. 2012; Olsen et al. 2013; Zazzo et al. 2013; Snoeck et al. 2014a; Van Strydonck et al. 2015).

Interpreting the oxygen isotopic composition ($\delta^{18}O$) of burned bone apatite carbonate is even more complex, as more exogenous sources of oxygen than carbon are available in the cremation environment, facilitating overprinting of in-vivo isotopic signatures (e.g. atmospheric O_2 and CO_2, oxygen from the flesh, soft tissues, and collagen, oxygen from the fuel, etc.; Munro et al. 2007; Harbeck et al. 2011; Snoeck et al. 2016). Still, useful information can be extracted from cremated bone; measuring the carbon and oxygen isotope ratios of cremated bone can provide insights into the cremation environment and process (instead of an individual's life history), including the presence or absence of fuel, oxygen availability during cremation linked to the size of the pyre and/or to the position of the pyre in the environment (e.g. within a building, in a pit, or in an open space), as well as the position of the body on the pyre (Zazzo et al. 2012; Snoeck et al. 2014a; 2014b; 2016; 2018; Salesse et al. in press).

In parallel, Fourier Transform Infrared Spectroscopy (FTIR) provides insight into the structural and compositional transformations of bone during cremation via a variety of well-documented infrared indices (Shahack-Gross et al. 1997; Thompson et al. 2009; 2011; 2013; Lebon et al. 2010; Snoeck et al. 2014b; Ellingham et al. 2015; 2016; Mamede et al. 2018; Minami et al. 2019; Gonçalves et al. 2020; Legan et al. 2020; Lemmers et al. 2020; Marques et al. 2021; SI 1). These indices have been developed based on the intensity and the position of various bands of the infrared spectra. Alterations that bones undergo during cremation destroy the organic components, including collagen and remove water, and, once fully calcined, only the inorganic fraction of the bone (often called bone apatite or bioapatite) remains. The inorganic fraction is a carbonated calcium phosphate mineral compound, with a composition and structure similar to hydroxyapatite [$Ca_5(PO_4)_3(OH)_2$]. Phosphate in bioapatite is partially replaced by carbonate (CO_3^{2-} known as type B carbonates) and carbonates also occupy some (if not most) of the hydroxyl group (OH^-) positions (known as type A carbonates; LeGeros et al. 1969; Skinner 2005; see Snoeck et al. 2014b for more details).

The infrared splitting factor (IRSF) provides information regarding bone apatite crystallinity. Crystallinity refers to the bioapatite crystallite size and the degree and range of structural order within the crystal lattice (Weiner and Bar-Yosef 1990). The carbonyl-to-carbonate ratio (C/C) compares two absorbance wavelengths associated to carbonate (the $ν_3CO_3$ band at 1450 cm^{-1} and the $ν_3CO_3$ band at 1415 cm^{-1}), while the carbonate-to-phosphate ratio (C/P) is used to assess the carbonate content in bone apatite relative to the phosphate content (Wright and Schwarcz 1996). The phosphate-to-phosphate ratio (P/P) is used to compare two absorbance wavelengths associated to phosphate (the $ν_3PO_4$ band at 600 cm^{-1} and the $ν_3PO_4$ band at 560 cm^{-1}; Lee-Thorp and Sponheimer 2003). The amide-to-phosphate ratio (Am/P) indicates the amount of organic matter and water still present in heated bones relative to the phosphate content (Trueman et al. 2004; Roche et al. 2010). The hydroxyl-to-phosphate ratio (OH/P) is used to describe the changes to hydroxyl groups in heated bone apatite (Snoeck et al. 2014b). Finally, the cyanamide-to-phosphate ratio (CN/P) is related to reducing-oxidation conditions (i.e. low oxygen availability; Zazzo et al. 2013; Snoeck et al. 2014b) and to the co-existence of ammonia during the burning process, which has been linked to the potential presence of garments worn by the deceased during cremation (Salesse et al. in press).

To investigate if the differences between Scheldt and Meuse basin observed in material culture and the types of cremation burials in the Metal Ages are also detected in the way cremation was performed, FTIR-ATR and stable carbon and oxygen isotope analysis of bone apatite carbonates were explored with linear discriminant analyses (LDA) and principal component analyses (PCA) as well as in more traditional, low-dimensional space (e.g., bivariate plots). The aim of this study is to assess the intra- and inter-basin variability in cremation settings (i.e temperature, time, fuel, pyre structure) from sites that date to the LBA and EIA. These sites are located both in the Scheldt (Velzeke and Blicquy) and the Meuse basins (Grand Bois and Herstal). Characterizing intra- and inter-basin variability helps to evaluate if cremation practices were homogeneous within, and between, basins. This will aid us in revealing if different members of a society were handled similarly after death. Further, this information can be used to characterize the skills and specialisation of those carrying out the cremations, allowing us to assess if, in the Metal Ages, the knowledge required to carry out cremations was shared and exchanged between different cultural groups.

Materials and methods

Sites and Samples

Four archaeological cremation cemeteries from different geographical areas of Belgium dating from the MBA to the EIA (ca. 1500-500/450 BCE) were selected (Figure 1; Table 1): two in the Scheldt basin (Velzeke and Blicquy) and two in the Meuse basin (Grand Bois and Herstal). In the cemetery of Velzeke/Paddestraat, 41 cremation graves were excavated during several archaeological campaigns in the 1970s (De Mulder & Rogge 1995), but only 14 urn graves could be investigated based on their preservation state. In these graves, the cremated remains were deposited either in a ceramic vessel or in a container made from organic material. The ceramic typology and radiocarbon dating indicate that the graveyard was in use from the LBA to the EIA (ca. 1200-500/450 BCE), and was abandoned during the EIA (De Mulder et al. 2007).

The cemetery of Blicquy/Ville d'Anderlecht was partially excavated during several campaigns in the 1990s. In total, 35 cremation graves have been found in the western and southern parts of the graveyard, although it seems that the graveyard extended farther to the north. Only the cremated remains of 14 graves were available for this study. The ceramic typology and radiocarbon dating show that the graveyard was in use from the later phase of MBA to the EIA (ca. 1500-500/450 BCE) (De Mulder et al. 2007).

The cemetery of Saint Vincent/Grand Bois is in the municipality of Saint-Vincent (Bellefontaine) in Belgium. In total, 87 cremation graves dating to the LBA-EIA (ca. 1200-500/450 BCE) were excavated in seven archaeological campaigns in the late 19th century (Mariën 1964). From the excavated graves, only 20 cremations were available for analysis.

Finally, the cemetery of Herstal/Pré Wigier is located near Liège, close to the eastern border of Belgium. In total, 30 cremation graves from the Metal Ages have been excavated in the 1970s (Alenus-Lecerf 1974). For the study of cremation conditions, the cremated remains of 21 graves were used. The grave goods and radiocarbon dating indicate that the cemetery of Herstal was in use from the LBA to the beginning of the EIA (1200-600 BCE) (Alenus-Lecerf 1974; Lecarme and Warmenbol 2014; Leclercq 2014; Sabaux et al. 2021).

Three different skeletal elements from different bone categories (cranium, diaphyses of the upper/lower limb long bones, ribs) were sampled from each selected individual to investigate how the cremation settings affect different skeletal elements usually located in different positions in the funerary pyre (Table 1). However, the sampling location on each bone was not consistent because of the high fragmentation of the Belgian cremated collections. In some cases, rib bones were extremely fragmented, and sampling was not possible.

Table 1. Samples from the studied Belgian sites dated to the Metal Ages (for osteoarchaeological report see SI 3 and Sabaux et al. 2021).

Site	Chronology	Samples	Selected Elements	Graves	Number of studied individuals	Sex	Age
Velzeke	Late Bronze/Early Iron age	35	14 Diaphyses 13 Cranium 8 Ribs	14	14	1 Female 11 Indeterminate 2 Not-applicable	11 Adults 2 Non-adults 1 Indeterminate
Blicquy	Middle/Late Bronze/Early Iron age	38	14 Diaphyses 14 Cranium 10 Ribs	14	15	2 Female 2 Male 10 Indeterminate 1 Not-applicable	12 Adults 1 Non-adult 2 Indeterminate
Grand Bois	Late Bronze/Early Iron Age	62	24 Diaphyses 24 Cranium 14 Ribs	20	20	1 Female 2 Male 17 Indeterminate 1 Not-applicable	8 Adults 1 Non-adult 5 Indeterminate
Herstal	Late Bronze/Early Iron age	73	32 Diaphyses 24 Cranium 17 Ribs	21	23	1 Female 2 Male 9 Indeterminate 11 Not-applicable	8 Adults 11 Non-adults 4 Indeterminate

Methods

Pre-treatment of calcined bone fragments

Fragments selected for isotope and infrared analysis (ca. 200mg) were pre-treated to remove secondary carbonates and any post-burial contamination using the following procedure. First, the visible sediment as well as the trabecular and the outer layers of periosteal and endosteal surfaces of cortical bone were mechanically removed using a diamond drill. Then, the samples were pre-treated following the procedure described in Snoeck et al. (2015) and in McMillan et al. (2019). The selected fragments were rinsed three times with MilliQ water in an ultrasonic bath for 10 minutes each time. Then, they were cleaned in the ultrasonic bath using 10mL of 1M acetic acid (CH_3COOH) for 10 minutes. A second round of three times rinsing with MilliQ water in an ultrasonic bath followed. Finally, the samples were left in an oven at 50°C to dry before being crushed with mortar and pestle.

Carbon and Oxygen isotope analyses of bone apatite carbonates

For carbon and oxygen isotope analysis, 30mg of bone powder was used and the samples were analysed in duplicates (15mg for each analysis). Prior to the analysis, the samples were placed in sealed glass tubes (exetainer® from Labco Limiter) and flushed using helium to remove any atmosphere from the tubes. Phosphoric acid was then added which reacts with the carbonates present in the bone and releases CO_2 that is analysed with a Nu Perspective IRMS (Isotope Ratio Mass Spectrometer) from Nu Instruments coupled with a Nu GasPrep automatic gas bench at the Vrije Universiteit Brussel (VUB). The stable isotope ratios are expressed as delta (δ) units, which measure deviation in isotope ratios from a particular standard value (McKinney et al. 1950, Mays 1998, Sharp 2007, Hoefs 2015). The results are reported as per mil (‰) deviation from VPDB reference standard. Three standards IA-R022 ($\delta^{13}C$ = -28.6‰ and $\delta^{18}O$ = -22.7‰), IAEA-603 ($\delta^{13}C$ = 2.5‰ and $\delta^{18}O$ = -2.3‰), and IAEA-CO_8 ($\delta^{13}C$ = -5.8‰ and $\delta^{18}O$ = -22.7‰) were used to calibrate the isotopic data. Over the course of all analyses, the analytical precision was better than ±0.30‰ and ±0.40‰ (1SD) for both $\delta^{13}C$ and $\delta^{18}O$ respectively based on repeated measurements of in-house cremated bone standard CBA (n=15; see De Winter et al. 2016).

Fourier Transform Infrared Spectroscopy in Attenuated Total Reflectance Mode (FTIR-ATR)

For FTIR-ATR measurements, the bone powder was sieved through two woven stainless-steel mesh sieves (50µm and 25µm). Only the 50-25µm fraction was analysed in triplicates as has been shown by Kontopoulos et al. (2018) to provide the most reliable and reproducible results (2-3mg of bone powder was used for each measurement). The infrared analyses were carried out at the VUB-AMGC research unit using a Bruker Vertex 70v FTIR spectrometer under vacuum (spectral range: 4000–400 cm^{-1}; number of scans: 32; spectral resolution: 4 cm^{-1}; mode: absorbance). After each measurement, the crystal plate and the anvil of the pressure applicator were cleaned using Isopropanol. Each sample was measured in triplicate and the indices reported in SI 2 represent the average of three measurements. Spectra were analysed using OPUS 7.5 software and all the indices were calculated after the baseline correction (see SI 1 for more details).

Statistical tests

A number of univariate statistical tests were performed using IBM SPSS Statistics version 26 to investigate the significance of the differences among measured attributes from the studied sites, including skeletal elements, sex, age, and cremation deposition with the infrared indices and the carbon and oxygen isotopic data. Before any statistical comparison, Gaussian Normality was checked using the Kolmogorov-Smirnov test. Most variables are not normally distributed and as a result, the non-parametric Kruskal-Wallis and/or a Mann-Whitney U test were performed to assess differences among populations. Statistical significance was set at $p \leq 0.05$.

Multivariate discriminant analysis was conducted on both FTIR (IRSF, C/C, BPI, C/P, P/P, OH/P, CN/P, and Am/P) and stable isotope ($\delta^{13}C$, $\delta^{18}O$) data to explore relationships within and among basins, sites, and skeletal elements. Discriminant analyses identify and extract gradients of variation that can be used to reduce the dimensionality of multivariate data among predefined groups (e.g., basins, sites, or skeletal elements). This is accomplished by condensing a number of variables into a new set of composite variables (i.e., canonical functions) that best describe hypothesized group membership (Mardia et al. 1979; Martinez & Kak, 2001; Venables & Ripley 2002). Linear discriminant analysis (LDA) uses linear combinations of variables to differentiate best among pre-defined groups and can also be used to predict the class of a given observation. We used LDA to examine the number of cases correctly classified (% correct) by canonical functions among our pre-defined groups (e.g., bone element, location) using a randomized test/training subsample, producing a statistic that indicates relative group uniqueness, integrity, and robustness (the higher the % correct, the more unique the groups). LDA tests were performed using the "MASS" package (Venables & Ripley 2002) in the R programming environment version 4.0.3 (R Core Team 2020). Prior to LDA analysis, scaling of variables, to meet parametric test assumptions, was assessed. Transformations were conducted automatically to best suit the data used in the model using the R package "caret" (Kuhn 2020).

Principal components analysis (PCA) was subsequently conducted using all variables as well as a subset of variables based on the weightings of variables in the LDA results. This was performed to assess if the results from the "supervised" LDA (i.e., with predefined groups) would produce a similar distribution using an unsupervised test (i.e., PCA, which does not include groupings as a prior). Principal components analysis uses orthogonal transformation to convert a set of observations of potentially correlated variables into a set of linearly correlated variables, or "principal components" (e.g, Mardia et al. 1979; Martinez & Kak, 2001; Jolliffe 2002; Venables & Ripley 2002). The first component exhibits the largest variance among individual observations, not predetermined groupings (hence "unsupervised"), and each subsequent component exhibits the highest variance possible while still being orthogonal to the preceding components. PCA was conducted using the prcomp() function, also in R. Like LDA, PCA is sensitive to the relative scaling of the original variables, so, prior to PCA analysis, all variables were also transformed using the function “caret” (i.e., the LDA and PCA tests were both performed on the same transformed dataset to reduce parametric test assumption violation).

Results

Isotopic and Infrared results

The $\delta^{13}C$ values of all the skeletal elements from the four sites range between -28.4 and -17.1‰ while $\delta^{18}O$ values range between -26.2 and -14.2‰ (Figure 2; SI 2). The median $\delta^{13}C$ values of the two sites from the Scheldt basin (Velzeke and Blicquy) are very close to each other as is the case for the two sites from the Meuse basin (Grand Bois and Herstal) (Figure 2; Table 2). There is, however, an average difference of about 1.6‰ in median $\delta^{13}C$ values between the samples of the two basins, which is statistically significant (Mann-Whitney U = 3488.0; p = 0.01). The $\delta^{18}O$ values observed in the Scheldt basin exhibit a narrower range compared to the samples from the Meuse basin; however, no statistical differences were observed between the basins (Mann-Whitney U = 4357.0; p = 0.19). For the different skeletal elements, diaphyses of long bones consistently present the highest variability in both $\delta^{13}C$ and $\delta^{18}O$ values, and ribs have the smallest (Table 3).

Table 2. Median values and interquartile range (IQR) of $\delta^{13}C$ and $\delta^{18}O$ of the studied sites, where the IQR is the difference between the third (Q3) and first quartile (Q1).

Site	Basin	IQR $\delta^{13}C$ (‰) Q3 Q1	Median $\delta^{13}C$ (‰)	IQR $\delta^{18}O$ (‰) Q3 Q1	Median $\delta^{18}O$ (‰)
Velzeke	**Scheldt**	-21.6 to -25.6	-24.0	-17.8 to -19.4	-18.3
Blicquy	**Scheldt**	-22.7 to -25.1	-24.0	-17.0 to -19.4	-18.2
Grand Bois	**Meuse**	-20.9 to -24.1	-22.3	-17.9 to -20.0	-18.9
Herstal	**Meuse**	-21.2 to -24.1	-22.5	-17.2 to -20.3	-18.3

Herstal was the only site with a high number of cremated non-adult individuals and therefore the only site where a comparison between adults and non-adults was possible. Kruskal-Wallis combined with a Mann-Whitney U statistical test among adults, non-adults, and indeterminate individuals indicated that in Herstal a statistically significant difference in $\delta^{13}C$ values exists between the adult and non-adult individuals (U = 248.0; p = 0.01). The median $\delta^{13}C$ value of the adults was higher (-22.0‰, IQR = -23.3‰ to -21.1‰) than the non-adults (-24.2‰, IQR = -25.2‰ to -22.3‰).

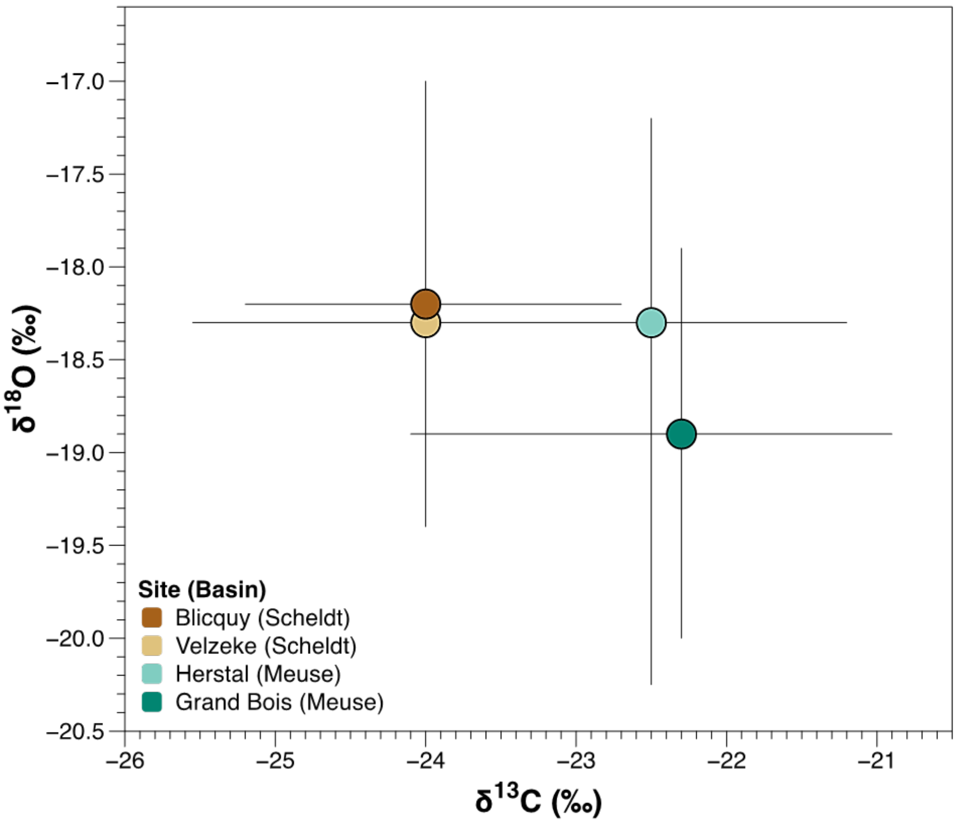

Figure 2. Median $\delta^{13}C$ to $\delta^{18}O$ values of the studied sites. The error bars indicate the 1st and the 3rd quartiles.

The median $\delta^{13}C$ values of different skeletal elements from the four sites indicate that rib fragments from Velzeke, Grand Bois, and Herstal have similar $\delta^{13}C$ values, while Blicquy presents a more depleted $\delta^{13}C$ value which differs by 1.5‰ on average and the highest median $\delta^{18}O$ value among them (Table 3). However, statistically significant differences are only observed between the ribs from Blicquy and Herstal (Mann-Whitney U = 42.0 ; p = 0.03 for $\delta^{13}C$ and U = 36.5; p = 0.01 for $\delta^{18}O$).

Table 3. Median values and interquartile ranges (IQR=Q3-Q1) of $\delta^{13}C$, $\delta^{18}O$, IRSF, and OH/P per skeletal element and site.

Median $\delta^{13}C$ values per skeletal element (Q3 to Q1) (‰)				
Skeletal element	**Velzeke**	**Blicquy**	**Grand Bois**	**Herstal**
Long bones	-21.8 (-20.9 to -25.1)	-23.4 (-20.6 to -24.2)	-21.4 (-20.8 to -23.5)	-22.5 (-21.1 to -23.7)
Cranium	-25.4 (-23.7 to -25.7)	-24.0 (-22.0 to -25.1)	-22.4 (-20.9 to -24.0)	-22.3 (-21.9 to -23.8)
Ribs	-23.3 (-22.4 to -24.1)	-24.6 (-23.5 to -25.3)	-23.4 (-22.0 to -25.0)	-22.7 (-21.4 to -24.6)
Median $\delta^{18}O$ values per skeletal element (Q3 to Q1) (‰)				
Long bones	-18.2 (-17.8 to -19.2)	-19.0 (-18.0 to -19.5)	-18.5 (-17.9 to -19.0)	-18.2 (-16.7 to -18.8)
Cranium	-18.3 (-17.8 to -19.3)	-17.5 (-16.8 to -18.5)	-19.2 (-17.9 to -20.2)	-18.1 (-17.2 to -20.4)

Ribs	-19.0 (-18.3 to -19.7)	-18.1 (-17.1 to -19.2)	-19.7 (-18.3 to -20.8)	-20.6 (-18.7 to -21.6)
Median IRSF values per skeletal element (Q3 to Q1)				
Long bones	5.8 (6.2 to 5.5)	5.9 (6.1 to 5.7)	5.3 (5.5 to 5.1)	5.2 (5.3 to 5.0)
Cranium	6.3 (6.5 to 6.0)	5.8 (6.2 to 5.5)	5.2 (5.4 to 5.0)	5.5 (5.7 to 5.2)
Ribs	5.8 (6.2 to 5.6)	5.8 (6.0 to 5.7)	5.2 (5.3 to 4.8)	5.0 (5.3 to 4.9)
Median OH/P values per skeletal element (Q3 to Q1)				
Long bones	0.37 (0.40 to 0.32)	0.37 (0.40 to 0.33)	0.41 (0.46 to 0.36)	0.46 (0.50 to 0.41)
Cranium	0.42 (0.46 to 0.39)	0.39 (0.40 to 0.32)	0.44 (0.46 to 0.42)	0.42 (0.45 to 0.36)
Ribs	0.39 (0.43 to 0.36)	0.39 (0.40 to 0.35)	0.47 (0.50 to 0.45)	0.48 (0.50 to 0.43)

The infrared indices (SI 2) show that the cremated bones from all the sites were likely exposed to high intensity burning as indicated by the extremely low values of the Am/P ratios (< 0.002), suggesting the absence of any organic matter or adsorbed water. In addition, the high crystallinity observed in bone samples from for all the sites (IRSF > 5) is consistent with fully calcined bones. Ribs present the lowest IRSF values of the studied skeletal elements, although the differences are not statistically significant (Table 3).

Regarding the infrared values, none of the investigated parameters showed significant difference between the two sites in the Scheldt basin (Velzeke and Blicquy) or between the sites in the Meuse basin (Grand Bois and Herstal). However, when comparing the results between the two basins, the IRSF values showed statistically significant differences (Mann-Whitney U = 1108.0; $p < 0.01$). All the skeletal elements of Velzeke and Blicquy show higher IRSF values and lower C/C values in comparison to Grand Bois and Herstal (Mann-Whitney U = 3180.5; $p < 0.01$) (Figure 3).

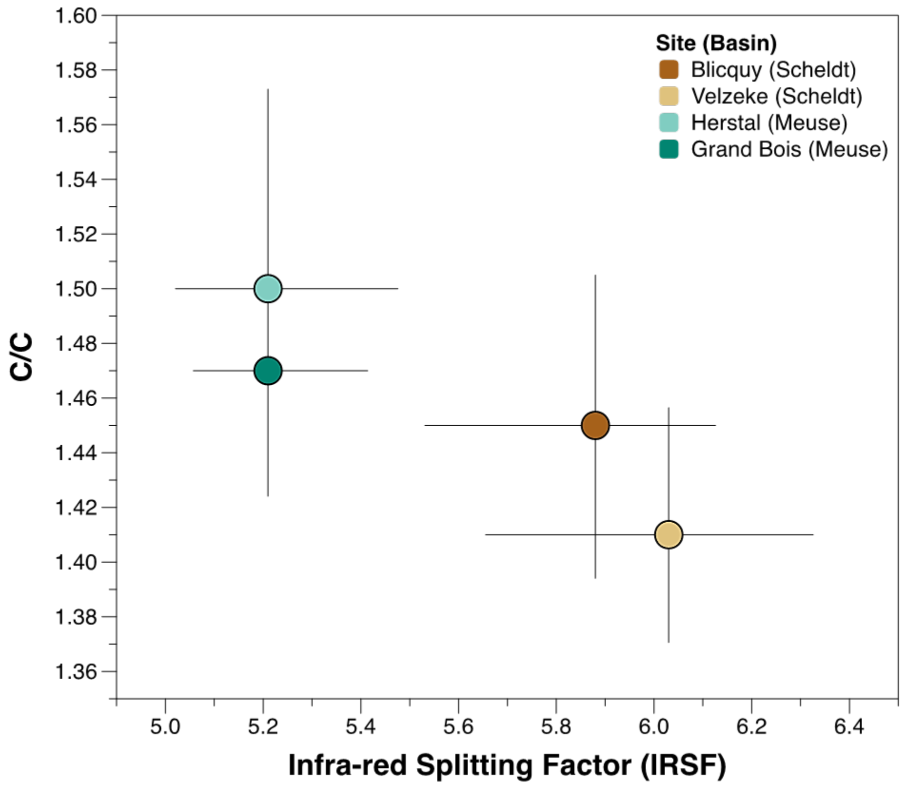

Figure 3. Median IRSF to C/C values of the studied sites. The error bars indicate the 1st and the 3rd quartiles.

An LDA model including all available variables (IRSF, C/C, BPI, C/P, P/P, OH/P, CN/P, Am/P, and $\delta^{13}C$, $\delta^{18}O$) and grouped by site, showed that the two basins can be differentiated using two discriminant functions, with minimal overlap between the basins, and with significant overlap between the sites in each basin (Figure 4A). An LDA test/training set of 20/80% of the same dataset grouped instead by basin also yielded ~95% of test cases accurately reattributed to the correct basin. An unsupervised PCA test run using the same variables showed a similar trend but with less distance between each basin and only 47% of summed variance explained by the first two components (Figure 4B). Less systematic difference among skeletal elements was identified by LDA using the same set of variables (Figure 4C), with a test/training set of 20/80% resulting in only 60% of cases accurately reattributed to the correct skeletal element. The weightings of variables for the first discriminant function in the LDA models trained to differentiate among investigated sites and basins were highest for infrared characteristics except Am/P and C/C. When the variables used in the PCA model were reduced to include only the five infrared variables with the greatest LDA weightings, BPI, C/P, OH/P, P/P, and IRSF, a similar trend became apparent as shown in Figures 4A and 4B (Figure 4D) except instead with 82% of summed variance explained by the first two components. Further, IRSF, OH/P, and P/P appear to create the largest differences among the basins based on the attitudes of PCA loadings in Figure 4D. In general, the multivariate statistical results suggest that the infrared data records the largest relative differences in samples from the two basins. When an LDA was conducted using only the isotopic variables by basin, a test/training set of 20/80% yielded 70% of samples accurately reattributed to the correct basin, and an LDA only using the infrared variables resulted in 92.5% of cases accurately reattributed to the correct basin.

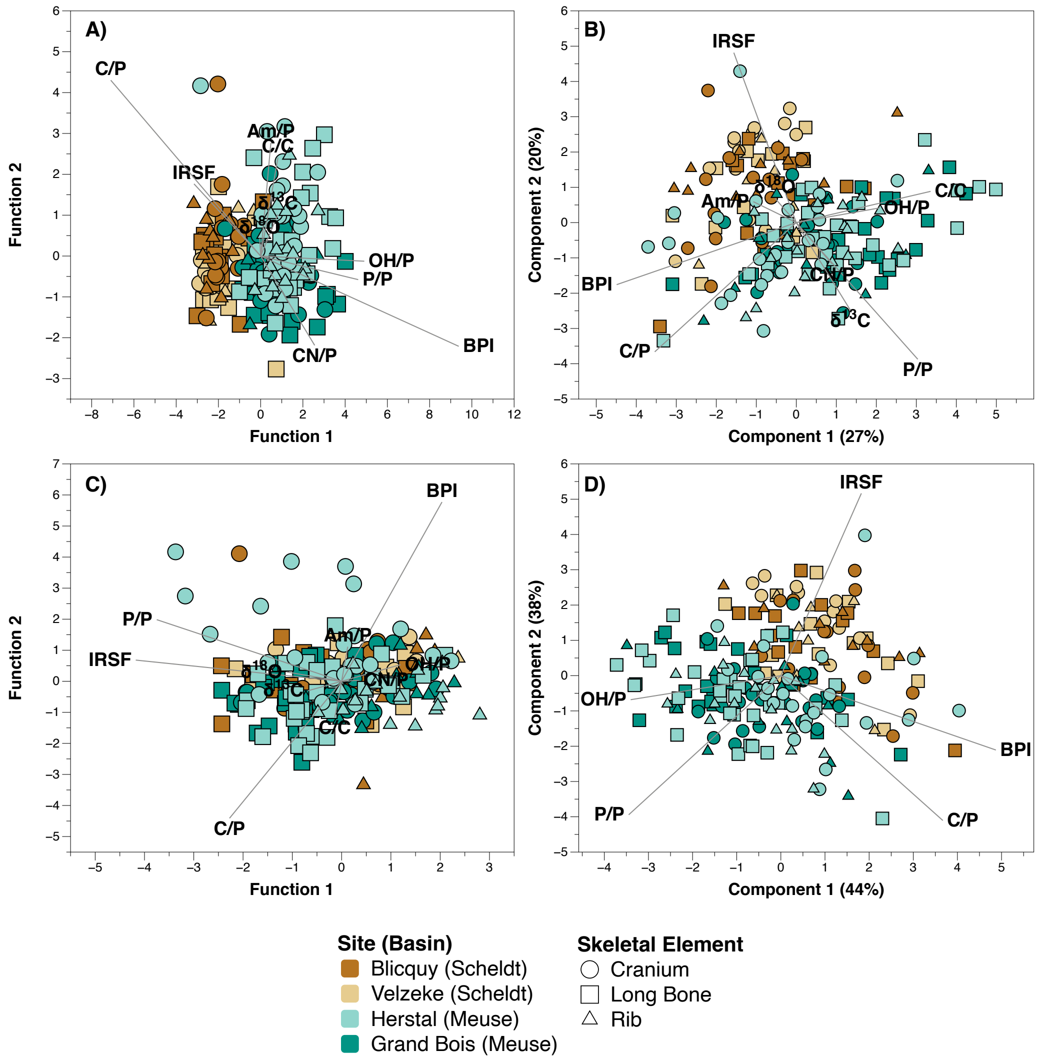

Figure 4. Linear discriminant analysis (LDA) and principal components analysis (PCA) results. Marker fill colours indicate site of origin, and marker shapes indicate bone elements. Gray vectors indicate weightings of variables in each model. Panel A: LDA among Velzeke, Blicquy, Grand Bois and Herstal using all available variables; Panel B: PCA among Velzeke, Blicquy, Grand Bois and Herstal using all available variables; Panel C: LDA among bone elements using all available variables; Panel D: PCA among Velzeke, Blicquy, Grand Bois and Herstal using a reduced set of infrared variables (BPI, C/P, OH/P, P/P, and IRSF).

All the skeletal elements from the sites of the Meuse basin present higher OH/P ratios than the skeletal elements from the sites of the Scheldt basin, except for the cranial bones from

Velzeke (Figure 5). The higher OH/P ratios in Herstal and Grand Bois are linked to lower IRSF values and the opposite was observed at Velzeke and Blicquy. It seems that there is a linear correlation between IRSF and OH/P (R^2 = 0.86) if the median value for the craniums from Velzeke is considered as an outlier. Comparing the results from Velzeke and Blicquy to those of Grand Bois and Herstal their difference in OH/P is statistically significant (Mann-Whitney U = 2442.0; $p < 0.01$).

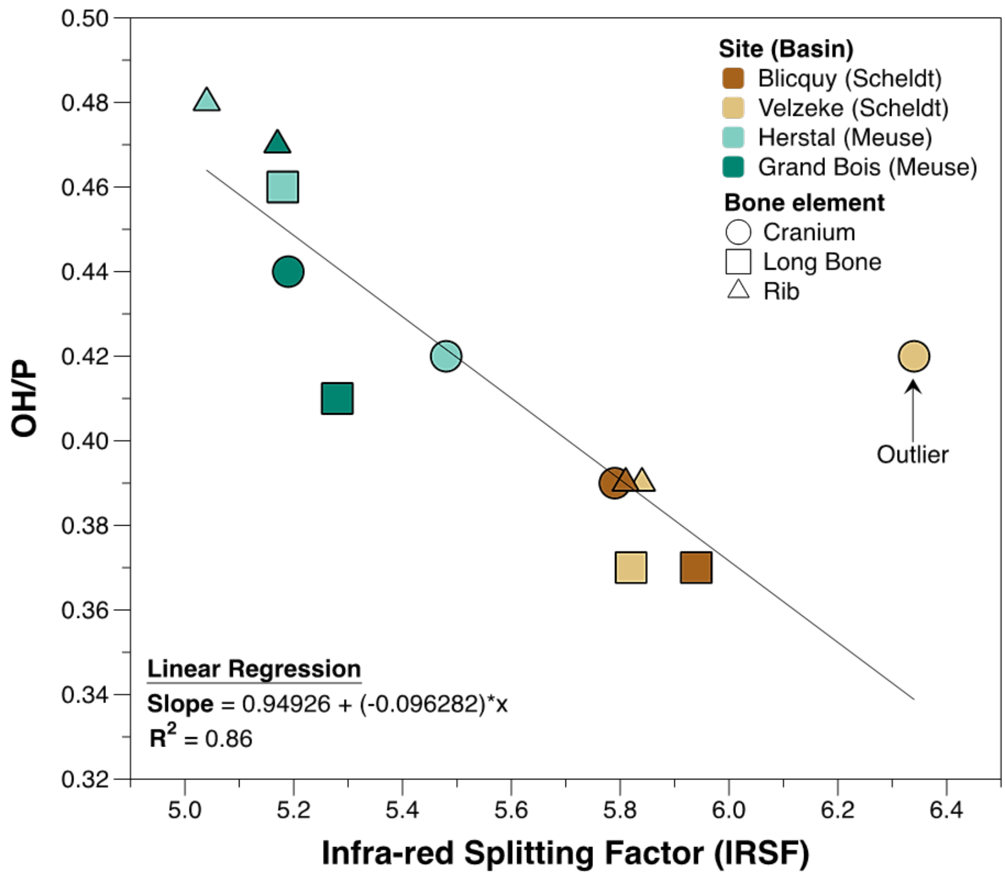

Figure 5. Median values of OH/P to IRSF for the different skeletal element categories of the studied sites.

Figure 6 shows the percentages of samples per skeletal element and site that have cyanamide content (CN/P) higher than 0.02. Regarding the infrared index of CN/P, a significance threshold of CN/P > 0.02 for detecting the presence of cyanamide (H_2CN_2) has been proposed by Salesse et al. (in press) based on an instrument comparison between an Agilent Technologies Cary 640 (no vacuum) and a Bruker Vertex 70v (under vacuum) on the experimental data of Snoeck et al. (2014b). Between the studied sites Grand Bois presents the highest percentage of samples containing cyanamide (55%) especially in long and cranial bones. Furthermore, when comparing the skeletal elements from all the sites, long bones always present the highest percentage of samples containing cyanamide and ribs the lowest (Mann-Whitney U = 1201.0; $p < 0.01$) with only one exception, the ribs from Velzeke.

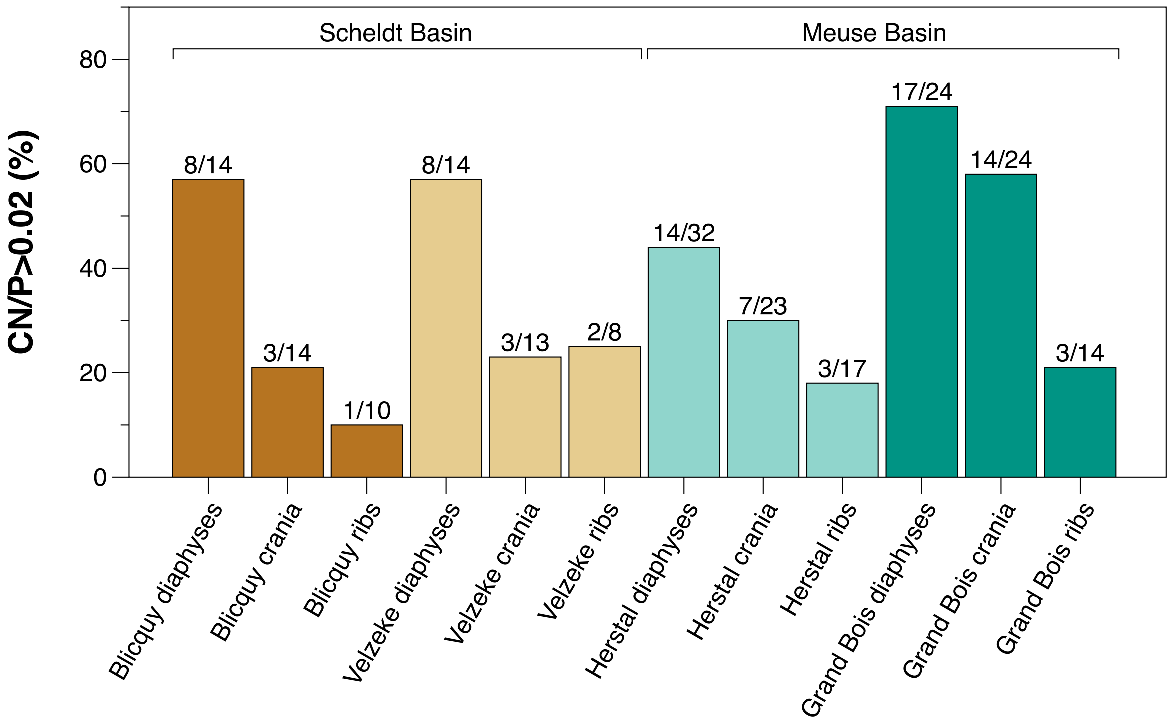

Figure 6. Percentages samples with CN/P>0.02 for the different skeletal element categories from Velzeke, Blicquy, Grand Bois and Herstal.

Discussion

Differences between adults and non-adults in Herstal

An interesting observation of this study is related to the relatively high number of non-adult individuals in Herstal (Table 1; see Sabaux et al. 2021) and the differences in the $\delta^{13}C$ values between adults and non-adults. As mentioned, the differences in cremation settings between adults and non-adults could only be examined at Herstal due to the very low number of identified non-adults in the other three sites (only one or two). The lower $\delta^{13}C$ values of the non-adults could be related to the lower density of the cortical bones and the different rate of bone turnover of non-adults in combination with the low body mass which allows bones to be exposed faster to the carbon exchange with the combustion atmosphere and mostly with the fuel that was used during cremation. An alternative explanation could be related to the use of a large amount of wood compared to the size of the body of non-adult individuals. In this case, it should be assumed that the person(s) who performed the cremation used the same amount of fuel for a child cremation as they would have used in an adult cremation. The last scenario could be related to the level of experience of the cremation operator(s) regarding the cremation of non-adult individuals, indicating that a different funerary ritual was carried out for non-adults. In any case, more research is necessary, especially in cremated individuals with determined ages to investigate whether age-at-death and the different rate of metabolism of the adult and non-adult bones influence the $\delta^{13}C$ and $\delta^{18}O$ values during combustion.

Differences within basins

The variability in cremation settings within the same basin is examined to investigate potential differences in the way cremation was performed on a local scale. Interestingly, there were no observable intra-basin differences in cremation conditions following the variables of biological sex and cremation deposit type.

Furthermore, no statistically significant differences on isotopic and infrared data were noticed between the sites of the Scheldt basin but only between the sites of the Meuse basin. Indeed, intra-basin variations between Herstal and Grand Bois were detected regarding the CN/P of different skeletal elements (Figure 6). Between the two sites, Grand Bois presents the highest cyanamide percentage in all the bone type categories. The presence of cyanamide (H_2CN_2) has been linked to reducing conditions (i.e. low oxygen availability), to the presence of ammonia during the combustion and/or to the position of the body on the pyre (Zazzo et al. 2013; Snoeck et al. 2014b; Marques et al. 2021; Salesse et al. in press). In experimental studies, higher CN/P have been linked to the presence of leather garments worn by the deceased during the burning process (Salesse et al. in press). This observation could suggest that in Grand Bois the individuals were burned wearing more and/or thicker clothes compared to Herstal. Another alternative would be that the pyres were slightly larger in Grand Bois than Herstal, which could lead to more reducing conditions and/or that the position of the body on the funerary pyre was different between the two sites. The slightly higher $\delta^{13}C$ values seen in Grand Bois, especially for the long bones, further indicate the possibility to have more reducing burning conditions in Grand Bois compared to Herstal. More research is needed to confirm this hypothesis. A more detailed multi-skeletal sampling strategy is necessary in order to investigate the position of the body on the pyre and/or the pyre size.

A possible explanation for the homogeneity observed in the Scheldt basin compared to the slight variability in the Meuse basin could be related to the distance between the studied sites in each area. The sites of the Scheldt basin are located very close to each other (only 37 km) while the sites of the Meuse basin are more than 130 km from one another. In the Meuse basin, the intra-basin variability is an indication that despite the fact that similar cremation settings (e.g. fuel, temperature) were used per basin the individuals of each site adjusted the funerary practices to their own socio-cultural identity or that the knowledge exchange about the cremation settings was affected by the longer distance.

Differences between basins

Belgium lies at the border between two important cultural groups. It represents a unique location to investigate the variations in pyre technology and body management and to examine the transfer of knowledge regarding the cremation techniques during the Metal Ages. The LDA and PCA analyses of the infrared and isotopic results clearly shows differences between the Meuse and Scheldt basins (Figure 4), for which the infrared variables are the best discriminators and exhibit the most variance between the basins. The variability in cremation settings between the two basins are probably linked to the co-existence during the Metal Ages of the Atlantic and Continental cultural traditions which influenced not only the material culture of Scheldt and Meuse basins but also the funerary practices.

Based on the carbon and oxygen isotope analysis, median $\delta^{13}C$ values are generally higher (ca. 1.6‰), and a higher degree of variability in $\delta^{18}O$ values is present in bones from the Meuse basin compared to the Scheldt basin (Figure 2). This higher $\delta^{13}C$ values seen in the Meuse basin could be related to the use of different fuels (different tree species or trees growing in different environments/conditions; Fyfe 2007; Bonafini et al. 2013), and/or different amounts of fuel used for the funerary pyre. The variability in the range of $\delta^{18}O$ values suggests differences in the oxidation conditions during cremation even within the same site. These differences could be related to many factors such as the amount of the fuel used during cremation, the position of the body on the pyre and the structure, the location of the pyre in the environment (e.g. on a hill, in a structure, in a pit) or even the variable amount of wind during cremation. The location of the two sites of the Meuse basin on slopes could be related to better ventilation conditions during the cremation process. More research is, however, needed to define how these factors affect the $\delta^{13}C$ and $\delta^{18}O$ values during the burning process.

The infrared analyses further show significant differences between sites from the Scheldt and Meuse basins, where the samples from the Scheldt basin present higher IRSF and lower C/C median values (Figure 3). These two indices have been shown to be temperature dependent (Thompson et al. 2009; Snoeck et al. 2014b; Reidsma et al. 2016; Marques et al. 2018; van Hoesel et al. 2019), suggesting that the temperature reached during cremation was generally higher in the Scheldt basin than in Meuse basin. The use of different kinds and/or amounts of wood for the funerary pyre between the two basins, as already suggested for the differences observed in the $\delta^{13}C$ values, or a better knowledge on the pyre management during the burning process could explain the higher temperatures of burning in the Scheldt basin.

Another difference between the sites from the Scheldt and Meuse basin is related to the OH/P to IRSF median values of all the skeletal elements (Figure 5). The strong linear but inverse corelation between the OH/P and IRSF median values is puzzling. During the burning process, carbonates are lost and (partially) replaced by hydroxyl groups (Snoeck et al. 2014b; Mamede et al. 2018) into the bioapatite structure. The hydroxyl groups are smaller than carbonates having as a result an increasing atomic order and a more compact structure with higher IRSF values. However, here, higher OH/P are linked to lower IRSF which seem to contradict the previous statement. Clearly, it is not yet fully understood to what extent factors such as the temperature, the duration and the fuel affect the correlation between IRSF and OH/P ratio and more experimental research is necessary.

For the first time, clear variations in the cremation funerary practices between different cultural regions have been demonstrated by studying the structural and chemical changes on cremated bones using a combination of analytical techniques. The cremation conditions vary significantly between different cultural groups, represented here by the Scheldt and the Meuse basins. It seems that even though cremation was the dominant funerary ritual for the whole Belgian area during the Metal Ages, each basin had established its own cremation settings using different types and/or amounts of wood, different pyre size, and different temperature during the burning process. The variations in pyre technology reflect differences in technological aspects of cremation which could be related not only to the wood availability in each area but also to the skills and/or the specialisation of the person(s) who was managing the funerary pyre. However, it is not yet known to what extent these differences were influenced by the connections that the basins had with the Atlantic and Continental cultural traditions and/or if these differences are related to the skills and the specialisation of the person(s) who carried out the cremation. Did this person(s) have specific knowledge on how to reach temperatures high enough to burn a human body? Did they just use the available wood resources of the region, or did they know which type of wood was the most appropriate to reach high temperature? These kinds of questions should be taken into account while examining the structural and chemical changes that occur during combustion.

As indicated in this study, the combination of infrared and isotopic results with LDA and PCA analyses provides new insights into our understanding of funerary practices, especially for a chronological period where the written sources are absent. To investigate further the pyre technology and the body management during cremation, and therefore to understand the cultural influences into the different ways cremation was performed, examining a large number of archaeological sites from different chronological periods and geographical regions where cremation was used as a funerary ritual is necessary. The combination of experimental work with the multi-proxy analyses proposed in this study, and a detailed multi-sampling strategy is also necessary to assess the structural and chemical changes occurring during the burning process in individuals of different sex and age who were burned in different cremation settings by operators with different skills and/or specialisation.

Conclusion

This study demonstrates the possibility to assess the inter- and intra-site variability in cremation settings using a multi-proxy analysis (FTIR-ATR, carbon and oxygen isotope analysis) on cremated bones. For the first time, the investigation of chemical and structural changes on archaeological cremated bones from the Metal Ages could be linked to differences in cremation funerary practices. This variability in cremation settings provides new insights about the funerary practices for a period when written sources are absent and the only available information is provided by the study of the material culture. Slight variability was detected between the two sites of the Meuse basin, despite the fact that in general their cremation conditions, such as the temperature and the kind and/or the amount of fuels used during combustion were similar. The intra-basin variability indicates that the long distance between the two studied sites of the Meuse basin probably affected the knowledge exchanges regarding cremation settings. The LDA and PCA analyses of the infrared and isotopic results clearly show differences among the sites of the Scheldt and Meuse basins. These differences are linked to a variation in the way cremation was performed between the two basins, indicating that during the Metal Ages the burning process was not homogeneous in the Belgian region. Instead, it seemed to confirm the different cultural influences observed in ceramics and bronze artifacts. Although the location of Belgium at the crossroads between two cultural traditions (Atlantic and Continental) would probably influence the way cremation was performed in each basin other factors such as the wood availability in each region as well as the skills and/or the experience of the cremation operator could also affect the variation in pyre technology. For this reason, the research area and chronological framework should be expanded to explore the changes of cremations through time and space and to investigate how cremation conditions were affected by the different cultural traditions.

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