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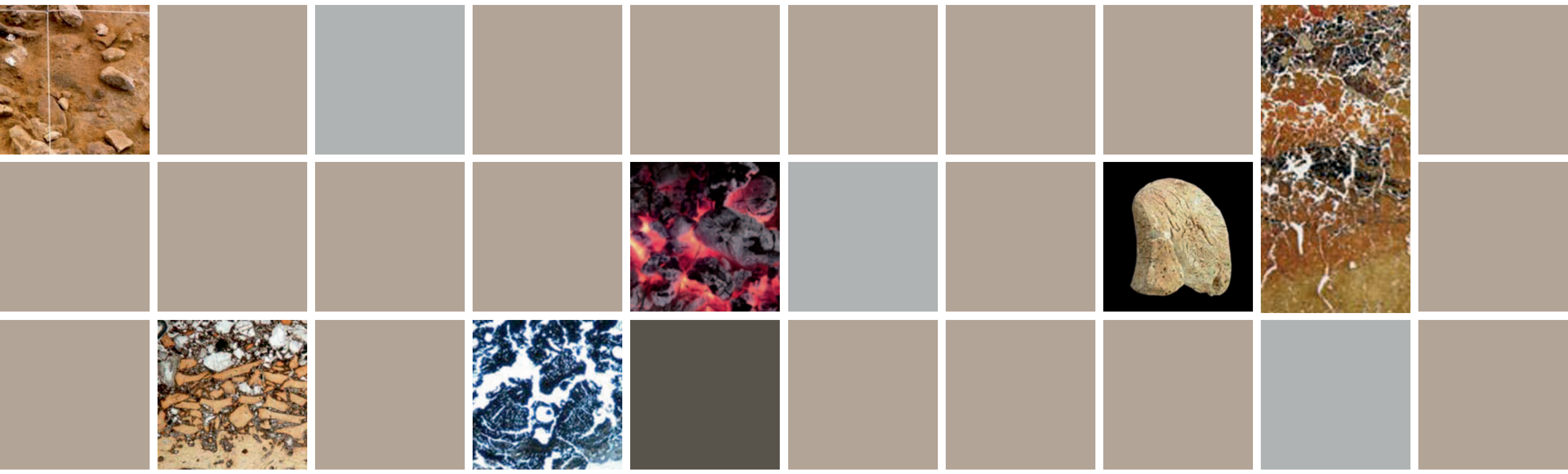
THE TAPHONOMY OF BURNED ORGANIC RESIDUES AND COMBUSTION FEATURES IN ARCHAEOLOGICAL CONTEXTS



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SUMMARY

INTRODUCTION

Isabelle THÉRY-PARISOT, Lucie CHABAL & Sandrine COSTAMAGNO

The articles in this volume are the result of a round-table entitled "The taphonomy of burned organic residues and combustion features in archaeological contexts", held on May 27-29, 2008 at the CEPAM (UPR 6130) laboratory in Valbonne, France. This encounter took place partly in the context of the Multidisciplinary Thematic Network, "Taphonomy", directed by Jean-Philip Brugal. The objective of this workshop was to review current and past taphonomic studies of burned organic remains and combustion features, to facilitate the sharing of knowledge and reference bases, as well as to initiate transdisciplinary collaborations and the development of collective research programs between different teams.

The burned "organic residues" at the heart of our reflections include diverse remains: human and animal bones, wood charcoal, seeds and fruits, phytoliths, etc. These can be the residues of intentional combustions (firewood, cremation, waste, for example) or accidental ones (proximity to a fireplace or wildfire). These remains document practices and knowledge and attest to technical choices and behaviors associated with a broad range of activities (funerary practices, food cooking, thermal treatment, waste management, etc.).

The materials that we study are the result of simultaneous or successive "processes" whose effects invariably lead to transformations, which, regardless of their nature, modify the assemblages and/or features in question and introduce biases that can influence our identification and/or interpretation of them. Having a greater knowledge of these "processes" and their effects is therefore a pertinent prerequisite to interpretation. But are we really speaking here of "taphonomy"?

The term Taphonomy (from the Greek *taphos*, burial, and *nomos*, law), first used by Ivan Efremov in 1940, designates in paleontology all the processes occurring after the death of an organism until its fossilization. Though these processes are rich and complex, the definition of the field of study is simple since it is traditionally considered as the transition of organics from the biosphere into the lithosphere. Archaeology has a wider definition of taphonomy, including not only the natural processes that modify the thanatocoenose, but also all the cultural choices and gestures that have an impact on the plant, animal or human materials, from their natural environment to their fossilization. This use of the term is not accepted by all members of the scientific community, some preferring to limit the field to its strict paleontological definition.

Meanwhile, in the case of burned organic archaeological residues and their associated features, is it pertinent to limit our studies to the combustion processes and post-depositional processes by which they are modified? The analysis of these materials indeed raises many questions. For example, it is necessary to take into account all the stages of the "life of a fireplace", even before the combustion itself. The intentional or unintentional burning of organic materials, fire maintenance methods, culinary practices and ash clearing, for instance, are all factors that are linked to the rhythms and actions that determine the final effects of the combustion on the remains. Therefore, isolating the strictly physical and chemical processes of combustion may prevent us from considering numerous other variables that also contribute to the nature of the final buried residues that we study.

And what about the gathering, splitting, storing and drying of wood? Shouldn't we expect these actions, also related to the making of fire, to be significant as well? In zooarcheology, should we take into account the carcass processes that occur before culinary actions or intentional bone burning? In carpology, an ensemble of human activities is associated with the unintentional burning of seeds and fruits (drying, roasting, culinary preparations), but must we look as far back as the threshing, storage and cultivation of seeds? In physical anthropology, should we consider the funerary rituals that occur before the act of cremation itself? In other words, when we study combustion, how far back into the process must our questions reach? In broader terms, we must not forget that the archaeological deposits that we attempt to understand are the result of numerous actions and natural processes whose effects cannot forcibly be isolated.

In addition, it is evident that the experiments we conduct to imitate taphonomic processes must also take into account the practices and actions associated with combustion. Whatever the combustion method employed, choices must be made concerning the manner of burning: for example, the type and duration of ignition, whether or not to refuel the fire, whether or not to concentrate the embers, etc. Even if we standardize our experiments by limiting the variables analyzed, we necessarily influence the results through our manner of proceeding.

There are thus many arguments in favor of considering the totality of processes that affect burned organic residues and combustion features, ranging from sociocultural choices to the effects of climatic-edaphic agents and of course the phase of combustion itself. For this reason, drawing on numerous archaeological examples and discussions inspired by experimentation, a significant goal of this publication is to clearly define what we can and what we wish to analyze.

This workshop was organized around three themes: "Combustion or waste discard structures", "Physical anthropology and taphonomy", "Archeobotany and taphonomy" and "Archeozoology and taphonomy".

Chapter 1, "Combustion or waste disposal features and taphonomy", presents four contributions. The article by B. Masson addresses interpretations of the morphology of combustion features. Based on modern examples of periglacial processes, archaeological examples and experiments, the author reveals formal convergences between periglacial structures and combustion features attributed to Mousterian groups at the site of Saint-Vaast-la-Hougue. The contribution by C.E. Miller, N.J. Conard, P. Goldberg and F. Berna proposes an interpretation of features in terms of the function and functioning of fireplaces. This work is based on a micromorphological analysis of experimental combustions whose results are applied to the study of the site of Hohle Fels in Germany. Two other contributions concern the detrital contents of combustion features. In their paper, D. Bosquet, A. Salavert and M. Golitko show how the floristic contents of Linearbandkeramik pits at three sites in Hesbaye (Liège Province, Belgium) can contribute information on the formation of pits and the formulation of hypotheses regarding the occupation duration of sites. Following a similar procedure, this time based on experimental data, G. Fiorentino and C. d'Oronzo study the formation of anthracological deposits and the contents of experimental combustion features to obtain information on the functioning of particular features (*escharon*) of the Sanctuary of Apollo at Hierapolis (Turkey) and the associated rituals.

Chapter 2, "Archeobotany and Taphonomy", includes five contributions. The first two present experimental studies of vegetal macroremains. The experimental analysis by M.-P. Ruas and L. Bouby addresses the effects of carbonization on carpological remains in function of diverse parameters such as temperature, heating duration, oxygenation and the condition of the seeds. The taphonomic biases generated by carbonization are evaluated for a range of cultivated and wild taxa, as well as other remains such as grain chaff. In their contribution, Théry-Parisot I., Chabal L., Ntimou M., Bouby L. and Carré A. present the results of wood combustion experiments in open fires. Through their analysis of the rate of disappearance of materials in carbonized residues and their fragmentation, they attempt to determine the degree of deformation of anthracological frequency spectra. In the domain of microremains, the contribution of C. Delhon proposes an experimental evaluation of the potential of phytoliths for the characterization of a carbonized ligneous biomass. This work analyzes the impact of ash dissolution processes, the difficulties associated with the taxonomic determination of the ligneous species phytoliths and the question of their origin (combustion or wood decomposition). The study of L. Marquer consists of a granulometric analysis of infra-millimetric wood fragments and proposes a quantification method based on image analysis, followed by its application to Paleolithic contexts in order to reveal the presence of these fine fractions in contexts in which wood charcoal fragments over 0.5 mm have disappeared through the actions of taphonomic processes. Finally A. Dufraisse, D. Sordoillet and O. Weller present an archeological, anthracological and micromorphological analysis of combustion features at the Neolithic site of *Poiana Slatinei* at Lunca (Neamt, Romania). In this work, the authors attempt to determine the origin, be it taphonomic or due to the salt production techniques employed, of the observed anatomical modifications of wood charcoals.

Chapter 3, "Archeozoology and Taphonomy", includes seven contributions. The first three specifically address the physico-chemical transformations of bones associated with heating. With the aid of various physico-chemical methods, I. Reiche describes modifications to the mineral phase of bone induced by heating and diagenetic processes. When applied to the archaeological remains from Chalain 19, these analyses reveal that analysis of the structural heterogeneities of of bones is an effective method for identifying the heating methods employed. Using infrared spectroscopy, M. Lebon proposes a new method based on the study of the $\nu_1\nu_3PO_4$ domain, which allows evaluation of three heating temperature ranges. This protocol can contribute to a more reliable identification of bones heated at low temperatures and a distinction of modifications related to heating from those of diagenesis in an archaeological context. The study conducted by A. Zazzo indicates that the mineral fraction of bones heated at high temperatures cannot be used for the reconstruction of diets due to a modification of the $\delta^{13}C$. On the other hand, calcined bone is a reliable material for radiocarbon dating, allowing us to test, in certain cases, the reliability of dates obtained on carbonized bones whose age can reduced in certain burial contexts. The four other contributions treat burned bones on a macroscopic scale according to two distinct approaches, one experimental Costamagno *et al*.; Gerbe), the other archaeological (Rillardon & Bracco; Morin). The objective of the experiments realized by M. Gerbe was to document the modification of burned bones exposed to the actions of atmospheric agents. She observed a high fragmentation of the materials, particularly for calcined, spongy bone fragments. In their paper S. Costamagno, I. Théry-

Parisot, D.Kuntz , F. Bon and R. Mensan analyze the impact of a prolonged combustion on bones used as fuel. An increase in the heating duration leads to an increase in the intensity of fragmentation and combustion, in particular for the spongy portions. Since these latter are more sensitive to the actions of certain processes, depending on the taphonomic history of the assemblage, a potential use of bone as fuel can be masked. The contribution of M. Rillardon and J.-P. Bracco presents a study of calcined bones found in a context unfavorable for the preservation of bones. Finally, E. Morin discusses the consequences of bone combustion for interpretations of skeletal part representations. He emphasizes the problem of equifinality in the use of bone as fuel and the differential preservation of bones.

A synthesis of the studies in each domain emphasizes the need to combine experimental approaches and analytical tools in order to reach a level of analysis that allows an integrated approach to combustion features and residues.

To conclude, we would like to express our gratitude to all of the protagonists of this project who contributed to its realization. First, both the organization of the workshop and the publication of these proceedings benefitted from the financial support of numerous institutions: the RTP Taphonomy, the CEPAM (UMR 6130, Sophia-Antipolis), LAMPEA (UMR 6636, Aix-en-Provence), TRACES (UMR 5608, Toulouse) and CBAE (UMR 5059, Montpellier) laboratories, the MSH's of Nice and Toulouse, ArScAn (UMR 7041, MAE Nanterre), the University of Neuchâtel/Laténium, the University of Liège, INRAP and the SRA Midi-Pyrénées. We also wish to thank all of the contributors who through their interest in this theme insured the scientific success of the workshop both in terms of the quality of the presentations and the richness of the discussions. The scientific organization of this encounter, as well as the significant editorial work, were realized with the active participation of the scientific committee, to whom we express our heartfelt thanks: Jean-Philip Brugal (LAMPEA, UMR 6636, Aix-en-Provence), Claire Delhon (CEPAM, UMR 6130, Valbonne), Henri Duday (PACEA, UMR 5199, Talence), Paul Goldberg (Boston University), Marie-Pierre Ruas (CBAE, UMR 5059, Montpellier), Brigitte Talon (IMEP, UMR 6116, Aix-en-Provence) and Benoit Devillers (UMR 5140, Lattes). Finally, we express our appreciation to the editorial committee of *P@lethnogie*, which very favorably welcomed our request to publish these proceedings, including Yann Beliez, who realized the layout, for the rigor of his editorial work, and Magen O'Farrell for the ensemble of French to English translations. We also thank Jeannine Francois and Françoise Trucas who efficiently aided us in the organization of the workshop, as well as Monique Clatot and Sabine Sorin for the realization of the editorial work.

CHAPTER 1

Combustion or waste disposal features and taphonomy

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COMBUSTION FEATURES AND PERIGLACIAL STRUCTURES: A NEW TAPHONOMIC ANALYSIS OF MOUSTERIAN COMBUSTION FEATURES AT SAINT-VAAST-LA-HOUGUE (50)

Bertrand MASSON

Abstract

The Mousterian site of Saint-Vaast-la-Hougue (Manche), excavated by Gérard Fosse in the early 1980's, has yielded around thirty combustion features. These features were excavated, described and interpreted without sufficient consideration of the periglacial processes that occurred during and after the human occupations. Based on observations of modern periglacial processes in active contexts, archaeological examples from sites in the Nord-Pas-de-Calais region and experiments conducted at high altitudes by A. Pissart (1973 to 1987) and researchers in the ACR program "Taphonomy of Middle Palaeolithic assemblages in periglacial contexts" and "The Palaeolithic in the Quercy" (2004-2007), we reveal evidence of formal convergences between the periglacial structures and the forms and functions of the combustion features attributed to the Mousterian at Saint-Vaast-la-Hougue.

Keywords : cryoturbation, cryoexpulsion, gelifluction (periglacial solifluction), Mousterian, periglacial, cryostatic pressure, solifluction, polygonal ground, combustion feature, taphonomy

Introduction

The Mousterian site of Saint-Vaast-la-Hougue is located in the Nord Cotentin, on the eastern coast of the Hougue peninsula (fig. 1). It was excavated by Gérard Fosse from 1978 to 1984. Several essential elements characterize this site, whose occupations extend from the end of the Eemian (stage 5e) to the beginning of the Weichselian periglacial (stage 4). First, the combustion features are exceptional for this period in terms of both their number and their formal diversity. Second, the site is spatially structured into two distinct zones separated by a granitic butte: one is a “habitat” zone, while the other is exclusively devoted to fire-related activities. Finally, a remarkable number of periglacial phenomena are present at this site. This last aspect was not considered in previous interpretations of the features and it thus appeared necessary to conduct new analyses of them in light of studies (in which the author of this paper is a participant) conducted in the context of the ACR programs “Taphonomy of Middle Palaeolithic assemblages in periglacial contexts”, directed by L. Vallin and “The Middle Palaeolithic in the southern Aquitaine: taphonomic studies in modern periglacial contexts”, directed by P. Bertran and J.-P. Texier.

Fig. 1 - Location of La Hougue Island.

Topographic situation and chronostratigraphy

Topographic situation

La Hougue was a small island until the end of the 17th century when a military engineering project reattached it to the mainland (fig. 2). It is composed of a granitic bedrock base covered with late Quaternary formations that surround and connect three granite outcrop buttes:

- to the north, an elongated butte, oriented N-S, 20 m NGF at its highest point;
- to the south, two buttes, one with a very steep profile, the other larger and with a weaker slope. Their highest points are 22 and 18 m respectively.

Fig. 2 - Areal view of La Hougue Island with the location of the two principal excavation sites.

The Mousterian site studied is located at the foot and to the southwest of the southernmost butte (fig. 3). The archaeological excavation is located at the top of the current beach on a strip of land (3 m wide) preserved by the 17th century construction work.

Chronostratigraphy

The excavation of around fifteen test pits distributed over the entire surface of the island (fig. 3) allowed J.-P. Lautridou, J.-P. Coutard, B. Van Vliet-Lanoë and J.-C. Ozouf of the *Centre de Géomorphologie du CNRS* in Caen to define the chronostratigraphy summarized in figures 4 to 8.

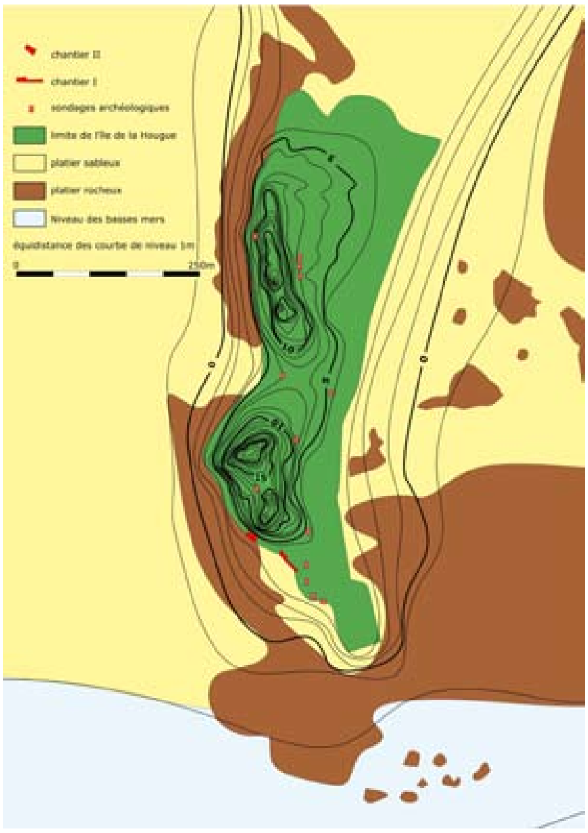

Fig. 3 - Location of the archaeological excavations overlaid on a map of La Hougue Island before the military construction work, reconstructed based on ancient maps (CAD Bertrand Masson).

The Mousterian occupations

With no apparent discontinuity, the Mousterian occupations are contained in the horizons from the top of the low level beach and the base of headlands D1-D2. These horizons correspond to the climatic degeneration during which periglacial conditions slowly developed at the beginning of the Weichselian. The state of preservation of the industry allowed the distinction of two large groups (Fosse 1982):

- the lower horizons, which contain a lithic assemblage with numerous cores and tested nodules, along with a few tools consisting mostly of notches, denticulates and scrapers. The objects have a thick, white patina and frequent alterations caused by freezing. The Mousterians knapped mediocre quality flint nodules from the beach. The features include a knapped flint concentration, unworked stones arranged in half circles and combustion features.
- the upper horizons, which contain an assemblage that is slightly modified by frost action and has a thin patina. The raw material is of a better quality and does not come from the beach, but from sedimentary contexts exposed by retreating sea levels. Scrapers are dominant in this Levallois-type industry, which can be attributed to the Typical Mousterian. The only features are knapped flint concentrations. The two *loci* studied (ChI et ChII) are near one another (around 40 metres apart) and are situated on either side of a small granite butte (fig. 9). Even if the narrowness of the excavated zones prevents us from reliably interpreting the site (fig. 10 and 11), a few ethnographic observations can be made. Though the occupations of the lower levels (beach horizons and C1) are similar, the occupations of level C2 are different. In Chantier (Site) I, the isolated combustion features are associated with knapped flint concentrations, numerous tools and granite blocks arranged in a half circle, suggesting a domestic activity zone and probably a habitat. The organization of Chantier II is different. Up against the dune (level C1) an ensemble of combustion features is superimposed over a depth of nearly one meter. Next to ashy pockets, there are true organized, which are bordered by granite stones. The lithic industry is poor and often reduced into debris by fire. This part of the site of “La Hougue” was dedicated to specialized activities related to fire (Fosse *et al.*, 1986), (Thiébault *et al.*, 1988).

The combustion features

Numerous combustion features were identified: 14 at Chantier I, over a surface of 18 m^2, and 20 at Chantier II, over 30 m^2. These are minimum numbers since several features, particularly at Chantier II, are interpreted as combustion complexes composed of several indistinct hearths. These features were classed into four types (Thiébault *et al.*, 1988).

Agglomerations of ash and charcoal

These are simple pockets of ash and charcoal materials. Some of these features are rather thin and

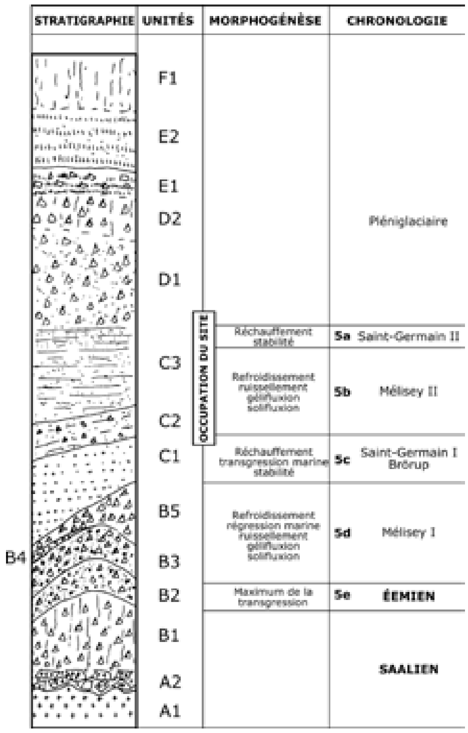

Fig. 4 - Synthetic stratigraphic sequence of the Saint-Vaast-la-Hougue peninsula, based on the work of J.-P. Lautridou, J.-P. Coutard, B. Van Vliet-Lanoë and J.-C. Ozouf of the Geomorphology Center of the CNRS in Caen.

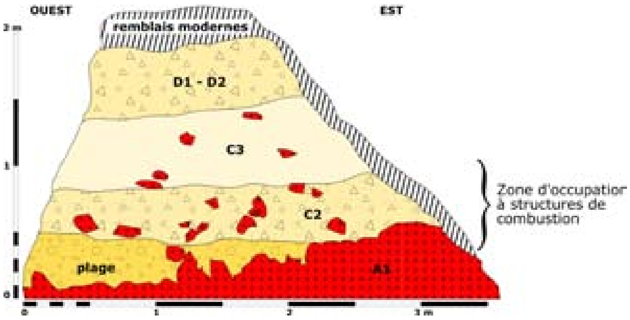

Fig. 5 - Stratigraphic sequence of Chantier I: the legends are in the text (CAD Bertrand Masson).

Fig. 6 - Photo of the stratigraphic profile of Chantier I (Photo Gérard Fosse).

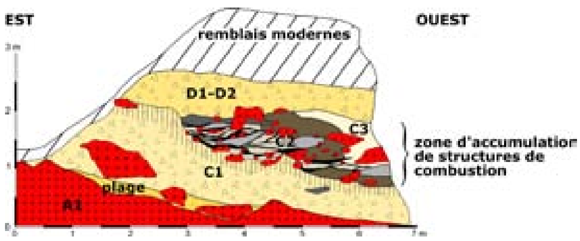

Fig. 7 - Stratigraphic sequence of Chantier II: the legends are in the text (CAD Bertrand Masson).

Fig. 8 - Photo of the stratigraphic profile of Chantier II (Photo Gérard Fosse).

Fig. 9 - View of Chantiers I and II taken from the beach (photo Gérard Fosse).

Fig. 10 - View of Chantier I in the process of excavation, showing the narrow surface of the preserved site. The excavated surface covers 18 m^2 (photo Gérard Fosse).

Fig. 11 - View of Chantier II in the process of excavation. The excavated surface is 30 m^2 (photo Gérard Fosse).

composed of sediments that are slightly darker than the surrounding sediments and contain wood charcoal (fig. 12 and fig. 14). The most evident agglomerations cover a surface of up to 1 m^2 and consist of a coalescence of different types of ashes containing varied amounts of charcoal (fig. 15 and fig. 16). The absence of a zone of rubified sediment under the ashes led the excavators to interpret these as dumping zones.

Fig. 12 - Agglomeration of thin ash and charcoal materials that are characterized by a darker and redder sediment colour and accompanied by wood charcoal fragments and balls of hard red sediment (photo Gérard Fosse).

Fig. 13 - Pocket of wood charcoal in an ashy gray silt (photo Gérard Fosse).

Fig. 14 - Pocket of large wood charcoal fragments in a whitish silt (photo Gérard Fosse).

Fig. 15 - Ensemble of white to grey ash zones (photo Gérard Fosse).

Fig. 16 - Zone of ashy silt (photo Gérard Fosse).

Simple hearth

These are hearths with no border, composed of an accumulation of combustion materials in a simple depression in the ground. These features have an ovular to circular form and are shallow (between 10 and 15 cm deep). They have a diameter of 30 to 100 cm (fig. 17 to fig. 20).

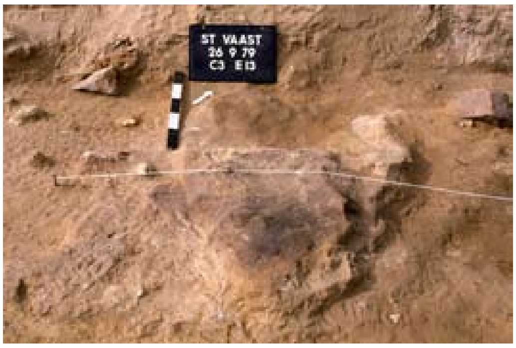

Fig. 17 - View of hearth DE13 when it was discovered. This is a large sub circular hearth approximately 1 m in diameter in a shallow (10 cm) intentionally dug pit (photo Gérard Fosse).

Fig. 18 - View of hearth at the end of its excavation: the ashy fill was removed (the solid line indicates the upper limits of the pit); the flint cobble in the middle was fractured by the fire. In the 1980 report, Gérard Fosse noted that “the whitish materials visible (to the right, outside of the hearth), have not yet been explained, but seem to be related to fire” (photo Gérard Fosse).

Fig. 19 - Small hearth in a pit: located on the border of the slope, half of the hearth was eroded by the sea. In the photo, the pit has been half emptied (photo Gérard Fosse).

Fig. 20 - Small hearth in pit D18: located on the border of the slope, half of the hearth was eroded by the sea (the arrow indicates an older combustion feature). All of the ash has been removed from the pit (photo Gérard Fosse).

Hearth with a stone border

Gérard Fosse (*in* Thiébault *et al.*, 1988) describes these hearths as “Hearths with a border of granite blocks simply pushed into place in order to obtain a central space that is ovular or circular and free of stones”. “This concerns nearly all the hearths constructed in the heterometric slope deposit of Chantier II; these fire places almost always include a deepened area” (fig. 21 to fig. 23).

Fig. 21- A constructed hearth in Chantier II : the granite blocks of the headland were pushed away in order to free a central space that has traces of combustion (ash and charcoal) (photo Gérard Fosse).

Fig. 22 - A constructed hearth in Chantier I with granite blocks pushed to the periphery and forming a circle (photo Gérard Fosse).

Fig. 23 - Hearth constructed in the headland of Chantier II (photo Gérard Fosse).

Combustion complex

These are extended combustion features with unclear limits, making them difficult to excavate and interpret.

They are composed of strongly imbricated and overlapping combustion zones. Only one such feature was found at Chantier I, while they are the most frequent type found at Chantier II (fig. 24, fig. 25 and fig. 26).

The periglacial processes

Solifluction

The features were excavated, described and interpreted without sufficient consideration of the periglacial processes that occurred during and after the human occupations at Saint-Vaast-la-Hougue. For example, Gérard Fosse, Dominique Cliquet and Gérard Villegrain wrote in 1986 that "...the archaeological levels were not disturbed by natural phenomena posterior to the human occupation, as they were in the silts of the northern Paris Basin, or were at most affected by "frost-creep", which resulted in only minor disturbances, and [...]the "features" were consequently preserved" (Fosse *et al*., 1986). Recent studies, especially since the experiments conducted by "TRANSIT" in modern, active periglacial contexts (Texier *et al*., 1998), have shown that modifications to prehistoric assemblages in periglacial contexts can be rapid and significant and that they can deform anthropogenic features both horizontally and vertically (Texier, 2001). The different processes, which rarely act alone, can result in the creation of anthropogenic pseudo-features (Texier, 2000). New analyses of the Palaeolithic sites of Baume-Vallée (Bertran, 1994), Combe-Capelle (Texier & Bertran, 1995) and La Ferrassie, (Texier, 2001) showed the presence of periglacial phenomena at these sites. Therefore, contrary to previous interpretations, these were not simple gravitational deposits and their development had consequences for the preservation of the archaeological levels.

At some point during their history, most Palaeolithic sites contemporary with periglacial climates met the conditions necessary for the functioning of periglacial processes that could modify their contents. A geomorphological study of Saint-Vaast-la-Hougue showed that this site does not escape this rule. It thus appeared necessary to conduct new analyses of its features in order to determine the possible effects of periglacial phenomena.

Fig. 24 - Combustion complex in Chantier I: extended combustion zone with numerous combustion pits that could have been used successively (photo Gérard Fosse).

Fig. 25 - Combustion complex in Chantier II (photo Gérard Fosse).

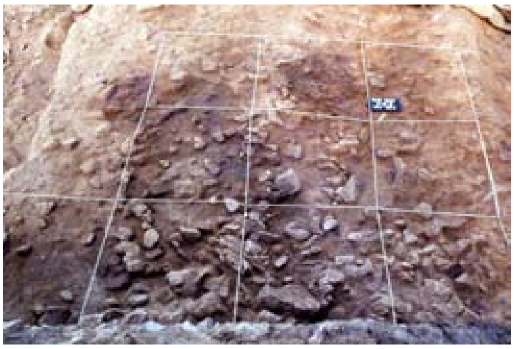

Fig. 26 - Combustion complex in Chantier II (cliché Gérard Fosse).

Definition and modern examples

Solifluction (figs. 29 and 30) is a slow, downslope mass movement of loose detrital materials under the action of freeze-thaw cycles (Bertran & Coutard, 2004). It is the result of several processes:

Frost creep: a mass movement of sediments that occurs when they are lifted perpendicular to the slope as a result of an increase in the volume of ice during its crystallization, followed by their more or less vertical subsidence as the ice thaws (Washburn, 1967).

Pipkrakes (needle ice): ice needles that form on the ground surface, perpendicular to the slope, under stones, vegetal fragments or blocks of sediment. These materials can be uplifted by the pipkrakes during their formation. A pipkrake can lead to the vertical tumbling an object in front of its original position, caused its displacement (Washburn, 1973, 1979). Pipkrakes are responsible for the movement of large materials on the surface of solifluction flows (fig. 27).

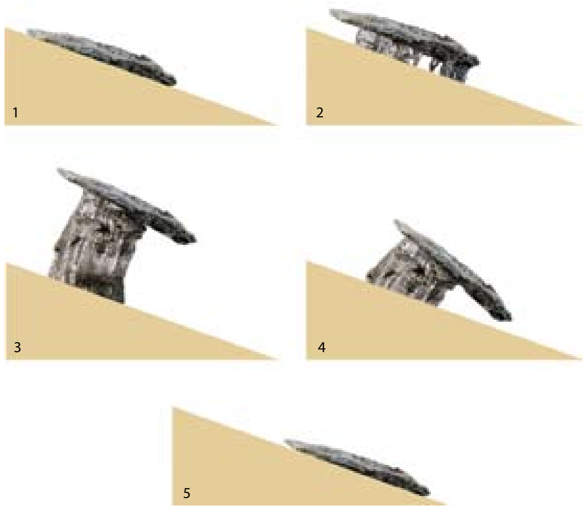

Fig. 27 - Schema explaining the movement of a stone by pipkrakes: 1 – initial position of the stone; 2 – beginning of the uplifting of the stone, perpendicular to the slope, by a pipkrake; 3 – end of the uplifting; 4 – thawing and vertical subsidence of the stone; 5 – final position of the stone, which was moved forward (CAD Bertrand Masson).

Periglacial solifluction (gelifluction): sediment flow that occurs during thawing when water is liberated faster than it can be drained (Baulig, 1956).

Solifluction flows that form on slopes between 2° and 35° progress slowly and in the same manner as the tracks of a tractor (Bertran, 2004). The distribution of movement rates, which are faster on the top and central part of the flow, than at its base, edges and front, result in a progressive rolling of the front into the form of a lobe (Bertran & Coutard, 2004; Coutard & Ozouf, 1996; Francou & Bertran, 1997) (fig. 28). One of the consequences of this type of movement is that stones can be stood up vertically at the front of the flow (figs. 31 and 38). At the front of the flow, we can thus expect to find semi-circular assemblages of vertically positioned stones.

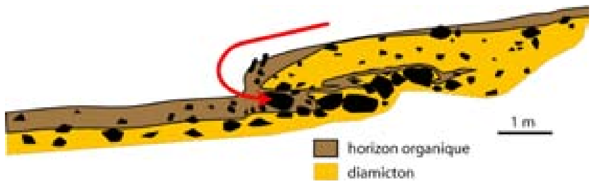

Fig. 28 - Schematic profile of a solifluction flow after Bertran & Coutard, 2004.

Fig. 29 - Lobed solifluction flow, Massif du Chambeyron, French Alps, altitude 2600 m, (photo Bertrand Masson).

Fig. 30 - Layered solifluction flow, Massif du Chambeyron, French Alps, altitude 2800 m, (photo Bertrand Masson).

Archaeological example: the Mousterian site of Saint-Amand-les-Eaux

This site was excavated by Philipe Feray in 2007. It is located on the eastern part of the Scarpe plain at the foot of a hill (Mont-des-Bruyères) composed of Eocene sands. Its maximum altitude is 30 m.

Since the study of this site is still in progress and the excavation has not yet been published, I will only briefly describe it here. According to current knowledge, it appears that the main function of this

Fig. 31 - Close-up of a solifluction flow showing the vertically positioned stones at the front of the flow, Massif du Chambeyron, French Alps, altitude 2600 m (photo Bertrand Masson).

Fig. 32 - Close-up of a solifluction flow showing the vertically positioned stones at the front of the flow, Massif de Rubren, French Alps, altitude 2600 m (photo Bertrand Masson).

Fig. 33 - View of the site of Saint-Amand. Note the front of the solifluction layers that traverse the site (photo Philippe Feray).

Fig. 34 - Profile view of the front of the solifluction layer at Saint-Amand-les-Eaux (photo Philippe Feray).

Fig. 35 - Close-up of the front of the solifluction layer at Saint-Amand-les-Eaux (photo Philippe Feray).

site was as a biface production workshop dated to stage 4 (Deschodt *et al.*, 2006). The average slope is 2.43° and it is traversed by a solifluction level (fig. 33). The meticulous excavation of this level revealed its organization, including the half-circle arrangement of stones at the front flow and the vertical position of sandstone blocks (figs.34 and 35).

Formal convergences

It is difficult, 30 years after an excavation, to re-examine a site from a different perspective as some data is forcibly lacking. The excavation of Saint-Vaast-la-Hougue was realized by successive horizontal levels, or *decapages*, a few centimetres deep and the ensemble of artefacts of natural and human origin were recorded and removed after each horizontal stripping. These conditions do not allow us to observe certain phenomena, such as a solifluction flow, in three dimensions. The object orientations (Bertran & Lenoble, 2002), which could have been used to distinguish the levels disturbed by solifluction, were not recorded. Nonetheless, comparisons of photos of the features at Saint-Vaast-la-Hougue with modern and ancient solifluction structures show formal convergences. Though not sufficient to deny the role of humans in the realization of partitions around the hearths, these formal convergences suggest the possibility of a natural process. Under certain conditions, such as those at Saint-Vaast-la-Hougue, natural phenomena can produce arrangements of vertical stones in a half-circle, which we can thus call "pseudo-features" (fig. 36).

Polygonal grounds

While archaeologists are beginning to recognize the role of solifluction in the disturbances of some prehistoric sites, the action of polygonal grounds in the redistribution of archaeological artefacts is still rarely considered, even if their effects can be quite significant (Masson & Vallin, in press).

Definitions and modern examples

The formation of polygonal grounds requires a very weak slope, a heterometric superficial formation and a high water content. Under these conditions, patterned grounds (in the geomorphological sense) result from the combined actions of the processes:

Segregation ice: in soils, water crystallizes in a discontinuous manner, forming ice lenses parallel to the freeze front. These lenses increase in size as water migrates toward the freeze front (cryosuction), thus causing the ground to swell (Pissart, 1987; Van Vliet-Lanoë, 1988). The ancient presence of segregation ice is shown by the leaflike structure of the sediment (fig. 38).

Cryoexplusion: in soils subject to freeze-thaw cycles, stones tend to be expulsed toward the surface (Pissart, 1987; Coutard & Van Vliet-Lanoë, 1994). The speed of the phenomenon is related to the number of freeze-thaw cycles and the size of the objects. The largest objects are cryoexpulsed the fastest, as has been shown in laboratory experiments by Coutard and Van Vliet-Lanoë (Coutard & Van Vliet-Lanoë, 1994).

Pipkrakes : see above.

Patterned grounds « appear as polygonal forms outlined by stony elements surrounding "islands" of finer materials. Within the rows of stones, which are often vertically positioned, there is sometimes a very clear granulometric sorting" (Pissart, 1987, p. 56), (fig. 39 and fig. 40). Experiments realized by Albert Pissart from 1968 to 1975 to identify the evolutionary processes of polygonal grounds showed that:

- the displacement rates on the surface due to the action of pipkrakes could attain 4 cm in 2 years;

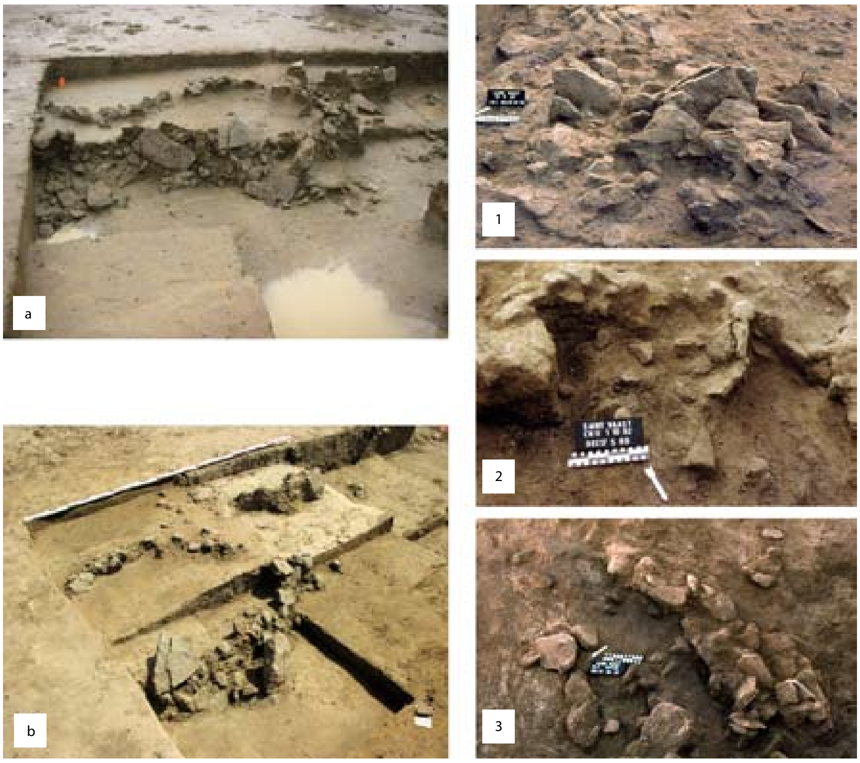

Fig. 36 - Comparison of the front of the solifluction layer at Saint-Amand-les-Eaux, on the left (photo Philippe Feray), and the constructed hearths at Saint-Vaast-la-Hougue (photo Gérard Fosse) on the right. Photo b clearly shows that a flat excavation in this type of level can result in the creation of false features.

- the speeds of the cryoexpulsion of buried stones was proportional to their size and attained 1 cm per year for gravels;
- a field of polygons destroyed by mixing could be reconstituted in 7 years.

Fig. 37 - Polygonal grounds on the Massif du Chambeyron, French Alps (photo Luc Vallin). Three generations of polygons are imbricated, the smallest measuring around 30 cm in diameter, the middle ones around 1 m and the largest between 2 and 3 m.

Fig. 38 - Saint-Amand-les-Eaux, profile of the archaeological level. The ancient presence of segregation ice is shown by the presence of iron oxides. The tag indicates the leaf-like structure of the sediment (photos Bertrand Masson).

Fig. 39 - Polygon of the Massif du Chambeyron, French Alps (Photo Luc Vallin). The vertical position of the stones in the walls of the polygon is clearly visible in this photo.

Fig. 40 - Polygon of the Massif du Chambeyron, French Alps (photo Luc Vallin). The granulometric sorting is visible in this photo.

Fig. 41 - Saint-Amand-les-Eaux, surface of the solifluction layer. The leaching of the fine sediments by rain allows us to observe the polygonal grounds that formed on the gravel surface of the solifluction flow (photo Philippe Feray).

Archaeological example: the Mousterian site of Saint-Amand-les-Eaux

There are numerous periglacial structures at the Mousterian site of Saint-Amand-les-Eaux. In addition to the solifluction level and segregation ice lenses already mentioned, several polygonal networks formed within the silt-sand sediment (fig. 42) at the surface of the solifluction level (fig. 43). These small polygons, 10 to 30 cm in diameter, modified the archaeological level (fig. 44).

Experimental demonstration

In order to better understand the redistribution of artefacts by polygonal grounds and to determine the speeds of these transformations, an experiment was realized at the Gavarnie Massif (Pyrenees) as part of the ACR program entitled « Taphonomy of Middle Palaeolithic lithic assemblages in periglacial contexts", directed by Luc Vallin (Masson &

Fig. 42 - Saint-Amand-les-Eaux, surface of the solifluction layer. Close-up of the polygons; the vertical position of the sandstone blocks and the granulometric sorting are clearly visible (photo Philippe Feray).

Fig. 43 - Saint-Amand-les-Eaux, surface of the archaeological level. Close-up of the polygon network of frost cracks (photo Philippe Feray).

Fig. 44 - Saint-Amand-les-Eaux, biface fabrication concentration disturbed by the polygonal network. The flakes are pushed into the walls of the polygons (photo Philippe Feray).

Vallin, in press). A pit measuring 1 m^2 and 10 cm deep was dug into the location of five small polygons (0.2 – 0.6 m diameter) and filled with silt (fig. 45). In 2005, 326 knapped flint flakes were regularly distributed over the surface of the pit so that they uniformly covered the silt fill (fig. 46). The object positions realized in 2006 show a reorganization of the objects that suggest the formation of polygons, more

Fig. 45 - Gavarnie experiment, initial polygonal ground: the polygons are small (20 to 60 cm diameter), the granulometric sorting is slight and the network is enhanced by the vegetation that is growing only in the walls (photo Luc Vallin).

Fig. 46 - Gavarnie experiment, september 2005, view of the silt pit covered with knapped flint (photo Luc Vallin).

or less following the ancient pattern (fig. 47). The average object displacement, 8.25 cm, is much higher than that recorded by Albert Pissart. This could be explained by the high frost susceptibility of silt and a large number of freeze-thaw cycles (between 30 and 45 freeze-thaw cycles recorded by the team directed by P. Bertran and J.-P Texier on their nearby experimental sites).

Fig. 47 - Gavarnie experiment, september 2006, view of the pit after around 40 freeze/thaw cycles. The artefacts have moved and are now located along the walls of the ancient polygons (photo Pascal Bertran).

Formal convergences

Revealing a polygonal pattern is not an easy task as it implies detecting not concentrations of objects, but the empty spaces around them. Visual discrimination remains the most reliable method (Masson & Vallin, in press). In figure 48, a comparison of modern and fossil polygonal grounds with the constructed hearths at Saint-Vaast-la-Hougue shows formal convergences and tends to invalidate the implication of humans in the realization of the circular, granite borders around the hearths. This invalidation is supported by the study of artefact distribution maps.

Analysis of the distribution of fire-modified granites near the bordered hearth (ST66) of Chantier II, shon in figure 48 (2), shows that there is no correspondence between the burned granites and the hearth borders. The burned granites are distributed over the entire excavated surface with no particular organization, which appears to indicate that the archaeological level was disturbed (fig. 49). On this same map, we can also see numerous empty zones surrounded by granite blocks. If we do not take into account the ash fillings, it is visually possible to fabricate other structures with forms equivalent to those of the bordered hearths (fig. 50). The ensemble of these pseudo structures belongs to a polygonal network (fig. 51).

Based on an analysis of the vertical distributions of refits, Gérard Fosse identified three occupation levels in the upper horizons of Chantier I (Fosse, 1983). The plans of the spatial distributions of artefacts in each level reveal several empty spaces outlined by artefacts (for example, level III, fig. 52). As in the preceding plan of Chantier II, it is possible to visually trace an ensemble of cells that form a polygonal ground (fig. 53). The walls of cells in this network are randomly constituted by worked flint and natural stones (granite, sandstone and schist).

These two examples show that the sub circular arrangements of stones found in several levels of Chantiers I and II, are independent of the elements that compose their walls or centre. A natural origin, such as a polygonal ground, is the best explanation for this type of artefact distribution.

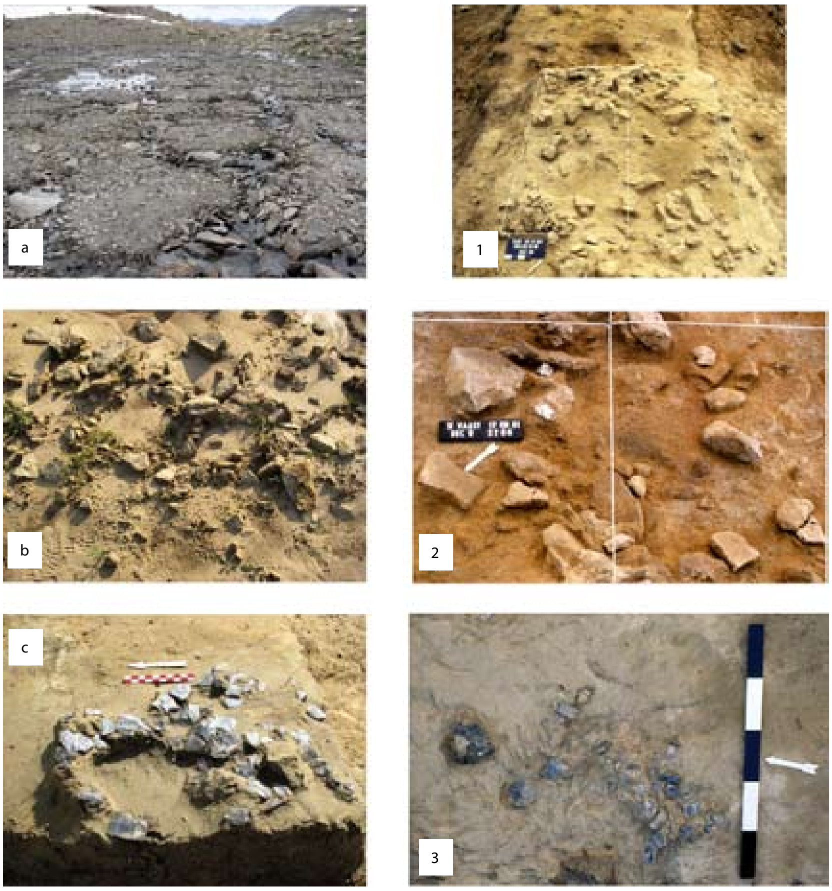

Fig. 48 - Comparison of natural (photo a) and fossil (photo b, Saint-Amand-les-Eaux, photo Philippe Feray) polygonal grounds and constructed hearths at Saint-Vaast-la-Hougue (photos 1 and 2, photos Gérard Fosse). The two bottom photos show the formal convergence between a concentration disturbed by the polygonal structures at Saint-Amand-les-Eaux (photo Philippe Feray) and a concentration at Saint-Vaast-la-Hougue (photo Gérard Fosse).

Cryoturbations: experiments by Albert Pissart (1973-1984)

Between 1968 and 1975, Albert Pissart conducted a series of experiments in a natural context (Massif du Chambeyron, 3000 m altitude) and in a laboratory with

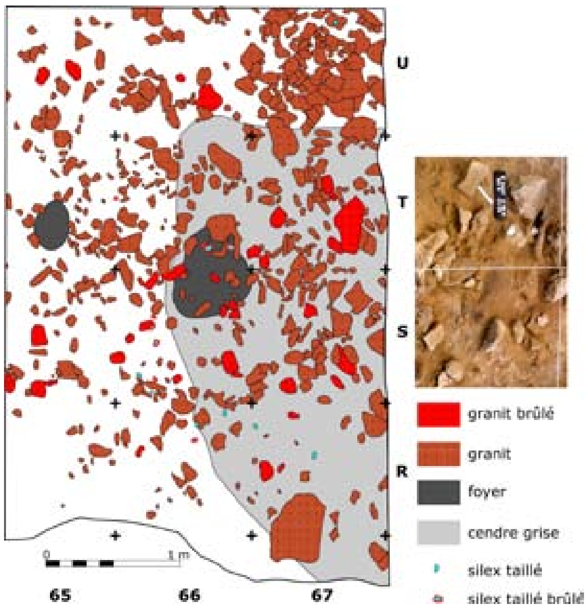

Fig. 49 - Plan of the hearths of decapage 9 at Chantier II at Saint-Vaast-la-Hougue (CAD Bertrand Masson).

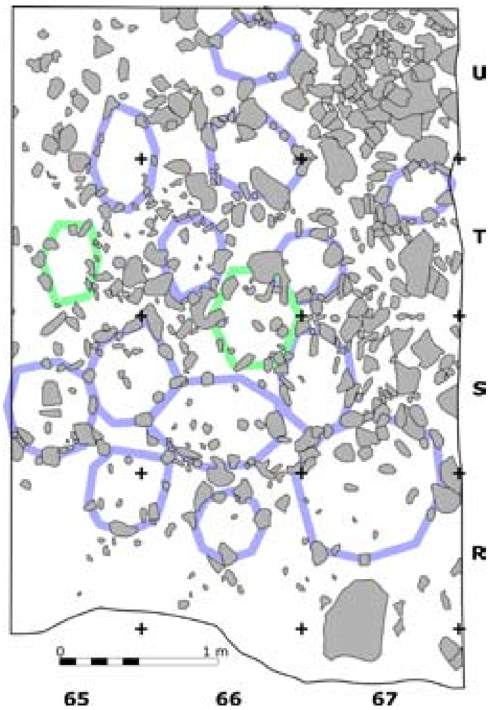

Fig. 50 - Plan of the artefacts (indistinctly represented) of decapage 9 at Chantier II at Saint-Vaast-la-Hougue, on which is indicated an ensemble of polygonal cells (in blue) identical to the hearth (in green) composed of an empty space surrounded by stones. This ensemble forms a polygonal network comparable to that recorded on the Massif du Chambeyron (fig.51), (CAD Bertrand Masson).

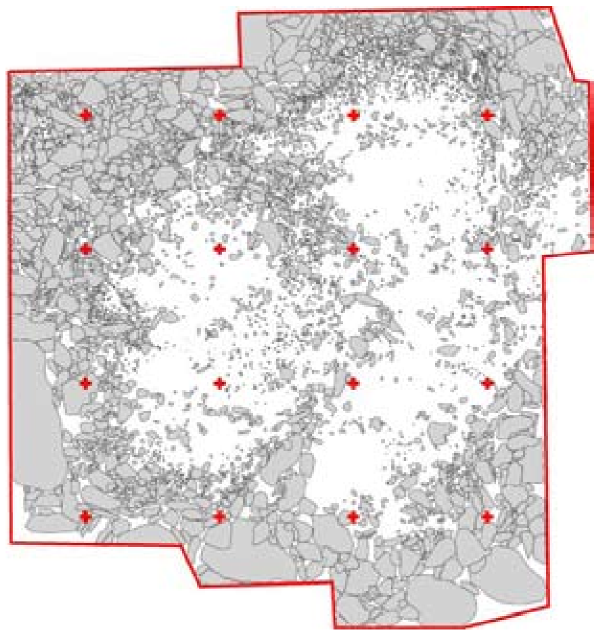

Fig. 51 - Plan of a polygonal ground on the Massif du Chambeyron (CAD Bertrand Masson).

the goal of identifying the processes of the formation and evolution of periglacial ground structures (Pissart 1987). The method of study he employed consists of observing the movements of coloured stones or the deformations of coloured silt blocks. In 1972 Albert Pissart thus buried levels of coloured earth, which were deposited horizontally in the centre of small, sorted polygons of the Chambeyron massif. This experiment showed that the modifications within polygons are not homogeneous and that the lateral intrusions at the limit between the fine and coarse materials, caused by freezing, more strongly deform these levels at the edges of the polygons (fig. 55A). Another similar experiment was realized in 1973 at the Col de la Gypière (2900 m, French Alps), on a slope of 3 to 6° within sorted soils (polygonal grounds modified along a slope, Fig. 54). Here, Albert Pissart in 1975, followed by Brigitte Van Vliet-Lanoë in 1984, observed the modifications made to a parallelepiped of coloured silt buried in the centre of a fine band of striated earth (fig. 55 B). This experiment showed that the rate of movements (maximum recorded by Albert Pissart: 7 cm/year) varies according to the depth of burial. The levels near the surface, which are subject to a greater number of freeze/thaw cylces, are the most deformed. Such cryoturbations could explain the form of some ashy levels on the periphery of hearths and the mixture of different types of ashes within them (fig. 55).

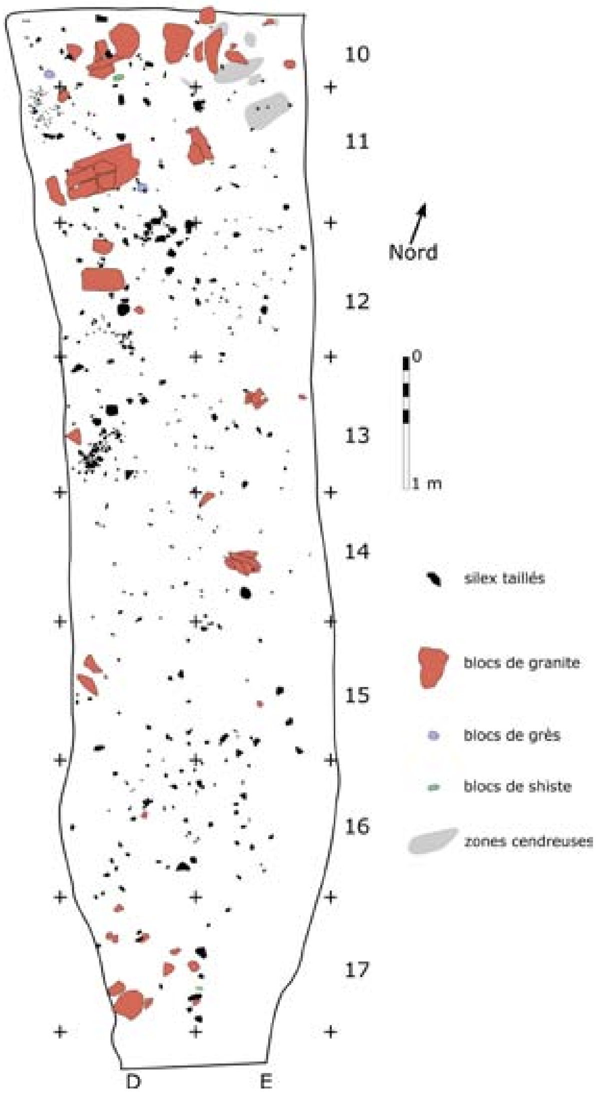

Fig. 52 - Spatial distribution map of the artefacts of level III of the upper horizons of Chantier I at Saint-Vaast-la-Hougue (CAD Bertrand Masson).

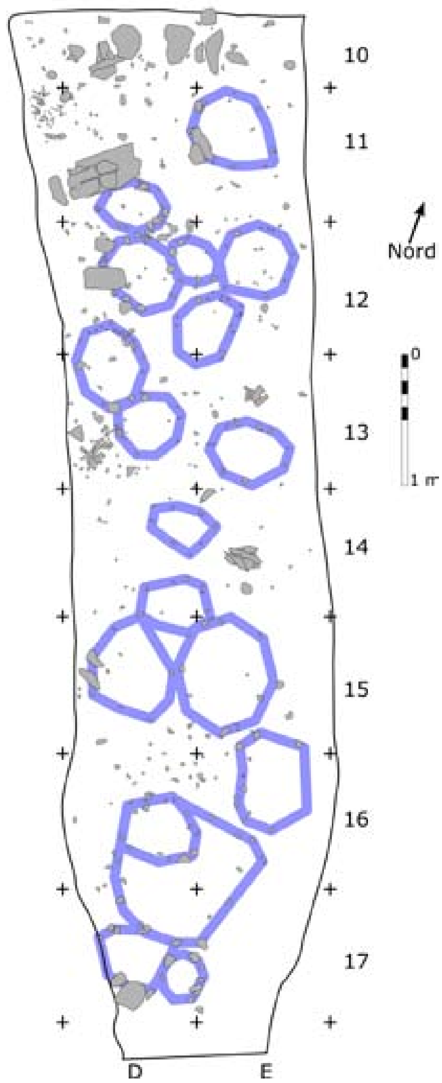

Fig. 53 - Spatial distribution map of the artefacts of level III of the upper horizons of Chantier I at Saint-Vaast-la-Hougue, on which an ensemble of polygonal cells (in blue) composed of an empty space surrounded by stones has been drawn (CAD Bertrand Masson).

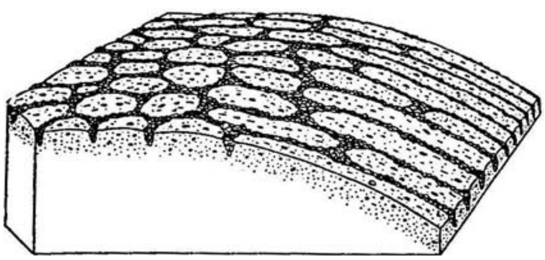

Fig. 54 -Passage, on a convex slope, from sorted polygons to striated sols (after Cotton, 1948, *in* Pissart 1987).

Cryostatic pressure

Experiments by Albert Pissart (1973–1984)

Still in the context of his work realized from 1973 to 1984, Albert Pissart conducted a series of experiments to demonstrate the unequal swelling of sediments with different granulometric dimensions during freezing. In a container with its walls inclined at 45° and covered with grease to avoid "wall effects", he deposited three cylinders of silt composed of six alternately coloured and non-coloured layers. One was placed on top of a layer of river sand and the two others on the bottom of the container and surrounded by 5 to 10 mm gravels (fig. 56 A and B). These experiments showed a progressive descent of the silt cylinders into the underlying sands. This descent increased with the number of freeze/thaw cycles (fig. 56 C).

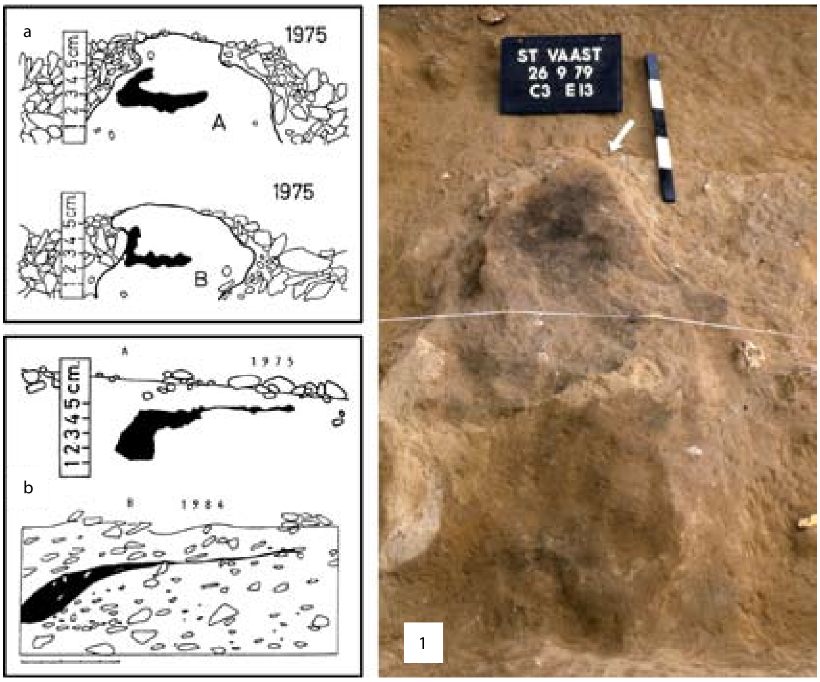

Fig. 55 - A – Profile of sorted polygons showing the deformation, after 2 years, of coloured layers deposited horizontally in 1973 (after A. Pissart, 1987). B – Deformation of a parallelepiped of coloured silt, placed vertically in 1973 within a fine band of striated soil, profile view (A. Pissart 1982 and B. Van Vliet-Lanoë 1988). 1 – Plan view of hearth DE13 when it appeared (photo Gérard Fosse). The forms of the ashy level, especially those on the periphery, could be explained by cryogenic deformations.

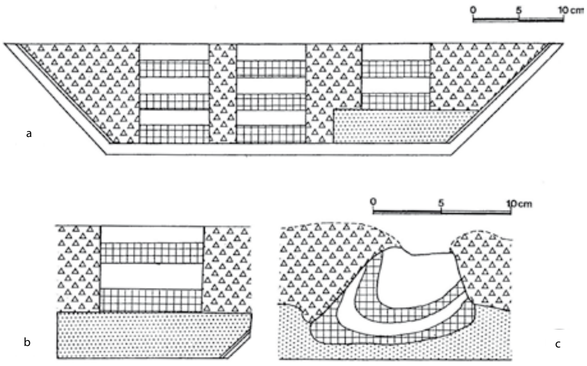

Fig. 56 - a – Schema showing the experimental apparatus employed by Albert Pissart to show the influence of cryostatic pressure in the deformation of soils (Pissart 1987). b – View of the silt cylinder, 73 mm in diameter, placed on coarse sand and surrounded by gravel before freezing.
c – Profile through the same cylinder after 15 freeze/thaw cycles, showing the descent of silt into the sand and the deformation of the coloured layers.

Archaeological examples

In the silts of northern France, examples of cryoturbated levels are frequent. We present here two examples that illustrate our topic. The first photo (fig. 57) shows a stratigraphic profile in levels attributed to the Weichselian early glacial period in Onnaing (Nord); despite a small granulometric difference, the gley soil horizons and those underlying them (humus-bearing at the base and loess at the top) are deformed by cryostatic pressure.

The second example comes from water-washed silts of the Weichselian early glacial at Hermies “Champ Bruquette”; the beds constituting these water-washed horizons, formed in humus-bearing and loess levels, were deformed by cryostatic pressure.

Formal convergences

Lacking an experimental reference base on the behaviour of hearths subject to freeze/thaw cycles, it is difficult to prove that cryostatic pressure can deform them. We saw before that small granulometric differences were sufficient for freezing to deform sediments. We can thus propose the hypothesis that the different sediments that compose a hearth—ashes, depending on their charcoal and organic material contents, rubified levels, and the surrounding sediments—do not have the same frost susceptibility, which could result in

Fig. 57 - Profile in the silts of the Weichselian early glacial at Onnaing (Nord), showing the cryostatic deformations suffered by the different horizons (photo Luc Vallin).

Fig. 58 - Profile in the water-washed silts of the Weichselian early glacial at Hermies «Champ Bruquette» (Pas-de-calais), showing the cryostatic deformations suffered by the different beds. The lightest beds are silty, the darkest are more clayey and humus-bearing (photo Luc Vallin).

their cryoturbation. As with the preceding examples, though a visual comparison is insufficient to affirm the role of freezing in the transformation of features, the formal convergences, visible in figures 59 and 60, show that a cryogenic origin can not be excluded.

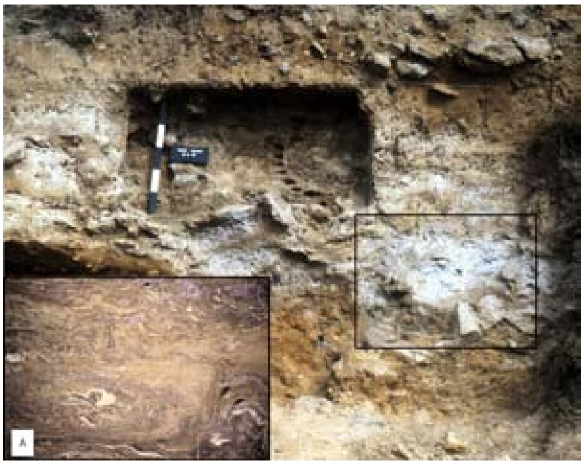

Fig. 59 - Comparison between a profile in the complex hearths at Saint-Vaast-la-Hougue (photo Gérard Fosse) and the water-washed and cryoturbated silts at Hermies "Tio-Marché", box A (photo Luc Vallin). We observe a similarity of form between the two boxes.

Conclusion

Researchers are now beginning to consider the influence of periglacial phenomena on lithic and osseous remains, notably in the context of research programs such as TRANSIT, experiments conducted in natural contexts (Gavarnie, Massif du Chamberyon) and in the laboratory as part of the "Taphonomy of Middle

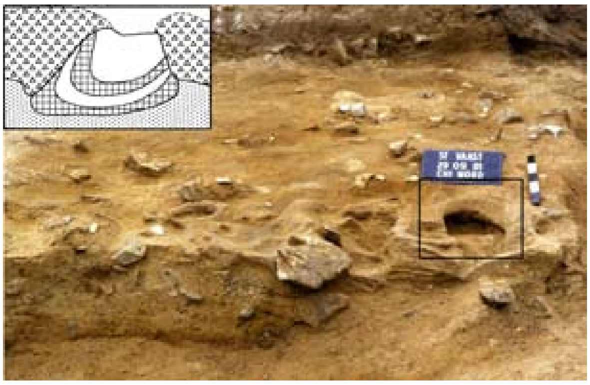

Fig. 60 - Comparison between a complex hearth at Saint-Vaast-la-Hougue (photo Gérard Fosse) and the forms obtained by Albert Pissart during laboratory experiments. The ashes of hearth D13 were emptied. The photo shows its hardened base composed of a coalescence of pits that were interpreted as successive reutilizations. Considering the formal convergence between the framed part, for example, and the forms obtained by Albert Pissart, a cryogenic origin cannot be excluded.

Palaeolithic assemblages in periglacial contexts" and "The Palaeolithic in the Quercy" research programs. However, to my knowledge, there have been no studies of the evolution of hearths in environments subject to freeze/thaw cycles. Due to the lack of an experimental reference base concerning the behaviours of combustion features subject to freeze/thaw alternations, it is not possible to make reliable interpretations of Mousterian combustion features in silt contexts in northern France.

Acknowledgements

I particularly thank Gérard Fosse (Regional Archaeology Service of Nord-Pas-de-Calais) who allowed me to study the hearths of Saint-Vaast-la-Hougue, as well as Philippe Feray (Inrap) who provided me with the unpublished data from Saint-Amand-les-Eaux. I also thank all the researchers who intervened in the context of the research programs "Taphonomy of Middle Palaeolithic assemblages in periglacial contexts" directed by L. Vallin and "The Palaeolithic in the Quercy" directed by P. Bertran an J.-P. Texier. Experiments concerning the influence of periglacial phenomena are difficult to realize as their analyses require enormous amounts of time and their results are available only after many years of effort.

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References

Akerman H. 1993 - Solifluction and creep rates 1972–1991, kapp linné, west spitzbergen. *In* : *Solifluction and climatic variation in the Holocene*. Essai : 225-250.

Baulig H. 1956 - Pénéplaine et pédiplaine. *Bulletin de la Société Belge d'Études Géographiques*, 25 : 25-58.

Bertran P. 1994 - Dégradation des niveaux d'occupation paléolithiques en contexte périglaciaire : exemples et implications archéologiques. *Paléo*, 6 : 285-302.

Bertran P. & Coutard J.-P. 2004 - Solifluxion. *Quaternaire*, 1 : 84-109.

Bertran P. & Lenoble A. 2002 - Fabriques des niveaux archéologiques : méthode et premier bilan des apports à l'étude taphonomique des sites paléolithiques. *Paléo*, 14 : 13-28.

Coutard J.-P. & Ozouf J.-C. 1996 - Modalités de la cryoreptation dans les massifs du Chambeyron et de la Mortice, Haute-Ubaye, Alpes françaises du sud. *Permafrost and Periglacial Processes*, 7 : 21-51.

Coutard J.-P. & Van Vliet-Lanoë B. 1994 - Cryoexpulsion et cryoreptation en milieu limono-argileux. expérimentation en laboratoire. *Biuletyn Peryglacjalny*, 33 : 5-20.

Deschodt L., Feray P. & Vallin L. 2006 - *Saint-Amand-les-Eaux (59) bas du mont des bruyères.* Rapport non publié, Sercice Régional de l'Arcéologie du Nord-Pas-de-calais, Villeneuve d'Ascq.

Fosse G. 1982 - Le gisement moustérien de Saint-Vaast-la-Hougue : structure et occupation du site. *In* : *Les habitats du Paléolithique supérieur*. Acte du colloque de Roanne-Villerest, (à paraître).

Fosse G. 1983 - Durée et périodicité d'occupation sur le site moustérien de Saint-Vaast-la-Hougue (Manche). *In : Séminaire sur les structures d'habitat*, Collège de France : 201-208.

Fosse G., Cliquet D. & Vilgrain G. 1986 - Le Moustérien du Nord Cotentin (département de la Manche) : premiers résultats de trois fouilles en cours. *In : Chronostratigraphie et faciès culturels du Paléolithique inférieur et moyen dans l'Europe du Nord-Ouest,* Tuffreau A. & Somme J. (eds). Actes du colloque international, Université des Sciences et Techniques de Lille, 2-7 septembre 1984. Supplément au Bulletin de l'AFEQ, 1986 : 141-155.

Francou B. & Bertran P. 1997 - A multivariate analysis of clast displacement rates on stone-banked sheets, cordillera real, Bolivia. *Permafrost and Periglacial Processes*, 8 : 371-382.

Masson B. & Vallin L. à paraître. Altération des sols paléolithiques par la formation de sols figurés sous climat périglaciaire : illustrations archéologiques et approche expérimentale. *Paléo*.

Pissart A. 1987 - *Géomorphologie périglaciaire. Textes des leçons de la Chaire Franqui Belge.* Laboratoire de Géomorphologie et de Géologie du Quaternaire de l'Université de Liège, Liège.

Texier J.-P. 2000 - A propos des processus de formation des sites préhistoriques. *Paléo*, 12 : 379-396.

Texier J.-P. 2001 - Sédimentogenèse des sites préhistoriques et représentativité des datations numériques. *In : Datation.* Barrandon J.-N., Guibert P. & Michel V. (eds). XXI[e] rencontres internationales d'archéologie et d'histoire d'Antibes. Editions APDCA : 159-175.

Texier J.-P. & Bertran P. 1995 - Les dépôts du site moustérien de Combe-Capelle bas (Dordogne) : leur signification dynamique et paléoenvironnementale. *Paléo*, 7 : 27-48.

Texier J.-P., Bertran P., Coutard J.-P., Francou B., Gabert P., Guadelli J.-L., Ozouf J.-C., Plisson H., Raynal J.-P. & Vivent D. 1998 - Transit, an experimental archaeological program in periglacial environment : Problem, methodology, first results. *Geoarchaeology*, 13 : 433-473.

Thiébault S., Fosse G. & Cliquet D. 1988 - Déterminations anthracologiques sur deux gisements moustériens du Nord-Cotentin en Normandie. *Bulletin du Centre de Géomorphologie du C.N.R.S.,* Caen, 35 : 9-19.

Van Vliet-Lanoë B. 1988 - *Le rôle de la glace de ségrégation dans les formations superficielles de l'Europe de l'Ouest. Processus et héritages*. Thèse de doctorat, Paris I-Sorbonne.

Washburn A. 1967 - Instrumental observations of mass-wasting in the mesters vig district, northeast greenland. *Meddelelser om Gronland*, 166 : 318.

Washburn A. 1973 - *Periglacial Processes and Environments*. Arnold, Londres.

Washburn A. 1979 - *Geocryology. A survey of periglacial processes and environments.* Edward Arnold, London.

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DUMPING, SWEEPING AND TRAMPLING: EXPERIMENTAL MICROMORPHOLOGICAL ANALYSIS OF ANTHROPOGENICALLY MODIFIED COMBUSTION FEATURES

Christopher E. MILLER, Nicholas J. CONARD,
Paul GOLDBERG & Francesco BERNA

Abstract

Six experimental fireplaces were constructed to investigate the ability of micromorphology to identify anthropogenic reworking of combustion features and to build a reference base of experimentally-derived conditions to calibrate micromorphological conditions. After burning, the fireplaces were either swept out, swept out and the material dumped, trampled, or a combination of these three. Micromorphological examination showed that these processes produce distinct characteristics readily identifiable at the microscopic scale. The application of this experiment to combustion-related features at the Paleolithic site of Hohle Fels in Germany showed that micromorphological examination of anthropogenic deposits—supported by experimental observations—provides an important context in which to evaluate other classes of artefacts.

Keywords : micromorphology, site-formation processes, combustion-related features, Hohle Fels

Introduction

As is apparent by the numerous contributions to this volume, research on combustion-related archaeology has intensified in the past decade. Our contribution provides an original point of view not discussed broadly in the literature: experimental micromorphology of combustion features. Although ethnographic and experimental studies have been a part of archaeological micromorphology for the past couple decades (e.g. Goldberg and Whitbread, 1993; Mallol *et al*., 2007), many interpretations of certain characteristics of microstructures found at archaeological sites are based on logical deductions reinforced by analogy with known geological processes. While such interpretations are perfectly valid when dealing with natural systems, any system that incorporates anthropogenic factors, such as the formation of archaeological sites, can become so complex that simple analogy with known natural systems may fail. Despite this problem, we think that certain human activities—especially those related to combustion—leave traces in the archaeological record and are readily visible at the microscopic scale (Courty *et al*., 1993). In fact, we believe that many single, discrete events are recorded not at the site- or even meso-scale, but occurred at and are recorded within the micro-scale. This has been one of the driving theoretical concepts in micromorphology since the publication of Courty *et al*. (1989). In this paper we provide some experimental results to test the effects of different human actions at the microscopic scale.

The inspiration for this experiment came from our excavations at Hohle Fels, a cave site located in the Swabian Jura of southwestern Germany (fig. 1). This cave site contains a stratified sequence of layers with archaeological material corresponding to Middle Paleolithic, Aurignacian, Gravettian, and Magdalenian occupations. Numerous features have been found, mostly within the Upper Paleolithic layers, and consist of lenses and laterally extensive layers of burnt bone, charcoal, and ash. The most striking feature at the site is the Gravettian layer 3cf which extends across more than 20 square meters and is locally up to 15cm thick. Schiegl et al. (2003) published a micromorphological study of the layer, interpreting this feature as a dumping zone. They noted that 3cf consisted mostly of angular sand-sized fragments of burnt and unburnt bone that were adjacent to one another. Although there was some weak bedding present, the bones were structured loosely and exhibited no evidence of graded bedding, ruling out water as a possible depositional agent. The open structure of the layer (exhibiting little to no compaction) and the lack of any in-situ crushed bone also suggested to the authors that 3cf was not extensively trampled. Altogether, the interpretation of 3cf produced by Schiegl *et al*. (2003) was that early Gravettian people at Hohle Fels used mostly bone as fuel and that the fireplaces that they constructed were located within a different part of the cave than the 3cf deposit, possibly closer to the entrance. They repeatedly built fires, removed the burnt waste from the main occupation area and dumped it elsewhere. These activities eventually formed layer 3cf. Although these interpretations explain all of the micromorphological observations, we wanted to experimentally test some of the ideas of anthropogenic deposition and modification, particularly related to dumping and trampling.

We specifically chose to test the effects of different types of anthropogenic, post-combustion activities on burnt material. These activities included sweeping out of hearths, trampling of hearths, dumping of hearth

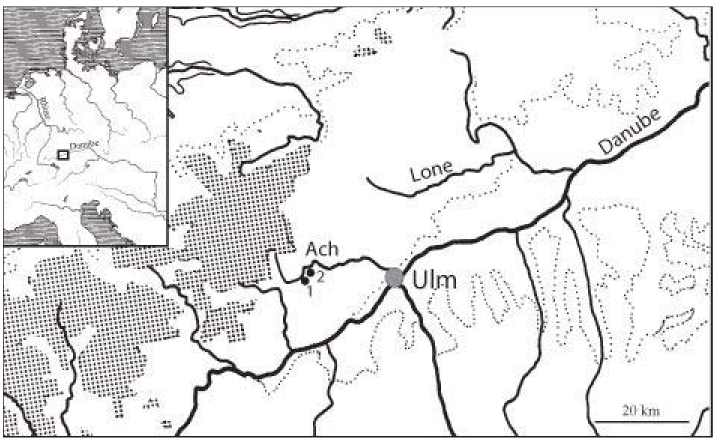


Fig. 1 - Location map of Hohle Fels, located within the Swabian Jura of SW Germany. Hohle Fels is indicated by number 1 on the map.

material, and combinations of these three activities. Many combustion features (not just at Hohle Fels), when investigated micromorphologically, do not appear intact. In other words, the simple presence of delimited lenses of charcoal at a site does not necessarily mean that the charcoal was produced exactly where it was excavated. Burnt material can be reworked by natural processes (Weiner *et al.*, 1998); however, it is possible that burnt material can be reworked and moved by humans (Meignen *et al.*, 2007). Although such anthropogenically reworked deposits are removed from their primary context, the action of removing or reworking burnt material can inform us about past behaviors, site maintenance, and use of space. An evaluation of the depositional history of a combustion-related feature also provides a better context in which to evaluate other classes of artifacts and their spatial distribution.

Experiment design and Method

We constructed six experimental fireplaces. The experimental areas were covered with a 3-5 cm-thick layer of reworked—and archaeologically sterile—cave sediment from Hohle Fels. Wood was collected from recently felled trees of the Schönbuch Forrest near Tübingen, Germany, which consisted mostly of beech and oak. The wood was dried in a 60° C oven overnight before the experiment. Each fire consisted of 5 kg of dried wood along with 2 kg of defleshed pork ribs and vertebrae, cut into 5-10 cm cubes. Although these bones were defleshed, some marrow, fat and meat were still attached. The fires were built using a small amount of dried leaves and grass as kindling; wood was stacked into a cone above the fire (fig. 2). Once the fire had started to burn, the bones were added on top of the wood. Except for the control hearth, the other fires were managed: pieces of unburnt wood and bone were moved into the flame to promote complete (or at least near complete) combustion of all material. The fires took approximately 1.5 to 2 hours to completely burn through the fuel (from lighting the fire to the point where no more flames were visible) (tab. 1). The fire experiment was conducted in November with a high temperature of 12° C during the day, and nighttime temperatures dipping below freezing. There was a mist on the day of the experiment, slowly turning into a light drizzle. After letting the experimental hearths cool overnight, we returned the next day to rework five of the six fireplaces (excluding the control). The reworking processes included trampling of a hearth (HT), sweeping out of a hearth (S), trampling of a swept-out hearth (ST), sweeping-out of a hearth, removing and dumping that material (D), and trampling of a similarly dumped hearth (DT). Trampling was carried out for a minute by two of the experimenters (fig. 3). They wore shoes with rubber soles and very little tread. Sweeping was conducted with a natural-grass hand-broom. We pushed the majority of the material out of the hearth and then swept the surface of the former hearth briskly, causing some of the finer combusted material to travel through the air as dust. The dumping of the hearths was carried out similarly to the sweeping action; however, the material was swept into a skin and carried to another experimental area, where it was quickly dumped by rapidly tossing the material to the ground. After the hearths were reworked, we waited a week to return and collect samples for micromorphological analysis.

We removed undisturbed sample blocks by excavating

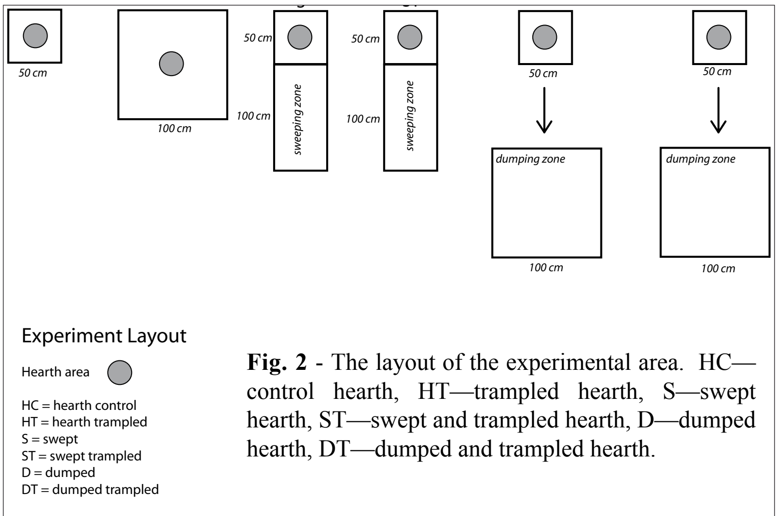


Fig. 2 - The layout of the experimental area. HC—control hearth, HT—trampled hearth, S—swept hearth, ST—swept and trampled hearth, D—dumped hearth, DT—dumped and trampled hearth.

Hearth Name	Hearth Type	Management
HC	Control	• Allowed to burn to completion without moving unburned materials to center • Incompletely burned wood and bone were placed in center of hearth to promote complete burning
HT	Trampled	• Burned to completion • Incompletely burned wood and bone were placed in center of hearth to promote complete burning • After cooling overnight, was trampled for a minute
S	Swept	• Burned to completion • Incompletely burned wood and bone were placed in center of hearth to promote complete burning • After cooling overnight, was swept out with a grass hand broom
ST	Swept	• Burned to completion • Incompletely burned wood and bone were placed in center of hearth to promote complete burning • After cooling overnight, was swept out with a grass hand broom • Was then trampled for a minute
D	Dumped	• Burned to completion • Incompletely burned wood and bone were placed in center of hearth to promote complete burning • After cooling overnight, burned material was swept into an animal skin, moved several meters away, and dumped on a patch of Hohle Fels sediment
DT	Dumped and Trampled	• Burned to completion • Incompletely burned wood and bone were placed in center of hearth to promote complete burning • After cooling overnight, burned material was swept into an animal skin, moved several meters away, and dumped on a patch of Hohle Fels sediment • Was then trampled for a minute

Tab. 1 - List of hearth name (as used in following figures), the type of hearth, and the specific management of the hearths.

around the desired location and covering them with plaster bandages. The blocks were moved to the micromorphology laboratory at the University of Tübingen, where they were dried for several days in an oven at 60° C. They were then impregnated with a mixture of unpromoted polyester resin (Viscovoss, Vosschemie Gmbh) that was diluted with styrene (VWR International). Methylethylketone peroxide (MEKP) was used as the polymerization catalyst. The samples were allowed to set for a week before being heated to 60° C overnight, causing full polymerization of the resin. Slices of the blocks were cut with a rock-saw and sent to Spectrum Petrographics (Vancouver, Washington, USA) to produce thin sections, 5 x 7.5 cm in dimension. These thin sections were analyzed using a standard, polarizing petrographic microscope, with magnification of 4-20 x. Nomenclature and descriptions follow that of Courty *et al*. (1989) and Stoops (2003).

Micromorphological Results

HT (trampled hearth)

We collected two slides from the trampled, *in situ* hearth (fig. 4 and 5; tab. 2). Both of these slides showed that the trampled hearth retained a typical hearth structure with a layer of charcoal overlying a rubefied base of sediment. Although the general hearth structure was preserved, there were several characteristics of sample HT that distinguished it as trampled. This included compaction of the underlying cave sediment, evident by a lack of void structure when compared with non-trampled samples. Furthermore, several larger pieces of bone and charcoal were pressed into the underlying sediment. Some of the pieces of bone appeared to be snapped and crushed. There appeared to be very little horizontal movement or displacement of components; most

Fig. 3 - Photographs of the various anthropogenic reworking activities. A) trampling of hearth ST, B) sweeping out of hearth D onto a skin, C) dumping of hearth D.

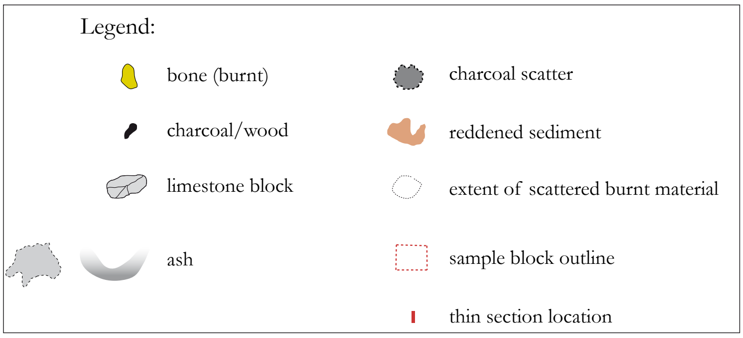


Fig. 4 - Legend for the plans of the hearths, as seen in figures 5-9.

non-rubefied sediment. Associated with the larger fragments of burnt bone and charcoal were some finer, mm-size clasts of rubefied sediment, presumably swept out with the larger burnt components. The overall structure of this sample is not realistic archaeologically, since successive periods of deposition and post-depositional alteration would most likely not preserve such an open structure.

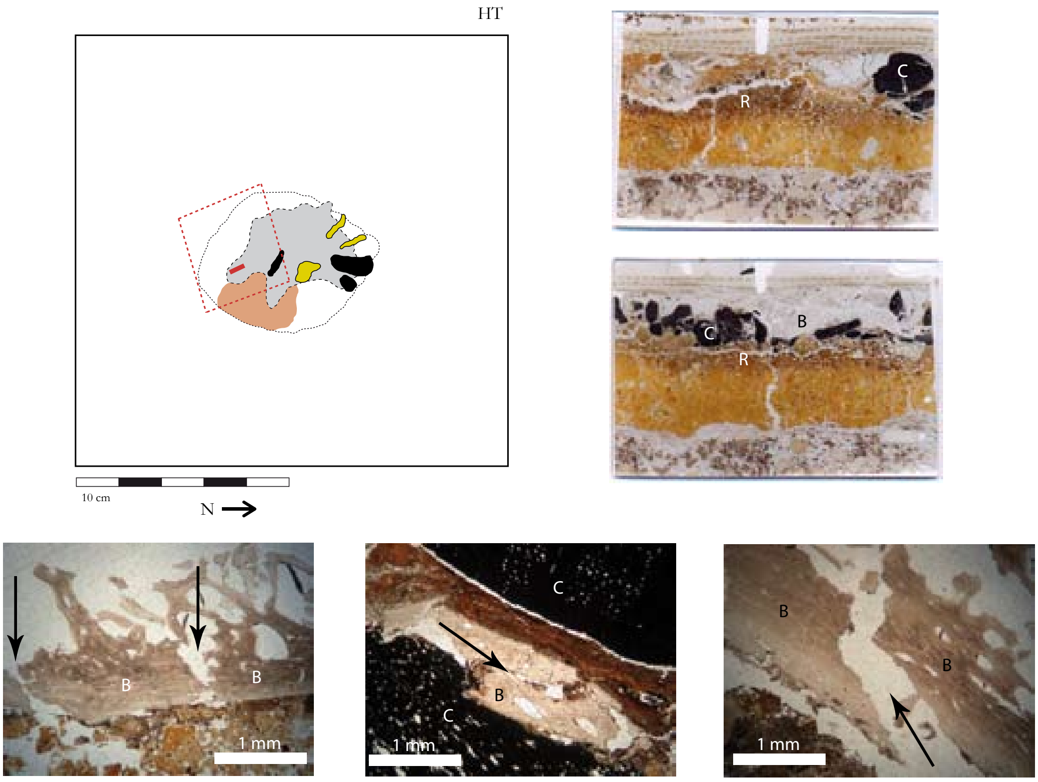


Fig. 5 - Trampled hearth (HT). See table 1 for a description of the types of hearths and table 2 for the macro- and microscopic descriptions. The plan view of the experimental area indicates where the samples were taken. Scans of the slides (dimensions are 5 x 7.5 cm) are provided in the upper right-hand corner of the figure. The lettering on the scans and the photomicrographs indicate: R—rubefied substrate, C—charcoal and B—bone. In the scanned slides, one can note that the charcoal and burned bone overlie a rubefied substrate. In the photomicrographs and the base of the figure, one can note (from left to right) a burnt bone snapped in several locations (indicated by arrows), a piece of burned bone crushed between two pieces of charcoal, and another snapped bone (indicated by the arrow).

movement was vertical, probably as a result of the compaction and pressure.

S (swept hearth)

A single slide was made from the swept sample (fig. 6). This slide consisted of large, cm-sized pieces of burnt bone and charcoal, very loosely organized, overlying a substrate of

ST (swept and trampled)

The single slide collected from the swept and trampled hearth showed generally similar characteristics to both HT and S (fig. 7). Like S, a layer of cm-sized pieces of burnt bone and charcoal overlie a non-rubefied substrate of cave sediment. Unlike S, the bone and charcoal components form a less-open

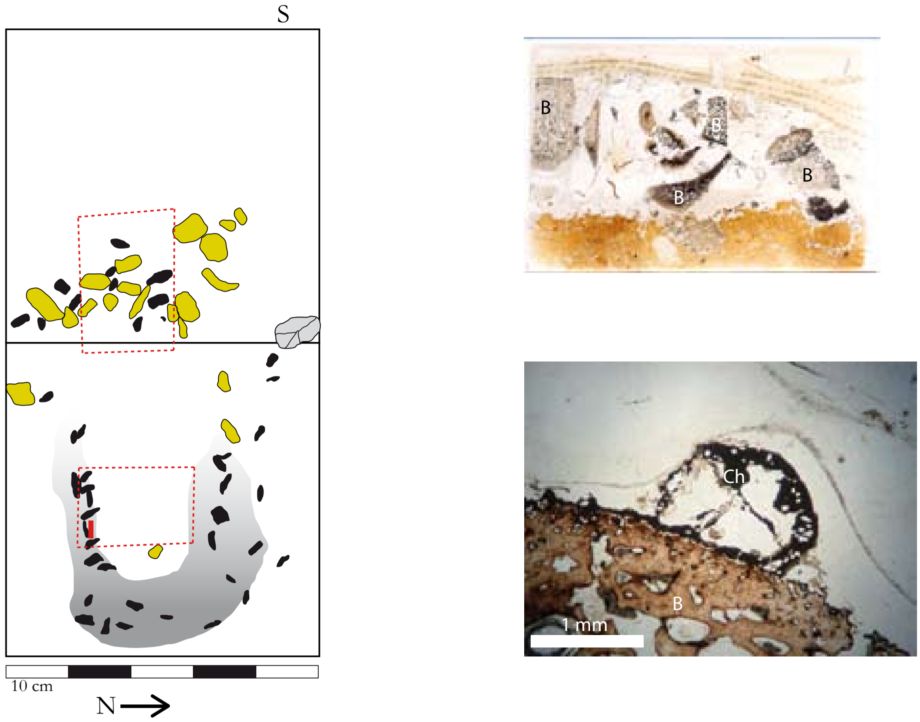


Fig. 6 - Swept hearth (S). Lettering on the scan and photomicrograph indicate: B—bone and Ch—char. Note the loose and open structure evident in the scanned slide (dimension of 5 x 7.5 cm). Also note that the burned bone and charcoal overlie a substrate that is not rubefied. The photomicrograph shows a piece of char attached to a burned bone.

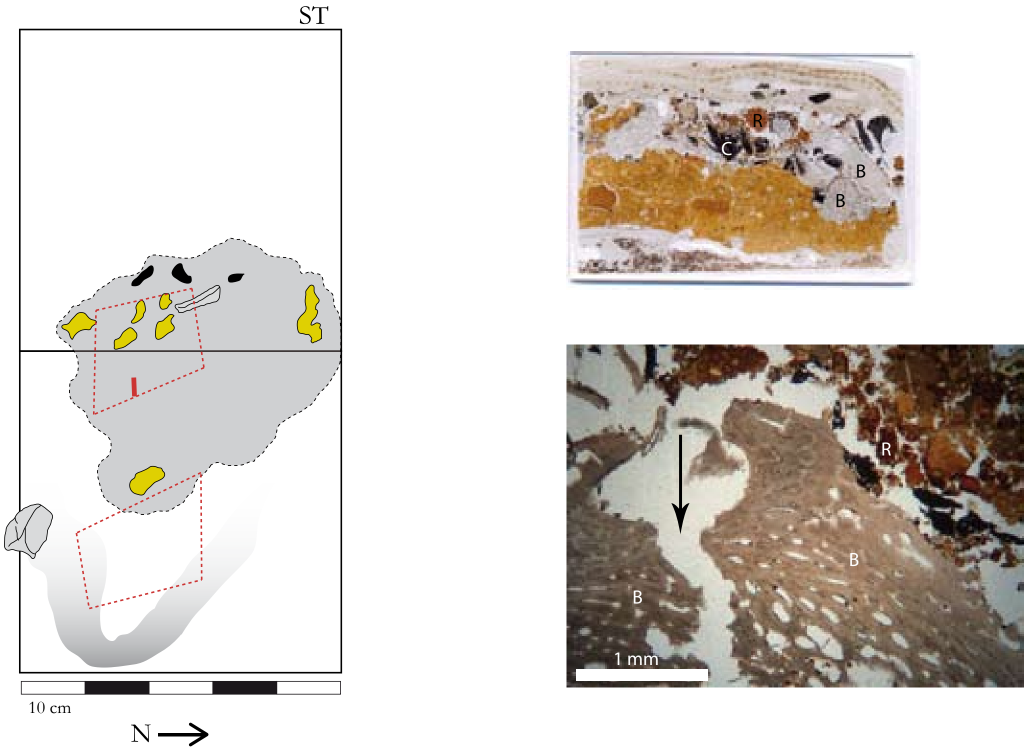


Fig. 7 - Swept and trampled hearth (ST). Lettering on the scan and photomicrograph indicate: C—charcoal, B—bone, R—rubefied clast. Note how more compact the burned material is in this scanned slide compared to that from the swept hearth (S—figure 6). Some of the larger pieces of bone are pressed into the underlying substrate, which is not rubefied. Some rubefied clasts, however, are incorporated into the reworked deposit. The photomicrograph shows evidence of a snapped bone (indicated by the arrow).

Hearth Name	Macroscopic observations	Microscopic observations
HT	• Circular outline of hearth area retained, similar to control • A patch of rubefied substrate was visible in the southeast corner • Larger pieces of burned bone and charcoal visible • Small pieces of charcoal and possible ash scattered around the central hearth area	• "classic" hearth structure visible—a rubefied base overlain by charcoal and burned bone • Larger pieces of bone and charcoal appear pressed into the underlying substrate, deforming the substrate • Some pieces of burned bone appear snapped in place, others appear crushed
S	• Burned material forms an elongated patch, oriented eastwards • The original outline and form of the hearth is no longer visible • Some coarser material (bone and charcoal) remain closer to the hearth center • Finer material is scattered further away (east) from the original hearth center	• Centimeter-sized pieces of charcoal and burned bone are loosely structured • They overlie sediment that has not been rubefied
ST	• Like S, this reworked hearth forms an elongated patch of burned material • Coarser material remained near the hearth center, whereas finer burned material is located further away from the center, forming an arc of sediment	• Centimeter-sized pieces of burned bone and charcoal overlie a non-rubefied substrate • The burned components are more compact compared to those from S • Clasts of rubefied material are found above and next to the pieces of charcoal and burned bone • The burned components are pressed into the substrate, deforming it • Some burned bones are snapped and/or crushed
D	• The burned material forms a patch slightly elongated in the northeast direction • A circular patch of charcoal was noted in the southwest portion of the patch • Larger pieces of burned bone are scattered throughout the patch	• Most pieces of charcoal and burned bone are finer (sub-centimeter) than in the previous hearths • The components are organized loosely and chaotically, especially the numerous sub-millimeter fragments of charcoal and burned bone • Sub-millimeter clasts of rubefied sediment are visible, scattered throughout the dumped deposit
DT	• The burned material here formed a more circular patch • Larger pieces of burned bone and charcoal were visible	• A loose, chaotically structured organization of the burned components was visible, although more compact • Larger pieces of burned bone and charcoal were pressed into the underlying sediment, deforming it • Some pieces of burned bone were snapped

Tab. 2 : Comparison of macroscopic and microscopic observations of the different hearths.des différents foyers.

structure. Within the layer of combusted material are several clasts and aggregates of rubefied sediment, obviously reworked from its primary context. It is not clear from these experiments if the rubefied material was reworked during the sweeping or the trampling, although both possibilities are plausible. Like HT, ST has evidence of several snapped and crushed bones.

D and DT (dumped and dumped & trampled)

Based on simple non-microscopic observation of the sample blocks from the dumped hearth, it is difficult to distinguish it from the swept-out material from the S hearth (Fig. 8 and Fig. 9). At a larger scale, the structure of the dumped deposit does not appear as elongated as the swept deposits, although this is probably a highly variable aspect of these deposits. Certainly one way of telling the swept hearths (S and ST) from the dumped deposits is that the dumped deposits (D and DT) are radically removed from any burned substrate. Microscopically there are some distinctions between the swept and the dumped deposits. The dumped deposits are organized more chaotically, with a wider range of size classes of charcoal and burnt bone adjacent. The dumped deposit that was not trampled also had a more open structure, similar to that of S.

Discussion

Looking at the results of the six fireplace experiments, there are several patterns that are applicable to the interpretation of archaeological samples. The first is the difference in the association between combusted material (bone and charcoal) and a rubefied substrate. For the control and the trampled hearth, the combusted material remained relatively in place: it lies directly above the rubefied substrate; even with trampling, the original structure and organization of the hearth was still visible. This could be a result of the short time that the samples were trampled (only for one minute); longer-term periods of trampling may have the effect of transporting the burnt material farther or significantly reworking the original structure of the hearth. Sweeping out of a hearth obviously disturbs this original structuring: in the thin section one notices that clasts of rubefied substrate have been reworked (similar to rip-up clasts) by the sweeping action. Furthermore, the deposit of combusted material overlies a layer of sediment that has not been affected by heating. The last situation examined here, the dumped deposits, are almost completely removed from any association with a reddened substrate. Some small (sub-mm) pieces of fire-reddened sediment were noted in the D and DT thin sections. However, their presence was negligible when compared to the swept or

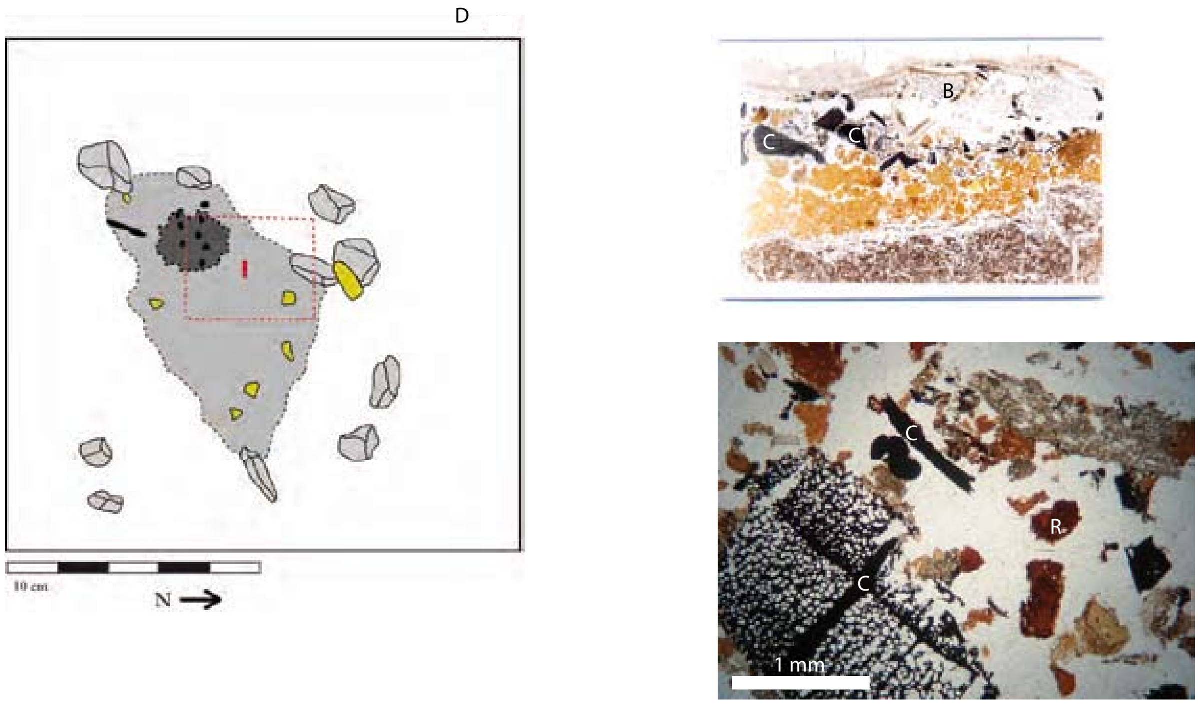


Fig. 8 - Dumped hearth (D). Lettering on the scan and photomicrograph indicate: C—charcoal, B—bone, R—rubefied clasts. Although some larger, centimeter pieces of charcoal are visible in the scanned slide, the matrix of the deposit consists of millimeter and sub-millimeter pieces of charcoal, burned bone, and rubefied clasts. In the photomicrograph one can note the open, loose and chaotic structuring of the sub-millimeter components.

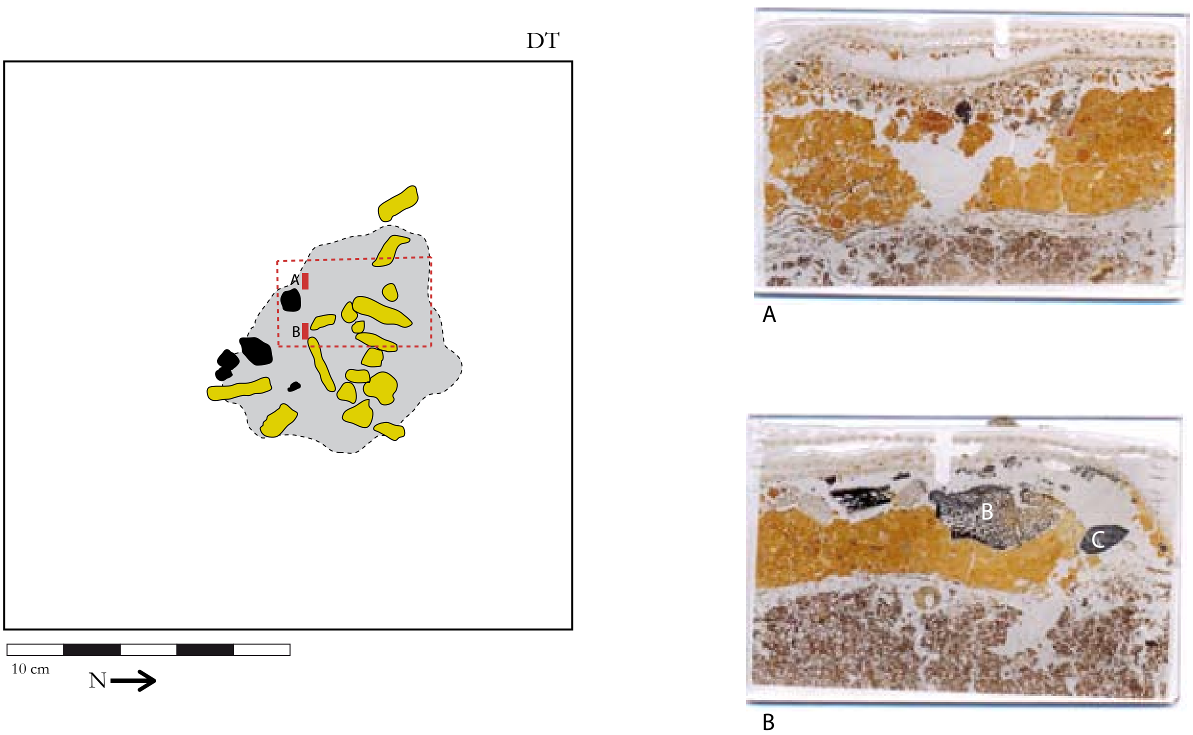
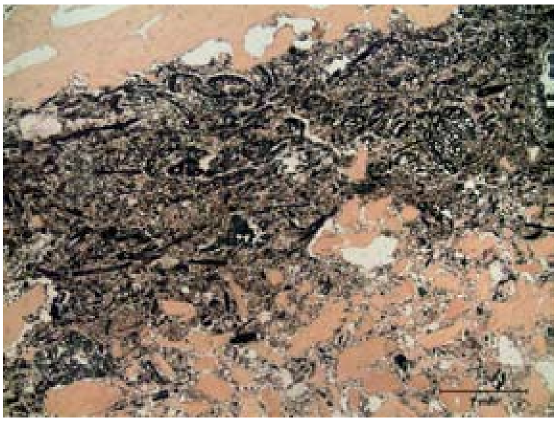


Fig. 9 -Dumped and trampled hearth (DT). Lettering on the scans indicated: C—charcoal and B—bone. Note in sample B that larger pieces of bone and charcoal have been pressed into the underlying substrate, which is not rubefied.

in situ samples. A lack of rubefication does not instantly suggest that a combustion-related feature is reworked: it is conceivable that some substrates may not redden in the presence of higher temperatures. However, the results from this experiment suggest that a lens of burnt material that is directly in contact with a substrate that is not rubefied—especially when it is known from experimentation that this sediment is commonly altered when subjected to heating—probably does not represent an *in situ* fireplace.

This experiment also showed that it is difficult to distinguish between swept and dumped material. One difference was that the grain-size distribution of burnt swept material was more homogenous compared to the grain-size distribution of dumped burnt deposits. This could be because sweeping causes a sorting of the material — especially if larger pieces of charcoal and burnt bone are removed by pushing to an area further away from the center of the hearth, while finer material is removed further from the hearth center by rapid sweeping motions. Since dumping is a more rapid movement—similar to a colluvial flow—it is not surprising that the material is more poorly sorted in terms of grain size. This observation, however, is cursory and needs further testing before it can be applied fully to archaeological material.

One of the most interesting results from this experiment was the very clear effect that trampling has on combustion features. The sediment was clearly compacted as a result of the trampling. In addition, burnt bones were snapped and also crushed. Such crushed and broken bones have been noted at several archaeological sites—including the South African Middle Stone Age site of Sibudu (Goldberg *et al*., forthcoming) and the French Middle Paleolithic site of Pech de l'Azé (Dibble *et al*., forthcoming; Fig. 10) — and have been reasonably assumed to represent trampling. This experiment shows that *in situ* snapped and crushed bone can occur as a result of only a minute of human trampling.



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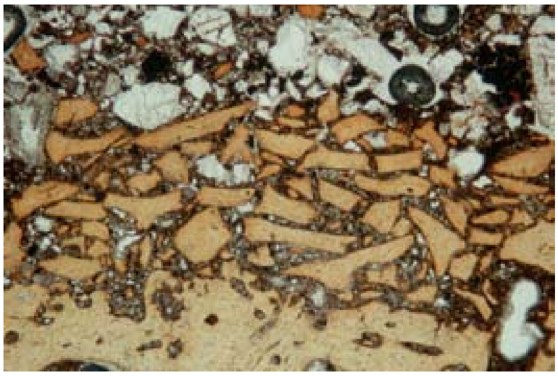

B

Fig. 10 - A) an example of what has been interpreted as bone crushed by trampling at the MSA South African site of Sibudu. B) Another example of crushed bone, from the Middle Paleolithic site of Pech de l'Azé, France. Width of view in this photomicrograph is approximately 6.2 millimeters.

Interpretation of Hohle Fels burnt bone layer (3cf) in the light of experimental results

We would like to provide a brief example of how this experiment is helping us interpret archaeological material from the site of Hohle Fels. A layer (3cf) of mostly sand-sized burnt bone, laterally extensive across the entire site and in some places up to 15 cm thick, was excavated within the Gravettian layers. Several hypotheses were proposed for the formation of this layer, including that it was possibly a sequence of *in situ* burning events, or that it may have been redeposited by flowing water. A micromorphological study of the layer (Schiegl *et al*., 2003) showed several distinctive characteristics (Fig. 11). There was no rubefication of the substrate and no fire-reddened clasts of sediment within the deposit. The deposit consisted almost completely of sand-sized burnt bone, with some calcitic ash, numerous lithic and organic artifacts, and faunal remains. The pieces of sand-sized burnt bone were organized in an open, chaotic structure, with fragments exhibiting varying degrees of burning adjacent to one another. The authors concluded that these characteristics demonstrated that the deposit was not *in situ* — but neither was it reworked

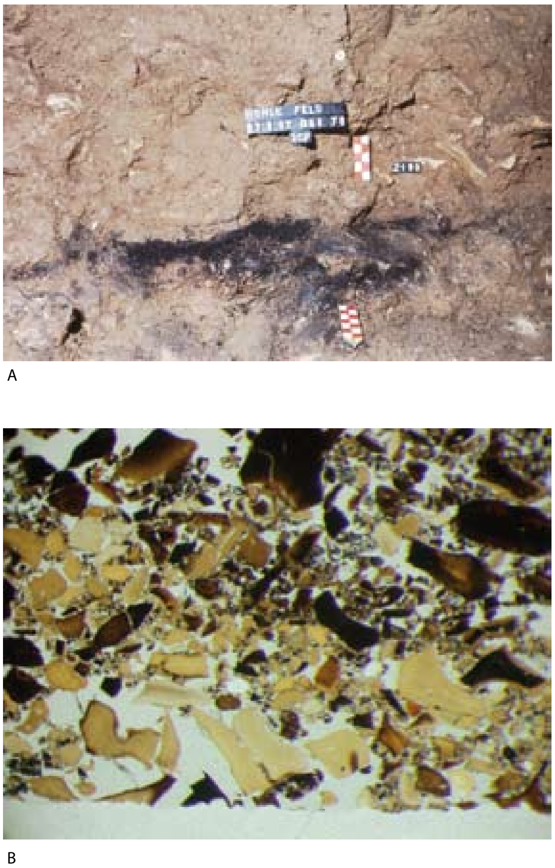

Fig. 11 - A) A field photograph of the Gravettian layer 3cf from Hohle Fels. B) A photomicrograph of layer 3cf in plane polarized light (PPL). Height of view here is 5 mm. Note the relatively loose, disorganized structure of the sand-sized fragments of burnt bone. Bone fragments of varying degrees of burning are adjacent. This layer is interpreted as a dumped layer. Compare this with the photomicrograph from hearth D, which shows a similar loose, chaotic structure.

by natural processes. Instead, they suggested that it was reworked by humans, who removed the material from the original hearth location and dumped it at this place in the cave. The thickness and lateral extent of the layer imply that this was done repeatedly over multiple periods of occupation. Furthermore, the open structure and the lack of snapped and crushed bone suggest little trampling, implying that, during the deposition of this layer, occupation was centered elsewhere within or near the cave while this area was used almost solely as a dump.

Several of these interpretations and observations have been demonstrated in this experiment, including the open and chaotic structure of dumped deposits and the fact that bones are commonly crushed when trampled. Understanding how deposits like this form—and understanding that these deposits are reworked anthropogenically — is very important for the interpretation of archaeological site formation processes. This understanding provides a context in which to interpret other classes of artifacts. For example, the burnt-bone layer at Hohle Fels contains numerous small flakes that are concentrated within several clusters (P. Kiesselbach, personal communication; Fig. 12); these flakes often refit. Without understanding how the burnt layer was deposited, it might be tempting to interpret these clusters of flakes as stone tool working loci representing *in situ* artifact scatters. However, because the micromorphological data show that

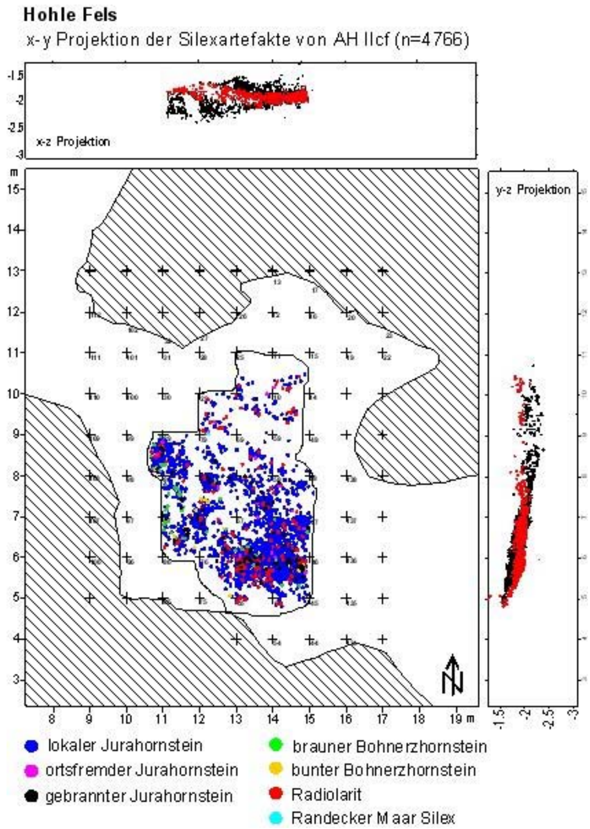

Fig. 12 - fig. 12 : A distribution map of lithic artefacts from Hohle Fels, layer 3cf (courtesy of P. Kiesselbach). Different types of local cherts (Hornstein) are indicated by different colors. Note that the distribution forms several clusters of artefacts. Based on the micromorphology of this layer, and supported by the experiments present here, these concentrations of lithic artefacts do not represent knapping loci or workshops, but most likely dumps of knapping by-products.

the flakes have been reworked, we therefore can conclude that these clusters do not represent *in situ* concentrations of flakes. Because the flake concentrations form clusters, and because there are so many refits, it seems that these concentrations represent byproducts of flaked stone tool production that, along with combusted material (burnt bone and ash) and other artifacts, were gathered together and dumped in a specific area of Hohle Fels cave.

Conclusion

In this study we presented results from six fireplace experiments. Excluding a control hearth, the other hearths were anthropogenically reworked, including a trampled hearth (HT), a swept-out hearth (S), a swept and trampled hearth (ST), a dumped hearth (D), and a dumped and trampled hearth (DT). Although some macroscopic differences were noted, micromorphological examination of the deposits provided clear evidence for the anthropogenic formation processes of the reworked deposits. These observations include:

1. Trampled deposits showed clear signs of compaction, such as bones and pieces of charcoal that were pressed into the underlying sediment and a less open structure within the reworked deposit itself.
2. All trampled deposits showed evidence of crushed and snapped bones. Similar features have been found in archaeological deposits and are interpreted as evidence for trampling.
3. Dumped deposits are typically more fine-grained than the other reworked deposits, and exhibit a loose, chaotic structure microscopically. Furthermore, a larger range of grain-sizes of burnt components are located throughout the deposit—resembling a colluvial deposit—compared to the swept samples.
4. Sweeping seems to cause a sorting of the burnt material, with finer-grained material located further out from the original hearth center. This conclusion is tentative, since this may be a result of the type of sweeping employed. More experiments should be conducted to test this.
5. Going from the trampled hearth to the dumped deposits, the association of the burned material with a rubefied substrate changes. In the trampled hearth (HT), the burned material was located directly above the rubefied substrate. In the swept samples, (S and ST), the burned material was not located above a rubefied substrate, although rip-up clasts of rubefied material were incorporated into the reworked deposit. In the dumped deposits (D and DT) some sub-millimeter-sized pieces of rubefied material were identified, although much less that those found in the swept deposits.

These microscopic observations show that distinct activities, such as trampling and dumping, are readily identifiable only at the microscopic scale. Although there are some distinctions between swept and dumped deposits, further experiments should strive to make these distinctions clearer. Further experiments should also aim to control natural taphonomic processes. This experiment was conducted outside, in a relatively moist environment. After waiting a week to collect the samples, most of the calcitic ash seemed to have blown away, or to have been dissolved. In a more protected cave setting, with a chemical environment that promotes at least short-term preservation of calcite, this would not be the case.

By using micromorphology to determine the depositional history of a combustion-related feature, we can begin to interpret how ancient people used fire, how they dealt with combusted material after it was no longer useful, and how ancient people organized their living space. Furthermore, a micromorphological investigation of combustion deposits at archaeological sites provides a context in which to evaluate other classes of artifacts, such as was show here with lithic concentrations within layer 3cf at Hohle Fels Cave.

We hope that this paper lays a foundation for future experimentation in micromorphology. It is through experiments like these that we can calibrate our interpretations made at the microscopic level and begin to unravel past human activities and behaviors preserved in anthropogenic deposits.

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References

Courty M.-A., Goldberg P. & Macphail R.I. 1989 - *Soil Micromorphology and Archaeology*. Cambridge, University Press, 344 pp.

Dibble H., Berna F., Goldberg P., Mcpherron S., Mentzer S., Niven L., Richter D., Thery-Parisot I., Sandgathe D. & Turq A. 2009 - Pech de l'Azé IV, layer 8 : A Case Study in Neandertal use of fire. *PaleoAnthropology* (soumis).

Gé T., Courty M.-A., Matthews W. & Wattez J. 1993 - Sedimentary formation processes of occupation surfaces. *In* : Goldberg P., Nash D.T. & Petraglia M.D. (eds). Madison, Wisconsin, Prehistory Press. Monographs in World Archaeology 17 : 149-163.

Goldberg P., Miller C.E., Schiegl S., Berna F., Ligouis B., Conard N.J. & Wadley L. 2009 - Bedding, hearths and site maintenance in the Middle Stone Age of Sibudu Cave, KwaZulu-Natal, South Africa. *Archaeological and Anthropological Sciences* (soumis).

Goldberg P., Schielg S., Meligne K., Dayton C. & Conard N. J. 2003 - Micromorphology and site formation at Hohle Fels Cave, Swabian Jura, Germany. *Eiszeitalter und Gegenwart*, 53 : 1-25.

Goldberg P. & Whithbread I. 1993 - Micromorphological studies of a Bedouin tent floor. *In* : Goldberg P., Nash D.T. & Petraglia M.D. (eds). Madison, Wisconsin, Prehistory Press. Monographs in World Archaeology 17 : 165-188.

Mallol C., Marlowe F. W., Wood B. M. & Porter C. C. 2007 - Earth, wind, and fire : ethnoarchaeological signals of Hadza fires. *Journal of Archaeological Science,* 34 : 2035-2052.

Meignein L., Goldberg P., & Bar-Yosef O., 2007 - The hearths at Kebara Cave and their role in site formation processes. *In* : O. Bar-Yosef & L. Meignen (eds). Cambridge, Massachusetts, Peabody Museum, volume 2 : 91-122.

Schiegl S., Goldberg P. & Conard N. J. 2003 - Palaeolithic burnt bone horizons from the Swabian Jura - Distinguishing between in situ fireplaces and dumping areas. *Geoarchaeology : an International Journal*, 18 : 541-565.

Stoops G. 2003 - *Guidelines for analysis and description of soil and regolith thin sections*. Madison, Wisconsin, Soil Science Society of America. 184 pp.

Weiner S. Xu Q., Goldberg P., Liu J., & Bar-Yosef O. 1998 - Evidence for the use of fire at Zhoukoudian, China. *Science*, 281 : 251-253.

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CHRONOLOGICAL SIGNIFICATION OF LBK WASTE ASSEMBLAGES: THE CONTRIBUTION OF ANTHRACOLOGICAL, TYPOLOGICAL AND STRATIGRAPHIC DATA FROM THREE LBK SITES IN THE HESBAYE (LIÈGE PROVINCE, BELGIUM)

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Abstract

Considerations on the chronological signification of Linearbandkeramik (LBK) waste assemblages are presented in this study based on data from anthracology, ceramic seriation and vertical distribution of pottery in pits. Interesting evidence concerning the occupation chronology is provided and a more complex vision of LBK refuse disposal is proposed than that generally accepted. In particular, it is argued that pits, as they appear today to archaeologists, represent no more than a few years of use. It is likely that refuse was first dumped in surface middens, and gradually displaced afterwards to the pits we excavate today.

Keywords : LBK in the Hesbaye (Liège, Belgium), chronology, anthracology, pottery analysis, taphonomy, waste disposal

Introduction

This article presents some thoughts on the crucial question of the chronological significance of Linearbandkeramik (LBK) waste assemblages. It is mainly based on the ongoing study of the pottery and charcoal at several LBK sites in the Hesbaye area (Belgium), but the questions considered and the lines of thought suggested far exceed this regional context. It follows a recently published work on the dynamics of the establishment of the first Neolithic groups in this region of Belgium, which led to the discovery of a pioneer phase during its settlement around 5000 BC (Bosquet *et al.,* 2008; Salavert, 2008). These results were themselves based on a comparison of typological and anthracological data, the latter appearing to be an element able to reliably support the relative chronology since observations were repeated on several sites presenting a similar spatial organisation (Salavert, 2008). During the presentation of these works at the Naumur Conference in November 2006, an important question was raised concerning the ceramic corpus: decorated vessels originating from a single pit covered virtually all of the styles recognised for the Belgian LBK and the Dutch Limbourg, or the equivalent, according to I. Jadin (2003), of 150 to 200 years of occupation (cf. § 2). How can we explain this fact, which is, moreover, repeated in other structures and at other sites, given that it is difficult to imagine pits in continuous use for such a duration?

In Belgium, all of the material used in absolute or relative dating originates from hollow structures—mainly pits, but also ditches and, very rarely, postholes—whether or not they are associated with houses and with the exception of burials, which are practically absent from the area. Strangely, even though the chronology is based on the study of their content, questions such as those related to the duration of the use of these pits, the stratigraphic distribution of the material they contain and the general organisation and taphonomy of the waste, are very rarely discussed, if at all. In the same manner, questions related to the management of waste by Neolithic people have been infrequently raised up to now. Some authors have nonetheless examined these issues (David and David-Hennig, 1971; Rulf, 1986; Pavlu, 1986; Kreuz, 1990a, 1990b; Staüble, 1990, 1997; Pavlu, 1998; Last, 1998; Pavlu, 2000; Birkenhagen, 2003; Staüble, 2005; Kreuz, 2007), and highlight various methods of disposal of pottery containers (Last, 1998) or a link between various types of fill and their content in vessels (Rulf, 1986; Last, 1998). Others conclude that the pits were in short-term use (Staüble, 1990, 1997, 2005) or that they were used prior to and alongside the construction phase of houses without continuing throughout their occupation (Birkenhagen, 2003; Kreuz, 2007). Concerning waste management and the use-life of pits in the LBK, it generally seems that the situation is quite variable from one site to another and certainly complex (Last, 1998). Yet within the scientific community, the idea persists that as soon as everyday objects became useless they were immediately and casually thrown into the pits bordering the dwellings, to the extent that one assumes that the duration of use of these structures corresponds more or less to the duration of occupation.

The study presented here agrees with the previous results, which tend to show that the management of waste by Neolithic people was more complex. In particular, we can argue that the assemblages as they come to us today correspond to a relatively short period of occupation (a few years at most) of the houses on these sites. If such is the case, the reconstructed assemblages correspond not to the duration of the occupation, but rather to the state of the corpus at the moment of disposal.

LBK chronology: basic principles

The chronology of the LBK is based on two types of data:

- 1. Radiometric dating, which provides absolute dates with very approximate accuracy, in particular for the period corresponding to the Hesbayen LBK, for which the calibration curve is affected by a marked plateau (Jadin, 2003, fig. 1).

- 2. The analysis of part of the archaeological material, mainly the decorated pottery, leading to the division of the LBK period into a certain number of successive phases of which the number varies depending on the author and the region. An equal duration is arbitrarily attributed to each of the phases, since the lack of precision of C14 prevents the reliable determination of their duration as an absolute value. The scientific community admits the imperfection of the system but has none better to suggest at present. The Hesbayen LBK period thus covers approximately 200 years, between 5150 and 4950 BC (Jadin, 2003) and divides, according to Modderman's (1970) typology, completed by Jadin (2003), into two periods of three and five phases respectively, or eight in total, each phase assumed to last around 25 years.

The decorative repertoire on which Modderman's chronology is based includes 33 principal or secondary patterns, which, according to their position on the containers, are also considered as discriminating factors in chronological terms. Nearly all of these patterns cover several of the eight identified phases, so the presence of one single pattern only rarely permits the attribution of the vessel to a single phase. Most often, only associations of pattern and form permit the narrowing of the typo-chronological window to a few phases, or even to one phase in some cases.

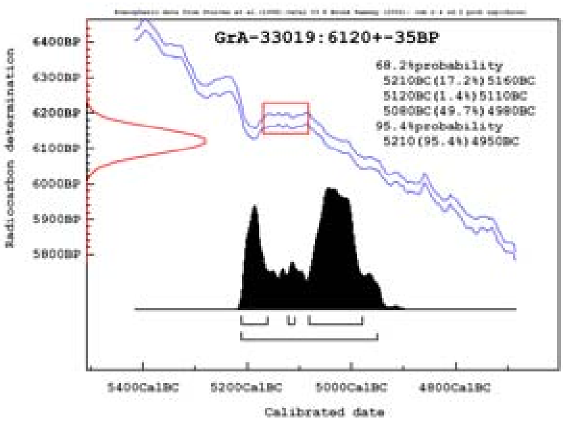

Fig. 1 - Fexhe-le-Haut-Clocher «Podrî l'Cortri». Radiocarbon date obtained on hazelnut tree bark, showing the plateau effect on the calibration curve (red box).

Context of the study

The study presented here is based on the analysis of nine pits divided between the sites of Remicourt «En Bia Flo» II (seven pits) and Fexhe-le-Haut-Clocher «Podrî l'Cortri» (two pits), excavated by the Walloon Region in collaboration with the Belgian Royal Institute of Natural Sciences along the TGV route 25 km to the west of Liège (Bosquet *et al.*, 2004) and a ditch excavated at the site of Waremme "Longchamps" on several occasions between 1988 and 2005 (Keeley *et al.*, 2005). The common feature of these structures is their production of both charcoal and decorated pottery, which is not always the case for pits. This contribution constitutes part of the publication of these sites in the form of multidisciplinary monographs.

Field methods

After drawing a plan at 1:50, the pits are excavated in squares in arbitrary levels of 10 cm. The archaeological material is labelled and packaged as it is discovered. During this operation, if concentrations of charcoal are encountered, they are removed immediately, like the other objects. Once the base of the structure is reached, the sections are carefully cleaned and photographed. The layers that comprise the fill are then highlighted with a stroke of the trowel and the section is drawn at 1:10 on graph paper.

The layers to be sampled are then selected according to various criteria, most often in collaboration with palaeoenvironmental specialists and are removed almost in their entirety in plastic bags for laboratory analysis. Each sample is indicated on the site plan at 1:10 and photographs of details are taken if necessary.

This method permits an excavation that is both rapid—a fundamental criterion in the rescue context—and that presents a sufficient degree of precision when situating the objects and samples stratigraphically (cf. § 7.4.3).

Laboratory methods

Pottery

After cleaning, each pottery sherd is marked (Site/pit/square/depth). The fragments are then attributed to a particular vessel on the basis of reconstructions, but also, when this is not possible, on the basis of technological criteria: decoration, clay, rim form etc. Thus, for each pit, a variable number of sherds cannot be reliably attributed to one or other vessel and are excluded from the final count. Next, the sherds of each vessel are numbered. The list by sherd notes, in addition to the provenance (Site/pit/square/depth/layer/vessel), the size of the fragment in cm² (cf. § 6).
It is important to make clear that the excavation method using arbitrary 10 cm levels does not allow the immediate attribution, in the field, of each sherd to one of the stratigraphic units representing successive waste episodes. The sherds are thus associated *a posteriori* with one of these layers by positioning them on the section drawings according to the excavation square and depth of discovery noted on the sherd. Clearly, this method implies a certain degree of inaccuracy, particularly for the non-horizontal layers covering all or part of the depth of the pit, but it appears that this case is quite rare and that it is more often possible to attribute the fragments accurately to a particular waste layer.

Charcoal

As the sediments removed during excavation were very clayey, samples were placed in an oven for 24 hours at 50 °C in order to dry the sediment and thus to facilitate its dispersion. They were then dispersed in a basin of water and sieved, also in water, with a 250 µm mesh. As a result, fragmentation of the material due to manual pressure is eliminated, since the charcoal is released easily from its silty matrix.
The charcoal was then fractured along three planes (transverse, longitudinal-tangential and longitudinal-radial) observed under a reflecting microscope and identified with the help of an identification atlas (Schweingruber, 1990) and reference collections from the IRNSB (Brussels) and the UMR 7041 (MAE, Nanterre, France).

Questions related to the taphonomy and stratigraphic distribution of the archaeological material

An initial question is related to taphonomy: are there vertical displacements of objects preserved in the pits according to their size? One could imagine, for example, that a sherd of 1 or 2 cm² is more likely to fall down through a hole than a sherd of 20 cm², which would lead to a possible vertical distribution according to size, with a higher proportion of small objects found at greater depth. If this is the case, we must conclude that the disturbances of all sorts that occurred over thousands of years have introduced a serious bias in a stratigraphic interpretation based on the position of sherds or lithic objects implicated in the reconstructions.
Two other questions are centred on the stratigraphic and planimetric distribution of the archaeological material, which have been examined according to two criteria. Firstly, how are the components of reconstructed lithic objects, or the sherds belonging to reconstructed vessels, distributed between the pits and within each of them? Next, is there a vertical distribution of the pottery that is consistent with the typology? If, as one tends to think, the pits were gradually filled throughout occupation, one might expect some sort of logic in the content in terms of vessels in the layers that make up the fill: the sherds that comprise the identified vessels should be distributed somewhat horizontally, according to the arrangement of the waste layers, and one should find the older vessels more towards the bottom of the pit, while the more recent ones should be concentrated further up.
It should be noted at the outset that, no matter which site is considered, the pits in question here have all suffered an average erosion of 70 cm, a value that represents at least a quarter of the fill of the original structure, and up to two-thirds. It is therefore important to remember that the layers of the final period and of the abandonment of

the structures, together with the archaeological material that they perhaps contained, are today completely absent. There is also often, though not always, a link between the depth of the pits and the quantity of material collected during an excavation. That said, since all of the pits on all of the sites in the region have suffered this type of damage to a relatively constant degree (between 60 and 80 cm of erosion), one could reasonably consider that the contents of the pits of equivalent depth may be studied and compared without risk of introducing a major taphonomic bias into the results. All the more so because, as we shall see later, the waste management method seems to have been relatively constant from one site to another. Nevertheless, this erosion must of course be taken into account as a potential gap in our estimation of the duration of activities and occupations.

The Remicourt «En Bia Flo» II site

Pits selected and quantity of material analysed

This site, excavated on several occasions between 1997 and 1998, is located 25 km west of Liège, along the E40 Brussels-Liège motorway (Bosquet *et al*., 2004).

Pits 10 and 141, associated with the external house, and 90, 113, 160, 234 and 235, situated within the enclosure, were selected for this study (fig. 2). These structures produced 594 sherds belonging to 135 vessels, of which 116, decorated, could be situated in Modderman's (1970) chronology. A total of 1376 pieces of charcoal were collected. Whether external or internal to the village, the material analysed - sherds, lithic objects and charcoals - originate from two very different layer types.

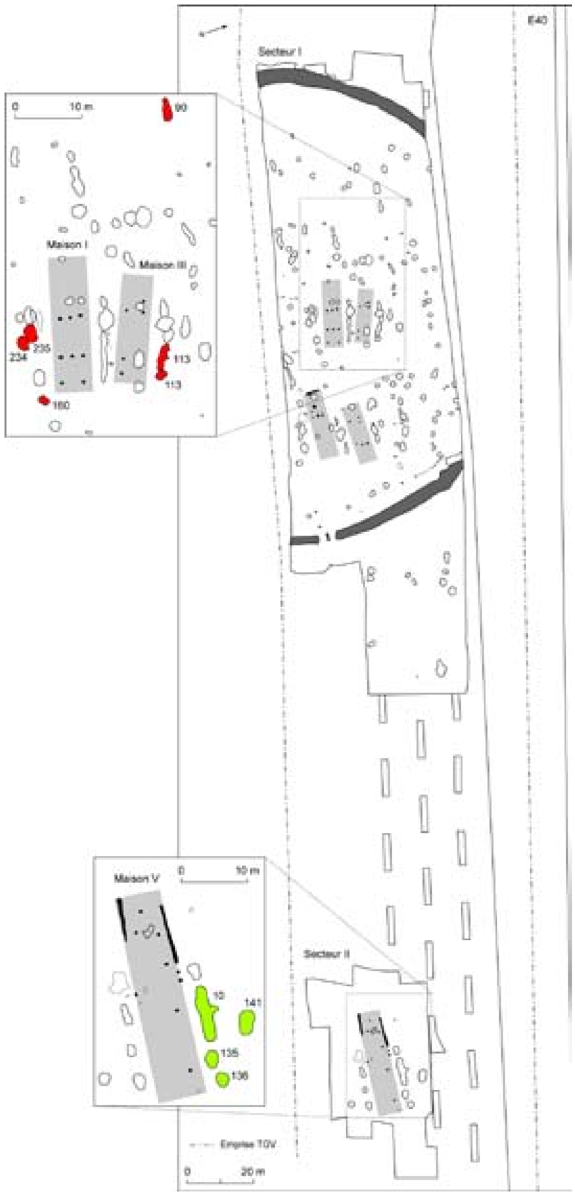

Fig. 2 - Remicourt «En Bia Flo» II. Location of the studied pits on the excavation plan (pits 135, 136, see chapter 7.2.2).

Description of type 1 and 2 layers

Type 1 layers (fig. 3, n° 1 to 6), present in six of the seven pits studied, share the following characteristics:

- these are layers with concentrated charcoal, around ten centimetres thick;
- they always lie near to or at the bottom of the pits;
- the only material present is charcoal with, in some cases, a few carpological remains and sparse fragments of burned earth;
- they most often contain a reduced number of taxa.

Type 2 layers (fig. 3, n° 1 to 6), present in six of the seven pits studied, share the following characteristics:

- they contain all types of everyday waste (carbonised vegetable remains, pottery, lithics, fragments of reddened earth, etc.), present in highly-variable proportions and accumulated in thicknesses of between 20 and 60 cm. In certain cases it is possible to discern several sub-layers within type 2 layers (fig. 4), each probably corresponding to a waste episode;
- charcoals is found dispersed in the sediment;

- type 2 layers often occupy the upper half or two-thirds of the pit, sometimes more, not including the thickness truncated by erosion;
- they contain a relatively high number of taxa.

Stratigraphic relationship between type 1 and 2 layers

In pits 10, 90, 160 and 234, where the two types of layer are present, two scenarios have been noted. Either a type 2 layer rests directly on the type 1 layer (fig. 3, n° 3 and 4), or there is a sterile fill layer between the two (fig. 3, n° 1, 2, 5 and 6).

Given the extent of erosive processes in a temperate climate on bare surfaces, Neolithic pits and ditches become filled naturally by collecting runoff mud or after the erosion and/or collapse of their walls as a result of weathering. A night of violent showers is sometimes sufficient to fill an excavation significantly (fig. 5); once again, this is a rapid phenomenon. An experiment recently carried out (Broes et Bosquet, 2007) showed that in one year a ditch became half-filled without any human intervention.

On the basis of these observations, we can say that where anthropogenic waste layers are in direct contact with each other, this means that the layers have quickly succeeded each other leaving no time for the natural filling process to develop. In the same way, given the rapidity of erosive phenomena, the presence of a sterile layer between two anthropogenic waste layers cannot be advanced as an argument to suggest a significant extension of the duration of use of the pits whose fill includes this type of layer. This is also true—and *a fortiori*—if the sterile layer indicates intentional filling carried out by Neolithic people with the aim, for example, of covering organic rubbish, probably regularly mixed with object debris.

Pottery

Stratigraphic distribution of sherds according to their size

Given the numerous bioturbations present in Neolithic pits—resulting from several millennia of biological activity—it is reasonable to imagine that a certain number of objects, particularly the smallest, will have been affected by these essentially vertical movements. In order to test the hypothesis of a vertical classification of sherds according to their size, induced by natural phenomena, a diagram showing the relationship between sherd size and their discovery depth has been drawn up for each pit (fig. 6). These diagrams show without doubt that there is no link between the two parameters: sherds of all sizes are spread from top to bottom in the pit, in no particular order.

Reconstructions between pits

For the pioneer house, although the reconstructions between pits are still at a preliminary stage, three individual pottery vessels link pits 10 and 141 and five lithic reconstructions link these same structures and pits 135 and 136 (Allard, pers. com.). It is therefore reasonable to suppose that, at least in part, these pits were jointly utilized. The presence of fragments of an individual pottery vessel in several pits is also an indication that the vessel, once broken or judged unusable, was disposed of in different places and thus very probably at different times, without here prejudging the lapse of time between each of these disposals.

For the village, the reconstructions between pits have not yet been carried out.

Reconstructions by pit

For both the exterior and interior of the village, the analysis of the reconstructions from each pit clearly show that the sherds comprising an individual vessel are distributed throughout the whole depth of the fill, very often in several layers (fig. 7). This is also true for the lithic reconstructions carried out for the pits belonging to the external house. If, for reasons linked to the excavation technique, doubts may persist as to the attribution of certain sherds to one or another layer (cf. § 5.1), there is little room for discussion regarding the reconstructions linking objects originating from the top and the bottom of pits.

We thus realize that the question concerning the existence of a stratigraphic logic according to typology, with the oldest objects at the bottom of the fill and the most recent at the top, becomes irrelevant.

As with the vessels present in several pits, it seems that those present in one pit were also disposed of at several different times.

Fig. 3 - - Types 1 (a) and 2 (b) layers and their organisation in some pits from Remicourt, with or without sterile layers (c).

Fig. 4 - Remicourt «En Bia Flo» II. Type 2 layer (b) containing sub-layers (underlined in white) indicating several waste dumping episodes.

Fig. 5 -Remicourt «En Bia Flo» II. The effects of one night of rainfall on an excavation square comparable to LBK pits: collapse of the vertical wall (a), erosion of the oblique wall and of the surface around the pit (colluvium).

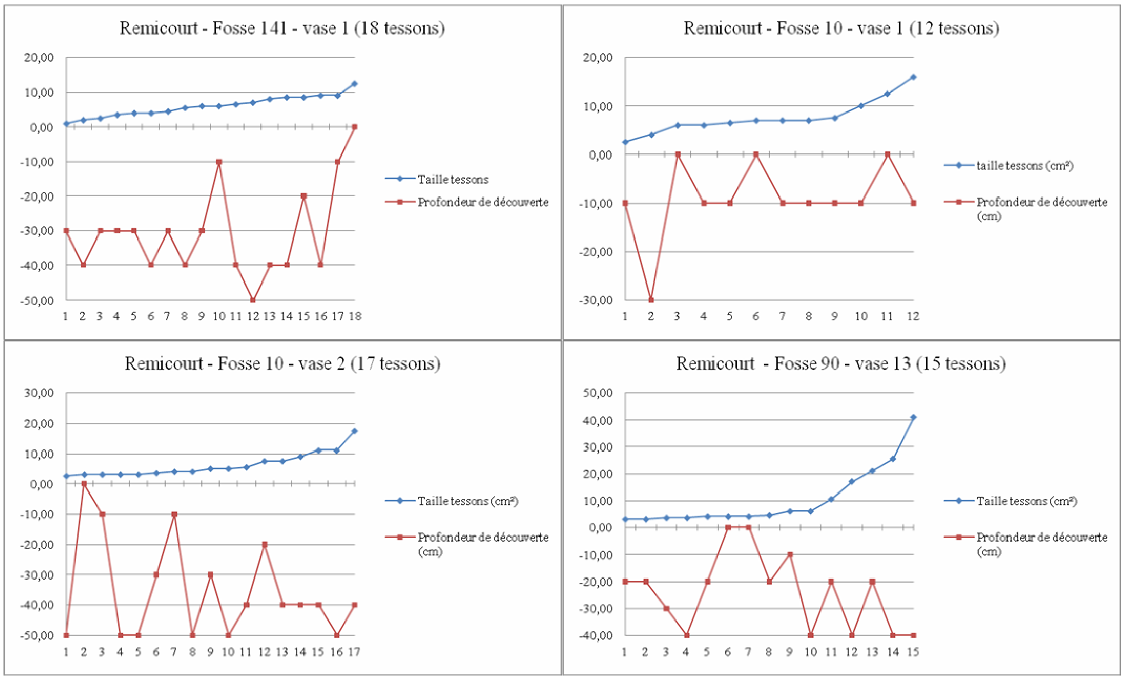

Fig. 6 - Graphics showing the absence of correlation between sherd size and discovery depth (pits 10, 141 and 90 from Remicourt).

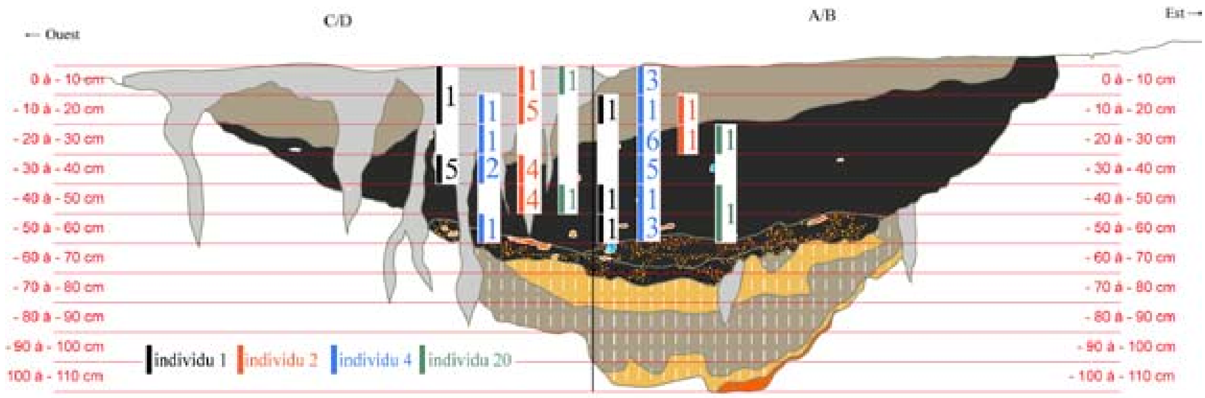

Fig. 7 - Remicourt «En Bia Flo» II, pit 141. Vertical distribution of ceramic sherds from 4 vessels (the number of sherds is indicated beside the coloured rectangles). All the vessels are distributed across the two major waste layers (type 2 layers), over 60 cm maximum. Light beige, grey and orange layers at the bottom of the pit are sterile, while the brown layer with orange dots corresponds to one or several layers of hearth cleaning waste (type 1 layer). It should be recalled that the pit is affected by erosion of 40 to 60 cm.

Chronology

The pioneer house

Pits 10 and 141 produced respectively 35 and 25 decorated vessels likely attributable to Modderman's (1970) phases and periods. The summary table of the typological attributions for the two pits (fig. 8) shows above all that no individual vessel can be attributed to only one of the eight phases, each vessel covering at least two (2 cases out of 60) of them, or at most six (6 cases out of 60), and the majority covering five (32 cases out of 60).

In addition, the two pits present a very similar image: the individual vessels that one could consider as the oldest—two of which are located exclusively in period I—are considerably fewer in number than the more recent vessels. Amongst these latter, 29 vessels are located between Id and IId, and

Fosses 10 et 141 - 60 individus (i)								
Individus	Période I			Période II				
	Ib	Ic	Id	IIa	IIb	IIc	IId	IId+
i1	x	x	x					
i2	x	x	x					
i3	x	x	x	x				
i4	x	x	x	x	x	x		
i5	x	x	x	x	x	x		
i6	x	x	x	x	x	x		
i7	x	x	x	x	x	x		
i8	x	x	x	x	x	x		
i9	x	x	x	x	x	x		
i10		x	x	x	x	x		
i11		x	x	x	x	x		
i12		x	x	x	x	x		
i13		x	x	x	x	x		
i14			x	x	x	x	x	
i15			x	x	x	x	x	
i16			x	x	x	x	x	
i17			x	x	x	x	x	
i18			x	x	x	x	x	
i19			x	x	x	x	x	
i20			x	x	x	x	x	
i21			x	x	x	x	x	
i22			x	x	x	x	x	
i23			x	x	x	x	x	
i24			x	x	x	x	x	
i25			x	x	x	x	x	
i26			x	x	x	x	x	
i27			x	x	x	x	x	
i28			x	x	x	x	x	
i29			x	x	x	x	x	
i30			x	x	x	x	x	
i31			x	x	x	x	x	
i32			x	x	x	x	x	
i33			x	x	x	x	x	
i34			x	x	x	x	x	
i35			x	x	x	x	x	
i36			x	x	x	x	x	
i37			x	x	x	x	x	
i38			x	x	x	x	x	
i39			x	x	x	x	x	
i40			x	x	x	x	x	
i41			x	x	x	x	x	
i42			x	x	x	x	x	
i43			x	x	x	x	x	
i44				x	x	x		
i45				x	x	x		
i46				x	x	x		
i47				x	x	x		
i48				x	x	x		
i49				x	x	x		
i50				x	x	x		
i51				x	x	x		
i52				x	x	x	x	
i53				x	x	x	x	
i54				x	x	x	x	
i55				x	x	x	x	
i56				x	x	x	x	
i57				x	x	x	x	
i58				x	x	x	x	
i59						x	x	
i60						x	x	

Fig. 8 - Remicourt «En Bia Flo» II. Extra muros pits: decorated ware seriation.

17 others exclusively in period II, between IIa and IIc or d. For the two pits, we also note the total absence of phase IId+, characterised by pivoted comb patterns.

These typological characteristics permit the association of these pits with a transitional phase between the regional Middle and Late LBK.

The village

The same table was created for the pits located inside the enclosure (fig. 9), comprising between 25 and 5 decorated vessels suitable for this purpose, out of a total of 56. Contrary to the observations for the pioneer pits, a majority of vessels may be attributed to a single phase (40 cases) while those covering from two to six phases are more rare (16 cases).

Once again, the oldest vessels are very clearly fewer in number than those attributed to later phases. As has already been indicated (Bosquet *et al.*, 2008), these pits correspond to the Late LBK period of the Hesbaye (IId), with a minor but clear presence of the final phase, IId+. The presence of a few vessels covering the same periods as those from the external pits allows for the possibility that, at least for a period, the two areas of habitation were jointly occupied.

Fosses 90, 113, 160, 234 et 235 - 56 individus (i)								
individus	Période I			Période II				
	Ib	Ic	Id	IIa	IIb	IIc	IId	IId+
i1	x	x	x	x				
i2	x	x	x	x				
i3	x	x	x	x				
i4	x	x	x	x				
i5	x	x	x	x	x	x		
i6	x	x	x	x	x	x		
i7		x	x	x	x	x		
i8			x	x				
i9			x	x	x	x		
i10			x	x	x	x	x	
i11				x	x	x	x	
i12					x	x	x	
i13					x	x	x	
i14						x	x	
i15						x	x	
i16						x	x	
i17							x	
i18							x	
i19							x	
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i46							x	
i47							x	
i48							x	
i49							x	
i50							x	
i51							x	
i52								x
i53								x
i54								x
i55								x
i56								x

Fig. 9 - Remicourt «En Bia Flo» II. Intra- muros pits: decorated ware seriation.

Duration of use of vessels and temporal inequality of typological phases

The typological analysis provides a decisive element in the context of the problem addressed: whichever pit or habitation zone is considered, the number of older vessels is always considerably less than that of those decorated with later patterns. This cannot be due to chance only since it is equally true for two pits analysed in the same way at the site of Fexhe-le-Haut-Clocher (Bosquet and Van Driessche, 2008).

Two possibly complementary phenomena explain this particularity in the typological composition of the assemblages: the use-life of the vessels and the unequal duration of the typological phases.

Various studies have been dedicated to the question of the use-life of pottery vessels in diverse ethnographic contexts (Mayor, 1994; Shott, 1996; Varien and Mills, 1997; Shott and Sillitoe, 2004), of which some (David and David-Hennig, 1971) were taken up by Last (1998) during his works on Miskovice. According to these studies, the use-life of vessels is very clearly linked to their size and thus often to their use. The oldest vessels are therefore large storage vessels, which are less mobile and thus less likely to be broken than cooking and serving vessels. For other vessel categories, the use-life varies in most cases between a few months and 15 years (Mayor, 1994; Shott, 1996, 476, table 5; Varien and Mills, 1997, 174-177, table A1), but small and medium vessels that are comparable in this respect to those studied here may sometimes significantly surpass these figures, being preserved for up to 50 years or more (Mayor, 1994, 192). In this context, we should recall that at the site of Fexhe-le-Haut-Clocher, some decorated vessels that had been entirely broken were repaired with birch bark tar by the Neolithic inhabitants (Bosquet *et al*., 2001).

The other element that may have an impact on the smaller number of older vessels is the unequal duration of the stylistic phases, the older phases having lasted for a shorter time. In support of this idea, one could consider the chronology of the sites of Darion "Colia" and Waremme "Longchamps" whose several phases of construction indicate the length of their occupation, but which have yielded only vessels attributed to period II, the vast majority being from IId and IId+ (Jadin, 2003).

Charcoal

Analysis of the taxa abundance in the type 1 and 2 layers of the pioneer house and the ditched village

In the pioneer pits (pits 10 and 141), two type 1 deposits and one type 2 deposit were considered (figs. 10, 11 and 12). They are each characterised by a low number of taxa and the exclusive presence of ash (*Fraxinus excelsior*), hazel (*Corylus avellana*) and oak (*Quercus* sp.). Only the latter is absent from the dispersed deposit in pit 10. Across the site, twelve taxa have been identified (Salavert, 2008), emphasizing the particularity of these taxa-poor *extra muros* deposits. Given the reproducibility of the results from one deposit type to another and from one pit to another, this cannot be due to the amount of charcoal analysed, nor caused by sampling bias (fig. 13).

In the village, the anthracological assemblage is much more diversified (fig. 14) than in the *extra muros* context. Ash, hazel and oak are included, together with Pomoideae (apple, pear, hawthorn and rowan family), elm(*Ulmus* sp.), willow/poplar (*Salix-Populus*), cherry/sloe (*Prunus* sp.), elder (*Sambucus* sp.), lime (*Tilia* sp.), buckthorn (cf. *Frangula* sp.) and maple (*Acer* sp.). The analysis is based on four type 1 layers and five type 2 layers. Among the type 1 deposits (fig. 10), three (pits 90, 160 and 235) are relatively poor in tree species, (three or four taxa) while another (pit 113) yielded a higher number of species (seven taxa). The type 2 deposits (fig. 11) generally contain five or more taxa. Only the deposit in pit 90 contains just two tree species, but the low number of charcoal pieces identified (N=16) explains this result. If we now compare all of the tree species identified in the type 1 layers with those of type 2 (fig. 12), we see that elm, buckthorn and maple are represented only in type 2 assemblages. However, the two latter taxa are rare at Remicourt and elm is not one of the dominant taxa. While each type 2 layer generally contains a higher number of tree species than each

COUCHES DE TYPE 1

Structure	10	141	90	113	160	235
Couche	**1**	**4**	**2b**	**2**	**3**	**2**
Profondeur en cm	**40-60**	**70**	**60**			
Carré	A1B1	B		B2/1		A2/B2 et D/A1
Poids non tamisé en kg	2	4,4	0,52	1,252	0,651	1
Poids charbons après tamisage en g.	4,94	4,0698		2,585	0,8569	0,349
N° Inventaire	A1753 A1756	A1807		A1788	A 1769	A1779
Localisation des structures	*extra muros*		*intra muros*			
TAXONS						
Quercus sp. (fc) - Chêne	11,4	3	79,54	53,78	64	84,61
Pomoideae - Pomoïdées	-	-	13,63	19,69	-	5,77
Fraxinus excelsior - Frêne	71	75	2,27	-	-	9,61
Corylus avellana - Noisetier	17,5	22,50	4,54	17,42	-	-
Ulmus sp. - Orme	-	-	-	-	-	-
Salix /Populus sp. - Saule/peuplier	-	-	-	3,03	-	-
Prunus sp. - Merisier/prunellier	-	-	-	0,75	-	-
Sambucus sp. - Sureau	-	-	-	0,75	36	-
Tilia sp. - Tilleul	-	-	-	4,54	-	-
Cf. Frangula sp. - Bourdaine	-	-	-	-	-	-
Acer sp. - Erable	-	-	-	-	-	-
Nombre de charbons	**114**	**80**	**44**	**132**	**50**	**52**

Fig. 10 - Remicourt «En Bio Flo» II. Anthracological results (%) for type 1 layers (concentrated).

COUCHES DE TYPE 2

Structure	10	90	113	160	234	235
Couche	**5**	**3**	**3**	**5**	**2**	**3**
Profondeur en cm	**20-30**	**30-40**			**couche sup.**	
Carré	A2	A2/B2	B2/1		A2/A3	A2/B2 et D/A1
Poids non tamisé en kg	2	0,43	1,272	1,352	0,298	3
Poids charbons après tamisage en g.	6,104	0,9297	1,272	1,093	0,9386	1,17
N° Inventaire	A1755 A1757	A1805	A1789	A1768	A1782	A 1780 A 1781
Localisation des structures	*extra muros*		*intra muros*			
TAXONS						
Quercus sp. (fc) - Chêne	-	93,75	35,63	45,11	61,90	77,06
Pomoideae - Pomoïdées	-	6,25	14,94	49,62	23,80	8,71
Fraxinus excelsior - Frêne	72,27	-	2,29	-	8,33	0,46
Corylus avellana - Noisetier	27,72	-	10,34	-	4,76	-
Ulmus sp. - Orme	-	-	27,58	0,75	-	-
Salix /Populus sp. - Saule/peuplier	-	-	1,14	-	-	6,42
Prunus sp. - Merisier/prunellier	-	-	8,04	2,25	1,19	4,59
Sambucus sp. - Sureau	-	-	-	0,75	-	0,46
Tilia sp. - Tilleul	-	-	-	-	-	0,92
Cf. Frangula sp. - Bourdaine	-	-	-	0,75	-	1,38
Acer sp. - Erable	-	-	-	0,75	-	-
Nombre de charbons	**101**	**16**	**87**	**133**	**84**	**218**

Fig. 11 - Remicourt «En Bia Flo» II. Anthracological results (%) for type 2 layers (dispersed).

Localisation des stuctures	*Extra muros*		*Intra muros*	
Types de couches	**TYPE 1 (concentrées)**	**TYPE 2 (dispersées)**	**TYPE 1 (concentrées)**	**TYPE 2 (dispersées)**
Nombre de structures	**2**	**1**	**4**	**5**
Poids non tamisé en kg	6,4	2,4	3,4	6,4
Poids charbons après tamisage en g.	9	6,1	3,79	5,4
TAXONS				
Quercus sp. (fc) - Chêne	X	-	X	X
Pomoideae - Pomoïdées	-	-	X	X
Fraxinus excelsior - Frêne	X	X	X	X
Corylus avellana - Noisetier	X	X	X	X
Ulmus sp. - Orme	-	-	-	X
Salix /Populus sp. - Saule/peuplier	-	-	X	X
Prunus sp. - Merisier/prunellier	-	-	X	X
Sambucus sp. - Sureau	-	-	X	X
Tilia sp. - Tilleul	-	-	X	X
Cf. Frangula sp. - Bourdaine	-	-	-	X
Acer sp. - Erable	-	-	-	X
Nombre de charbons	**194**	**101**	**278**	**538**

Fig. 12 -Remicourt «En Bia Flo» II. Comparison of taxonomic lists (X = presence, - = absence) between the two layers types in the two zones (extra and intra muros).

type 1 layer, the list of taxa identified for each type is fairly homogenous. It therefore appears, at this stage of analysis, that the determining factor in the abundance in taxa in the samples is their *intra muros* or *extra muros* location, once sample size has been accounted for. The deposit type is of only secondary significance in explaining taxa abundance. Based on this analysis, it is possible to consider the possible causes for the number of taxa, their nature and proportions in the samples.

What time-span, what environment type and what activities can the intra and extra muros pits indicate?

Basic principles

When an anthracological assemblage contains a low number of taxa, the deposit may potentially correspond to one or two collections of firewood, particularly if the sample originates from contexts rich in charcoal. On the contrary, waste deposits accumulated over time are more appropriate contexts for suggesting palaeoenvironmental interpretations since they are likely to represent several collections of firewood (Chabal, 1994, 1997). Starting from these basic principles, several questions arise: what information in terms of occupation duration can be extricated from the analysis of the taxonomic abundance of type 1 (concentrated in charcoal) and type 2 layers (dispersed) and—in terms of the differences observed in the anthracological assemblages of the pioneer house and the ditched village—what were the woodland types exploited, how did they develop and which activities do they indicate?

Waste duration

The type 1 layers, whether *intra* or *extra muros* and characterised by a major concentration of charcoals have, in four cases (*intra muros* pits 10 and 141; *extra muros* pits 160 and 235), low or somewhat low taxonomic diversity in terms of the number of charcoal pieces analysed (fig. 13). For the *extra muros* pits, we have seen the link between this lack of diversity and their situation in the pioneer house, whatever the deposit type (cf § 7.5.1). For the pits in the village, their concentrated nature is involved. We can estimate that deposits of type 1 in pits 160 and 235 (*extra muros*) represent a rapid waste episode with a very short timescale, particularly as the waste is fine, regular and there is no archaeological material. However, the type 1 waste of pits 90 and 113 (*intra muros*) have an increased taxa abundance when compared with the amount of charcoal analysed (fig. 13). Not all of the concentrated deposits are taxa-poor. This demonstrates a classic anthracological observation: it is highly likely that concentrated charcoal deposits (such as hearths) reflect a short activity duration (with few taxa represented),but they may also prove to be abundant, and thus represent a certain number of collections, sufficient to result in reasonable representation of the firewood used over time.

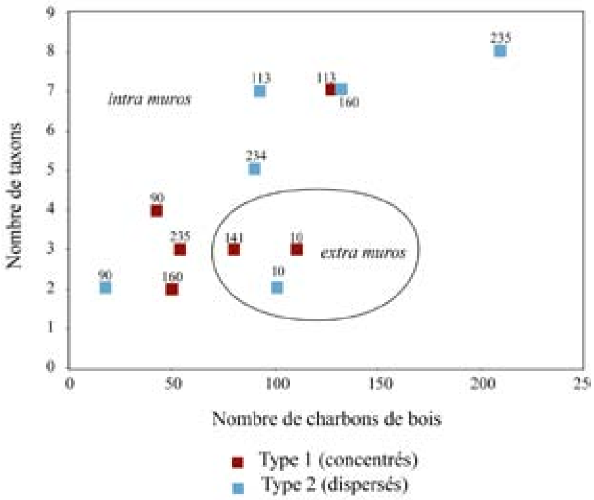

Fig. 13 - Remicourt «En Bia Flo» II. Number of taxa in comparison with the number of pieces of charcoal analysed for each sample. We note that in the extra muros pits, the low number of identified taxa is not linked to a methodological problem as the phenomenon appears in the three features and, in addition, for a comparable amount of charcoal analysed in the intra muros pits, the number of taxa is higher. Finally, the type 1 layers (in red) are in general poorer in taxa (up to 4) than those of type 2 (in blue).

Being characterised by a dispersion of charcoal, the type 2 layers in the village have a high taxonomic diversity. It is thus possible to estimate based on anthracological analysis that these deposits represent a longer time-span than the type 1 layers, particularly as the waste is thick and contains a varied archaeological material. In addition, spatial analysis of the pottery indicates that it is likely that a few years were required to accumulate these detritus layers.

As we saw in § 7.5.1, if the spatial analysis of the pottery and lithic material is to be believed, a diffuse *extra muros* deposit (pit 10), while representing the same use-duration as in the interior of the village, presents a dearth of taxa comparable to that of the concentrated layers.

This taxonomic difference between the diffuse *extra* and *intra muros* layers cannot be explained by the duration of the waste episodes, but by the close link between certain activities carried out by the LBK people and the diversity of the environment and its development. This link is a function either of the spaces occupied or of the occupation phases, whether pioneer or principal.

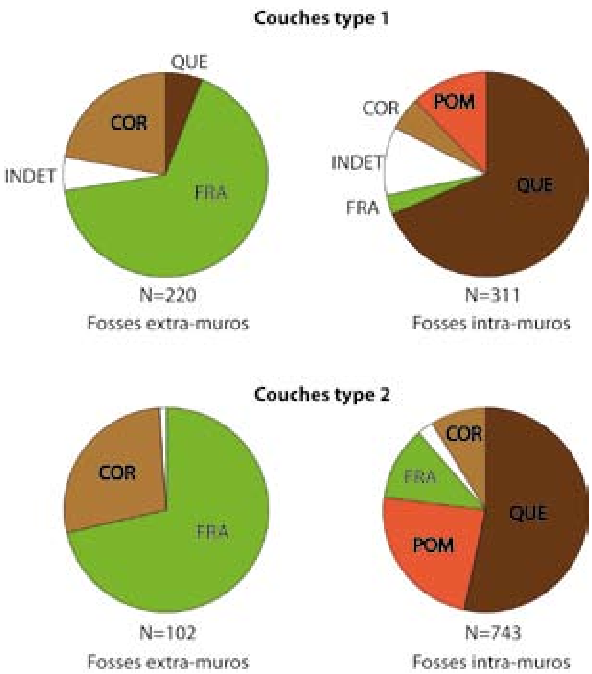

Fig. 14 - Remicourt «En Bia Flo» II. Anthracological spectra: on the left results from the two extra muros pits, on the right spectrum from intra muros pits. In the latter case, only major taxa present in the village are represented (Salavert, 2008), in order to illustrate the transition from ash, which dominates in the extra muros area, to oak dominating intra muros.

Ecology and development of the environment

In the three deposits studied in the extra muros pits, ash dominates significantly (more than 70%), followed by hazel (between 17 and 27%) and oak (between 3 and 11%), present only in the two deposits concentrated in charcoal. Ash is a heliophilous taxon adapted to humid edaphic conditions. At Remicourt, the village was established at the base of the slope, immediately adjacent to the bottom of the «valley of Bia Flo». It is difficult to know with certainty whether at that time a watercourse ran at the bottom of the valley in question, but it is very possible that the «pioneers» would have exploited the banks of the watercourse in order to bring the future village closer, as well as providing accessibility to the banks for the creation of fields and pastures for domestic animals. The presence of hazel, which is adapted to the shade of underbrush and also develops in hedges and the edges of woods on fertile soils, indicates the presence of woodland openings, perhaps maintained by the Neolithic people.

In the village, oak and the Pomoideae also dominate the assemblages by some margin. These are two heliophilous tree species that tolerate half-shade (Rameau *et al.*, 1989). Oak develops in copses on flat land and Pomoideae prefer woodland margins and hedges. In these formations, they can be accompanied by a great number of tree species (Bissardon *et al.*, 1997) such as Prunoideae, elder, elm, maple and hazel, all present in the *intra muros* assemblages. It thus seems that the inhabitants of the village exploited the mature woodlands as much as their margins, but were not particularly limited by watercourses and diversified their collections compared to the inhabitants of the first occupation.

Woodland resource management

In general, we should consider two activity types that are linked to the occupation period:

1. Clearance: deforestation is aimed both at creating open spaces and obtaining tree-trunks in the first phases of an occupation. It provides:

- timber comprising high forest species used for construction and rope (bark), or other carpentry elements;
- wood suitable for wattle, particularly taxa such as hazel;
- waste wood for use as fuel.

2. The everyday exploitation of fuel for use in various hearths linked to daily and craft activities, having taken place before, during, and after, the clearances and the construction of buildings.

While some selection of species is probable for construction purposes, it is generally recognize that for the supply of domestic hearths, most collection is more of an opportunistic nature. In collections specifically for fuel, the material «on hand» is used, or failing that, material that is available closer to home. For craft hearths, some selection is conceivable, though, at least from a strictly technical viewpoint, it is not really necessary, based on the ethnographic data and experiments devoted mainly to pottery firing.

If we apply these principles to the results obtained from Remicourt, it is tempting to see in the type 1 layers (concentrated charcoal) the waste from hearths fed to a large extent with construction waste, since the tree species usable for this purpose dominate significantly (ash and oak), whether outside or inside the village. The fact that these layers are consistently found close to the bottom of the pits, i.e., associated with the start of digging, reinforces this hypothesis. The *intra muros* presence, in two concentrated deposits, of Pomoideae, a taxa *a priori* inappropriate for construction, particularly in the case of hawthorn (although possible if rowan), does not contradict this idea; it simply illustrates the fact that hearths maintained during the construction of the houses were not exclusively fed with waste products.

The composition of the *intra muros* type 2 diffuse layers that contain a diversified number of taxa corresponds well to the idea we have of opportunistic collections carried out over a certain period. However, this is not the case with the only *extra muros* diffuse layer (pit 10), which does not show any taxonomic development in comparison with type 1 layers, even though it accumulated during a time span equal to that of the *intra muros* diffuse layers (cf. § 7). At least two explanations are possible. This may also be an opportunistic collection, but in an environment that was still little modified since the construction of the first house, so that, whatever the use—fuel or construction—the species are the same, and the wood is taken from the dominant species. This is not to say that other species, including Pomoideae, are not already present, but rather that they do not dominate sufficiently to be included in opportunistic collections. According to another hypothesis, this may indicate collection linked to a specific activity. Clearances immediately come to mind, which one can easily imagine to be one of the main tasks of the first colonists. While ash is not very resistant to insect attack (Bakels, 1978), both it and oak do have trunks of the size and diameter suitable for the construction of LBK houses. Due to its suppleness, hazel can be used in wattle. Thus by carrying out woodland clearance, the LBK builders also provided themselves with construction timber and fuel. The very obvious predominance and recurrence of these taxa in the pits analysed illustrates their abundance near the site during the first stage of colonisation. In addition, it is entirely possible that during clearances certain species judged useful for food were spared or even favoured. This may particularly be the case for the Pomoideae, whose appearance in the anthracological assemblages of the village shows that at this point in the occupation these taxa dominated sufficiently around the habitation to be gathered during opportunistic collections. The abundance of these species may be due to a natural development because of the previously created openings. In the Pomoideae group, several types such as rowan or hawthorn are heliophilous and linked to woodland margins, clearings or light woodland. These environments may exist naturally but their particular abundance, as is the case here, can be hypothetically linked to the recolonisation by these taxa of deforested spaces, and thus to previous thinnings or clearances. We see no other explanation, either technological or in terms of fuel, for their abundance. This thus demonstrates the anthropogenic impact on the area around Remicourt between the pioneer and secondary occupations. This is all the more probable since this situation is repeated across several villages where the pioneer settlement combines, in each case, a reduced assemblage that is free from Pomoideae, with an older pottery assemblage than that of the secondary settlement, but also with a noticeably different method of supply in terms of ceramic and lithic raw materials (Bosquet *et al.*, 2008; Salavert, 2008; Golitko *et al.*, 2009). This is the case at the two sites presented in the following section: Fexhe-le-Haut-Clocher "Podrî l'Cortri" and Waremme "Longchamps".

Fexhe-le-Haut-Clocher «Podrî l'Cortri», and Waremme «Longchamps»

Stratigraphic distribution and typology of individual pottery vessels

For the Fexhe site, only two pits have been analysed according to the same methods, but the two structures concentrate 60% of the pottery collected, i.e., 1045 sherds forming 192 individual vessels (Bosquet et Van Driessche, 2008).

In both pits, the sherds of a few vessels are grouped by layer. These are fragments of containers composed of eight sherds at most, of which some were clearly broken after burial, judging by the presence of fresh breaks. The other vessels are composed of sherds distributed throughout the depth of the pits, as at Remicourt (fig. 15).

At Waremme, a section of the enclosure ditch has been studied. It yielded 66 decorated vessels whose sherds are once again vertically distributed over more than 1 metre and between several waste layers.

From a typological point of view, the situation at Fexhe is comparable with that of Remicourt: in the pioneer pits, some old vessels are associated with more recent ones, with no combed patterns, while in the village combed patterns appear—stroked in this case. At Waremme, the pioneer house is attributed to IIc, while the village develops in IId. In this case, the comb is omnipresent, but in very different proportions: between 5 and 10% in the pioneer house and 68% for the village (Bosquet *et al.*, 2008). We must add to this the fact that a very clear difference is consistently observed between the pioneer and

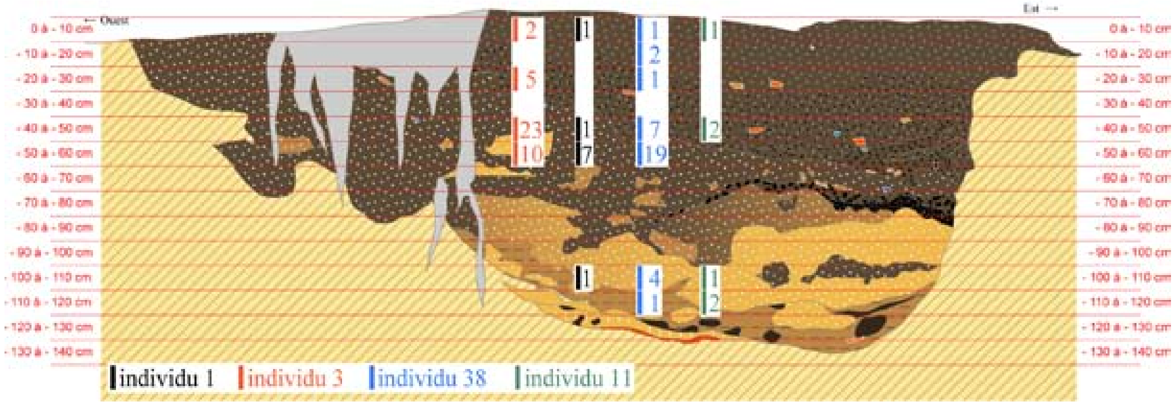

Fig. 15 - Fexhe-le-Haut-Clocher «Podrî l'Cortri», pit 314. Vertical distribution of ceramic sherds from 4 pots (the number of sherds is indicated beside the coloured rectangles). As at Remicourt, the vessels are distributed over a considerable depth (60 to 120 cm), corresponding to at least two waste episodes. Here again the pit is affected by 40 to 60 cm of erosion.

principal establishments in terms of the supply of lithic and ceramic raw materials (Bosquet *et al.*, 2008; Golitko *et al.*, 2009).

The observations made for these two sites during their study thus clearly confirm the results obtained at Remicourt and tend to show that the use duration of vessels, the method of waste management and the periodicity of disposal are relatively constant from one village to another.

Composition of the anthracological assemblages

At both Fexhe and Waremme, the assemblages from the pioneer settlements are characterised by a small number of taxa always consisting of oak, ash, hazel and elm. Lime is also present, particularly at Fexhe. This occurrence supports the age of the occupation since lime characterises the forests that developed at the time of the arrival of LBK people in Middle Belgium (Bakels, 1992). Pomoideae are represented by only one piece of charcoal out of the 560 identified in the pioneer house at Waremme. However, in the villages, the collection of firewood diversifies. Pomoideae, although not reaching the percentage attained at Remicourt (Salavert, 2008), are well represented (between 7 and 8%) given that the rate of cherry/sloe, which also prefers woodland margins, reaches 15% at Waremme. Therefore, the same observations may be proposed for the three sites at which at least two occupation phases have been identified. Pomoideae characterise the secondary occupations, suggesting that these tree species are favoured by human activities.

Discussion/conclusion: waste management, periodicity of disposal and occupation duration documented by the LBK pits

The type 1 *intra* and *extra muros* layers, exclusively composed of charcoals, correspond to a few disposals probably made during construction of the houses, that is during a very short or rather short period. In four cases out of six they are composed of a low number of tree species, and in two cases out of six—but only in the village—of a number of taxa equal to that of the type 2 layers (the abundance being reflected in the amount of charcoal analysed). These two cases do not bring into question the shortness of the fill period since in anthracology we see the same type of ambiguity in hearth abandonments, for example. This indicates that a short activity duration generally produces few taxa, but may, given the randomness of wood collection, produce a higher number of tree species.

In the type 2 layers (mainly present in the LBK pit fill), though they are sometimes composed of superimposed sub-layers, it seems that the majority of the pottery, used lithic material and hearth waste that they contain was not immediately disposed of upon breakage, wear or successive uses. The objects were first moved elsewhere, probably in the open-air and perhaps to be used, with other materials and over some time, in recycling contexts such as we still see in ethnographic cases (Beck and Hill, 2004). When the mass of waste was considered too intrusive and/or the remaining fragments were unusable, all or part of the midden was removed to the available pits and/or to pits dug for this purpose. After some period of additional accumulation, the next disposal into a pit or pits occurred, forming the following layer and so on until the abandonment of the site. This is the method of waste management that can best explain the mixture observed here. The numerous associations between sherds and flint flakes originating from layers sometimes located at the bottom and the top of the pits, also show that these layers would have accumulated quite rapidly since it is difficult to imagine that several fragments of a single vessel or a lithic reconstruction would have been consistently disposed of tens of years apart. However, if this waste was disposed of over a few years at most, this would indicate that the material originating from the pits does not reflect the total duration of occupation, but rather the state of the corpus over the duration of the accumulation of waste; at most a few years. It seems rather unlikely that the occupation duration of an LBK house does not exceed a few years. If this is indeed the case, we must admit that only a small proportion of the waste material is accessible to us through excavation, the rest having been washed away by erosion after the abandonment of the site, whether from the upper part of the pits or the residual middens.

Consequently, as we might expect, the presence in these pits of containers covering nearly the entire regional chronology is not due to the fact that the houses were occupied for 150 or 200 years, but rather to the durability of a small number of old vessels and to the unequal duration of the typological phases, the oldest probably having been shorter than the more recent, at least in the Hesbaye.

Regarding the positioning of the waste heap(s) in the villages, we can hypothesise that they occupied some of the zones surrounded by waste pits and without post holes present in all the habitations, such as in the village *Dalupa* analysed by Beck and Hill (2004, 304, fig. 2) during their study of the management of middens in the Philippines. These are generally explained as the locations of houses whose foundations have been entirely destroyed by erosion. While this possibility remains valid, it is not unreasonable to consider that at least some of these areas were open-air rubbish zones.

In terms of the archaeobotanical aspect of this study, we must accept the evidence that type 1 layers are not able to provide certain information on the state of forest coverage during the LBK occupation. On the other hand, the type 2 waste layers more consistently represent a long timescale and thus contexts more appropriate for palaeoecology (Chabal, 1997; Asouti et Austin, 2005). In addition, the clearest difference that appears is not between the type 1 and 2 layers, but between the pits of the pioneer house and those of the village. It is clear that the charcoal from the pioneer house, whether concentrated or dispersed, originates from specific supplies, which we have attributed to a phase of clearance, both of short duration and of local character (exclusive representation of ash, hazel and oak).

Therefore, at the three sites presented, the development of the forest cover between the pioneer phases and the main occupations has been revealed through analysis of the layers that accumulated over a time span estimated at a few years based on the results of spatial analysis of the pottery and, in one case, the lithic evidence. According to these results, if the main occupation phase quickly followed the pioneer phase—which is not yet proven—we can conclude that the impact of human activities on the original forest environment was rapid. We thus once again raise here the question of the chronological relationship between the two occupations, which is directly linked to that of the minimum time necessary to induce the modifications of the forest cover observed at the three sites between the two occupations; this is information that we do not currently possess.

This recognition of a more complex waste management method than that generally envisaged and the resulting chronological interpretation of the waste assemblages could not have been achieved without integration of the archaeological and anthracological data from several sites. These latter are often limited to strictly palaeoenvironmental interpretations, though in this case they contribute elements for the determination of occupation duration represented by the different types of waste materials present in the LBK pits, as well as for the wood supply specific to an activity and/or a habitation phase.

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References

Asouti E. & Austin P. 2005 - Reconstructing woodland vegetation and its relation to human societies, based on the analysis and interpretation of archaeological wood charcoal macro-remains. *Environmental Archaeology*, 10 : 1-18.

Bakels C.C. 1978 - Four linearbandkeramik settlements and their environment : a palaeoecological study of Sittard, Stein, Elsloo and Hienheim. *Analecta Praehistorica Leidensia,* 11.

Bakels C.C. 1992 - The botanical shadow of two early Neolithic settlements in Belgium : carbonized seeds and disturbances in a pollen record. *Review of Palaeobotany and Palynology,* 73 : 1-19.

Beck M. E. & Hill Jr. M. E. 2004 – Rubbish, relatives, and residence : The Family use of Middens, *Journal of Archaeological Methods and Theory*, 11 : 297-333.

Birkenhagen B. 2003 - *Studien zum Siedlungswesen der westlichen Linearbandkeramik,* Habelt Bonn, Saarbrücker Beiträge zur Altertumskunde, 75.

Bissardon M., Guibal L. & Rameau J.C. 1997 - *Nomenclature Corine Biotope : Types d'habitats français,* ENGREF. Museum National d'Histoire Naturelle, Ministère de l'Agriculture, de la Nature et de la Pêche, 217 pp.

Bosquet D., Regert M., Dubois N. & Jadin I. 2001 - Identification de brai de bouleau sur quatre vases du site rubané de Fexhe-le-Haut-Clocher « Podrî l'Cortri ». Premiers résultats. *Notae Praehistoricae*, 27 : 119-127.

Bosquet D., Fock H., Goffioul C., Preud'homme D. & Marchal J.-P. 2004 - La néolithisation en Hesbaye (Belgique) : apport des fouilles récentes (1996-1999) sur le tracé du TGV oriental et le domaine militaire d'Alleur. *In* : *Section 9 et 10. Néolithique et âge du cuivre. Sessions générales et Posters*. Actes du XIV^{ème} Congrès UISPP, 2-8 septembre 2001, Liège. Oxford, BAR International Series 1303 : 81-94.

Bosquet D., Golitko M. & Salavert A., avec la collaboration de Beugnier V., Deligne F., Jadin I., Martin F. & Quick R. S. 2008 - Une phase pionnière à l'origine du peuplement rubané de la Hesbaye liégeoise (Belgique). *In* : *Fin des traditions danubiennes dans le Néolithique du Bassin parisien et de la Belgique (5100-4700 BC). Autour des recherches de Claude Constantin,* Burnez-Lanotte L. (ed). Actes du colloque de Namur, 24-25 novembre 2006, Paris, Mémoires de la Société préhistorique française, XLIV : 301-315.

Bosquet D. & Van Driessche A. 2008 (inédit) - *Convention pour l'étude et la diffusion des découvertes préhistoriques du TGV et pour expertise en contexte préhistorique en Wallonie*. Rapport final (Bilan scientifique), Institut royal des sciences naturelles de Belgique, 1 vol., 90 pp, 74 fig.

Broes F. & Bosquet D. 2007 - Fabrication d'outils de terrassier et creusement des fossés rubanés : de la théorie à la pratique. *Notae Praehistoricae*, 27 : 131-149.

Chabal L. 1994 - Apports de l'anthracologie à la connaissance des paysages passés : performances et limites, *Histoire et Mesure*, IX-3/4 : 317-338.

Chabal L. 1997 - *Forêts et sociétés en Languedoc (Néolithique final, Antiquité tardive) : l'anthracologie, méthode et paléoécologie.* Paris, Éditions de la Maison des Sciences et de l'Homme, Documents d'Archéologie Française, 63, 189 pp.

David N. & David-Hennig H. 1971 - Zur Herstellung und Lebensdauer von Keramik. *Bayerisches Vorgeschichtsblatter*, 36 : 289-317.

Golitko M., Bosquet D., Keeley L. H., Jadin I. & Goemaere E. 2009 – *Chemical analyse of Linienbandkeramik ceramics and clay samples from the Hesbaye region of Belgium by LA-ICP-MS.* Poster presented at the 74nd annual meeting of the Society for American Archeologist, April 22-26, Atlanta, Georgia, Chicago, University of Illinois.

Jadin I. 2003 - *Trois petits tours et puis s'en vont…La fin de la présence danubienne en Moyenne Belgique*, 2ème édition. Etudes et Recherches Archéologiques de l'Université de Liège, 109, 726 pp.

Keeley L. H., Bosquet D., Quick R. S., Jadin I. & Golitko M. 2005 - Nouvelles fouilles sur le site rubané de Waremme-Longchamps, Rapport de la campagne 2005. *Notae Praehistoricae*, 25 : 153-161.

Kreuz A. 1990a – *Die ersten bauern mitteleuropas - Eine archäobotanische untersuchung zu umwelt und landwirtschaft der Ältesten Bandkeramik*. Leiden, University of Leiden, Analecta Praehistorica Leidensia, 23, 256 pp.

Kreuz A. 1990b - Searching for «single-activity refuse» in Linearbandkeramik settlements. An archaeobotanical approach. *In* : *Experimentation and reconstruction in environmental archaeology,* Robinson & David E. (eds). Roskilde, Denmark, 1988, Oxbow Books, Symposia of the Association for Environmental Archaeology, 9 : 63-74.

Kreuz A. 2007 - Archaeobotanical perspectives on the beginning of agriculture north of the Alps. *In* : *The origins and spread of domestic plants in southwest Asia and Europe,* Colledge S. & Conolly J. (eds). Left Coast Press, Walnut Creek/UCL Insitute of Archaeology publications : 259-294.

Last J. 1998 - The residue of yesterday's existence : settlement space and discard at Miskovice and Bylany. *Bylany Varia*, 1 : 17-46.

Mayor A. 1994 - Durée de vie des céramiques africaines: Facteurs responsables et implications archéologiques. *In* : *Terre cuite et société, document technique, économique, culturel,* Audouze F. & Binder D. (eds). Rencontre internationale d'Archéologie et d'Histoire d'Antibes. Juan-les-Pins, A.P.D.C.A., CNRS : 179-198.

Modderman P. J. R. 1970 - *Linearbandkeramik aus Elsloo und Stein.* Leiden, University of Leiden, Analecta Praehistorica Leidensia, 3 (2 vol.), 218 pp, 18 fig., 232 pl.

Pavlu I. 1986 - The principles of analysis of settlement refuse. *In* : Theses on the Neolithic site of Bylany, Pavlu I., Rulf J. & Zapotocka M. (eds). *Pamatky Archeologicke*, 77 : 310-314.

Pavlu I. 1998 - Linear pottery settlement area of the Miskovice 2 Site (Distr. Kutna Hora). *Bylany Varia*, 1 : 53-82.

Pavlu I. 2000 - *Life on a Neolithic site*. Prague, Institute of Archaeology, 340 pp.

Rameau J.C., Mansion D. & Dumé D. 1989 - *Flore forestière française. Plaines et collines*. Développement Forestier, 1, 1789 pp.

Rulf J. 1986 - Analytical programme of the structure of features and finds at a Linear pottery culture (LnK) site. *In* : Theses on the Neolithic site of Bylany, Pavlu I., Rulf J. & Zapotocka M. (eds). *Pamatky Archeologicke*, 77 : 291-310.

Salavert A. 2008 - Analyse anthracologique de Remicourt « en Bia Flo II » (Rubané, Hesbaye). Résultats préliminaires. *In* : *Fin des traditions danubiennes dans le Néolithique du Bassin parisien et de la Belgique (5100-4700 BC). Autour des recherches de Claude Constantin,* Burnez-Lanotte L. (ed). Actes du colloque de Namur, 24-25 novembre 2006, Paris, Mémoires de la Société préhistorique française, XLIV : 317-326.

Schweingruber F. H. 1990 - *European wood anatomy*. Bern, Verlag Paul Haupt, 800 pp.

Shott M. J. 1996 - Mortal Pots : On use life and vessel size in the formation of ceramic assemblages. *American Antiquity*, 61-3 : 463-482.

Shott M. J. & Sillitoe P. 2004 - Modeling use-life distributions in archaeology using New Guinea Wola ethnographic data. *American Antiquity*, 69-2 : 339-355.

Stäuble H. 1990 - Die ältestbandkeramische grabenanlage in Eitzum, Ldkr. Wolfenbüttel. Überlegungen zur verfüllung und interpretation von befunden. *Jahresschrift für Mitteldeutsche Vorgeschichte*, 73 : 331-344.

Stäuble H. 1997 - Häuser, gruben und fundverteilung. *In* : *Ein siedlungsplatz der Ältesten Bandkeramik in bruchenbrücken, stadt Friedberg/Hessen,* Lüning J. (ed). Habelt Bonn, Universitätsforschungen zur prähistorischen Archäologie, 39 : 17-150.

Stäuble H. 2005 - *Häuser und absolute datierung der Ältesten Bandkeramik*. Habelt Bonn, Universitätsforschungen zur prähistorischen Archäologie, 117 pp.

Varien M. D. & Mills B. J. 1997 - Accumulation research : Problems and prospects for estimating site occupation span. *Journal of Archaeological Methods and Theory*, 4-2 : 141-191.

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AN ARCHAEOBOTANICAL AND EXPERIMENTAL APPROACH TO IDENTIFYING SUCCESSIVE FIRE EVENTS IN HEARTH STRUCTURES IN THE SANCTUARY OF APOLLO IN HIERAPOLIS (TURKEY)

Girolamo FIORENTINO & Cosimo D'ORONZO

Abstract

We use anthracological and experimental approach for decoding fire refuses and thermal alterations of soil in an area of the Sanctuary of Apollo in Hierapolis (Turkey). Results obtained from experimental hearth structures show that the escharon is the result of a series of ground-level hearths, pit hearths and secondary ash deposits. Important ritual implications derive from the contextual identification of these fire events, that shed new light on the Apollo cult in the region.

Keywords : Apollo Temple, Hierapolis, Turkey, earth structures, cultural function, experimental design, anthracology

Introduction

The study of hearth structures is highly complex since it involves numerous aspects of human behaviour which do not always leave clear traces in archaeological deposits. The traces allowing us to identify the use of fire are combustion residues, thermal alteration of soil and structures . Over the years, the analysis of hearth structures has sought to go beyond the purely descriptive level in an attempt to reconstruct both formal and functional aspects (Clarke, 1968). Studies of activities linked to the use of fire have been borrowed from ethnography – despite the risks linked to the use of analogy in the interpretation of archaeological contexts (Cazzella, 1989; Henry *et al.*, 2009; Joly *et al.*, in press; Moutarde, 2006; Ntinou, 2002; Orme, 1981; Solari, 1992). However, studies of activities have also benefited from the application of multidisciplinary analyses (Leroi-Gourhan, 1943; 1945; 1964; 1965; 1973; March, 1996) and experimental reproduction. In the first case, numerous chemical-physical and pedological analysis techniques have been used to isolate traces and establish the relationships of cause and effect affecting depositional and post-depositional agents (Wood, Lee Johnson, 1978; De Guio, 1988; Leonardi,1992). The experimental approach entails the reproduction of the structure and the measurement of certain parameters held to be important for understanding various of its aspects.

In some cases a combination of both approaches may produce results that are difficult to manage, leading to the loss of the main objective of the study of prehistoric cultures, i.e., an understanding of the behaviour of a group, deducible from a contextual reading of the traces in archaeological deposits.

However, a careful assessment of the archaeological context and the nature of the hearth structures currently cannot do without the experimental approach. Experimental archaeology has recently undergone a radical transformation, becoming a sophisticated and rigorous research tool which is entirely compatible with hard science. Experimental archaeology has passed from the concept of imitation (Ascher, 1961) to one of replication (Coles, 1973), developing cyclical formulae (Reynolds, 1978; 1979) and other more sophisticated models based on complex epistemological principles (Malina, 1980; 1983). The combined application of hypothetical deductive and hypothetical inductive logical models can corroborate hypotheses developed in the course of studying a phenomenon (Popper, 1970).

These contributions confirm the importance of the contextualisation of data, experimental control and replicability. Also needed are experimental protocols designed to achieve deeper knowledge of the phenomena (Begoňa, 2003; March, 1992; Théry-Parisot, 1998).

In this paper we propose a reading of some aspects of the use of fire in a cult context in South-western Turkey. Since 2002, an area with a high concentration of ash-rich sediment and burnt organic matter, located next to the Temple of Apollo at Hierapolis and interpreted as an *escharon* (Semeraro, 2005; 2007), has been subject to special excavation strategies and archaeobotanical analyses designed to throw light on how these distinctive archaeological deposits were formed in relation to religious practices (Fiorentino, Solinas, 2009).

Archaeological Problems

Traditionally, the *escharon* is a place used for dumping the residues of material burnt in religious structures, although it may itself be the object of particular rituals (fig. 1). Therefore it may contain secondary deposits of combustion residues. In this case, the secondary deposits are made up of a layer of ash covering the area (SU 372), while the primary deposits are contained in two pits (SUs 486, 488, 754, 425, 458, 531).

However, the association in this context of combustion residues with a thermally altered substrate indicates combustion activities *in loco*, although the formation dynamics of the primary deposit are hard to read. Specific features of the context and data from the

Fig. 1 : The Sanctuary of Apollo in Hierapolis (Turkey).

The effects of the heat produced by the combustion of a solid (wood) on a substrate have been studied as part of forestry research (Wells *et al.,* 1979; Wright, Bailey, 1982; DeBano, 1991), but these studies have paid little attention to the morphology of thermal alteration of the soil, an aspect fundamental to archaeological research (Gasco, 1985; Canti, Linford, 2000).

Experimental design ought to take account of the variables that the observer believes to be important in the behaviour of a phenomenon (b, fig. 2). In addition, it should ensure experimental control and enable the researcher to reach a higher level of knowledge of the phenomenon being studied.

microstratigraphical and archaeobotanical analyses raise a number of questions concerning the reading of these structures, and have given rise to a series of hypotheses (fig. 2).

Methods and materials

Given the complexity of the archaeological context, we sought to decode the events that may have led to the formation of the deposit by means of an experimental approach. Initially, we identified the elements in the archaeological deposit that could provide clues to a reading of the phenomenon: the combustion residues (ash and charcoal remains) and the thermal alteration of the substrate (a, Fig. 2). Combustion is a chemical process that produces energy in the form of light and heat. The heat energy produced tends to cause a series of transformations in objects that are near the heat source or in direct contact with it. The passage of energy between two objects may induce chemical-physical transformations such as calcination and variations in colour and magnetism (Humpheys, Craig, 1981; Marshall, 1998; Canti, Linford, 2000; Gose, 2000; Çengel, 2005; Berna *et al.,* 2007), or mechanical transformations such as thermoclastic fractures (Lintz, 1989; Petraglia, 2002; Anderson-Ambrosiani, 2002; Pagoulatos, 2006).

To verify certain hypotheses (c, d fig. 2), two combustion cycles were conducted in the open air to test the behaviour of a hearth at ground level (EXP_C, EXP_D). In each cycle, the ground-level hearth was subject to five combustions at intervals of 24 hours. Subsequently two pits were dug in the hearth, called EXP_Cα (EXP_Dα in the second cycle) and EXP_Cβ (EXP_Dβ in the second cycle). In EXP_Cα and Dα, five combustions were performed, and in EXP_Cβ and Dβ only one. The parameters measured included the flame temperature, the temperature of the soil at four depths (-2, -7, -12 and -18 cm), atmospheric temperature and humidity near the fire and 4 m away, and the wind speed and direction at 2 m and 10 m above the ground.

The temperature of the soil was measured by type k thermocouple every 30 seconds, while the atmospheric parameters were measured every 5 minutes.

Semi-arid wood was used as fuel, a different type being used in each combustion (*Olea europaea*, *Pinus halepensis*, *Cupressus sempervirens*, *Quercus ilex*, *Quercus coccifera*, *Quercus cerris*, *Prunus armeniaca*, *Pyrus communis*, *Ficus carica*, *Vitis vinifera*). The dimensions of the branches (diameter from 5 mm to 80 mm) and the wood taxa were chosen on the basis of the archaeobotanical evidence found in the context (tabs. 4-5).

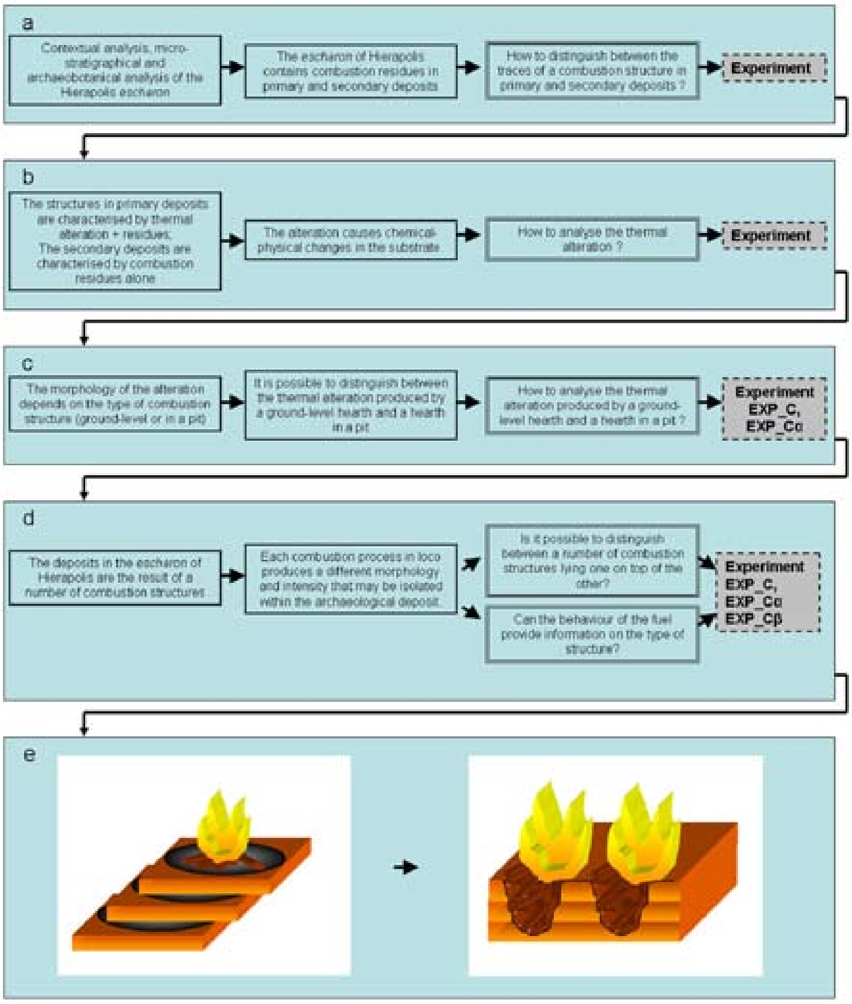

Fig. 2 : Logic model to decoding the deposit of Hierapolis.

11% of the total weight (Tabs 1 and 2). This difference is probably related to the different number of combustions in the pits. It cannot be ruled out that the charcoal produced by the initial and intermediate combustions were burned off by subsequent combustions. Indeed, while fresh fuel was being loaded on the fire, this was observed to cause lateral spreading of the deposit, which in some cases exposed charcoal fragments left over from previous combustions, leading to their complete combustion. In contrast, in other cases, the combustion residues near the edge of the hearth were partly covered by material falling on them from the walls of the pit, which seems to have shielded the fragments from subsequent combustions (tab. 3).

Results

The average percentage of ash residues produced by the combustion of 16 kg of fuel used to feed the ground level fires was 2% and 3% for EXP_C and EXP_D respectively. In addition, the two cycles produced 56.4 g and 32 g of charcoal. In the first cycle the species that produced the most residues was *Pinus halepensis*. In other cases, given the small number of replicas, the behaviours observed did not indicate significant tendencies. However, it is interesting that *Quercus ilex* produced little or no charcoal (tab.1).

In contrast, the behaviour of the pit hearths was completely different (tab. 2). In EXP_Cα and EXP_Dα, 80 kg of wood was burned, the residues amounting to 2.5% of the total weight, while in EXP_Cβ and EXP_Dβ 16 kg of wood was burned, the residues amounting to

The anthracological analyses of the combustion residues again highlight the different behaviour of a hearth structure used just once with respect to a structure used many times. In EXP_Cα and EXP_Dα, unlike the first and the last combustion episode, there was little material left from the second (figs. 3, 5).

The first levels of the ash deposit contain charcoal fragments from the last combustion episode, but few from the fourth and second combustion. This may depend on factors intrinsic to the fuel used. In addition, both pits had a deposit on the bottom composed of charcoal fragments of more than 60 mm in length belonging to the first combustion episode.

In contrast, the assemblages in pits EXP_Cβ and EXP_Dβ appear to be more complex, since they are composed of residues of every single load of wood placed on them, which partly follow the order of deposition of the fuel (figs. 4,6). In this case the material at the bottom of the pits had a low fragmentation index.

Cycles	*Prunus armeniaca*	*Pinus halepensis*	*Olea europaea*	*Quercus ilex*	*Quercus cerris*
		Ash			
Cycle EXP_C	380 g	637 g	415 g	371 g	336 g
Cycle EXP_D	448 g	429 g	370 g	759 g	410 g

Tab. 1 : Weight of combustion residues in ground hearth structures.

Structures with 5 combustion events: EXP_Cα (charcoal: 62,7 g; Ash: 2036,5 g)										
Weight (g)	US 16	US 17	US 18	US 19	US 20	US 21	US 22	US 23	US 24	US 25
Charcoal	2,4	0,5	0,2	0,5	2,8	0,8	4,6	2,6	13,1	35,2
Ash	0	196,9	93,7	177,6	582,9	146,8	164,5	83,4	520,7	70
Structures with 1 combustion events: EXP_Cβ (charcoal: 247,8 g; Ash: 1746,3 g)										
Weight (g)	US 3	US 4	US 5	US 6	US 7	US 8	US 9	US 10	US 11	US 12
Charcoal	33	1,3	0,5	7,2	8,5	1,6	9,6	117,7	67,2	1,2
Ash	0	141,6	114,6	218,3	0	0	333,8	456,6	58,1	423,3

Tab. 2 : Weight of combustion residues in pits hearth structures (Cycle C).

Structures with 5 combustion events: EXP_Dα: (charcoal 34 g; ash: 2033,5 g)													
Weight (g)	US 57	US 58	US 59	US 60	US 61	US 62	US 63	US 64	US 65	US 66	US 67	US 68	US 69
Charcoal	13,9	0,1	0,5	1,1	1,3	0,3	1,8	0,8	2,4	1,9	3,1	2,9	3,9
Ash	0	99,7	238,1	148,5	200,7	47,1	420	75,4	109,1	211,1	49,4	41,2	393,2
Structures whit 1 combustion events: EXP_Dβ: (charcoal 138,4 g; ash 1530,9 g)													
Weight (g)	US 41	US 42	US 43	US 44	US 45	US 46	US 47	US 48	US 49	US 50	US 51	US 52	US 53
Charcoal	6,9	0,1	0,2	0,1	0,6	0,9	2,1	0,9	1,5	4,7	7,4	6,1	106,9
Ash	0	253,6	202,6	5	27,3	128	97,6	0	93,8	77,8	16,7	0	628,5

Tab. 3 : Weight of combustion residues in pits hearth structures (Cycle D).

The survival of some taxa rather than others could be due to the calibre, age and humidity of the branches (Trabaud, 1976; Théry-Parisot, 1993; 1998), but may also result from the way in which the flow of heat is propagated and from the properties of the surrounding atmosphere (oxidant/reductant) during the combustion. An aspect of this is the survival on the bottom of the first loads of fuel, apparently because the combustion process was interrupted by the reduced circulation of oxygen as a result of subsequent loads of fuel being placed on top and of material falling on to the fire from the walls of the pit.

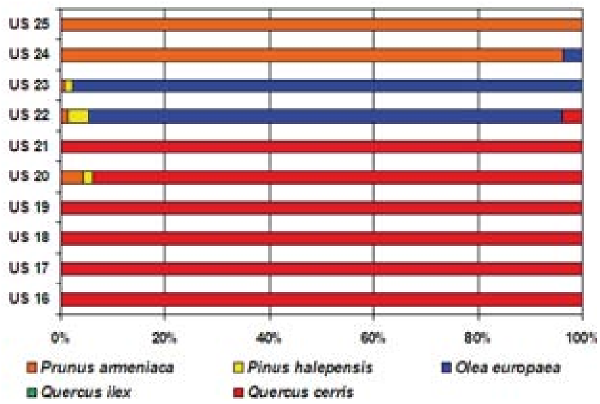

Fig. 3 : Anthracological diagram of EXP_Cα.

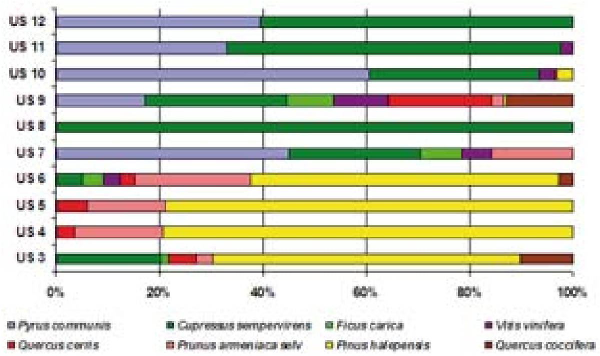

Fig. 4 : Anthracological diagram of EXP_Cβ.

During the experiment, the extent of the thermal alteration and variation in colour of the sediment was observed by using the soil colour codes table in Cailleux . Before lighting the fires in the ground-level hearths (EXP_C, EXP_D), the sediment was brown (P51). After the first combustion the substrate was dark grey (T73) with some parts of a lighter colour (R31). At the end of the experiment, 80% of the surface was orange (N60), while along the edges, near and below the stone circle it was black (T92). In cross section, the thermally altered substrate was thinner at the edges (10 mm thick on average) and thicker in the centre (up to 20 mm). The colour at depths of 0 to -0.5 cm was orange (N39), while between -0.5 cm and -2.5 cm the intensity of the orange colour tended to diminish (N59). Below this intensely altered level was a layer of sediment (0.4

Ground level hearth structure	
Hearth structure	Hearth delimitated by stone circle Internal diameter: 50 cm
Five combustion phases	
Wood fuel	Semi-arid wood of : *Prunus armeniaca*, *Pinus halepensis*, *Olea europaea*, *Quercus ilex*, *Quercus cerris* Total weight : 16 kg for every taxon
Parameters	Flame temperature Temperature of soil at four different depths (-2, -7, -12, -18 cm) Atmospheric temperature (near the fire and 4 m away) Humidity (near the fire and 4 m away) Wind speed (at 2 m and 10 m above the ground) Wind direction (at 2 m and 10 m above the ground).
Instruments:	Thermocouple Type K Multichannel T-C 08 Picologger Weather station Anemometer Chronometer
Time for registration	Flame and soil temperatures : 30 s Atmospheric parameters: 5 min
Time for re-loading fuel	2 kg of wood fuel every 15 minutes
Procedures	Load fuel – lighting – charring – reloading fuel. Every 15 minutes the hearth structure was reloaded with another 2 kg of wood. The experiment was monitored for 3 hours, and the ash deposit was excavated after 24 hours.

Tab. 4 : Experimental design for ground hearth structure.

Oval hearth structures	
Hearth structure	Two oval pits 40 X 90 cm, delimitated by stone circles, 30 cm deep
EXP_Cα : Five combustion phases	
Wood fuel	Semi-arid wood of : *Prunus armeniaca*, *Pinus halepensis*, *Olea europaea*, *Quercus coccifera*, *Quercus cerris* Total weight of fuel : 16 kg for every taxon
EXP_Cβ : One combustion phase	
Wood fuel	Semi-arid wood of : *Pyrus communis*, *Cupressus sempervirens*, *Ficus carica*, *Vitis vinifera*, *Quercus cerris*, *Prunus armeniaca*, *Pinus halepensis*, *Quercus coccifera* Weight of each load : 2 kg for every taxon
Parameters	Flame temperature Temperature of soil at four different depths (-2, -7, -12, -18 cm) Atmospheric temperature (near the fire and 4 m away) Humidity (near the fire and 4 m away) Wind speed (at 2 m and 10 m above the ground) Wind direction (at 2 m and 10 m above the ground).
Instruments:	Thermocouple Type K Multichannel T-C 08 Picologger Weather station Anemometer Chronometer
Time for registration	Flame and soil temperatures : 30 s Atmospheric parameters: 5 min
Time for re-loading fuel	2 kg of wood fuel every 15 minutes
Procedures	Load fuel – lighting – charring – reloading fuel. After 15 minutes the hearth structures is reloaded with others 2 kg of wood fuel. The experiment is monitored for 3 hours. After five combustions another oval pit was excavated. In this second pit one combustion was performed. The ash deposit was excavated after 1 month.

Tab. 5 : Experimental design for pit hearth structure.

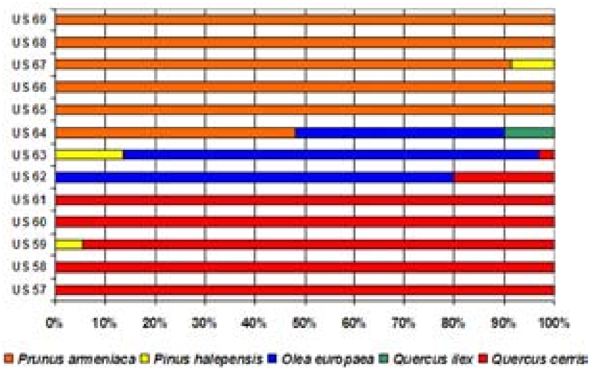

Fig. 5 : Anthracological diagram of EXP_Dα.

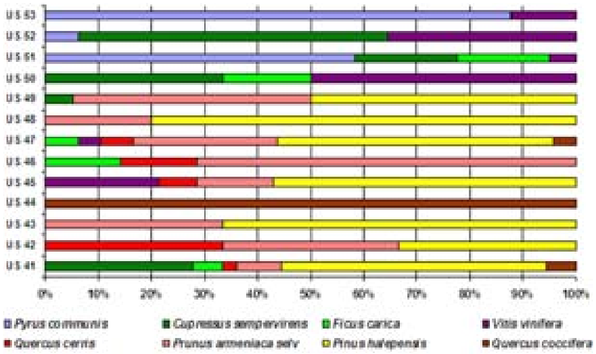

Fig. 6 : Diagramme anthracologique de EXP_Dβ.

cm thick) subject to little or no alteration which was dark brown in colour (S91) with shades of black. Below this, the levels had no variations in colour.

The colour variations of the substrate of combustion pits EXP_C*β* and EXP_D*β* were similar to those of the ground-level hearths but with different formation times. The colour of the substrate next to the fire after the first loads of fuel was black (N73), due to the gases released by carbonisation. At the end of the first combustion episode, a semicircular variation in colour was seen on the walls of the central part of the pit. The outer edges were dark brown (S50) while the intermediate area was darker (T51), with shades of black (T92). The central part, closest to the fire itself, was orange (N45). After five combustions the central part displayed a more intense degree of alteration, to the point that it was almost red (N17). This was delimited first by a semicircular dark brown band (T30) and then by an outer black band (T92).

Discussion

The studies conducted in the *escharon* of the sanctuary of Apollo and the experimental reproduction highlight a number of events linked to the use of fire. The experimental reproduction made it possible to separate the outcomes of the use of fire: thermal alteration and combustion residues. Analysis of the former shows a correlation between the morphology of the altered soil and the structure that produced it. In contrast, the relationship between the colour of the soil and the temperature of the hearth is more complex. The orange-red colour seems to be determined not so much by the exposure of the soil to high temperatures as to the absence of an insulating "deposit" between the source of heat and the substrate (Canti, Linford, 2000). In the experimental cycles the shift from the natural colour of the soil to red was seen in two cases: when there was direct contact between the source of heat and the substrate (especially in the pit hearths) and when the ground-level hearth was reused after being cleaned. However, the variation in colour of the substrate may also depend on its mineral composition and the percentage of organic residues contained in it. It is no coincidence that the sediments rich in ferrous minerals tended to take on a red colour even at low temperatures (Frandsen, Ryan, 1986; Cornell, Schwermann, 1996; Fitzpatrick, 1988; Canti, Linford, 2000).

It is clear that the structure of the *escharon* in Hierapolis is the result of a series of ground-level hearths, cleaned after the formation of the ash deposit before being re-used each time, in which a series of pits were subsequently dug (e, fig. 2).

Lastly, the interaction between the archaeobotanical analyses and the experimental reproduction was shown to be a useful tool for decoding thermally altered deposits. In the case of Hierapolis reference was made to hypotheses based on the simultaneous presence, observed during the excavations, of the archaeological results of a number of separate events or behaviours linked to various religious practices. Although with our current level of knowledge it is not possible to fully interpret their symbolic meaning, it was possible to differentiate between practices which involved the secondary dumping of ashes in an area different from that of the original combustion and other ritual activities demonstrated by thermal alterations in the substrate and by ash and carbon-rich residues resulting from direct combustion *in loco*.

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References

Anderson-Ambrosiani P. 2002 - Man-Fire-Stone. Fire-cracked stones as a cultural expression. *In* : *Fire in Archaeology*, Gheorghiu D. (ed). Papers from a session held at the European Association of Archaeologist Sixth Annual Meeting in Lisbon 2000, British Archaeological Reports, S1089 : 125-131.

Ascher R. 1961 - Experimental archaeology. *American Anthropologist*, 63 : 793-816.

Begoňa S. L. 2003 - Estudio de las estructuras de combustion prehistoricas : una propuesta experimental : Cova Negra (Xàtina, Valencia), Ratlla del Bubo (Crevillent, Alicante) y Marolles-sur-Seine (Bassin Parisien, France), Deputacion de Valencia.

Berna F., Behar A., Shahack-Gross R., John Berg J., Boaretto E., Gilboa A., Sharon I.,Shalev S., Shilstein S., Yahalom-Mack N., Zorn J. R. & Weiner S. 2007 - Sediments exposed to high temperatures: reconstructing pyrotechnological processes in Late Bronze and Iron Age Strata at Tel Dor (Israel). *Journal of Archaeological Science*, 34 : 358-373.

Canti M.G. & Linford N. 2000 - The effects of fire on archaeological soils and sediments: temperature and colour relationships. *Proceedings of the Prehistoric Society*, 66 : 385-395.

Cazzella A. 1989 - *Manuale di Archeologia. Le società della preistoria*. Roma, Editori Laterza.

Çengel Y.A. 2005 - *Termodinamica e trasmissione del calore*. Milano, McGraw-Hill.

Clarke D.L. 1968 - *Analytical Archaeology*. London.

Coles J. 1973 - *Archaeology by Experiment*. London.

Cornell R.M., Schwermann U. 1996 - *The Iron Oxides*. Weinheim : VCH.

De Guio A. 1988 - Unità stratigrafiche come unità operazionali: verso le archeologie possibili degli anni '90. *In* : *Archeologia stratigrafica dell'Italia settentrionale*, Uboldi M. (ed). Como, Edizioni New Press : 9-22.

DeBano L.F. 1991 - *The Effect of Fire on Soil Properties*. United States Department of Agriculture Forest Service General Technical Report, INT-280 : 151-156.

Fiorentino G. & Solinas F. 2009 - Micro-stratigraphical and archaeobotanical approaches to investigating ash deposits in the Apollo Sanctuary in Hierapolis. *In* : *Paper of 1st International Symposium on Oracle in Antiquity and the Cults of Apollo in Asia Minor*, Izmir 17-19 August, 2005, Arkeoloji Dergisi, Special Issue.

Fitzpatrick R.W. 1988 - Iron compounds as indicators of pedogenetic process : examples from the Southern hemisphere. *In* : *Iron in Soil and Clay Minerals*, Stucki J.W., Goodman B.A., Schwermann U. (eds), Dordrecht : Reidel, Nato ASI, 217 : 351-96.

Frandsen W.H. & Ryan K.C. 1986 - Soil moisture reduces belowground hear flux and soil temperatures under a burning fuel pile. *Canadian Journal of Forest Research of Botany*, 14 : 243.

Gascó J. 1985 - *Les installations du quotidien. Structures domestiques en Languedoc du Mésolithique à l'Age du Bronze d'après l'étude des abris de Font-Juvénal et du Roc-de-Dourgne dans l'Aude*. Documents d'Archéologie Française, 1. Paris. Ed. de la Maison des Sciences de l'Homme.

Gose W. 2000 - Paleomagnetic studies of burned rocks. *Journal of Archaeological Science,* 27 : 409-421.

Henry, A., Théry-Parisot, I. & Voronkova, E., 2009. La gestion du bois de feu en forêt boréale: problématique archéo-anthracologique et étude d'un cas ethnographique (Région de l'Amour, Sibérie). *In* : *Gestion des combustibles au Paléolithique et au Mésolithique: nouveaux outils, nouvelles interprétations*, Théry-Parisot I. & Henry A. (Eds). Proceedings of worshop 21, UISPP 13, XV congress, Lisbon, 4-9 septembre 2006. Archaeopress. British Archaeological Reports International, 1914 : 17-37.

Humpheys F.R., Craig F.H. 1981 - Effects of fire on soil chemical, structural and hydrological properties. *In* : *Fire and the Australian Biota,* Gill A.M., Groves R.H., Noble J.R. (eds). Camberra, Australian Academy of Science : 177-200.

Joly D., March R.J., Marguerie D. & Yacobaccio H., sous presse. Gestion des combustibles dans la province de Jujuy (Puna, Argentine) depuis l'Holocène ancien: croisement des résultats ethnologiques et anthracologiques. *In* : *Gestion des combustibles au Paléolithique et au Mésolithique : nouveaux outils, nouvelles interprétations*, Théry-Parisot I., Costamagno S. & Henry A. (eds). Proceedings of worshop 21, UISPP 13, XV congress, Lisbon, 4-9 septembre 2006. Archaeopress. British Archaeological Reports International, 1914 : 39-56.

Leonardi G. 1992 **-** Il deposito archeologico: bacini, processi formativi e trasformativi. *In* : *Atti del Seminario internazionale Formation processes and excavation methods in archaeology: perspectives,* Leonardi G. (ed). Padova 15-27 Luglio 1991. Saltuarie dal laboratorio del Piovego, 3 : 13-47.

Leroi-Gourhan A. 1943 - *L'homme et la matière*. *Evolution et techniques I*. Paris, Ed. Albin Michel.

Leroi-Gourhan A. 1945 - *Milieu et techniques*. *Evolution et techniques*. Paris, Albin Michel.

Leroi-Gourhan A. 1964 - *Le geste et la parole*. *Technique et Langage I*. Paris, Ed. Albin Michel.

Leroi-Gourhan A. 1965 - *Le geste et la parole*. *La mémoire et les rythmes II*. Paris, Ed. Albin Michel.

Leroi-Gourhan A. 1973 - Structures de combustion et structures d'excavation. *In* : *Séminaire sur les structures de combustion*. Collège de France. Ethnologie Préhistorique, CNRS, 52.

Lintz, C. 1989 - Experimental thermal discoloration and heat conductivity studies of caliche from Eastern New Mexico. *Geoarchaeology*, 4 : 319-346.

Malina J. 1980 - *Metody experimentu v archaeologii*. Praha, Accademia.

Malina J. 1983 - Archaeology and Experiment. *Norwegian Archaeological Review*, 16, 2 : 69-85.

March R.J. 1992 - L'utilisation du bois dans les foyers préhistoriques: une approche experimentale. *Bulletin de la Société Botanique de France*, 139, Actualités Botaniques, 2-4 : 245-253.

March R. J. 1996 - L'Etude des structures de combustion préhistoriques: une approche interdisciplinaire. *In* : *The Lower and Middle Palaeolithic,* Bar-Yosef O., Cavalli-Sforza L. L., March R.J., Piperno M. (eds), Colloquium X UISPP. Forlì, A.B.A.C.O. : 251-275.

Marshall A. 1998 - Visualising burnt areas: patterns of magnetic susceptibility at Guiting Power 1 round barrow (Glos., UK). *Archaeological Prospection,* 5 : 59-177.

Moutarde F. 2006 - *L'évolution du couvert ligneux et de son exploitation par l'homme dans la vallée du Lurin (côte centrale du Pérou), de l'Horizon Ancien (900 –100 av. J.-C.) à l'Horizon tardif (1460-1532 ap. J.-C.). Approche anthracologique*. Université de Paris 1, Paris, 2 vol, 317 pp.

Ntinou, M., 2002. *La Paleovegetación en el norte de Grecia desde el Tardiglaciar hasta el Atlántico : formaciones vegetales, recursos y usos*. Oxford, British Archaeological Reports, S XVI. Archaeopress, 268 pp.

Orme B. 1981 - *Anthropology for Archaeologists : an introduction*. London.

Pagoulatos P. 2006. Experimental burned rock studies on the Edwards Plateau : a view from Camp Bullis, Texas. *North American Archaeologist*, 26 : 289-329.

Petraglia M.D. 2002 - The heated and the broken: thermally altered stone, human behaviour, and archaeological site formation. *North American Archaeologist,* 23 : 241-269.

Popper K. 1970 - *Logica della scoperta scientifica*. Torino, Einaudi.

Reynolds P.J. 1978 - *The Experimental Storage of Grain in Underground Silos*. Unpublished PhD thesis, Leicester University.

Reynolds P.J. 1979 - The nature of experiment in archaeology. *In* : *Experiment and Design – Archaeological Studies in Honour of John Coles*, Harding A.F. (ed.). Exter, The Short run Press : 156-162.

Semeraro G. 2005 - Per un approccio contestuale alla lettura delle immagini. Le ceramiche a rilievo di Hierapolis di Frigia. *MEFRA*, 117, 1 : 83-98.

Semeraro G. 2007 - Ricerche archeologiche nel Santuario di Apollo (Regio VII) 2001-2003. *In* : *Hierapolis di Frigia I, Le attività delle Campagne di scavo e restauro 2000-2003,* D'Andria F., Caggia M.P. (eds). İstanbul, Turkey, Ege Yayinlari : 169-209.

Solari, M.-E., 1992. Anthracologie et ethnoarchéologie dans l'archipel du Cap Horn (Chili). *Bulletin de la Société Botanique de France*, 139, 2-4 : 407-420.

Théry-Parisot I. 1993 - *Les déformations anatomiques des charbons de bois : implications archéologiques (sites des Canalettes et des Usclades, Aveyron)*. D.E.A., Université de Paris I.

Théry-Parisot I. 1998 - *Économie du combustible et Paléoécologie en contexte glaciaire et périglaciaire, Paléolithique moyen et supérieur du sud de la France, Anthracologie, Expérimentation, Taphonomie*. Thèse de doctorat, Université de Paris I.

Trabaud L. 1976 - Inflammabilité et combustibilité des principales espèces des garrigues de la région méditerranéenne. *Eocologia Plantarum*, 11, 2 : 117-136.

Wells C.G., Campbell R.E., DeBano L.F., Lewis C.E., Fredriksen R.L., Froelich R.C. & Dunn P.H. 1979 - *The effects of fire on soil : a state of knowledge review*. National fire effects workshop, Denver, Colorado, April 10-14, 1978. United states Forest Service General Technical Report WO-7.

Wood W.R. & Lee Johnson D. 1978 - A Survey of disturbance processes in archaeological site formation. *In* : *Advances in archaeological method and theory,* Schiffer M.B. (ed) : 315-381.

Wright H.A. & Bailey A.W. 1982 - Temperature and heat effects. *In* : *Fire and Ecology: United States and Southern Canada,* Wright H.A., Bailey A.W. (eds). New York : 8-23.

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CHAPTER 2

Archeobotany and taphonomy

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CARBONIZATION, PRESERVATION AND DEFORMATION OF CARPOLOGICAL REMAINS

Marie-Pierre RUAS & Laurent BOUBY

Abstract

In archaeological sites in temperate climates and with aerobic conditions, carbonized seeds represent the majority of preserved carpological remains. Among these, cereals, legumes and certain fruits are the most frequent. In this paper, we present a selection of experiments concerning the effects of carbonization on the deformation of seeds and fruits and on the differential preservation of carpological assemblages. These experiments explored the influence of parameters such as temperature, heating duration, oxidizing or reducing conditions and the initial state of the seeds in on the modifications of their forms and dimensions.They were conducted on hulled or naked caryopses, seeds of pea, apple and wild and cultivated grape seeds. Other experiments focused on the rapidity of destruction of the anatomical parts of ripe cereals (stem, rachis, glumes, caryopses) and acorns (casings, pericarps, cotyledons), and of the seeds of various wild or cultivated plants according to their physical and biological constitution. The results allow us to evaluate the taphonomic biases created by carbonization, which are detrimental to the specific identification of seeds and the interpretation of archaeological assemblages.

Keywords : seeds, carpological assemblages, preservation, deformation, experimentation, diagnosis, morphometry, cereals, legumes, grape, acorn

Introduction

Carpological remains seeds, fruits, tubers, cereal ear elements, etc. are characterized by a diversity of forms, dimensions and anatomical and chemical compositions. Their preservation in archeological sites is determined by both their state at the moment of deposition and the physical and chemical conditions of the context in which they are buried. The anaerobic conditions of waterlogged sites and contexts often preserve plant remains in a sub-fossil, humid (saturated) state. In dry sites and contexts, in temperate climates, aerobic microorganisms degrade these types of remains if they are not fossilized by carbonization or mineralization. Carbonized seeds, which are the most often preserved in these sites, have constituted the basis for the study of the origins and diffusion of cultivated plants (Zohary & Hopf, 2000).

Palethnobotanical studies conducted at sites in arid environments in the Near East and in temperate environments in Europe have shown that the carbonization of seeds can have several causes: the use of agricultural residues as a combustible material; the use of a combustion structure in the transformation or cleaning of plants (crop processing, alimentary preparation, combustible) or habitat, storage or cultivation (refuse burning, sterilization of silos, burning of vegetation) zones, or; accidental fires in occupation zones (Knörzer, 1971; Hillman, 1981; Miller, 1996; Charles, 1998; Van der Veen, 2006). Carpological studies indicate that on a site where seeds are preserved in these three states, some categories of plants are rarely present in a carbonized state: the seeds of green vegetables, spices and fruits with pits or pips (seeds). The majority of carbonized assemblages are thus composed of cereals, legumes and a few shelled fruits (Green, 1979; Ruas, 1992, Ruas *et al.*, 2006). The strictest spectra of carbonized assemblages include the plants that are processed for consumption through the use of a heat source, or those whose easily stored seeds (farinaceous seeds, shelled fruits) are exposed to fires in habitat zones (Van der Veen, 2006). Meanwhile, cereals and legumes also constitute the basis of vegetal productions in preindustrial European societies.

In this paper, we present a selection of studies that address the taphonomic effects of carbonization on the deformation of seeds and the composition of carpological assemblages. In addition to these experiments, two ongoing projects concerning grape seeds (Bouby & Terral, unpublished) and acorns (Bouby & Ruas, unpublished) are discussed.

Deformation of seeds by carbonization

Carbonization results in modifications to the form and size of seeds that archeobotanists have known for a long time (Helbaek, 1952; Hopf, 1955 cited par Renfrew, 1973, 9-15). These modifications must thus be taken into account in all morphometric analyses and can constitute a serious handicap for taxonomic identifications, particularly at the infra-specific level, in the distinction between cultivated and wild forms of the same species, for example.

The analysis of the effects of carbonization is essentially addressed through experimentation. The first experimental reproductions in laboratory muffle furnaces were realized in the 1950's on apples (Helbaek, 1952 cited by Renfrew, 1973) and in the 1970's on grape seeds (Logothetis, 1970) and cereal caryopses (Renfrew, 1973). Since the 1990's, researchers have attempted to vary parameters such as temperature, heating duration, oxygen intake, the humidity of seeds at the beginning of the experiment and the taxon studied, in order to better understand the influence of the conditions of carbonization on modifications to the configuration of seeds.

The experiments conducted with grape seeds by H. Smith and G. Jones (1990) are representative of this type of approach (Mangafa & Kotsakis, 1996; Bouby *et al.*, 2006). Grape constitute a favorable research topic since the distinction between wild grape seeds (*Vitis vinifera* subsp. *sylvestris*) and cultivated grape seeds (*V. vinifera* subsp. *Vinifera*)—and thus the dating of the beginnings of viticulture in a given region—are generally based on morphometric characteristics. Wild grape seeds are usually small, with a globular to cordiform shape and have a short point, while domestic grape seeds are generally bigger, ovoid to pyriform and have a long point (Levadoux, 1956) (fig. 1A, 1B).

The reduction in size is accompanied by a deformation; the retraction and carbonization variably affect the dimensions of the seed (fig. 2). The seeds tend to take on a more spherical

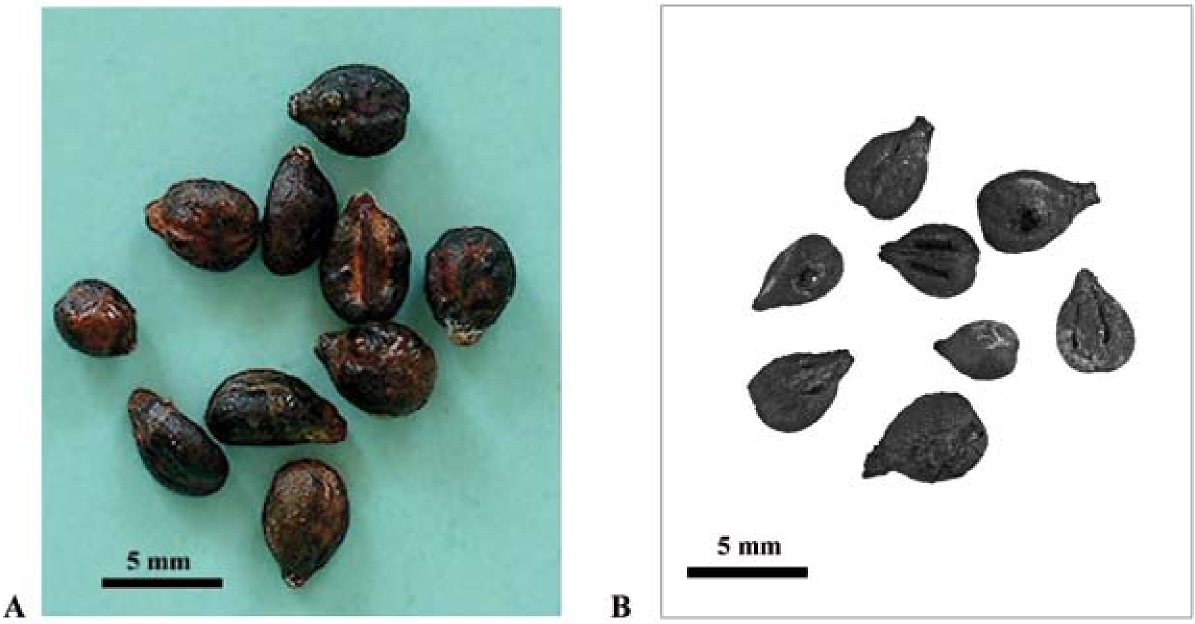


Fig. 1 : Modern fresh seeds of wild grapevines (*Vitis vinifera* subsp. *sylvestris*), Grésigne Forest (Tarn) (Photo L. Bouby); B – Carbonized seeds of cultivated/wild grapevines (*Vitis vinifera* subsp. *vinifera*) from the site of Castellu (Corte, Haute-Corse), 5th to 4th centuries. The assemblage includes cultivated seeds with a pronounced point and elongated body and wild seeds with a small point and rounded body (Photo M.-P. Ruas).

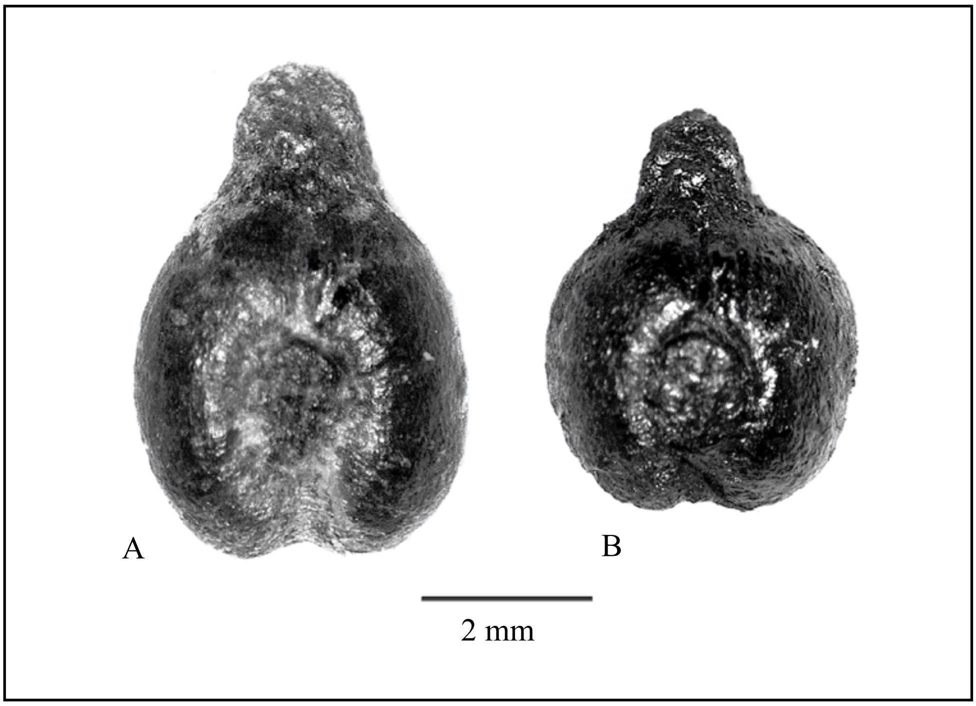


Fig. 2 : Effect of carbonization on a cultivated grape seed (*Vitis vinifera* subsp. *vinifera*), « Roussanne » cultivar (Photos L. Bouby).
A - before carbonization;
B - after carbonization at 350°C in an oxidizing atmosphere for 30 minutes. The carbonization resulted in a longitudinal retraction (especially the point) and a dorsal-ventral swelling of the seed.

form, which makes that of cultivated grapes more like that of the wild morphotype. These modifications also depend on the conditions of carbonization. Temperature is the principal parameter: the higher the temperature, the greater the deformation. The influence of oxygen intake and the heating duration is moderate. The initial humidity level of the seeds does not have a significant effect, except perhaps at high temperatures (Smith & Jones, 1990; Mangafa & Kotsakis, 1996; Bouby *et al*., 2006). These experiments show that use of a reference base of wild and modern cultivated seeds allows their attribution to one or the other of the sub-species with a satisfying degree of precision even after carbonization (Mangafa & Kotsakis, 1996).

The results of similar studies of wheat are now available (*Triticum aestivum* & *T. dicoccum*) (Braadbaart *et al*., 2004 ; Braadbaart & Van Bergen, 2005; Braadbaart, 2008). They also show a general reduction in size of the caryopses; their length is further reduced as the temperature increases. This occurs independently of the width, which until 300 °C, tends to augment, after which it diminishes at higher temperatures. As a result, the form index, expressed simply as the length/width ratio of the seed, varies according to the temperature. A similar observation was made for pea seeds (*Pisum sativum*) (Kislev & Rosenzweig, 1991; Braadbaart & Van Bergen, 2005).

The modifications of the form and dimensions of wheat also vary according to whether the caryopses are covered by hulls or not, particularly for emmer wheat (*T. dicoccum*), whose glumes are more robust and adhere more strongly to the seed than those of naked wheat.

The experimental results also reveal a few elements concerning the conditions of carbonization, which contribute to our understanding of the processes by which the fossil assemblage was constituted. The experiments conducted with wheat show that protrusions are produced on the surface of the caryopses, especially when the temperature rises rapidly. But as Braadbaart (2008) points out, this relationship has not been verified at high temperatures (600 °C) and the presence of such protrusions is not mentioned in the archeobotanical literature.

According to experiments realized with *Panicum miliaceum* (Lundström-Baudais *et al*., 2002), a lateral fold appears only on the caryopses carbonized in their glumellae and the apex of some of them take on a pointed form. These types of stigmata are not present on seeds carbonized without a hull. Unfortunately, they seem to be observable on hulled grains within only a very small temperature range, between 210 °C and 230 °C. No experiments have been realized at temperatures over 250 °C.

Effects of carbonization on carpological assemblages

Other studies have addressed the biases created by differential destructions caused by carbonization within carpological assemblages, relative to their initial composition (Boardman & Jones, 1990; Van der Veen & Jones, 2006). Most concerned cereal assemblages that are probably the residues of crop processing activities. Some address the variations of assemblage compositions preserved on the floors of domestic units (Gustafsson, 2000; Guarino & Sciarrillo, 2004).

Experimental carbonizations of whole cereal ears and hulled and dehusked grains analyzed the resistance of the different elements (rachis, glumes, glumellae, seeds: fig. 3) at different temperatures and exposure duration (Bowman, 1966; Jenkinson, 1976 cited by Boardman & Jones, 1990). They demonstrated the significant effects of two factors on the differential destruction of anatomical parts. While their color becomes only slightly browner at low temperatures, higher temperatures carbonize the seeds and more rapidly destroy the rachis and glumes. A. Bowman (1966) observed that with Einkorn wheat (*Triticum monococcum*), the hulls are carbonized at higher temperatures than are the seeds (fig. 4A, 4B).

D. Wilson (1984) and T. Märkle & M. Rösch (2008) studied the seeds of different wild and cultivated species according to their chemical composition and reserves (carbohydrates, lipids), as well as their dimensions and form (compact, flat). Their study included the oleaginous seeds of flax (*Linum usitatissimum*), poppy (*Papaver somniferum*) and crucifera, small millet (*Setaria italica*

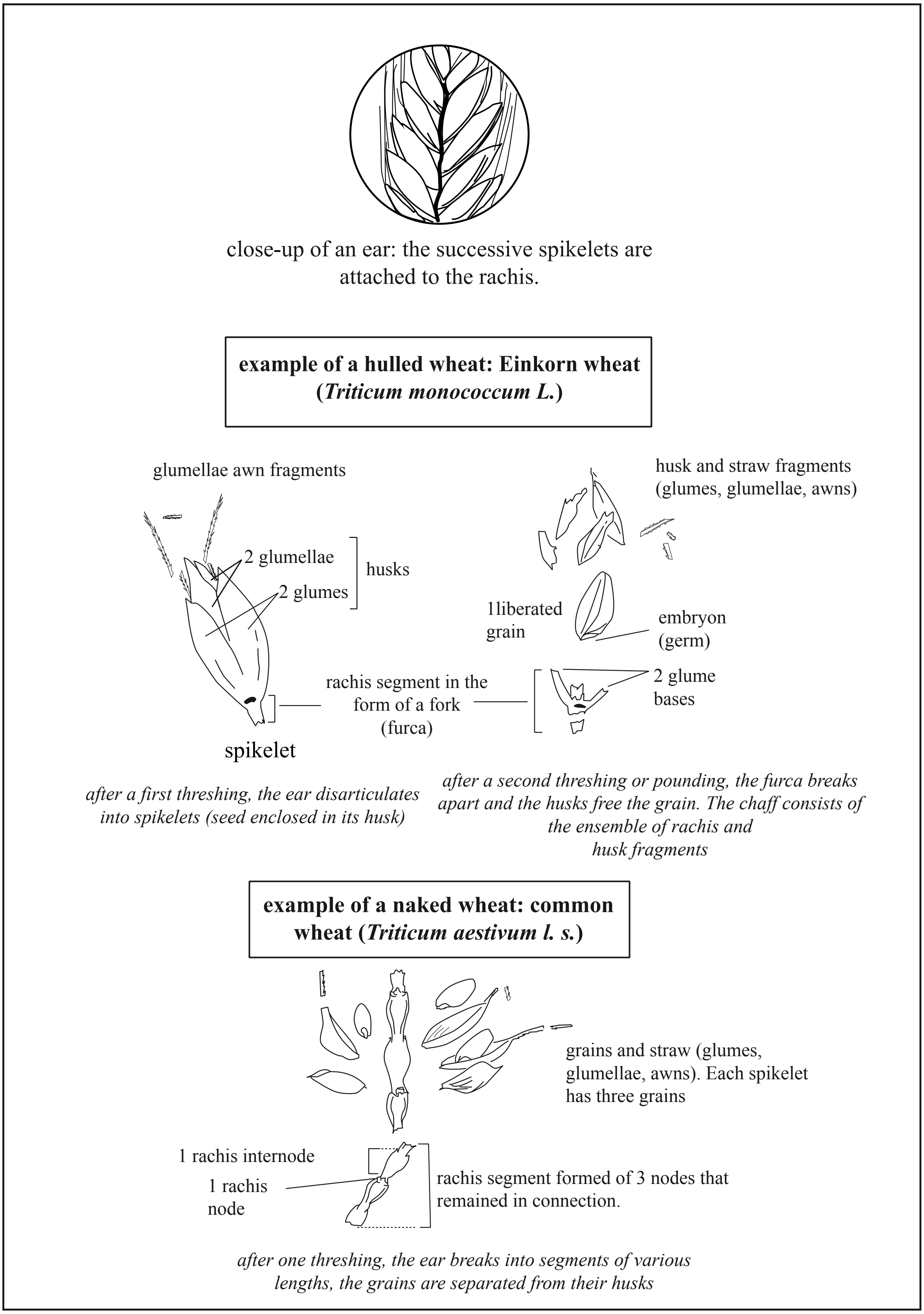
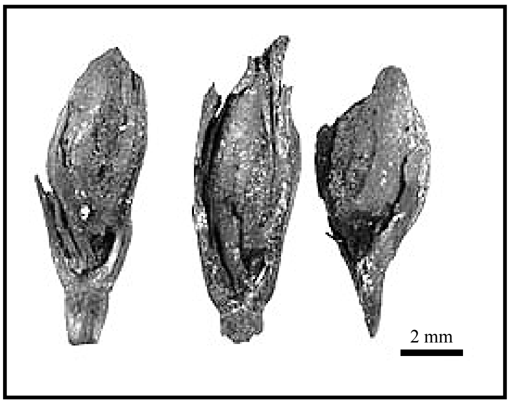


Fig. 3 : Composition of hulled and non hulled cereal ears. The threshing and dehusking operations separate the seeds from the chaff (hulls, barbs, rachis). Depending on the combustion conditions, these anatomical elements are destroyed at variable rates or are maintained in a carbonized state. (drawing M.-P. Ruas after Ruas 2002 modified).

A



B

Fig. 4 : Carbonized remains of Einkorn wheat (*Triticum monococcum*), La Gravette granary (Isle-Jourdain, Gers), 11th century (photos M.-P. Ruas).
A – Carbonized spikelets: the bases of glumes (furcas), the glumellae and the seeds are preserved.
B – Seeds without hulls following carbonization.

et *Panicum miliaceum*) and several weed species. The seeds rich in lipids tended to burst when heated and were less resistant than the seeds with amylacea reserves. Compact legume seeds covered with a thick tegument burn slower than flat seeds (flax) or the smallest seeds (weeds, millets). These studies permitted evaluations of the loss of different taxa, which affect the assemblages at the moment of carbonization.

The degree of humidity of the seeds (Wright, 2003) and the presence/absence of a floor during carbonization were also considered. Experiments were recently conducted in contexts reconstructed to resemble sites in Sweeden (Gustafsson, 2000) and Italy (Guarino & Sciarrillo, 2004), such as Protohistoric houses and fireplaces. These were compared to combustions in laboratory ovens. The depth of burying and the nature of the ground were taken into account in the deformations and disappearance of seeds. In the Italian cases, few variations in the degree of carbonization were observed between seeds within the same family (*Poaceae, Vitaceae, Fabaceae*) according to their positions in the ground. On the other hand, remarkable differences were observed in the loss of seeds belonging to different families. These result principally from the sensitivity of different seed types to heating. Cereal grains, for instance, are very sensitive to high temperatures (between 200 and 400°C) and are rapidly carbonized, while grape seeds are completely carbonized starting at 450°C and legume seeds (lentils, chick peas and broad beans) are carbonized starting at 500°C due to the protection provided by their thick tegument.

Over past twenty years, the carbonized residues of cereal stocks have been interpreted based on observations of non-mechanized agricultural procedures (*crop processing sequences*) for cleaning hulled or non-hulled cereals. The proportion of chaff (by-products of cereal processing: awns, barbs, rachis), caryopses and seeds of weed species are the variables taken into account for the identification of the degree and stage of the cleaning of the harvest (Hillman 1981, 1984; Van der Veen, 1992). The modifications of the composition of such assemblages can differ significantly depending on the conditions of carbonization previously described (cf. §1). The quantitative analysis (relative proportions of remains) of such assemblages must take into account the bias created by the differential disappearance of these elements before the stage in theprocessing sequence can be determined. The parameters were studied at variable temperatures and combustion durations for the components of several hulled or non-hulled cereals (straw, glumes, caryopses) (Boardman & Jones, 1990). One of the conclusions emphasizes that the first components to disappear are precisely those that are the least often represented in carbonized archeobotanical assemblages and those that characterize the first cleaning stages of non-hulled cereals (straws and rachis). These authors focused on the kinetics of carbonization and the destruction of the caryopses of several hulled and non-hulled cereal species in function of the heating duration, temperature and quantity of available oxygen. In order to have a descriptive scale of the physical state of carbonized caryopses and a tool for the analysis and interpretation of the combustion conditions of archeological seeds, they employed, after modifications, the scales of "Preservation" and "Distortion" of Hubbard (1977 cited by Boardman & Jones, 1990, 4: table 1). Their experiments show that the state of deformation ("Distortion") is a better descriptor than the degree of preservation ("Preservation") for determining the conditions in which the seeds of fossil assemblages were burned.

CODE P	PRESERVATION
P 1	Perfect; hairs, etc. preserved
P 2	Epidermis virtually intact, hairs, etc. occasionally preserved
P 3	Epidermis incomplete
P 4	Fragments of epidermis remaining, other features generally unobserv
P 5	Gross morphology only observable
P 6	Vesicular (hollowed), heavily pitted or "clinkered"
CODE D	**DISTORTION**
D 1	Very little distorsion
D 2	Slight distorsion
D 3	Clearly distorsion
D 4	Gross distorsion
D 5	Grain partially destroyed or fused into solid lump

Tab. 1 : Scales used to record the state of preservation and deformation of carbonized seeds (adapted from Boardman & Jones 1990, 4).
The codes are defined following experimental furnace combustion of the non-hulled caryopses of cereals (wheat, barley, rye). The points of destruction of the seed are P5/L or D5; at these stages of experimental combustion, the seed is generally difficult or impossible to determine.

Acorn shelling or the result of carbonization?

The differential preservation of carpological assemblages directly influences interpretations of the function of combustion features and the nature of deposits. However, thermal treatments applied to harvests and food stocks during storage and culinary preparation procedures modify the composition of these assemblages by eliminating certain elements.
Among the farinaceous foods, the acorns of diverse oak species (*Quercus* spp.) also played an important role (Maurizio, 1932; Aurenche, 1997), as is illustrated by discoveries in Protohistoric and historic storage or roasting contexts in both northern (Jorgensen, 1977) and southern (Coularou *et al.*, 2008) Europe. Their frequent discovery in a shelled or non-shelled state raises the question of the representivity of the preserved and non preserved elements (cotyledons, pericarps and casings) following their combustion. We thus conducted carbonization experiments with sets of whole acorns of evergreen oak and durmast oak (*Quercus ilex* and *pubescens*) (fig. 5A, 5B). The results of this work in progress will allow us to more reliably interpret the nature of acorn deposits discovered in habitats by evaluating the contributions of shelling treatments and destructions related to their carbonization. Our

A

B

fig. 5 : Experimental carbonization of durmast oak (*Quercus pubescens*) acorns, whole and dry at 550°C for 8 minutes (photos L. Bouby).
A – in reducing conditions: all the elements are carbonized, the pericarp is nonetheless roasted in some areas (brownish color);
B – in oxidizing conditions: the cotyledons are reduced to ashes, while the casing and the pericarp are carbonized.

objective is to clarify the alimentary role of these fruits for human populations and/or their domesticated animals: the practice of the *glandée*, well illustrated in Medieval western Europe (Mane, 2006, 336), often leads to the conclusion that the consumers of acorns were forcibly swine.

Conclusion

While the carbonization of seeds provides an opportunity to record information that would otherwise have disappeared, it also creates biases in the preservation of carpological assemblages, which all analyses must take into account. These biases concern two principal aspects: size and form of seeds and the qualitative and quantitative composition of carpological assemblages. The examples cited in this paper illustrate the essential role of experimentation in the analysis of these processes. Though it is difficult to precisely mimic archaeological conditions, experimentation is an effective method for testing the effects of different parameters, which must always be defined based on an archeobotanical problematic. It would be of little interest to study preservation by carbonization by itself. It is on the other hand, essential to integrate its effects into methodological procedures that address questions raised by carpological assemblages. The variations in the size and form of seeds can be of great interest in studies of domestication and the relationships between wild and cultivated species. Part of this variability could have a taphonomic origin and be linked to carbonization. While it is necessary to determine if this is the case, this is possible only if the pertinent morphometric criteria have been identified beforehand and if their living variability has been defined through adapted methods and reference bases.
The impact of carbonization on the composition of carpological assemblages can be apprehended only in the context of a broader taphonomic approach integrating the role of human activities, which themselves must be identified. The references in this case are ethnographic or, once again, experimental.

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References

Aurenche O. 1997 - Balanophagie : mythe ou réalité ? *Paléorient*, 23, 1 : 75-85.

Boardman S. & Jones G. 1990 - Experiments on the Effects of Charring on Cereal Plant Components. *Journal of Archaeological Science*, 17 : 1-11.

Bouby L., Terral J.-F., Ivorra S., Marinval P., Pradat B. & Ruas M.-P. 2006 - Vers une approche bio-archéologique de l'histoire de la vigne cultivée et de la viticulture : problématique, choix méthodologiques et premiers résultats. *Archéologie du Midi Médiéval*, 2-24, 2005-2006 : 61-74.

Bowman, A. R. A. 1966 - *Studies on the heat induced carbonisation of cereal grains*. Unpublished undergraduate dissertation. Department of Agricultural Botany, University of Reading (Grande-Bretagne).

Braadbaart F. 2008 - Carbonisation and morphological changes in modern dehusked and husked *Triticum dicoccum* and *Triticum aestivum* grains. *Vegetation History and Archaeobotany*, 17 : 155-166.

Braadbaart F. &t van Bergen P. 2005 - Digital imaging analysis of size and shape of wheat and pea upon heating under anoxic conditions as a function of the temperature. *Vegetation History and Archaeobotany*, 14 : 67-75.

Braadbaart F., Boon J.J., Veld H., David P. & van Bergen P. F. 2004 - Laboratory simulations of the transformation of peas as a result of heat treatment : changes of the physical and chemical properties. *Journal of Archaeological Science*, 31 : 821-833.

Charles M. 1998 - Fodder from dung: the recognition and interpretation of dung-derived plant material from archaeological sites. *Environmental Archaeology*, 1 : 111-122.

Coularou J., Jallet F., Colomer A. & Balbure, J. (eds) 2008 - *Boussargues. Une enceinte chalcolithique des garrigues du Sud de la France*. Toulouse, Archives d'Ecologie Préhistoriques, 337 pp.

Green F. J. 1979 - Collection and interpretation of botanical information from medieval urban excavations in southern England. Festschrift Maria Hopf. *Archaeo-Physika,* 8 : 39-55.

Guarino C. & Sciarrillo R. 2004 - Carbonized seeds in a protohistoric house : results of hearth and house experiments. *Vegetation History and Archaeobotany*, 13 : 65-70.

Gustafsson S. 2000 - Carbonized Cereal Grains and Weed Seeds in Prehistoric Houses - an Experimental Perspective. *Journal of Archaeological Science*, 27 : 65-70.

Helbaek H. 1952 - Preserved apples and Panicum in the prehistoric site at Nøore Sandegaard in Bornholm. *Acta Archaeologica,* 23 : 107-115.

Hillman G. C. 1981 - Reconstructing Crop Husbandry Practices from Charred Remains of Crops. *In* : *Farming Practice in British Prehistory*, Mercer R. (ed.). Edinburgh, Edinburgh University Press : 123-162.

Hillman G. C. 1984. Interpretation of archaeological plant remains : the aplication of ethnographic models from Turkey. *In* : *Plants and Ancient Man. Studies in Palaeoethnobotany*, van Zeist W. & Casparie W.A. (eds). Balkema, Rotterdam : 1-41.

Hopf M. 1955 - Formveranderungen von Getreide-Körner beim Verkohlen. *Berichte der Deutschen Botanischen Gesellschaft*, 68 : 191–193.

Jenkinson R. 1976 - *Carbonization of seed material: some variables at work*. Unpublished undergraduate dissertation, Department of Archaeology and Prehistory, University of Sheffield (Grande Bretagne).

Jorgensen G. 1977 - Acorns as a food-source in the later Stone Age. *Acta Archaeologica*, 48 : 233-238.

Kislev M. E. & Rosenzweig S. 1991 - Influence of experimental charring on seed dimensions of pulses. *In* : Palaeoethnobotany and archaeology, Hajnalova E (ed). International Work Group for Palaeoethnobotany, 8th symposium Nitra-Nove Vozokany, Tchécoslovaquie 1989. Nitra, *Acta Interdisciplinaria Archaeologica* 7 : 143–157.

Knörzer K.-H. 1971 - Pflanzliche Grossreste aus der Rössenerzeitlichen Siedlung bei Langweiler, Kreis Jülich. *Bonner Jarhrbücher*, 171 : 9-33.

Levadoux L. 1956 - Les populations sauvages et cultivées de *Vitis vinifera*. *Annales de l'Amélioration des Plantes*, 6 : 59-117.

Logothetis B. 1970 - The development of the vine and of viticulture in Greece based on archaeological findings in the area (in Greek with English and French summaries). *Epistimoniki Epetiris tis Geoponikis kai Dasologikis Sholis*, University of Thessaloniki, 17 : 5-286.

Lundström-Baudais K., Rachoud-Schneider A.-M., Baudais D. & Poissonnier B. 2002 - Le broyage dans la chaîne de transformation du millet (*Panicum miliaceum*) : outils, gestes et écofacts. *In : Moudre et Broyer. I - Méthodes*, Procopiou H. & Treuil R. (eds). Paris, CTHS : 181-209.

Mane P. 2006 - *Le travail à la campagne au Moyen Âge. Etude iconographique*. Paris, Picard, 471pp.

Mangafa M. & Kotsakis K. 1996 - A new method for the identification of wild and cultivated charred grape seeds. *Journal of Archaeological Science*, 23 : 409-418.

Märkle T. & Rösch M. 2008 - Experiments on the effects of carbonization on some cultivated plant seeds. *Vegetation History and Archaeobotany,* 17 : 257–263.

Maurizio A. 1932 (traduit du polonais) - *Histoire de l'alimentation végétale depuis la Préhistoire jusqu'à nos jours*. Paris, Payot, 663 pp.

Miller N. 1996 - Seed eaters of the ancient Near East: human or herbivore? *Current Anthropology*, 37 : 521-528.

Renfrew J. M. 1973 - *Palaeoethnobotany : the prehistoric food plants of the Near East and Europe*. New York, Columbia University, 248 pp.

Ruas M.-P. 1992 - The archaeobotanical record of cultivated and collected plants of economic importance from medieval sites in France. *In :* Festschrift for Professor Van Zeist*,* Pals J.-P., Buurman J. & Van der Veen M. (eds). Amsterdam, Elsevier Publishers Sciences, *Review of Palaeobotany and Palynology. An International Journal*, 73, 1-4 : 301-314.

Ruas M.-P. 2002 - *Productions agricoles, stockage et finage en Montagne Noire médiévale : le grenier castral de Durfort (Tarn)*. Paris, MSH, Documents d'Archéologie Française, 93, 231pp.

Ruas M.-P., Bouby L. & Pradat B. 2006 - Les restes de fruits dans les dépôts archéologiques du Midi de la France (V^e^-XVI^e^ siècle). *In* : Dossier Fructiculture, Ruas M.-P. (ed). *Archéologie du Midi Médiéval*, 23-24 : 145-193.

Smith H. & Jones G. 1990 - Experiments on the effects of charring on cultivated grape seeds. *Journal of Archaeological Science*, 17 : 317-327.

Van der Veen M. 1992 - *Crop Husbandry Regimes. An Archaeobotanical Study of Farming in northern England 1000 BC - AD 500*. Sheffield, University of Sheffield, Sheffield Archaeological Monographs, 3, 227 pp.

Van der Veen M. 2006 - Formation processes of desiccated and carbonized plant remains - the identification of routine practice. *Journal of Archaeological Science,* 20 : 1-23.

Van der Veen M. & Jones G. 2006 - A re-analysis of agricultural production and consumption: implications for understanding the British Iron Age. *Vegetation History and Archaeobotany,* 15 : 217-228.

Wilson D. G. 1984 - The carbonisation of weed seeds and their representation in macro-fossil assemblages. *In* : *Plants and ancient man. Studies in Palaeoethnobotany.* Van Zeist W. & Casparie W. A. (eds). Actes du 6^e^ symposium de l'I. W. G. P., Groningen, Pays-Bas, 1983, Groningen-Balkema : 201-206.

Wright P. 2003 - Preservation or destruction of plant remains by carbonization ? *Journal of Archaeological Science*, 30 : 577–583.

Zohary D. & Hopf M. 2000 - *Domestication of plants in the Old World.The origin and spread of cultivated plants in West Asia, Europe and the Nile Valley*. 3e edition, Oxford, Oxford University Press, , 316 pp.

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FROM WOOD TO WOOD CHARCOAL: AN EXPERIMENTAL APPROACH TO COMBUSTION

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Abstract

The anthracological deposit as it appears in archaeological contexts is the result of successive taphonomic agents intervening at many stages from the gathering of wood, to combustion and post-depositional processes. These taphonomic agents constitute successive filters between the past vegetation and the charcoal studied. Therefore, the interpretation of charcoal remains should take into account the potential deformation between the anthracological spectra and initially burned wood.

This paper presents the methodological issues and the first results of an experimental cycle whose aim was to study one of these taphonomic agents: the combustion process. Does combustion involve differential preservation of burned wood species? Does charcoal quantification reflect the real proportion of burned species? Is it necessary to define a correctional index for anthracological data?

In order to answer these questions, 110 experiments were carried out under standardized laboratory conditions and more than 295,000 charcoals were studied. Such factors as wood density or temperatures are often considered to play an active role in the fragmentation process of charcoal during combustion. As expected, the results indicate a differential behaviour of species, but one which proved to be independent of the expected factors, tending to show that the parameters that interact in the combustion process are of different nature. These factors increase the difficulty of defining a correctional index. Nevertheless, it seems that the quantification of charcoal reflects in a satisfactory manner the initial proportion of each of the burned species.

Keywords : experimentation, carbonization, fragmentation, wood charcoal, representivity

Introduction

Anthracological studies of archaeological wood charcoal are mainly based on paleoecological or economic interpretations of residues originating from incomplete wood combustions collected in archaeological sediments or fireplaces. If we believe the supposed occupation durations, these residues represent a very small proportion of the wood mass burned and probably a variable proportion depending on the nature of the sites and deposits, in other words, depending on the modes of fuel management, fire maintenance, fireplace cleaning, habitation cleaning, etc. Being ignorant of these *habitus*, we know nothing of the rate of disappearance of material from the time the wood was lit on fire until the residues were finally collected. We therefore cannot attribute any significance to the absolute quantities of wood charcoal from each species. For instance, a scarcity of residues does not necessarily indicate a scarcity of wood in the environment. Few wood charcoals are found in certain periods of the Paleolithic due to a rarity of forests or freeze/thaw cycles in the deposits; but this is also true during Antiquity because fireplaces and occupation floors were swept clean. For this reason, an anthracologist cannot reason based only on the list of taxa identified and their relative frequencies in a sample. It is necessary to know what these relative frequencies represent.

As with any archaeological assemblage, wood charcoals result from a succession of events that occur starting in their original environment until the deposits that we study. The succession of taphonomic processes that affect them can be studied in order to evaluate the degree to which the information they contain has been transformed, whether in the context of past forest environments, fire-related social and cultural practices or interactions between the economy of wood and afforestation (Chabal 1997, Théry-Parisot 2001, Théry-Parisot *et al*., 2010). In this succession of factors, there is one, combustion, which merits closer scrutiny since it plays a major role in the disappearance of wood materials. Should we thus consider that combustion acts as a bias in the representivity of the taxa present and modifies our image of the vegetation or any other factor that we wish to understand?

In anthracology, the measurement unit most often used to determine the frequencies of taxa is the number of wood charcoals present (Chabal, 1982, 1988; 1992, 1997; Heinz, 1990; Badal-Garcia, 1992; Bourquin-Mignot *et al.*, 1999). This quantification method requires the existence of a statistical process of fragmentation that is the same for all the taxa of an archaeological sample. Therefore, the state of fragmentation observed in an archaeological sample of wood charcoal, meaning the distribution of the individual masses of fragments, always corresponds to a Poisson distribution whose parameters depend on the method of sieving employed, but not the taxon considered (Chabal 1989, 1992, 1997). This permits us to affirm that the unit constituted by “one wood charcoal” has a meaning in statistical terms and allows us to compare different taxa, in other words, to express their relative frequencies. A specific question that interests us here is the following: are the frequencies of taxa expressed in this manner a valid expression of their frequencies among the intentionally burned wood materials? In effect, fragmentation and mass reduction are two processes that are simultaneous, but independent in their results. Until now, anthracology has been based on the hypothesis that all species behave in the same manner in an identical fire in terms of mass reduction: in other words, the ratio of proportionality between the quantity of wood charcoal and the quantity of wood burned would be identical for all species. This hypothesis is founded on (i) the reproducibility of frequency spectra between synchronous levels of the same site—this would not be the case if the combustion created a variable distortion of the frequency of taxa—and (ii) the paleoecological representivity of archaeological samples of wood charcoal and the coherence of anthracological diagrams between themselves and through time—this would not be the case if the hierarchy between dominant taxa and subordinate taxa was modified by a differential mass reduction. The existence of a single statistical law of fragmentation for all taxa justifying our unit of measure is largely demonstrated (since it is based on observed archaeological samples). Inversely, the hypothesis according to which all species behave in the same manner in an identical fire is still justifiably questioned since it has not yet been experimentally validated. Therefore, probably due to the complexity of their realization, quantifications of the frequencies of taxa following experimental combustions are few and sometimes present discordant results, which merit discussion.

Starting in the 1970-80's, experiments addressing the processes of fragmentation and the reduction of the mass of burned wood have been conducted. The results of these experiments vary greatly, however, depending on the authors who generally opposed the intrinsic and extrinsic variables of combustion as factors determining the proportions of residues. According to some researchers, the proportions of residues depend on the physical, chemical and mechanical properties of the species in question (Rossen & Olson, 1985; Smart & Hoffman, 1988; Loreau 1994; Braadbaart and Poole, 2008), while others defend the predominant role of extrinsic variables (type of combustion feature, temperatures, oxygenation) (Scott & Jones, 1991; Belcher *et al.,* 2005; Vaughan & Nichols 1995). Rossen and Olson (1985) argue that low density woods produce less charcoal than hard woods, but Loreau (1994) believes the opposite is true. According to this latter author, a high humidity level in the wood clearly reduces the proportion of residues, while our own results tend to minimize the role of this parameter (Théry-Parisot *et al*., 2010). For some authors the caliber of the wood determines the proportion of residues (Smart and Hoffman, 1988). Lingens *et al.* (2005) suggest that the differences in the proportion of remains are more closely linked to the chemical composition of wood than to its density. The work of Belcher *et al.,* (2005) and Scott & Jones (1991) indicates that the proportions of residues reflect a concomitant effect of the combustion temperature and oxygenation. Finally, according to Vaughan & Nichols (1995), the temperature attained in a fireplace determines the size, density and morphology of the residues.

Faced with these diverging results and in the context of a broader exploration of taphonomic factors, it appeared useful to us to pursue and develop analytic approaches to combustion.

- Do different species behave differently when burned?
- Is there an over or under-representation of certain taxa following combustion?
- What are the experimental variables that determine the behavior of fuel materials in fire?
- Can we and should we define correction indices of the frequencies of taxa that can be used in anthracology?

Experimentation is the most appropriate method for addressing these types of questions.

Experimentation: procedure and protocol

The experiments were realized in an open fireplace under laboratory conditions in order to limit the known effects of external factors (wind, atmospheric humidity, fireplace form, etc.) on the combustion process (fig. 1). The form, size and maintenance of the fire of course have a direct effect on its thermal behavior and, consequently, on the residues themselves. But there exist so many possible combinations that it would be illusory to think that we are capable of experimentally reproducing all past situations. Therefore, by eliminating (to the greatest degree possible) all variation linked to external factors and by standardizing the experiments as much as possible, the impact of a combustion process on its combustibles can be studied independently of the technical gestures. With an understanding of the effects of combustion, and in particular the behavior of different species in a fire, it will then be possible to consider the over-representation of a taxon due to other causes, such as the form of the fireplace or the practices and gestures related to the use of fire. Given that these aspects are of great interest to us, and that we must not risk false interpretations of them, it is all the more clear that the combustion process must be known beforehand.

The experiments (fig. 2) were conducted with eleven taxa commonly represented in anthracological assemblages: *Quercus pubescens; Betula pubescens;*

Olea europea; Corylus avellana; Carpinus betulus; Ostrya carpinifolia; Pinus pinaster; Pinus halepensis; Pinus sylvestris; Populus alba; Populus tremula. In order to account for intra-specific variability, the lots of each taxon were collected from two geographically distinct sources (except for *Populus alba*, *Populus tremula*, *Carpinus betulus* and *Ostrya carpinifolia* for which only one source was sampled). The wood was unsplit, dry and had a humidity level between 12 and 14%. Prior to combustion, the characteristics of the logs within each lot were recorded (mass, volume, surface, caliber and humidity level).

All of the combustions were realized following a strictly identical protocol: each fireplace was composed of 6 calibrated logs and was constructed and maintained in an identical manner (fig. 1). Each fire was lit using a blowtorch to avoid introducing small caliber wood fragments.

For each taxon and each source, 6 combustion replicas (or sometimes only 5) were realized in order to control and record the variability of similar experiments, called "intra-individual" variability.

In an open structure, temperature is a highly fluctuating measure that is conditioned by the position of the sensor in the fireplace and a high number of sensors are thus necessary to obtain an exploitable measure. The temperatures were thus simultaneously recorded using 12 sensors distributed within the fireplace. The total duration and the duration of combustion with flames were also recorded. After the combustion, all of the residues were sieved with four meshes in order to separate five fractions (>4 mm, 2 to 4 mm, 1 to 2 mm, 0,5 to 1 mm and < 0,5 mm), which were weighed and counted (for the >4 and 2 to 4 mm classes). The ashes were reserved for a quantitative study of the phytoliths (Delhon, this volume). The results we present here concern 110 combustions, represented by 295,688 residual wood charcoals. In this paper, we present the results of the analysis of residues greater than 2 mm (2 and 4 mm sievings).

Fig. 1 - Experimental combustion structures: from wood to wood charcoal.

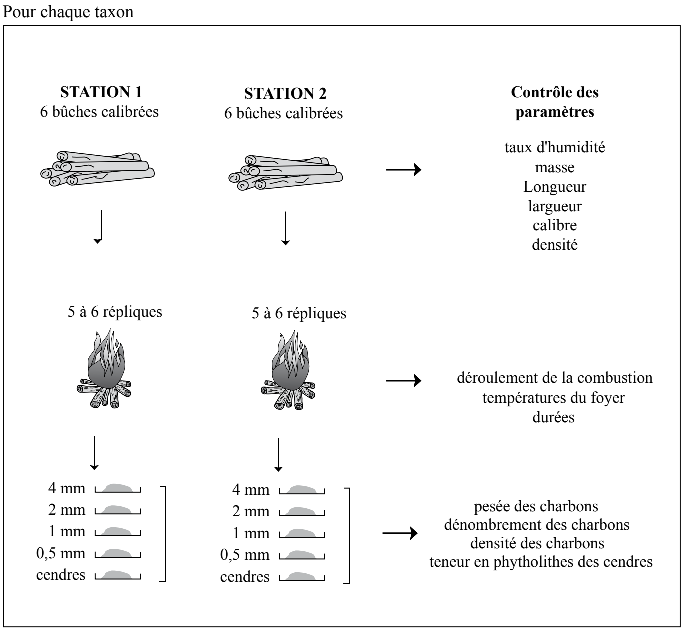

Fig. 2 - Experimental protocol.

Is the number of residual charcoal fragments dependent on the quantity of wood burned?

The first factor measured was the relationship between the quantities of wood put into the fires, expressed as a volume, and the residual number of wood charcoal fragments, expressed as the number of fragments: is the residual number of wood charcoals proportional to the quantity of wood put into the fire, regardless of the taxon considered? We express the quantity of wood as a volume since the combustions were realized with logs lots having the same volume[1], but a highly variable mass depending on the density of the species[2]. Reasoning in terms of wood masses would thus have meant working with fires that were not standardized between species and whose measures would thus not be comparable.

In figure 3, each point represents a combustion and indicates that the number of charcoals logically tends to increase with the volume of wood, but the regression shows the statistical independence of this relationship (R^2=0.04): the number of wood charcoals is not dependent on the quantity of wood put into the fire when the parameters of species and source are varied. It is therefore not possible to deduct the quantity of wood burned in a fire based on the quantity of charcoal found in an archaeological context. We can remark that this lack of correlation between the quantity of wood burned and the quantity of carbonized residues is demonstrated under standardized combustion conditions; it is thus *a fortiori* true and certainly even more significant under real conditions where parameters such as fire aeration, the caliber of the wood pieces and the form of the combustion structure my vary. On this graph, we can observe a clustering effect: the grouping of points of the same color, which represent a single taxon individualized according to its source, shows the reproducibility of the measure, indicating that the replicas within a single experiment behave in a nearly identical manner. This reproducibility of results tends to validate the protocol and suggests the existence of a correlation between the taxa and the residual number of wood charcoals.

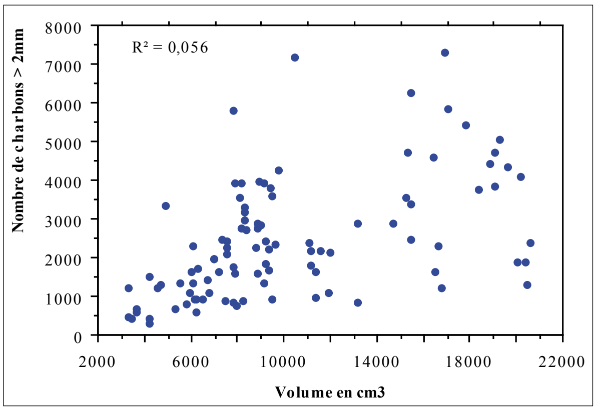

Fig. 3 - Relationship between the volume of wood put into a fire and the number of residual wood charcoals >2mm.

Source variability

The results of our experiments are expressed as proportions of residues in order to standardize and allow comparisons of the experiments[3] (fig. 4).

[1] - Stabilizing the volume of wood and the caliber of the logs is important in order to standardize the aeration of the fire, which would not be possible with log lots of equal masses.

[2] - It is for this reason that firewood is commercially sold by the cubic meter and not by the ton.

[3] - Number of wood charcoals over 2 mm over the initial volume of wood.

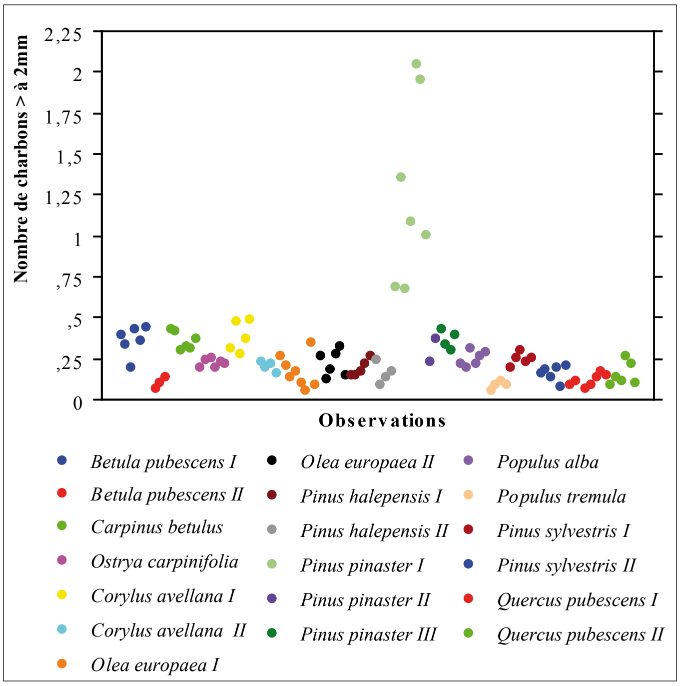

Fig. 4 - Projection of the number of wood charcoals >2mm for each source.

According to the graph, the replicas within a single experiment produce very similar results for 18 of the 19 sources tested[4]. Only the *Pinus pinaster* I station is distinguished by a clearly higher residue proportion and significant variations between the replicas. Except for *Pinus pinaster* I, the reproducibility of the measure in the replicas for 18 sources (10 species) validates the experimental results. The variability among the sources is evaluated through a comparison of the results obtained for the two sampling sources of each species (fig. 5). On each graph, the average proportion of residues for the two sources is represented. The source variability is not significant for *Quercus pubescens*, *Olea europaea* and *Pinus halepensis*, the two lots leaving an equivalent average proportion of residues. Inversely, it is significant for *Pinus*

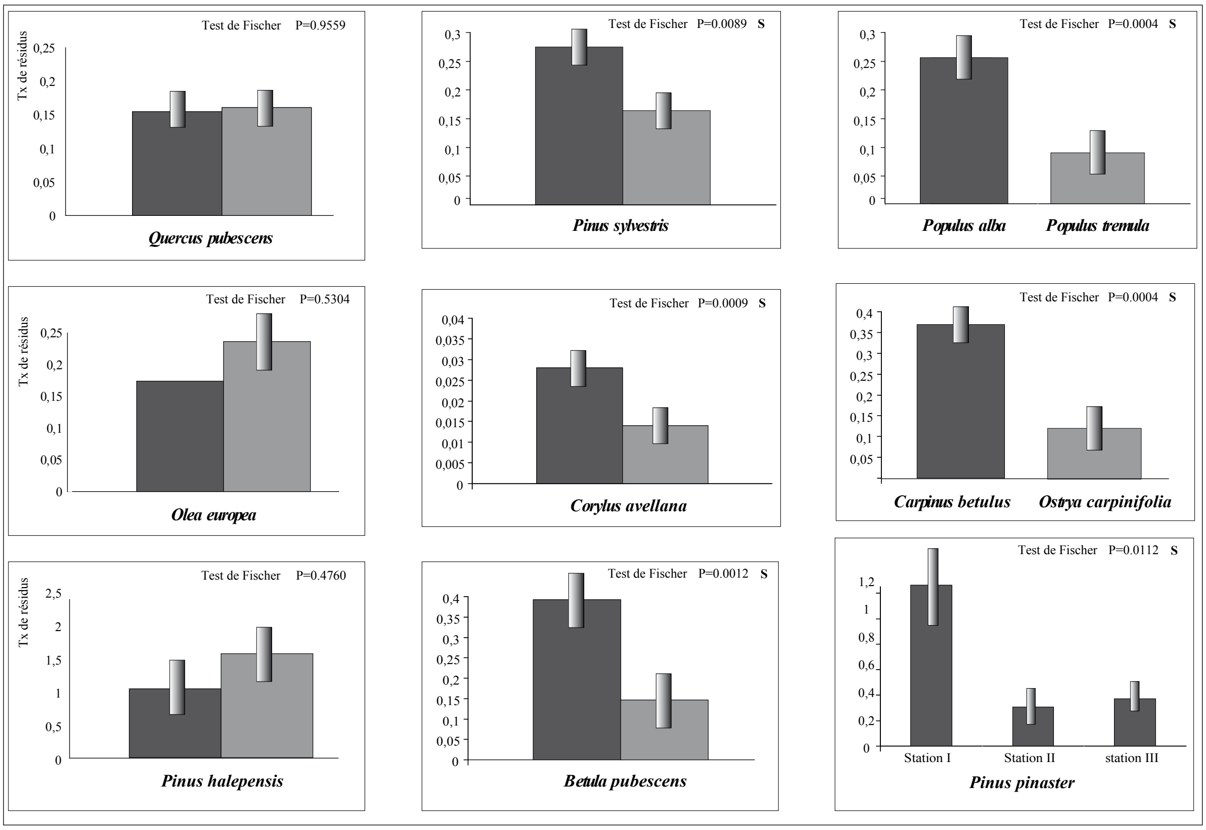

Fig. 5 - Variance analysis of the proportion of wood charcoals >2 mm “source effect”.

[4] - The points of the same color represent the replicas within a single experiment, meaning all of the experiments realized for a taxon with the same geographic origin (source).

sylvestris, *Corylus avellana* and *Betula pubescens,* which present a variable proportion of residues depending on the source. This variability could be due to factors of environmental growth, which may have determined certain characteristics of the wood that could have an influence on its behavior when burned. For example, it is possible that source variations in the density or chemical composition of wood are determinant in this "environmental" variability.

Finally, three taxa raise particular problems. The *Pinus pinaster* I station presents an abnormal dispersion of the residue proportions in six of the experiments realized. A new series of six experiments conducted with the lots originating from a third source confirm the abnormal behavior of the *Pinus Pinaster* I (PpI) lot. Contrary to source I, sources II and III present residue proportions that are comparable and coherent with the other results (they remain within the scatter plot). A rapid microscopic observation of the residual charcoals shows that the bark of PpI represents a high proportion of the residues analyzed[5]. However, (i) bark is often poorly represented in anthracological assemblages, (ii) and when it is, it is rarely possible to determine the species. It would thus be necessary to recalculate the residue proportions without the bark so that it represents only the identifiable charcoals, which was not possible due to the very high number of remains (9.500 fragments). Therefore, the source variability of *Pinus pinaster* could be (i) real, but remains to be determined, and/or (ii) result from a quantification bias due to the over-representation of bark. Since we do not yet have a response to this question, the data from the PpI source are excluded from this study and only sources II and III are taken into account. In addition, the source variability could not be evaluated for the *Populus* and *Carpinus* lots due to a problem during the procurement of the wood: the *Populus* lots originate from two species *P. alba* and *P. tremula*. However, these two groups present statistically distinct residue proportions. Similarly, the two *Carpinus betulus* sources, which are also discriminated by their residue proportions, correspond in reality to one *Carpinus betulus* source and one *d'Ostrya carpinifolia* source. Consequently, lacking a comparison source, it is not possible to reach a conclusion concerning the source variability of *Populus alba*, *Populus tremula*, *Carpinus betulus* and *Ostrya carpinifolia*. At most, we assume the existence of a taxonomic variability (intra-generic for *Populus*, specific for the others).

According to our results, the source variability, though it clearly exists, is not constant for all the species analyzed. The question of specific variability is then raised. If the inter-specific variability is greater than the intra-specific variability (source), this latter can be considered negligible and the residue proportion must be analyzed as a variable related to the species. If the intra-specific variability is greater, on the other hand, we must conclude that there is no average species/genus behavior, but rather a high variability of behavior in fire of populations, of an environmental nature, within a single taxon.

Specific variability

Figure 6 shows the proportion of residues recorded for each taxon, with the two sources of each species now being grouped, except for the *Carpinus betulus* and d'*Ostrya carpinifolia* and *Populus alba* and *Populus tremula* lots, each of which originate from only one source (see above).

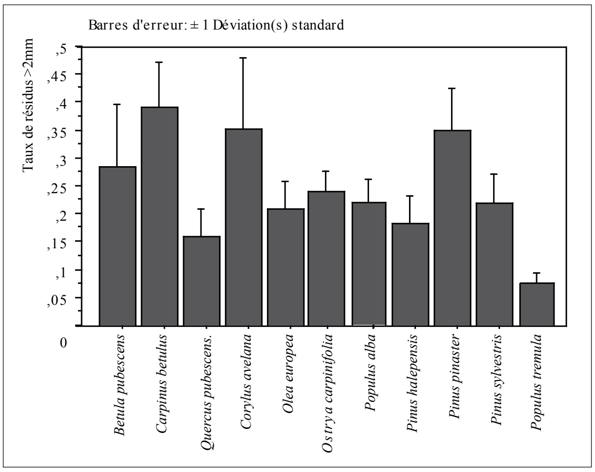

Fig. 6 - Variance analysis of the proportion of wood charcoals >2 mm "species effect".

[5] - Observations of the characteristics of the wood lots of source I also show that the bark is very thick (average 3.5 cm).

Variance analysis shows that, despite source variability, the species "effect" is significant with a value of $p < 0.0001$. Three groups of taxa are statistically discriminated ($F= 23.8$; $p<0.0001$) (fig. 7). Group 1, composed of *Betula pubescens, Carpinus betulus, Corylus avellana* and *Pinus pinaster,* leaves on average more residues than the two other groups ($TR_{es\,=}$ 0.334). Group 2, composed of *Pinus sylvestris, Olea europea, Populus alba* and *Ostrya carpinifolia*, has an average residue proportion (TR_{es}=0.222) that is close to the statistical average (TR_{es} =0.248). Group 3, composed of *Pinus halepensis, Quercus pubescens* and *Populus tremula,* leaves, on average, fewer residues than the two other groups ($TR_{es\,=}$ 0.155). Group 1 leaves on average two times more residues than group 2.

This signifies that, after combustion, and if we refer to group 2 (*Pinus sylvestris, Olea europea, Populus alba* and *Ostrya carpinifolia*), for which we can say that the burning behavior is close to the statistical average, the *Betula pubescens*, *Carpinus betulus*, *Corylus avellana* et *Pinus pinaster* taxa are over-represented in the number of wood charcoals, while *Pinus halepensis, Quercus pubescens, Populus tremula* and *Ostrya carpinifolia* are under-represented.

These results reveal specific variability in the residue proportions whose cause must now be analyzed: why are the effects of combustion different for close species? How can we explain, for example, that the variability of residue proportions is so high between two species of the same genus (cf. *Populus alba* and *P. tremula*, *Pinus pinaster* and *P. halepensis*)?

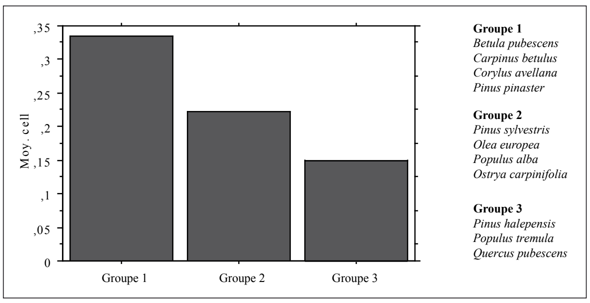

Fig. 7 - Variance analysis of the taxa "group effect".

The effect of density

We first considered the effect of wood **density**, which some authors believe could have an influence on residue proportions (Loreau, 1994; Rosen & Olson, 1985). In effect, the density, via the porosity, is related to the inflammability of wood. Figure 8 shows the residue proportions and average density of each taxon. We observe that the density has no clear relationship with the residue proportions with a coefficient of determination that is nearly null (R^2=0.04). For example, *Quercus pubescens* and *Carpinus betulus*, which are both high density wood, are represented in the extreme groups (1 and 3). The same is true for *Populus tremula* and *Pinus pinaster*, which are both low density woods. Even more so, *Populus tremula* and *Populas alba*, whose theoretical density is nearly the same, have very different residue proportions (fig. 9). This factor thus does not explain the species specific residue proportions.

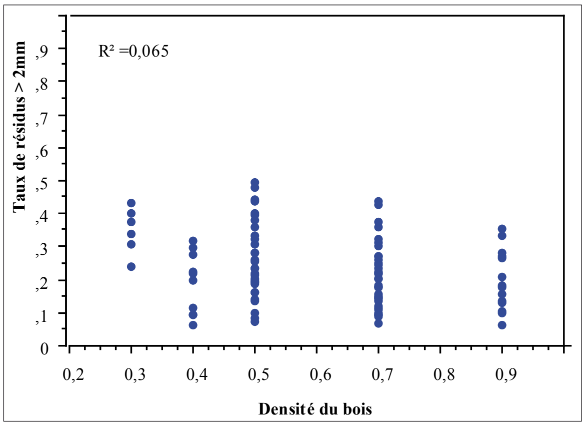

Fig. 8 - Regression of wood density vs proportion of wood charcoals >2mm.

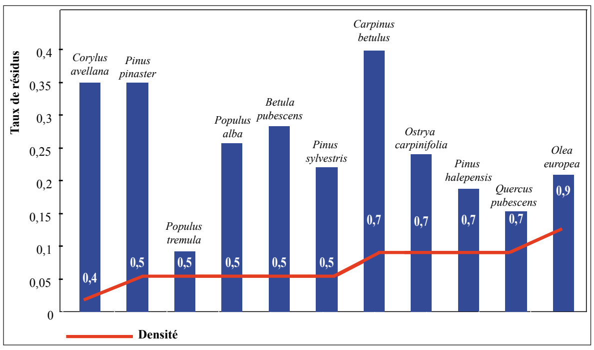

Fig. 9 - Residue proportions and wood density.

The effect of the taxonomic proximity of the species

Among the four species of group 1 (high residue proportions), three present evident anatomical similarities (*Betula pubescens*, *Corylus avellana* and *Carpinus betulus*) (Schweingrüber, 1978). The genera *Betula, Corylus* and *Carpinus* are in fact currently classed in the same family, the Betulaceae. Group 2 (low residue proportions), on the other hand, are composed of both Gymnosperms (conifers) and Angiosperms (hardwoods). Furthermore, the three species of the genus *Pinus* are not represented in the same group. Therefore, if the **anatomy**, or even chemistry (also linked to the taxon), of wood had an influence on its burning behavior, why aren't the species with the greatest number of taxonomic affinities in the same group? It thus appears that the burning behavior of the different species is not linked to a simple relationship with the anatomy of the wood considered from the perspective of taxonomic affinity.

The effect of temperatures

The recording of **temperatures** during combustion allows us to address the recurring question of the relationship between temperature and the proportion of residues. A regression analysis shows the independence of these two variables (R^2=0.1) (fig. 10). The maximal average temperatures recorded, which vary in our experiments from 780 to 916°C, are independent of the residue proportions (fig. 11). There is thus no

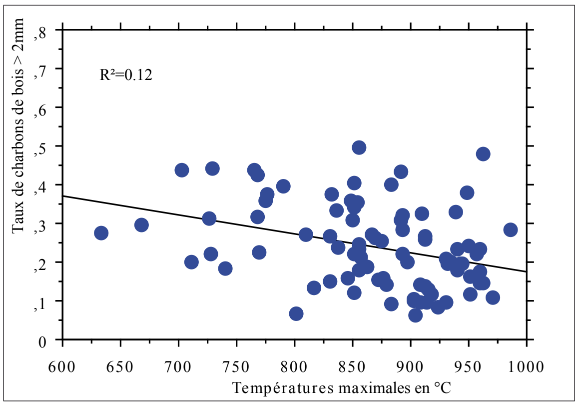

Fig. 10 - Regression of maximal temperatures vs proportion of wood charcoals >2mm.

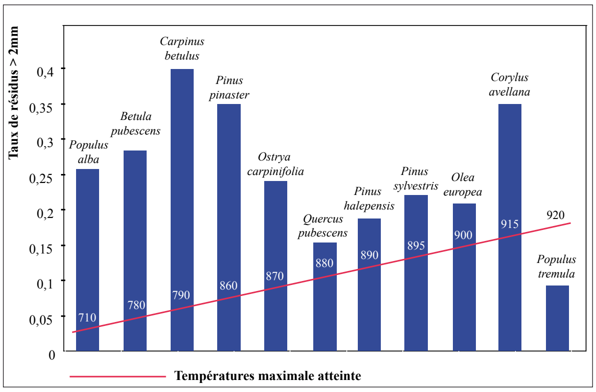

Fig. 11 - Residue proportions and maximal temperatures attained during combustion.

relationship between the temperature curve of a species and its residue proportion. In addition, we can question the pertinence of this measure, which, even if it consists of an average calculated from 12 sensors per combustion, can vary from 100 à 200°C on average depending on the position of the sensors within the structure.

The effect of the combustion duration

In our experiments, the combustion **duration** varied from 4 to 15 hours with no relationship between the quantity of wood put into the fire and the total duration (fig. 12) (R^2=0.033).

The combustion duration varied little from one taxon to another with the exception of *Carpinus betulus* and *Quercus pubescens,* which have longer combustion durations (fig. 13). However, the residue proportion of *Quercus pubescens* is the lowest, while that of *Carpinus betulus* is one of the highest. The variable of the combustion duration of each species thus does not explain its residue proportion.

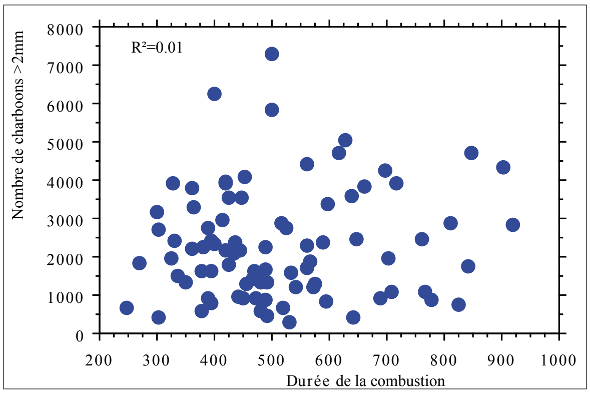

Fig. 12 - Regression of combustion duration vs proportion of wood charcoals >2mm.

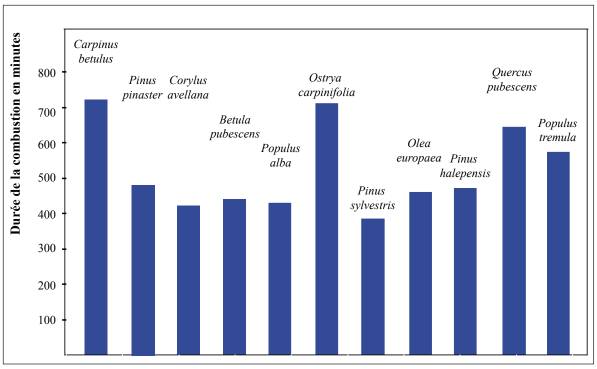

Fig. 13 - Residue proportions and combustion duration.

Discussion and conclusions

Our results appear to be validated by the reproducibility of measures within the same experimental modalities. They show that (i) the proportion of residues is not due to a simple proportional relationship with the quantity of wood put into a fire, even under standardized combustion conditions; (ii) that the behavior of different sources (intra-specific variability) in a fire is random, and; (iii) that there is an inter-specific variability that discriminates three groups of taxa. This specific variability is not explained by any of the variables tested (density, anatomical proximity, temperatures or combustion duration). These experiments thus underline the difficulty of detecting a relationship between variables whose influence appears *a priori* clear, and the proportions of residual wood charcoals. For example, temperatures, combustion durations, or wood density for the two *Populus* species (*P. alba* et *P. tremula*) clearly show a statistic independence between wood density and residue proportions.

In order to understand the relationship of taxon/residue proportion, it will be necessary to consider other variables whose influence is seemingly less evident than those tested, such as microporosity[6], chemical composition or a global thermal synthesis that is not limited to the optimal temperatures.

The groups identified are opposed relative to the statistical average, with some taxa tending to be over-represented (*Carpinus betulus*, *Betula pubescens*, *Corylus avellana* and *Pinus pinaster*) and others under-represented (*Quercus pubescens*, *Populus tremula* and *Pinus halepensis*). But what about the "real" proportionality, meaning one that compares all of these data? In our analyses, the representation of residues is evaluated relative to a statistical average that is calculated based on the individual data (replicas) of each taxon. The Anova test evaluates the dispersion of each taxon around this statistical average. But another possibility is to globally evaluate (all experiments together) the relationship between the initial proportion of wood burned for each taxon and the proportion of residual charcoals. This consists of nothing more than an anthracological diagram presenting the *pre*- and *post*-combustion proportions (fig. 14). These species were of course not burned together and this is only a graphic representation.

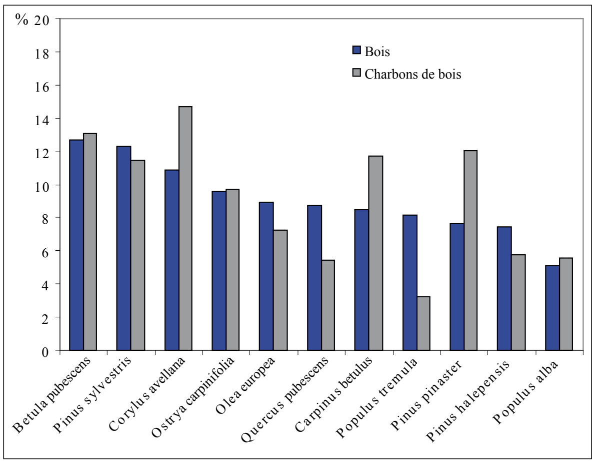

Fig. 14 - Proportionality relationship between the quantity of wood put into a fire and the quantity of residual wood charcoals.

We observe that the proportionality ratio is respected for six of the eleven taxa analyzed (difference < to 2%): *Betula pubescens, Pinus sylvestris, Ostrya carpinifolia, Olea europaea, Pinus halepensis* and *Populus alba.* Three taxa are over-represented (*Corylus avellana, Carpinus betulus* and *Pinus pinaster*), while two are under-represented (*Quercus pubescens* et *Populus tremula*).

[6] - The porosity of wood could be measures by the density of the vessels and the vascular conductivity, two variables that are considered in quantitative ecoanatomy, and which vary according to the species and climatic parameters (source variations) (Bourquin-Mignot *et al.*, 1999, p. 101).

Therefore, the proportions of wood charcoals are generally an accurate reflection of the initial proportions of species put into a fire and there is no inversion of majority or minority values. We should nonetheless note that the residue proportions of a few taxa are poor reflections of the proportion of wood put into a fire. In addition, these results do not integrate the potential and probable interactions that would occur between different species if they were burned together.

In archaeological contexts, there is also the question of the anatomical distinction of species whose behavior in a fire is not similar. For example, though it is theoretically possible, the anatomical discrimination between *Pinus pinaster* and *P. halepensis* can sometimes be problematic. The same is true for *Populus alba* and *Populus tremula*, which though they were discriminated in our experiments, are anatomically comparable. It would thus be difficult to evaluate the quantitative representivity of these taxa based only on our experiments.

The results we have presented here remain incomplete and many other observations must be realized based on the same data. For example, there is the question of the representivity of different size classes of wood charcoals, separated by different mesh sizes in a sieving column. Does one of the fractions better represent the initial proportion of each species burned? The data were analyzed here in a global manner (fraction above 2 mm) even though our data would allow us to treat the N > 4 mm and 2 < N > 4 classes separately. Furthermore, the expression of residues in terms of mass, rather than as the number of fragments, was not addressed. Therefore, questions related to the relationship between the number and mass of residual charcoals, the representivity of these two measures and the proportional relationship between the volume of wood burned and the residual mass of wood charcoal, remain unanswered. Our results should also be compared with those of combustions realized in outside, in open-air conditions—or inside but with the introduction of variables such as a controlled aeration of the fire—in which extrinsic factors dominate over intrinsic factors and could override and nullify the effects recorded in our experiments.

To finish, we must remember that considering combustion as a possible agent in the distortion of anthracological diagrams is not sufficient. It is of course necessary to integrate the impact on assemblages of wood gathering practices before combustion and depositional and post-depositional processes after combustion. Meanwhile, in our current state of knowledge, experimental measures addressing the differential behavior of species are rare. Research in progress on this subject should greatly contribute filling this void.

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References

Badal-Garcia E. 1992 - L'anthracologie préhistorique : à propos de certains problèmes méthodologiques. *Bulletin de la Société Botanique de France*, 139 : 167-189.

Belcher C.M., Collinson, M.E. & Scott, A.C. 2005 - Constraints on the thermal energy released from the Chicxulub impactor: new evidence from multi-method charcoal analysis. London, *Journal of the Geological Society*, 162 : 591-602.

Bourquin-Mignot M. ***et al.***, 1999 - *La botanique*. Collection Archéologiques, Ferdière. Paris, éditions Errance, 207 pp.

Braadbaart F. & Poole I. 2008 - Morphological, chemical and physical changes during charcoalification of wood and its relevance to archaeological contexts. *Journal of Archaeological Science*, 35 : 2434-2445.

Chabal L. 1982 - *Méthodes de prélèvement des bois carbonisés protohistoriques pour l'étude des relations homme-végétation*. D.E.A., Université de Montpellier II., 54 pp.

Chabal L. 1988 - Pourquoi et comment prélever les charbons de bois pour la période antique : les méthodes utilisées sur le site de Lattes (Hérault). *Lattara*, 1 : 187-222.

Chabal L. 1989 - Perspectives anthracologiques sur le site de Lattes (Hérault). *Lattara*, 2 : 53-72.

Chabal L. 1992 - La représentativité paléo-écologique des charbons de bois archéologiques issus du bois de feu. *In :* Les charbons de bois, les anciens écosystèmes et le rôle de l'Homme, Vernet J.-L. (ed). Actes du Colloque International Montpellier, 10-13 septembre 1991. *Bulletin de la Société botanique de France*, 139, 2-4 : 213-236.

Chabal L. 1997 - Forêts et sociétés en Languedoc (Néolihique final, Antiquité tardive). L'anthracologie, méthode et paléoécologie. *Documents d'Archéologie Française*, 63, 189 pp.

Delhon C. ce volume - Phytolithes et taphonomie, apport de l'expérimentation à la quantification des phytolithes dans les cendres de bois. *In* : Tapononomie des résidus organiques brûlés et des structures de combustion en milieu archéologique, Théry-Parisot I., Chabal L. & Costamagno S. (eds). Actes de la table ronde, Valbonne, 27-29 mai 2008. *P@lethnologie*, 2 : 97-109.

Heinz C. 1990 - Dynamique des végétations holocènes en Méditerranée nord occidentale d'après l'anthracoanalyse de sites préhistoriques : méthodologie et paléoécologie. *Paléobiologie continentale,* XVI, 212 pp.

Lingens A., Windeisen E. & Wegener G. 2005 - Investigating the combustion behaviour of various wood species via their fire gases. *Wood Science and Technology*, 39 : 49-60.

Loreau P. 1994 - *Du bois au charbon de bois : approche expérimentale de la combustion*. Université de Montpellier II, Montpellier, 64 pp.

Rossen J. & Olson J. 1985 - The controlled carbonisation and archaeological analysis of SE U.S. wood charcoals. *Journal of Field Archaeology*, 12 : 445-456.

Scott A.C. & Jones T.P. 1991 - Microscopical observations of Recent and fossil charcoal. *Microscopy and Analysis,* 24 : 13-15.

Smart T.L. & Hoffman E.S. 1988 - Environmental interpretation of archaeological charcoal. *In* : *Current paleoethnobotany*, Hastorf C.A. & Popper V.S. (eds).Chicago and London, University of Chicago press : 165-205.

Schweingrüber F.H. 1978 - *Anatomie microscopique du bois*. Institut fédéral de Recherche Forestière, Zurcher A. G., 126 pp.

Théry-Parisot I. 2001 - *Economie des combustibles au Paléolithique. Expérimentation, anthracologie, taphonomie*. D.D.A. n° 20, CNRS-Editions, 195 pp.

Théry-Parisot I., Chabal L. & Chrazvzez J., 2009 - Anthracology and taphonomy, from wood gathering to charcoal analysis. A review of the taphonomic processes modifying charcoal assemblages. *Palaeogeography, Palaeoclimatology, Palaeoecology,* 291, 1 : 142-153.

Vaughan A. & Nichols G.J. 1995 - Controls on the deposition of charcoal: Implications for sedimentary accumulations of fusain. *Journal of Sedimentary Research*, A65 : 129-135.

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PHYTOLITHS AND TAPHONOMY, THE CONTRIBUTION OF EXPERIMENTATION TO THE QUANTIFICATION OF PHYTOLITHS IN WOOD ASHES

Claire Delhon

Abstract

Ashes, the mineral residues of wood combustion, contain siliceous particles that can be preserved for long periods in archaeological sediments. Phytoliths can thus be useful indicators of combustion activities whose biodegradable or soluble remains have disappeared.

In this paper, an experimental evaluation of the potential of phytoliths for the quantitative and qualitative analysis of carbonized ligneous biomasses is presented. The results show: 1) that only a very small portion of ash is capable of resisting dissolution phenomena, 2) that phytoliths originating from ligneous tissues are only slightly characteristic from a taxonomic perspective, and 3) that it is not possible through a routine microscopic analysis to differentiate phytoliths derived from combustion and phytoliths liberated following a slow decomposition of organic material. It thus appears that strong concentrations of "wood" phytoliths can be an indicator of combustion, but that phytolithic analysis does not allow taxonomic identification of the ligneous combustible or evaluation of the quantity of biomass burned.

Keywords : phytolithic analysis, AIF, ligneous combustible, experimentation

Introduction

Wood combustion results in the fragmentation and reduction of the mass of ligneous material and produces two types of remains that are distinguished according to their size and chemical composition: charcoal and ash. Charcoal is produced by the incomplete combustion of wood: if the combustion process is completed, only ashes remain (Chabal *et al*., 1999). The residues are then mineral, composed principally of carbonates, phosphates, sulfates and siliceous compounds (Wattez, 1988; Olanders & Steenari, 1995). If the temperature continues to rise after this calcination phase, the ashes are fused (Olanders & Steenari, 1995).

Charcoal and ash are chemically stable compounds that can be preserved over long periods, notably in archaeological contexts. Charcoal, however, is easily fragmented and can thus gradually disappear into the sedimentary matrix. The ash fraction is composed partly of particles that are soluble in water or acid pH solutions (mainly carbonates) and partly of slightly soluble particles with a neutral or acid ph, the majority of which are silicates. These silicates are not produced by combustion. They are particles of opal silicate, called phytoliths. As they are present in vegetal tissues and are non combustible, they can be found concentrated in combustion residues. In these unfavorable preservation conditions, When preservation conditions are unfavourable, after charcoal has disapperaed, at least at macroscopical scale, and the carbonated ashes have dissolved, phytoliths are the last remaining traces of the carbonized biomass (Schiegl *et al*., 1994, 1996; Albert *et al*., 2000).

However, data are currently lacking to evaluate to what degree it is possible to reconstruct the ligneous fuel based on a phytolithic assemblage, whether qualitatively (identification of the combustible) or quantitatively (evaluation of the quantity of wood burned). In addition, since phytoliths are not produced by combustion, but simply concentrated by it, we also lack techniques for distinguishing between "burned" phytoliths and those liberated from their organic gangue by other processes.

This paper presents a first contribution to the quantification of microremains (i) resulting from the combustion of ligneous tissues, (ii) which can be preserved over long periods in archaeological contexts, (iii) and that can be identified through a microscopic analysis of the sediment. The goal of this quantification is first to determine the degree to which the insoluble ash fraction can be used to identify combustion zones that have disappeared at the macroscopic scale, and, second, to verify if it is possible to quantitatively reconstruct the use of ligneous fuel in terms of the volume of wood burned.

Materials and methods

Materials

In the context of combustion experiments realized over several years at the CEPAM laboratory (UMR 6130, CNRS) under the direction of Isabelle Théry-Parisot[1], the ashes, defined as the fraction under 500 µm, were systematically collected and weighed. It appears that ashes represent an extremely variable proportion of the residues depending on the species of burned wood, attaining up to 80 with oak%, for example (Thery-Parisot & Chabal, this volume).

The corpus studied corresponds to 16 fires burned with eight different taxa (two softwood and six hardwood trees): Aleppo pine (*Pinus halepensis*), maritime pine (*p pinaster*), pubescent oak (*quercus pubescens*), olive (*olea europaea*), hazelnut (*corylus avellana*), poplar (*populus sp*.), birch (*betula pendula*) and hornbeam (*carpinus betulus)*. For each taxon, two fires corresponding to wood originating from two different stations were taken into account and several parameters were recorded (Théry-Parisot & Chabal, this volume), including the volume of wood put into the fire, the mass of charcoal (fractions 0,5 – 4 mm and >4 mm) and the mass of ash (fraction <500 µm).

[1] – « Économie des combustibles au Paléolithique. De l'expérimentation à la modélisation » (« Economy of combustibles in the Paleolithic. From experimentation to modeling »): Funded by ACI-jeunes chercheurs, Ministry of Research 2000-2003 & APN, CNRS 2000-2003.

Methods

Extraction of the Acid Insoluble Fraction (AIF), density < 2,4

It was decided that the quantitative parameters that would allow an evaluation of the loss of material between the combustible and the ash residues would be the volume of wood put into the fire and the weight of the ashes. In effect, for wood, the volume is less influenced than the mass by variations of humidity, which is a parameter that is difficult to control. In contrast, since ash is more sensitive to packing, weighing it appeared to be the only way to quantify the residues of each combustion in a reproducible and comparable manner.

For each fire, around 1 gram of the ash fraction was collected and weighed at a precision of 0.1 mg. The ashes were then treated following the method proposed by R. M. Albert (Albert *et al*., 1999) with a hot mixture of nitric and hydrochloric acids, and then with hot hydrogen peroxide. These treatments allow us to destroy the carbonates, phosphates and organic material, thus concentrating only the particles that are less sensitive to dissolution, and to collect the *Acid Insoluble Fraction*, or AIF, as defined by R.M. Albert (Albert *et al*., 1999). In the sediments, this fraction is the chemically most stable; only very alkaline pH, which are rarely encountered, can affect its preservation.

However, if this extraction method is applied in this manner to the sediments, a large number of acid insoluble particles with a non vegetal origin will also be collected, in particular quartzes. Quartzes and phytoliths are thus separated based on their different densities. The density of phytoliths is less than 2.4, while quartzes are heavier; flotation on a liquid with a controlled density allows us to separate biogenic silica and geological silica (Fredlund & Tieszen, 1994). So that our experimental results could serve as a reference base in the analysis of archaeological sediments, we separated our samples in this manner, using a solution of sodium polytungstate (d = 2.4). This operation allowed us to collect that which will be designated below as “light AIF”.

Microscopic analysis of ash residues

The light AIF contains various formless or figured particles, identifiable or unidentifiable, including the phytoliths themselves.

The phytoliths are amorphous particles of opal silicon that form in living vegetal tissues. A differential production exists: the monocotyldons in general and the Gramineae in particular produce many more than the gymnosperms and dicotyldons (Carnelli *et al*., 2001). In addition, the silica is deposited more easily in the chlorophyllian and transpiration organs (green leaves and stems) than elsewhere in the plant and the identifiable forms often originate from the silicification of the epidermic cells. In the internal tissues, we find mainly granular forms, which might correspond to a neutralization of elements that are toxic to the vegetal organism (Lewin & Reimann, 1969; Jones & Handreck, 1965, Sangster & Hodson, 2001) and particles with a poorly defined morphology, probably originating form more diffuse silica deposits. The only “wood” phytoliths that provide taxonomic information are those with a spherical form and a rough or smooth surface. Most authors agree that these phytoliths are produced in the ligneous tissues of dicotyledons (Scurfield *et al*., 1974; Kondo *et al*., 1994; Alexandre *et al*., 1997). For pines, a rather variable morphotype appears to be characteristic; these are forms with an alveolated surface (Delhon, 2002, 2005). All the other particles are considered to be non classable as they resemble debris, partial silicifications of unidentifiable cells or siliceous aggregates, amalgams of phytoliths themselves and other mineral particles (fig. 1).

The observation of residues by transmission optic microscopy (magnification 400 X to 1000 X) allows evaluation of the proportion of phytoliths contained in the light AIF. This semi-quantitative evaluation is rather delicate and can be subjective. For this reason, a range of proportion was defined (for example: 1/2 to 1/3 of the particles present are phytoliths). The phytoliths belonging to an identifiable morphotype were then counted, as well as those with no standardized morphology. These categories correspond respectively to the “consistent morphology” and “variable morphology” of R. M. Albert (Albert *et al*., 1999).

Comparative data

To our knowledge, the only published data concerning the AIF content in wood ash are those presented by Schiegl and collaborators (1996) in a study with the objective of evaluating the volume of ash that contributed to the sedimentary deposits of Hayonim and Kebara Caves (Israel, Paleolithic). These data concern eight species, all sclerophylls, only one of which, olive, corresponds to our corpus. This study involved experimental combustions of 5 kg of dry wood. The mass and volume of ashes obtained and the proportion of the AIF were recorded. The volume of wood initially put into the fires is unknown. The relationship between the mass and volume strongly depends on the species and water content and it is not possible to pass from one to the other given these unknown factors. On the other hand, data are available concerning the weight of the ashes. The analyses conducted by I. Théry-Parisot show that for most species, there is a regression between the weight of ash obtained and the initial volume of wood (Thery-Parisot, personal communication). Though it is imperfect, this correlation can used to evaluate the volume of wood put into the fires in the experiments by S. Schiegl.

Results

Quantity of AIF

Experimental results

Following the acid treatments, the quantity of residues obtained is rather variable, with an average of 19.2 mg for 1g of treated ash, or nearly 2% of the weight (tab. 1, fig. 2a). Even if we exclude the particularly high value obtained for one of the combustions with Maritime Pine (78.8 mg/g), which corresponds to a sample very

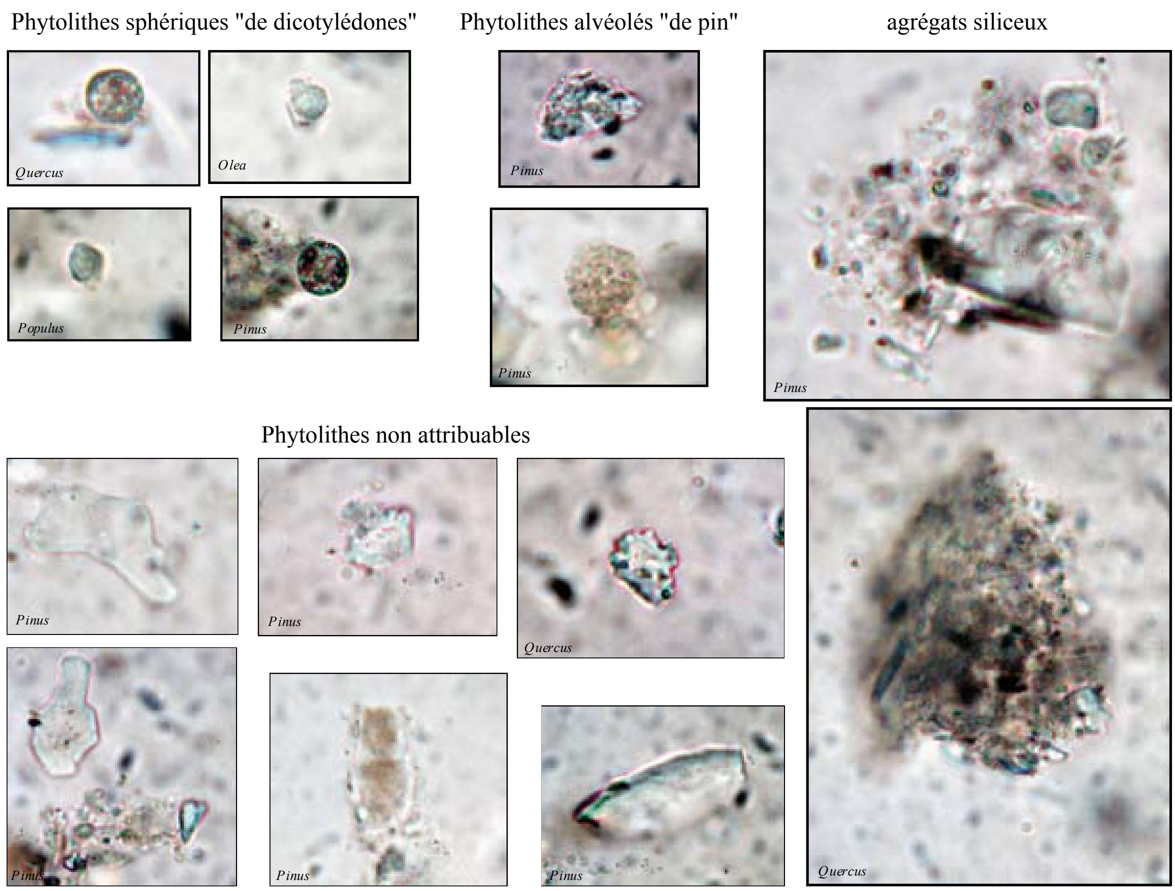

Fig. 1 - Siliceous particles observed in the wood ashes (transmission optic microscopy, magnification 1000 X) (C. Delhon).

	volume de bois mis au feu (1)	masse totale de cendres <0,5 mm (1)	masse de cendre traitée (1)	masse AIF (1)	masse AIF légère (1)	proportion de phytolithes dans l'AIF légère (2)		masse AIF par g de cendre (3)	masse AIF par cm³ de bois (3)	masse AIF légère par g de cendre (3)	masse AIF légère par cm³ de bois (3)	masse phytolithes par g de cendre (3)		masse phytolithes par cm³ de bois (3)	
	cm³	g	g	mg	mg	proportion		mg/g	mg/cm³	mg	mg	mg/g		mg/cm³	
						estimation basse	estimation haute					estimation basse	estimation haute	estimation basse	estimation haute
Quercus pubescens I	6502	179,50	0,9983	23,0	13,0	1/3	2/3	**23,0**	**0,636**	**13,0**	**0,360**	**4,3**	**8,7**	**0,120**	**0,240**
Quercus pubescens II	3482	105,69	0,8777	5,0	1,7	2/3	3/4	**5,7**	**0,173**	**1,9**	**0,059**	**1,3**	**1,5**	**0,039**	**0,044**
Pinus pinaster I	7544	15,46	0,8165	64,3	28,6	1/4	1/3	**78,8**	**0,161**	**35,0**	**0,072**	**8,8**	**11,7**	**0,018**	**0,024**
Pinus pinaster II	5559	31,31	0,9532	9,7	4,1	1/3	3/4	**10,2**	**0,057**	**4,3**	**0,024**	**2,9**	**3,2**	**0,016**	**0,018**
Olea europaea I	4207	35,70	0,9605	7,1	6,6	1/3	1/2	**7,4**	**0,063**	**6,9**	**0,058**	**2,3**	**3,4**	**0,019**	**0,029**
Olea europaea II	5324	42,50	0,9295	5,9	2,6	1/3	1/2	**6,3**	**0,051**	**2,8**	**0,022**	**0,9**	**1,4**	**0,007**	**0,011**
Pinus halepensis I	6107	36,58	1,0402	13,5	6,2	2/3	3/4	**13,0**	**0,078**	**6,0**	**0,036**	**4,0**	**4,5**	**0,024**	**0,027**
Pinus halepensis II	6199	40,00	0,954	10,7	7,3	2/3	3/4	**11,2**	**0,072**	**7,7**	**0,049**	**5,1**	**5,7**	**0,033**	**0,037**
Populus sp. I	7535	48,24	0,9176	26,1	11,0	2/3	3/4	**28,4**	**0,182**	**12,0**	**0,077**	**8,0**	**9,0**	**0,051**	**0,058**
Populus sp. II	7532	39,04	1,0179	16,4	8,6	2/3	3/4	**16,1**	**0,083**	**8,4**	**0,044**	**5,6**	**6,3**	**0,029**	**0,033**
Carpinus betulus I	8877	45,28	0,8641	16,5	4,8	1/3	1/2	**19,1**	**0,097**	**5,6**	**0,028**	**1,9**	**2,8**	**0,009**	**0,014**
Carpinus betulus II	9029	50,15	0,9164	14,2	5,7	1/2	2/3	**15,5**	**0,086**	**6,2**	**0,035**	**3,1**	**4,1**	**0,017**	**0,023**
Betula pendula I	8297	20,56	0,9244	32,1	11,1	1/3	1/2	**34,7**	**0,086**	**12,0**	**0,030**	**4,0**	**6,0**	**0,010**	**0,015**
Betula pendula II	18419	48,02	1,0119	23,6	13,7	1/4	1/3	**23,3**	**0,061**	**13,5**	**0,035**	**3,4**	**4,5**	**0,009**	**0,012**
Corylus avellana I	7920	50,00	0,9361	5,6	33,6	2/3	3/4	**6,0**	**0,038**	X	X	X	X	X	X
Corylus avellana II	7012	44,00	0,9242	8,3	6,0	2/3	3/4	**9,0**	**0,056**	**6,5**	**0,041**	**4,3**	**4,9**	**0,027**	**0,031**
MOYENNE								**19,2**	**0,124**	**9,5**	**0,065**	**4**	**5,1**	**0,029**	**0,041**
ECART-TYPE								**18,0**	**0,144**	**7,9**	**0,083**	**2,2**	**2,9**	**0,028**	**0,056**

Tab. 1 - Absolute and relative content of wood ash in insoluble residues. 1/direct measures (weights), 2/semi-quantitative estimation, 3/indirect data (calculations based on the preceding). Gray: measuring error, X missing data (C. Delhon).

rich in bark, the range of variation extends from around 6 mg to 35 mg of ash, with an intraspecific variation that can be high (oak: 5.7 – 23 mg/g; poplar: 16.1 – 28.4 mg/g; birch: 23.3 – 34.7 mg/g). The combustion conditions, which are comparable, do not seem to be the cause of these variations, but this must be verified since it is difficult to control the parameters that influence combustions in open-air fireplaces.

Since the combustion of different species does not produce the same quantity of ash, it is necessary to refer not to the weight of the ashes but to the volume of wood put into the fire (fig. 2b). The values are much less dispersed in this case and it appears that one cubic centimeter of wood produces around 0.1 mg of AIF (average of 0.124 mg/cm^3).

Comparison with the data of Schiegl *et al.*, 1996

Despite the heterogeneity of the corpus, the results converge with an average ash AIF content very close to 2% (1.9 versus 1.92 in our study) (tab. 2 fig. 2a). Two species, oak and olive, were measured twice, confirming the intraspecific variability that we observed in our corpus (Olive: 1 – 3.6 %; pubescent oak: 1.1 – 2.7 %). Moreover, for the taxon analyzed in both studies (Olive), the lowest value obtained by Schiegl *et al.*, 1%, is relatively close to ours (which are coherent: v 6.3 and 7.4 mg/g, or 0.63 and 0.74%), but the other one, 3.6, is clearly different.

Referring to the volume of wood put into the fire (fig. 2b), even if it is an extrapolation in this case, also permits a reduction of the differences between individuals and species and to calculate an average that is coherent with our results of approximately 0.1 mg of insoluble residues per cm^3 of wood (0.097 mg/cm^3 versus 0.124 mg/cm^3 in our study).

Quantity of light AIF per volume of wood

The process of extracting phytoliths from the sediment requires us to work with only the light AIF fraction (d<2.4) in order to obtain a reference base that can be used in archaeological contexts. This light fraction globally represents a little more than half of the total AIF (tab. 1, fig. 2c), meaning an average of a little less than 1% of the weight of the ashes (9.5 mg/g). One value clearly diverges from this average (35.9 mg/g), but it corresponds to a recording error (hazelnut I: the "light" AIF fraction was measured in a greater quantity than the total AIF) and will no longer be taken into account. Referred to the volume of wood (fig. 2d), the average is 0.065 mg/cm^3, but the results are far from homogeneous. While the total AIF content for Maritime Pine could appear aberrant, its light AIF content is close to that of other samples (0.0718 mg/cm^3). On the contrary, one of the values obtained for pubescent oak (0.3595 mg/cm^3) is really above the average.

Once the insoluble fraction with a density lower than 2.4 is extracted, it is possible to identify the phytoliths through a microscopic analysis and thus maybe to interpret the assemblage as originating from wood ash. In our experimental samples, the proportion of phytoliths on the slides, evaluated in a semi-quantitative manner, varies between "1/4 to 1/3" and "2/3 to 3/4" of the particles observed (tab. 1). On figure 3, there are thus 2 points for each slide, representing the upper and lower limits of the proposed range. We thus estimate the average quantity of phytoliths in one gram of wood ash to be between 4 and 5.1, which represents approximately 0.5% of the mass.

In relation to the volume (fig. 3b), the average quantity of phytoliths is not higher than 0.04 mg per cm^3 (low value average: 0.03 mg/cm^3; high value average: 0.04 mg/cm^3).

Phytolithic analysis of the light AIF

The phytolithic analysis of the experimental ashes shows that the great majority of the amorphous silica of wood has no characteristic form (tab. 3). Only a minority of forms can be attributed to dicotyledons or pine (always below 20% of the particles observed), but the values are relatively stable. Two groups are identifiable among the dicotyledons. The first, which is richer in identifiable phytoliths than the second, includes oak and olive (an average of 14.7% of the identifiable phytoliths), while the second group includes all the other species (an average of 3.7% of the identifiable phytoliths). It is possible that the growth rate (slow for the first group, rapid for the second)

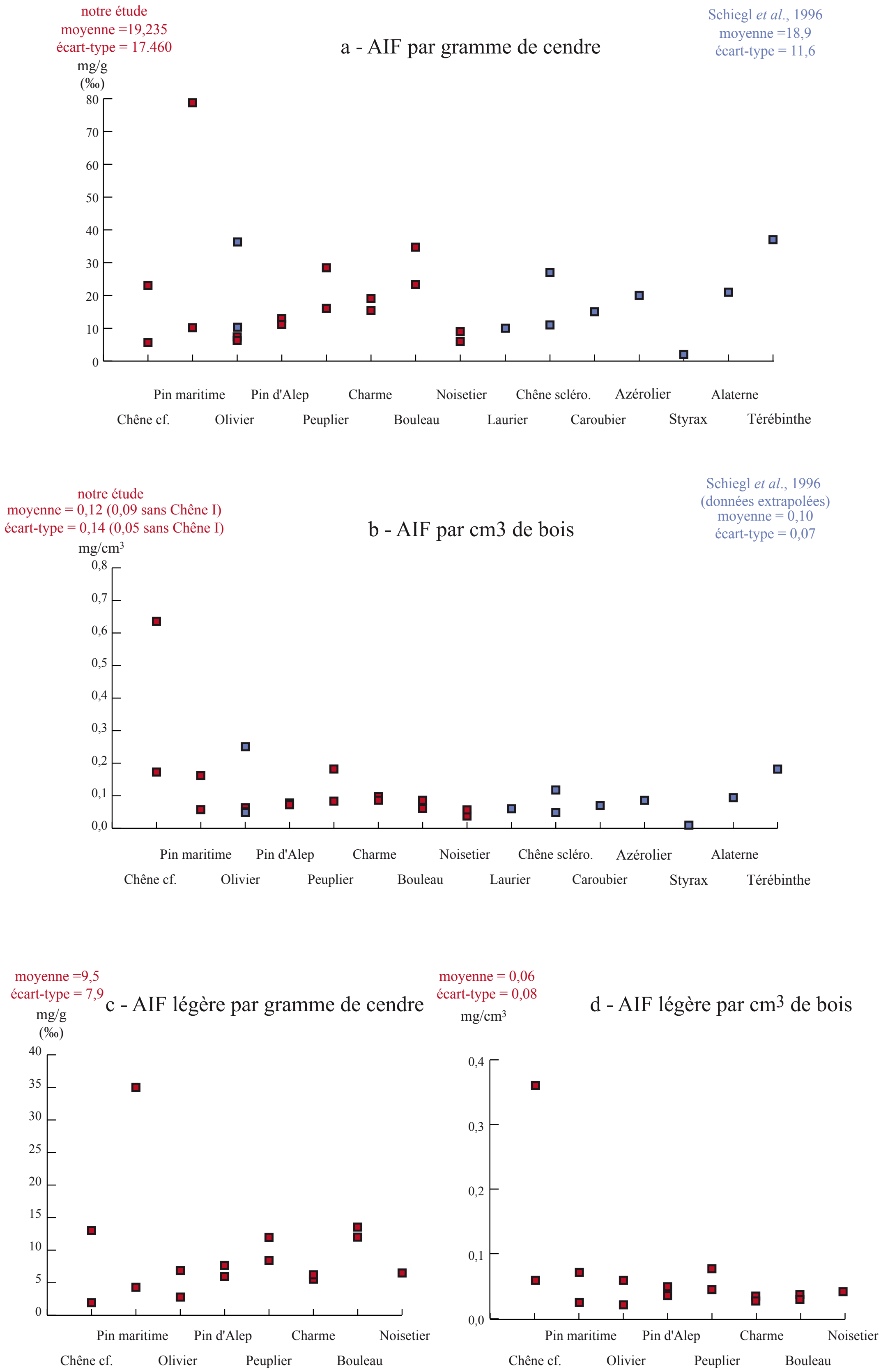

Fig. 2 - AIF (2a & 2b) light AIF (2c & 2d) content correlated with the mass of ash obtained (2a & 2c) and the volume of wood pout into the fire (2b & 2d) (C. Delhon).

	p = poids de cendres (1)	v = volume de bois (2)	proportion d'AIF dans la cendre (1)	AIF / cm^3 de bois
	g	cm^3	% du poids	mg/cm^3
Olea europaea	45	6455,75	**3,6**	**0,251**
Olea europaea	112	23205,75	**1,0**	**0,048**
Laurus nobilis	57	9455,75	**1,0**	**0,060**
Quercus calliprinos	187	41955,75	**1,1**	**0,049**
Quercus calliprinos	226	51705,75	**2,7**	**0,118**
Ceratonia silica	134	28705,75	**1,5**	**0,070**
Crataegus azarolus	268	62205,75	**2,0**	**0,086**
Styrax officinalis	99	19955,75	**0,2**	**0,010**
Rhamnus palaestinus	174	38705,75	**2,1**	**0,094**
Pistacia palaestina	102	20705,75	**3,7**	**0,182**
MOYENNE			**1,9**	**0,097**
ECART-TYPE			**1,2**	**0,071**

Tab. 2 - absolute and relative content of wood in the study by Schielg *et al.* (1996) in insoluble residues. 1/data of Schiegl *et al.*, 1996, table 2b, 2/extrapolation based on the data of de Schiegl *et al.*, 1996, table 2b and according to the formula: v = (p-19.77)/0.004 (Théry-Parisot, personal communication).

	morphotypes (%)				
	non attribués	attribués aux dicotylédones		attribués au pin	
	informes	sphériques rugueux	sphériques lisses	alvéolés	effectifs
Quercus pubescens I	83,3	10,8	5,9	0,0	204
Quercus pubescens II	83,4	14,2	2,4	0,0	211
Pinus pinaster I	86,9	1,9	0,5	10,7	206
Pinus pinaster II	81,2	3,8	2,3	12,7	213
Olea europaea I	86,8	12,3	0,9	0,0	212
Olea europaea II	87,5	11,1	1,4	0,0	216
Pinus halepensis I	83,3	2,7	1,4	12,7	221
Pinus halepensis II	80,9	4,3	2,4	12,4	209
Populus sp I.	96,8	3,2	0,0	0,0	220
Populus sp. II	96,1	3,9	0,0	0,0	205
Carpinus betulus I	96,3	2,8	0,9	0,0	216
Carpinus betulus II	96,9	3,1	0,0	0,0	227
Betula pendula I	94,5	5,0	0,5	0,0	201
Betula pendula II	95,1	3,9	1,0	0,0	205
Corylus avellana I	97,4	2,6	0,0	0,0	190
Corylus avellana II	97,1	2,4	0,5	0,0	208

Tout le corpus	**90,2**	**6,8**	X	**MOYENNE**
	6,5	**5,0**		**ECART-TYPE**
Pins	**83,1**	**4,8**	**12,1**	**MOYENNE**
	2,8	**2,0**	**1,0**	**ECART-TYPE**
Dicotylédones	**92,6**	**7,4**	X	**MOYENNE**
	5,6	**5,6**		**ECART-TYPE**
Quercus & Olea	**85,3**	**14,7**	X	**MOYENNE**
	2.2	**2,2**		**ECART-TYPE**
Autres dicot.	**96,3**	**3,7**	X	**MOYENNE**
	1,0	**1,0**		**ECART-TYPE**

Tab. 3 - Results of the phytolithic analysis of ash (C. Delhon).

and the life duration (long for the first group, shorter for the second) are at the origin of these differences, the accumulation of phytoliths in perennial organs being a progressive process. It is not possible to make more precise determinations based on the morphology of wood phytoliths. We can note that in the pine ashes we find up to 6.7% of the forms commonly attributed to dicotyledones, for approximately 12% of forms characteristic of pine, which can falsify the interpretation of fossil when we do not know if the assemblage is mono- or multi-specific. It appears, indeed, that pines can contain more dicotyledone-type phytoliths (average 4.8%) than the less productive hardwoods (only 3.7% in average).

Discussion

The AIF and phytoliths as quantitative records of fire?

Though the quantity of ash produced by the combustion of a given volume of wood can be highly variable (Théry-Parisot & Chabal, this volume), this variation is mainly explained by the proportion of soluble particles (carbonates). The insoluble fraction (AIF), relative to the volume of wood put into the fire, is much more constant (fig. 2). There is a linear regression between these two variables (fig. 4-a), allows us to determine the volume of wood put into the fire when we know the quantity of AIF.. This regression is nonetheless difficult to apply in archaeological contexts since the sediment itself contains insoluble particles that will enrich the AIF and distort the extrapolation. It is thus necessary to work with the light AIF, which concentrates particles of a vegetal origin and , more specifically, with phytoliths, which in AIF we are capable of identifying as truly being of a vegetal origin. Unfortunately, when we focus on these AIF fractions, it is no longer possible to propose a regression (fig. 4-b and c).

It is therefore impossible to propose a transfer function that would allow an evaluation of the volume of wood burned based on the quantity of the light AIF or the phytoliths contained in the sediments. We can, however,

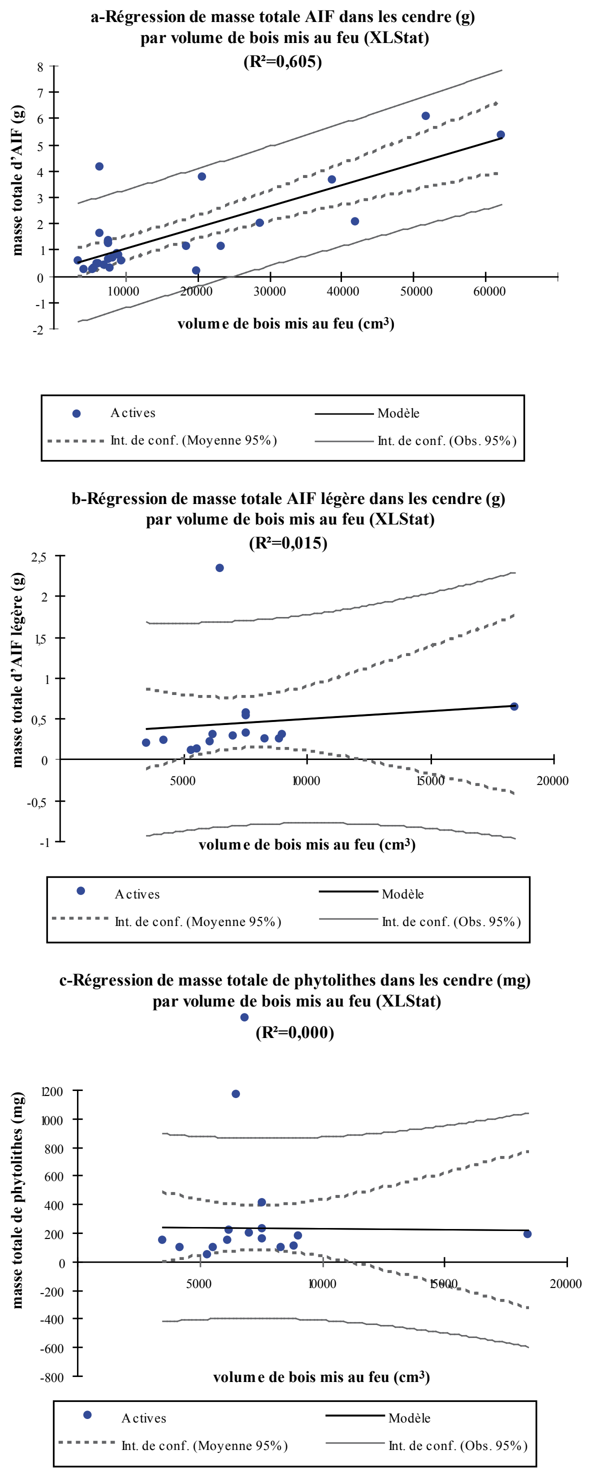

Fig. 4 - Regression curves of the residues (4a-AIF, 4b-light AIF, 4c-phytoliths) for the volume of wood put into the fire. Only the first one (total AIF mass per volume of wood put into the fire) shows a correlation between the variables (C. Delhon).

evaluate the average quantities of these weakly soluble residues produced by combustion activities. For each experiment, we calculated the quantities of AIF, light AIF and phytoliths produced by a "standard" fire (fig. 5) composed of 6 logs of 1500 cm^3 (approximately 8 cm diameter and 30 cm long). This quantity of wood produces flames for 4 to 12 hours depending on the species and combustion conditions. This type of fire produces approximately 1 gram (average 1.1 gram) of acid insoluble residues, which can be preserved in sediments over a long period, of which 60% (average 0.6 grams) have a density lower than 2.4 and can be collected using the phytolith extraction techniques (fig. 5-a). Only a part of them (0.3 to 0.4 grams) can be identified as "phytoliths" (fig. 5-b). It thus appears that a fire with 6 logs would add to the sediment only one half of a gram of microscopic particles indicative of the contribution of wood.

Identification and interpretation of the phytolithic spectra of "wood"

The phytolithic analysis of combustion residues shows that the great majority of particles has no diagnostic value for the type of wood burned. On the contrary, some interpretations could be distorted by the ubiquity of forms usually attributed to dicotyledons and that we found in the gymnosperms (fig. 6). In the phytolithic assemblages obtained, we also note the absence of "silicified tracheid" a morphotype frequently attributed to wood (Piperno, 2006), and which have a vessel morphology with ring or spiral ornamentations. It has not been proven that these forms correspond to hardwood vessels or conifer tracheides. Their morphology also suggests that they could be elements of silicified phloem, which would not exclude their production by monocotyledons. This form is rarely, but not exceptionally, found in archaeological phytolithic assemblages. Their total absence from the sixteen assemblages observed in this study tends to prove that they are not produced in "wood" tissue.

The recognition of "burned" phytoliths is also important for the identification of combustion zones. Our observations lead us to conclude that phytoliths extracted from ash have no specific characteristic indicative of their exposition to high heat: burned phytoliths, once extracted from the ashes, have the same appearance as non burned phytoliths. In other words, it

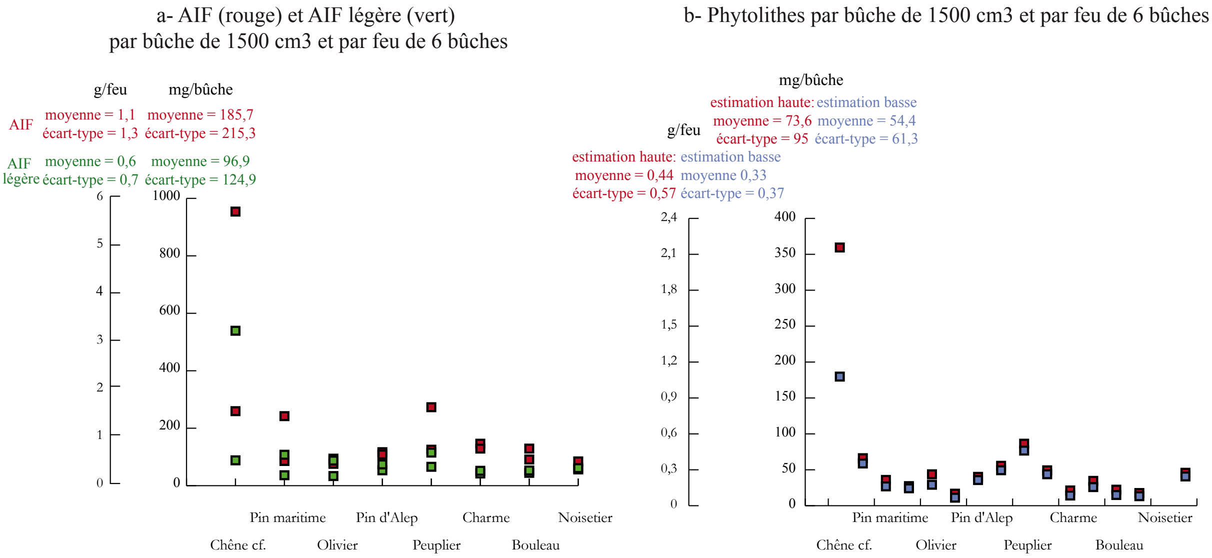

Fig. 5 - Quantity of AIF, light AIF (5a) and phytoliths (5b) per "standard" log of 1500 cm3 and per fire with 6 logs (C. Delhon).

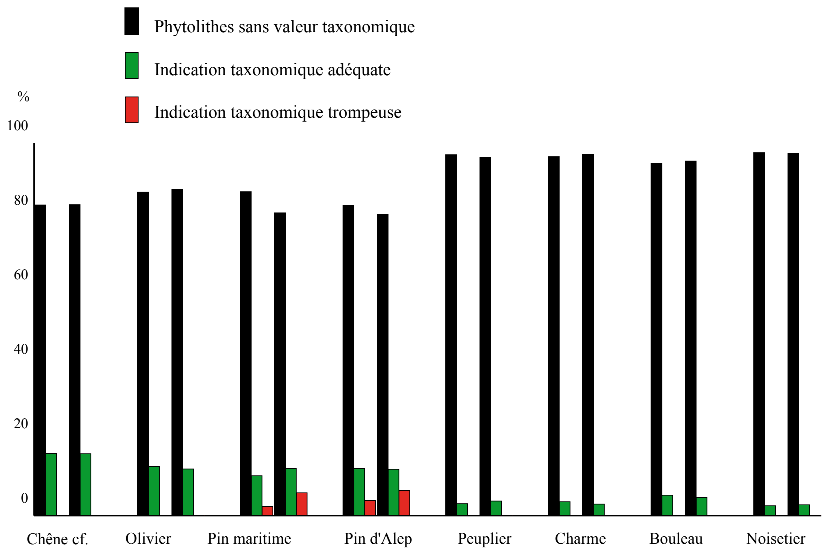

Fig. 6 - Results of the phytolithic analysis of ash: relative proportions of phytoliths to which no taxonomic value is attributed (black), phytoliths to which an adequate taxonomic value is attributed (green) and phytoliths to which an inadequate taxonomic value is attributed (red), for each studied species (C. Delhon).

is not possible to attribute a fossil assemblage to fire residues based on the appearance of the phytoliths alone. Our observations counter the common idea that burned phytoliths are black or brown (Kealhofer & Penny, 1998; Parr & Carter, 2003; Piperno & Jones, 2003; Piperno, 2006), which is perhaps related to the abusive incorporation of certain micro charcoals in the category of "phytoliths". It is probable that certain reactions, related for example, to pedogenetic mechanisms, are more likely the cause of blackish organic deposits on the surface of phytoliths. In most cases, however, the extraction of phytoliths through the use of several acids should result in the disappearance of this type of deposit.

Conclusion

Ash is a highly soluble substance, most of which (around 98%) rapidly disappears into sediments by dissolution. Though the fraction that can be preserved over the long term appears, in contrast, to be very stable and is produced in proportions that vary only slightly from one species to another, it represents only a minute proportion of the wood (approximately 0.1 mg per cm^3 of wood). Due to the potential compaction of ash, intra and interspecific variations and the difficulty of isolating vegetal particles from the sediment, it is impossible to make quantitative reconstructions of the biomass burned in a fire.

The great majority of phytoliths contained in wood are formless. The morphotypes commonly attributed to the wood of dicotyledons or pine represent only a small portion of the biogenic silica. It is thus impossible to identify these taxa, even if the presence of wood is detectable, since the phytolithic spectra of other tissues (leaves) is different (presence of epidermic cells and silicified stomata). In the past, the formless particles were used to detect the presence of wood, particularly by R. M. Albert under the name of "variable morphology". Though this method is reliable when phytoliths are present in large quantities, we must consider i) the difficulty of identifying these particles, defined as having an unidentifiable form, ii) their production, even if in lower proportions, by all vegetal tissues, from ligneous to herbaceous, including gramineae, and iii) the fact that, on one hand, the particles most sensitive to dissolution belong to this category, and, on the other, re-precipitations of silica with sediment can create secondary siliceous aggregates that also belong to this category.

It is thus clear that due to the low production of phytoliths by ligneous tissues only large accumulations of wood ash are detectable through phytolithic analysis.

In addition, it is impossible to distinguish phytoliths liberated from vegetal materials following combustion from those liberated following a slow decomposition. The presence of wood phytoliths thus directly signals the presence of wood, but only indirectly that of fire. When preserved charcoal is lacking, it appears that only physical-chemical analyses of sediments can determine the presence of ash. Phytoliths can then be one element within a cluster of indices: a high concentration of wood phytoliths would confirm the presence of a wood derivative already having a concentration of phytoliths. The decomposition of a simple branch *in situ* does not appear sufficient to significantly enrich the wood phytolith content of the sediment and ashes are therefore the most probable component with a high concentration of phytoliths in most contexts. Nonetheless, we must not forget that other organic materials, such as dung, have high phytolith concentrations and can be accumulated in archaeological contexts (Delhon *et al.*, 2008).

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References

Albert R. M., Tsatskin A., Ronen A. & Lev-Yadun S. 1999 - Mode of occupation of Tabun cave, Mt Carmel, Israel during the Mousterian period : a study of the sediments and phytoliths. *Journal of Archaeological Science,* 26 : 1249-1260.

Albert R. M., Weiner S., Bar-Yosef O. & Meignen L. 2000 - Phytoliths in the middle Palaeolithic deposits of Kebara Cave, Mt. Carmel, Israel : study of the plant material used for fuel and other purposes. *Journal of Archaeological Science*, 27 : 931-947.

Alexandre A., Meunier J. -D., Lézine A. -M., Vincens A. & Schwartz D. 1997 - Phytoliths : indicators of grassland dynamics during the late Holocene in intertropical Africa. *Palaeogeography, Palaeoclimatology, Palaeoecology,* 136 : 213-229.

Carnelli A. L., Madella M. & Theurillat J. -P. 2001 - Biogenic silica production in selected alpine plant species and plant communities. *Annals of Botany*, 87 : 425-434.

Chabal L., Fabre L., Terral J. -F. & Théry-Parisot I. 1999 - L'anthracologie. *In* : *La botanique*. Collection « Archéologiques », Paris, Errance : 43-104.

Delhon C. 2002 - Phytolith and pedoanthracological analysis of an Holocene pedosedimentary sequence from the middle Rhone valley (France). *In* : *Charcoal analysis, methodological approaches, palaeoecological results and wood-uses*, Thiébault S. (ed). BAR international series : 169-178.

Delhon C. 2005 - *Anthropisation et paléoclimats du Tardiglaciaire à l'Holocène en moyenne vallée du Rhône : études pluridisciplinaires des spectres phytolithiques et pédo-anthracologiques de séquences naturelles et de sites archéologiques.* Thèse de doctorat, Université Paris I, 843 pp.

Fredlund G. & Tieszen L. 1994 - Modern phytolith assemblages from the North American Great Plains. *Journal of Biogeography,* 21 : 321-335.

Jones L. & Handreck K. 1965 - Study of silica in the oat plant – II. Uptake of silica from soils by the plant. *Plant and soil,* 23 : 79-96.

Kealhofer L. & Penny D. 1998 - A combined pollen and phytolith record for fourteen thousand years of vegetation change in northeastern Thailand. *Review of Palaeobotany and Palynology,* 103 : 83-93.

Kondo R., Chils C. & Alkinson I. 1994 - *Opal phytoliths of New Zealand*. Manaaki Whenua Press, Lincoln, 85 pp.

Lewin J. & Reimann B. 1969 - Silicon and plant growth. *American Review of Plant Physiology,* 20 : 289-304.

Olanders B. & Steenari B.-M. 1995 - Characterization of ashes from wood and straw. *Biomass and Bioenergy*, 8 : 105-115.

Parr J. F. & Carter M. 2003 - Phytolith and starch analysis of sediment samples from two archaeological sites on Dauar Island, Torres Strait, Northeastern Australia. *Vegetation History and Archaeobotany,* 12 : 131-141.

Piperno D. 2006 - *Phytoliths. A comprehensive guide for archaeologists and palaeoecologists*. Landham, Altamira, 238 pp.

Piperno D. & Jones J. 2003 - Palaeoecological and Archaeological implications of a late Pleistocene/Early Holocene record of vegetation and climate from the pacific costal plain of Panama. *Quaternary Research,* 59 : 79-87.

Sangster A. & Hodson M. 2001 - Silicon and aluminium codeposition in the cell wall phytoliths of gymnosperms leaves. *In* : *Phytoliths, applications in Earth Science and Human History,* Meunier J. -D. & Colin F. (eds). Lisse, A. A. Balkema : 343-355.

Schiegl S., Lev-Yadun S., Bar-Yosef O., El Goresy A. & Weiner S. 1994 - Siliceous aggregates from prehistoric wood ash : A major component of sediments in Kebara and Hyonim caves (Israel). *Israel Journal of Earth Sciences*, 43 : 267-278.

Schiegl S., Goldberg P., Bar-Yosef O. & Weiner S. 1996 - Ash deposits in Hayonnim and Kebara caves, Israel : macroscopic, microscopic and mineralogical observations, and their archaeological implications. *Journal of Archaeological Science*, 23 : 763-781.

Scurfield G., Anderson C. & Segnit E. 1974 - Silica in woody stems. *Australian Journal of Botany,* 22 : 211-229.

Wattez J. 1998 - Contribution à la connaissance des foyers préhistoriques par l'étude des cendres. *Bulletin de la Société préhistorique française*, 85 (10-12) : 352-366.

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FROM MICROCHARCOAL TO MACROCHARCOAL: RECONSTRUCTION OF THE "WOOD CHARCOAL" SIGNATURE IN PALEOLITHIC ARCHAEOLOGICAL CONTEXTS

Laurent MARQUER

Abstract

The wood charcoal recovered during archaeological excavations represents only a partial image of the anthracological materials initially produced by human activities. Once buried, these objects are subject to diverse post-depositional processes that fragment them. While macrocharcoals (>500 µm) can be collected one by one and recorded within a coordinate system, or can be extracted by flotation and sieving during excavation, smaller fragments, such as "mesocharcoals" (500-160 µm) and micro charcoals (<160 µm), can be isolated from the sediments only through adapted procedures. A method of extraction and quantification through image analysis has thus been developed in order to record and evaluate the significance of the elements present in the finest sedimentary fractions. Such analyses have been applied in a Paleolithic context at the Magdalenian site of Grand Abri on the co*teau de La Garenne* (La Garenne hillside) (Saint-Marcel, Indre, France). This work shows that the quantity of charcoal found in the very fine sedimentary fractions (500-160 µm and <160 µm) is greater than that of the macrocharcoals. These quantifications allow us to reconstruct a "charcoal signature" from the macroscopic to the microscopic scale, and thus to identify, in situ, the smallest charcoal fractions resulting from the taphonomic processes that modified the archaeological site.

Keywords : charcoal, microcharcoal, taphonomy, Paleolithic, coteau de La Garenne (La Garenne hillside)

Introduction

The remains of combustion activities in Paleolithic contexts consist essentially of wood charcoal, burned bones, burned stones and ash. They originate from the remains of fireplaces that are more or less structured or dispersed with an archaeological level. In many Upper Paleolithic sites, wood charcoal absent or scarce, while other artifacts resulting from combustion, especially burned bones, are often abundant (Perlès, 1977; Théry-Parisot, 1998; 2001; Costamagno *et al.*, 1999; Théry-Parisot, 2002a; 2002b; Villa *et al.*, 2002; Théry-Parisot *et al.*, 2005; Yravedra *et al.*, 2005). Some believe that the high quantity of burned bone in fireplaces, compared to wood charcoal, could be explained by a limited collection of wood due to the low arboreous biomass during the last glacial period. The presence of charcoal in prehistoric contexts spanning the last 40,000 years has nonetheless been recorded in diverse cultural and bioclimatic contexts in Europe from the end of the Middle Paleolithic to the Final Paleolithic. Consequently, even if paleoenvironmental evolutions during the last glacial period in Europe (de Beaulieu & Reille, 1984; 1992a; 1992b; Reille & de Beaulieu, 1988; 1990; Guiter *et al.*, 2003; Beaudouin *et al.*, 2007; Naughton *et al.*, 2007) may have influenced the economy of combustibles, they cannot alone explain the absence or near absence of charcoal. The use of bone in fireplaces for economic, cultural and/or energetic reasons was then supposed (Théry-Parisot, 1998; 2001; Théry-Parisot, 2002a; 2002b; Villa *et al.*, 2002; Théry-Parisot & Costamagno, 2005; Théry-Parisot *et al.*, 2005).

Since wood is necessary to light a fire (Costamagno *et al.*, 2005; Théry-Parisot *et al.*, 2005), its combustion residues should be present in prehistoric occupation levels. Archaeological excavations only rarely consider the wood charcoal present in sediments finer than one millimeter. The smallest charcoal fragments produced on the surface of wood (or herbaceous plants) during combustion, then secondarily following the refragmentation of the largest particles, are thus not recorded. It is thus possible that some information concerning the presence of charcoal is lost.

Diverse quantification methods have been developed for the microparticles of burned vegetal materials, most of which are undeterminable, and are included in the category of microcharcoals. These quantifications were multiplied and perfected at the end of the 1980’s in the context of quaternary sediments (Clark, 1984; Patterson *et al.*, 1987; Clark, 1988; McDonald *et al.*, 1991; Clark & Hussey, 1996; Rhodes, 1998; Carcaillet *et al.*, 2001; Vannière, 2001; Thevenon, 2003; Carcaillet, 2007; Daniau, 2008). Based on these analytical approaches, I have developed a study protocol for fireplace residues, from microscopy to macroscopy, in order to evaluate the potential loss of two combustion signatures (charcoal signature and burned bone signature) in the finest sediment fractions.

Methodological approach

Extraction procedure for macro- meso- microcharcoals

Several samples are collected depending on their presence in the sediment and the archaeological feature studied. They are collected throughout the feature in order to obtain the best representivity possible. One sample corresponds to a 100 to 500 cm^3 volume of sediment. The sample is then treated in a sieving column (fig. 1). Water sieving was favored over dry sieving because by diffusing water homogeneously and at a very low pressure over the sediment it is possible to remove the other micro-particles that can be deposited on the macro-, meso- and microcharcoals, which can hinder observations during optical analyses. For sediments with a high clay content, a deflocculation agent can be used (sodium hexametaphosphate: $Na_6O_{18}P_6$). Sieving meshes of 500 µm and 160 µm were employed in order to extract the charcoal fragments contained in the macroscopic (>500 µm: lowest limit possible for taxonomic identification in most cases), “mesoscopic” (500-160 µm) and microscopic (<160 µm: starting limit at which charcoal micro-particles are analyzed on palynological slides) sedimentary fractions (Hounslow & Chepstow-Lusty, 2002; 2004; Marquer *et al.*, 2008a; Marquer, 2009). In these three fractions, burned bone fragments are found along with the charcoal

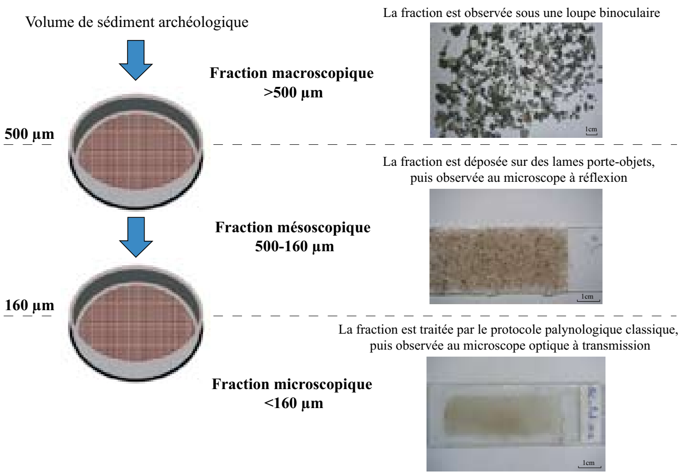

Fig. 1 - Extraction of the three sedimentary fractions: macroscopic (> 500 µm), mesoscopic (500-160 µm) and microscopic (<160 µm).

fragments. They were thus analyzed in the same manner as the charcoals to allow a direct comparison of the data concerning the relative presence of these two types of combustion remains.

The sediment of the macroscopic fraction was conserved in a Petri dish before being sorted with the aid of a binocular magnifier. The mesoscopic fraction was successively quartered and a final volume of sediment retained. This volume was placed on a microscope slide that was specially adapted for analysis by reflected light microscopy. Finally, a volume of the microscopic sediment fraction was treated using the classic palynological procedure (Cour, 1974; Faegri & Iversen, 1989) in order to eliminate the diverse mineral and organic fractions of the sediment that can hinder microscopic observations (Asselin & Payette, 2005). However, the chemical treatment used can bias the charcoal/burned bone proportions as the acids may partially dissolve the microfragments of burned bones. This is particularly true of hydrochloric acid, which attacks the carbonated elements (Marquer *et al.*, in press). Consequently, on the microscopic slides, only the quantifications of microcharcoals are considered.

Discrimination by microscopy

Different procedures were carried out with each of the sediment fractions in order to clearly discriminate the black burned bone fragments from the charcoal particles (Stiner *et al.*, 1995; Cain, 2005). To define the main criteria of identification, experimental macroscopic samples of wood and bone were submitted to diverse combustion temperatures, and then crushed and observed in each of the sedimentary fractions.

The macrocharcoals (>500 µm)

Charcoal and burned bone fragments are discriminated based on multiple criteria observed with a binocular magnifier: colorimetric (black for charcoal and diverse gradients of color for burned bone), morphological/anatomical (with observations of vegetal fibers for charcoal and cancellous elements for the bone tissues) and textural (fragment density).

The macrocharcoals possess three observable planes of wood anatomy and are thus potentially determinable at the species, gender or family level. The precision of the identification depends on the taxon and the condition of the fragment.

The "mesocharcoals" (500-160 µm)

The mesoscopic fraction is observed with the aid of a reflected light microscope in order to discriminate the charcoal from burned bone based on differences in the reflection of their superficial structures, which allows identification of the ligneous or herbaceous vegetal fibers (fig. 2).

These vegetal meso-particles have two to three anatomical planes, in certain cases allowing determinations that generally remain imprecise (Monocotyledons, Dicotyledons or Gymnosperms).

The microcharcoals <160 µm

The microcharcoals present in the microscopic fraction (<160 µm) are observed with an optical transmission microscope. The minimal dimension for the discrimination of vegetal micro-particles retained in our analyses is approximately 20 µm. These micro-elements are produced during combustion and then secondarily by the refragmentation of macrocharcoals and mesocharcoals. Based on their shape and structure,

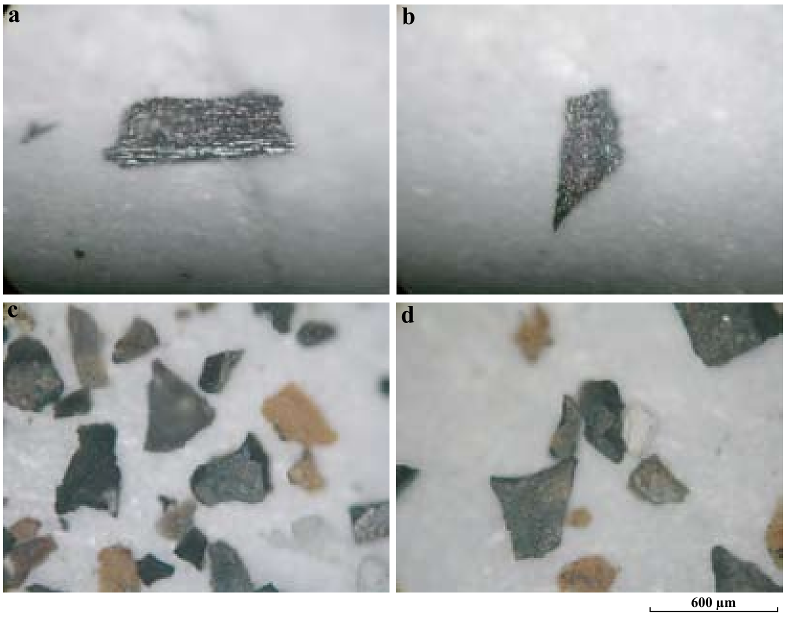

Fig. 2 - Mesoscopic fraction (500-160 µm) observed with a reflected light microscope: fragments of charcoal (a, b) and burned bone (c, d).

observed. These latter may correspond to longitudinal fragmentations with no visible characteristic vegetal structures. In addition, these micro-particles are generally opaque in transmitted light due to the thickness of the fragments, and probably to the degree of combustion as well.

The discrimination of vegetal micro-particles in this microscopic fraction is thus realized based on optical density and morphological criteria: black color, opaque, angular form, sometimes with the presence of well defined vegetal cells (Patterson *et al.*, 1987; Clark, 1988).

it is sometimes possible to identify cellular elements in one of the anatomical planes of wood or other vegetal organs (fig. 3; fig. 4).

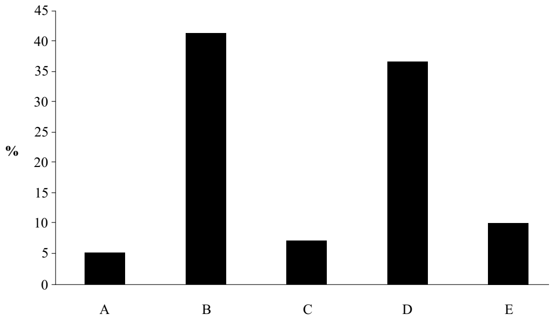

Fig. 3 - Percentages of the different types of microcharoals (<160µm), observable on palynological slides (study conducted with ground wood charcoal) A: opaque microparticles with cellular remains, B: opaque microparticles with an angular form, C: opaque microparticles with an elongated form, D: other types of opaque microparticles, E: translucent microparticles.

Very few micro-particles with cellular forms are observable on the microscopic slides. This is probably because most of the macro and mesocharcoals are fragmented along a longitudinal axis, rather than a transversal one. Some internal parts of vessels with intervascular punctuations can also be identified and correlated with a longitudinal fragmentation. Meanwhile, opaque black particles with an angular form constitute the largest proportion of the vegetal micro-elements

Quantification of the charcoal signature

Quantifications of charcoals are most often expressed as numbers of charcoal objects. These counts, which represent a final state of fragmentation, are influenced by the hazards of fragmentation (Chabal, 1992; 1997; Chabal *et al.*, 1999). In paleoecological analyses of microcharcoals, the results are calculated as a surface concentration of charcoal (cm^2) per volume of sediment treated (cm^{-3}). These global quantifications in the form of a surface do not take into account the final state of fragmentation and thus ignore the hazards of the fragmentation. In effect, if we take the example of a sample containing a charcoal whose largest surface is 20 mm^2, fragmented into four 5 mm^2 elements, the value attributed to the sample is 4. This value is dependant on the fragmentation processes. On the other hand, in the case of surface measures, whatever the number of resulting fragments, the original surface will be reconstituted ($4 \times 5\ mm^2 = 20\ mm^2$). These measures thus allow us to compare the different sedimentary fractions with each other and to add them to obtain a total, while eliminating the hazards linked to the fragmentation processes, with the goal of reconstituting the global charcoal signature present in the sediments. We have applied this quantification scale to the three

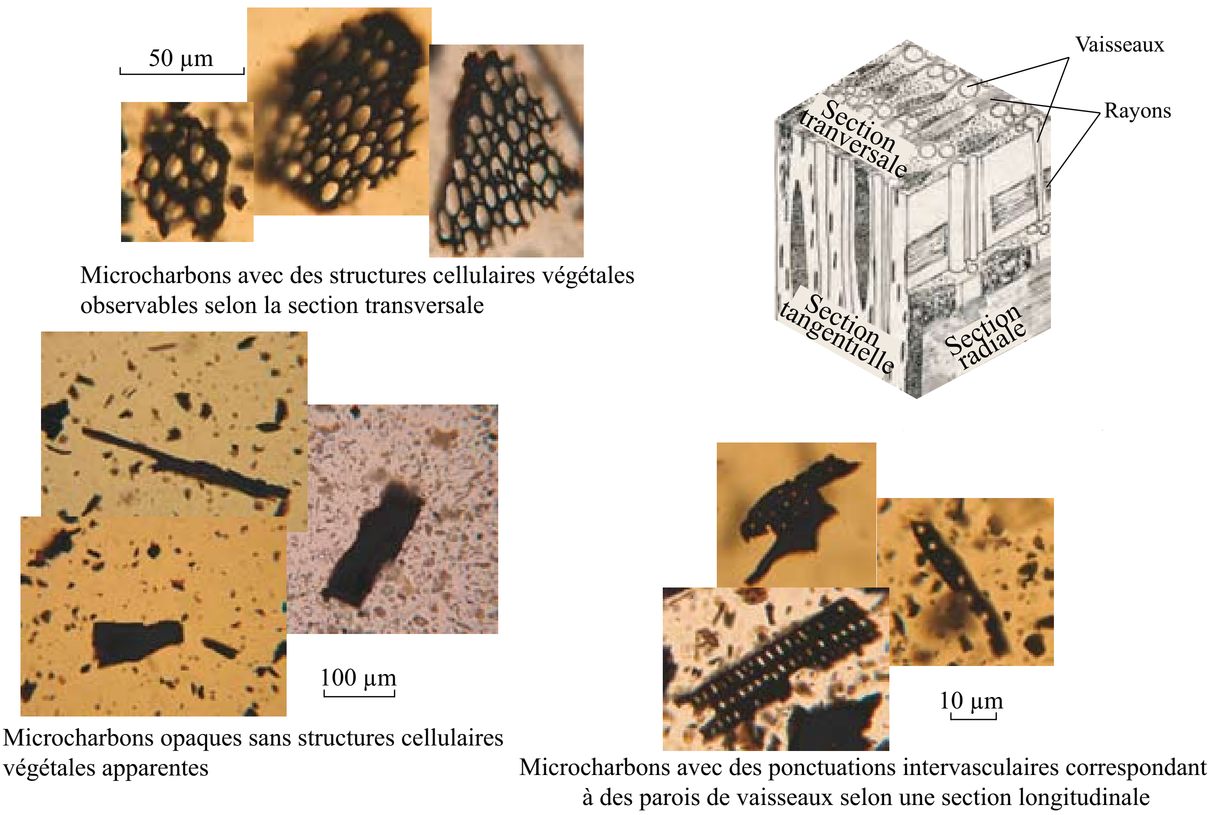

Fig. 4 - Different types of microparticles present on pollen slides observed through optical transmission microscopy (observations made with ground wood charcoal).

sedimentary fractions, allowing us to measure the concentrations on the macroscopic, mesoscopic and microscopic scales. It is important to note that the quantification of surfaces underestimates the relative quantity of macrocharcoals relative to microcharcoals. This is because the macrocharcoals have a third side (thickness that we do not see) that is on average larger than the micro-particles. The surfaces thus give an approximation of the volumes that overestimates the small fragments relative to the larger ones. Meanwhile, our analyses were applied to sites characterized by a great scarcity or absence of macrocharcoals and the few macrocharcoals found have very small volumes, thus minimizing this bias.

An image analysis method that allows an automatic quantification of charcoal surfaces was developed following Marquer *et al.* (2008b). This method calculates the total surfaces of each size class (>500µm; 500-160µm; <160µm). The samples are scanned with a camera connected to a computer. For each sample, the field of numeric observations is captured and analyzed with an image analysis program (©*Image J.1.41*) that allows identification of the burned particles based on their gray levels (fig. 5). A threshold of the image is then manually set by the analyst after identification of the nature of the particles. Several calculation parameters can then be obtained for all of the numeric fields of a sample, such as the average surface of a particle and the total surface of all the particles present. Based on these measures, we calculated for each sample the average surface of a particle (cm^2, mm^2 or μm^2 depending on the sedimentary fraction analyzed) and the concentrations ($cm^2\ cm^{-3}$) for each sediment fraction.

First applications in an archaeological context

We present here the case of the “Grand Abri” rock shelter located on the *coteau de La Garenne* (La Garenne hillside) (Saint-Marcel, Indre, France), which contains archaeological levels dated to the Middle Magdalenian (fig. 6; for a summary of the archeological data of this site, see Allain, 1985 and Despriée *et*

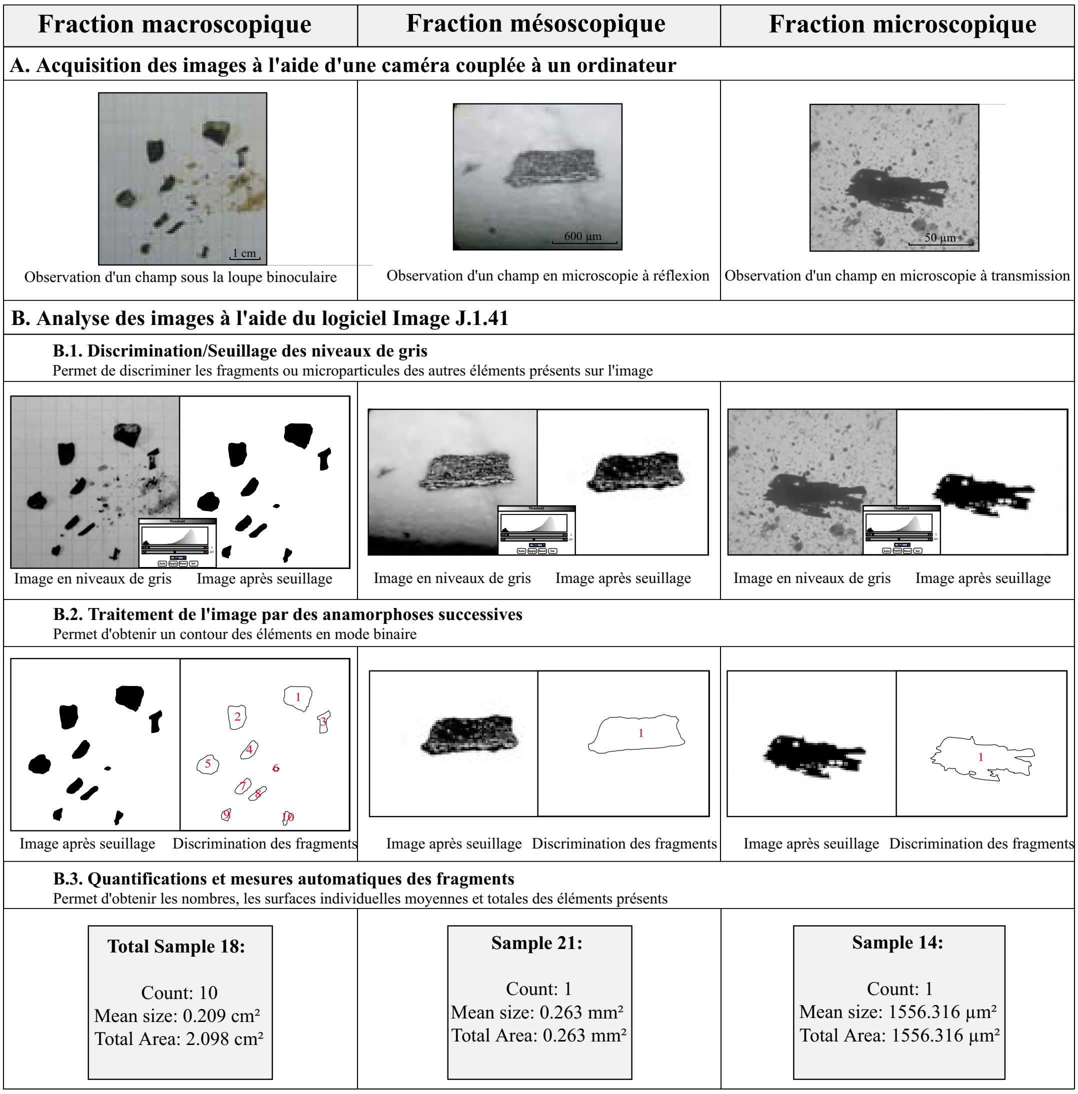

Fig. 5 - Synthesis of the procedure for quantifying macro-, meso- and microcharcoals though image analysis.

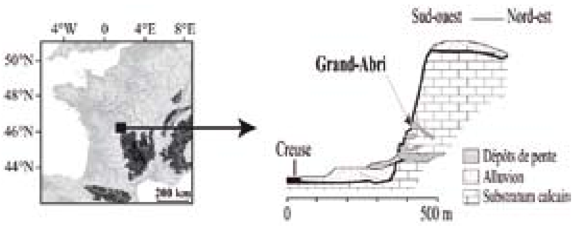

Fig. 6 - North-south profile of the coteau de La Garenne (Saint-Marcel, Indre, France): location of the Grand Abri rock shelter.

al., 2004). Two hearths in stratigraphic position in archaeological levels (A and B) were fully excavated. Descriptions of the combustion features at La Garenne are available in the field notes of Dr. Jacques Allain (Musée Argentomagus at Argenton-sur-Creuse) and in the publication by Allain (1953).

During the excavations directed by Dr. Allain (1946-1976), a near absence of wood charcoal was noted. Our search for charcoal remains within the fine sediment fractions confirms these first observations (fig. 7). The charcoal concentrations are indeed very low and only a few very small macroremains are preserved. Intensive fragmentation is likely responsible for the alteration of the macrocharcoals. In addition, we find the signature of these remains almost solely in the mesocopic and microscopic fractions. Charcoals are thus present, but are too small to have been extracted during excavation, suggesting a loss of information concerning hearth residues.

Pre- and post-depositional processes that modify wood charcoal must thus be considered. The main pre-depositional factors linked to combustion and that can

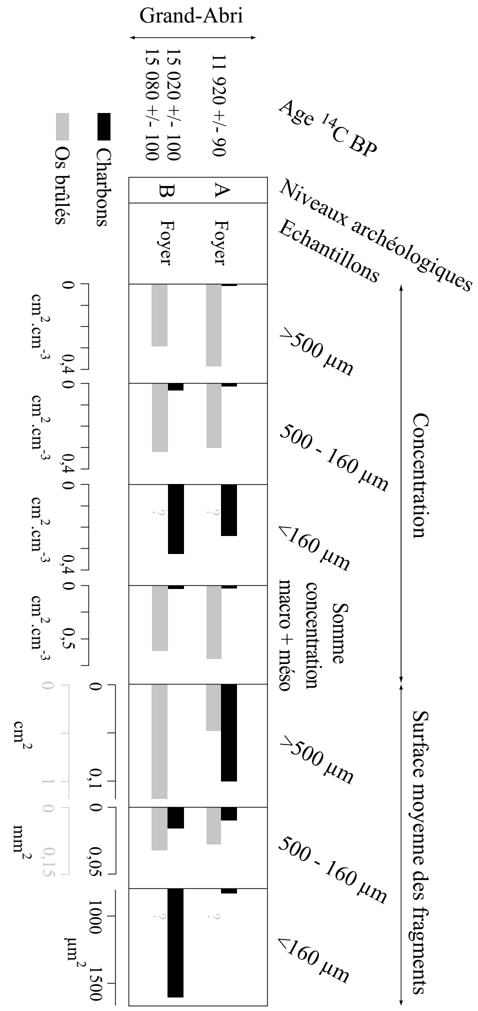

Fig. 7 - Results of the quantifications of combustion signatures (charcoal and burned bone) in the hearths of archaeological levels A and B of the Grand Abri. The micro-fragments of burned bone observed on the pollen slides are not quantified because they are partially altered by the chemical procedure employed in palynology.

influence the preservation of charcoal are the nature of the wood (dead, green or drift wood) (Théry-Parisot, 2001; Théry-Parisot & Texier, 2006), the vegetal species collected and the combustion duration and temperatures related to the functioning of the hearth (Théry-Parisot, 2002a). Dr. Allain (1953) supposed that the near absence of charcoal in the archaeological levels of La Garenne could be due to a nearly total combustion of wood. The presence of bones in the hearths, which increases the combustion duration (Théry-Parisot, 2002a), could have played an important role in the reduction of the macrocharcoal mass, which would partially explain their low frequencies. Post-depositional processes, such as climatic and/or edaphic factors (freeze/thaw, sediment humidity, sediment compaction), biological activities (roots, fungi) and numerous human activities repeated near the hearth (Thinon, 1992; Théry-Parisot, 1998), can favor the "elimination" of charcoals. Charcoals do not appear to be altered by chemical and/or biochemical agents in the soils (Thinon, 1992; Nichols *et al.*, 2000), even if certain authors speak of elements such as oxidation, which could nuance this hypothesis Cohen-Ofri *et al.*, 2006; 2007).

During his excavations, Dr. Allain also noted high quantities of burned bone elements. This second combustion signature, which we have quantified, confirms that this abundance remains constant in the mesoscopic fraction. Burned bones are subject to the same combustion effects and post-depositional processes as charcoal. However, the different physico-chemical constitution of burned bones must be taken into account as burned bone fragments are denser and thus probably more resistant to taphonomic processes than wood charcoal, which is more friable. The quantity of burned bones relative to charcoals could thus increase in function of the intensity of the taphonomic processes.

Conclusion

The procedure of extraction/quantification by image analysis defined experimentally and then applied to the Magdalenian hearths of the Grand Abri has revealed the presence of macrocharcoals (>500 µm). However, since the concentrations are low and the surfaces of the macrofragments are very small, it is impossible to extract them during the archaeological excavation of this site without an adapted procedure. The fact that the entire charcoal signature is present in the very fine sediment fractions (<160 µm) suggests a significant that the wood charcoal was fragmented. The resulting "micro-remains" thus constitute the main source information concerning the presence of combustion residues, and for this reason must be recorded.

Following the numerous taphonomic processes that can modify an archaeological level, the differential fragmentation of wood charcoal and burned bones could result in an overestimation of the burned bone fragments. However, the total concentrations of charcoal present in all the sedimentary fractions, from the coarsest to the finest, thus integrating the refragmented fractions, remains inferior to the sum of the concentrations of macro- and meso-fragments of burned bones. This suggests that burned bones were indeed abundant in the hearths at La Garenne, on the condition that they were submitted to a rate of refragmentation or disappearance equal to that of the wood charcoal, which is not certain due to their greater mechanical resistance. These first analyses of the microremains of combustion contribute important information and raise numerous questions concerning the presence of residual combustion elements in hearths. It will thus be beneficial to continue, develop and enlarge their application to the ensemble of identifiable residual combustion elements, such as phytoliths, seeds, manures and lignins.

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References

Allain J. 1953 - Foyers et dallages dans le Magdalénien de la Garenne à Saint-Marcel (Indre). *L'Anthropologie*, 57 : 284-294.

Allain J., Desbrosse R., Kozlowski J.K., Rigaud A., Jeannet M. & Leroi-Gourhan Arl. 1985 - Le Magdalénien à navettes. G*allia Préhistoire*, 28 : 37-124.

Asselin H. & Payette S. 2005 - Detecting local-scale fire episodes on pollen slides. *Review of Palaeobotany and Palynology*, 137 : 31-40.

Beaudouin C., Jouet G., Suc J.P., Berne S. & Escarguel G. 2007 - Vegetation dynamics in southern France during the last 30 ky BP in the light of marine palynology. *Quaternary Science Reviews*, 26 : 1037-1054.

Beaulieu de J.L. & Reille M. 1984 - A long upper-Pleistocene pollen record from Les Echets near Lyon, France. *Boreas*, 13 : 111-132.

Beaulieu de J.L. & Reille M. 1992a - Long Pleistocene pollen sequences from the Velay Plateau (Massif Central, France). *Vegetation History and Archaeobotany*, 1 : 233 - 242.

Beaulieu de J.L. & Reille M. 1992b - The last climatic cycle at La Grande Pile (Vosges. France) : a new pollen profile. *Quaternary Science Reviews*, 11 : 431-438.

Cain C.R. 2005 - Using burned animal bone to look at Middle Stone Age occupation and behaviour. *Journal of Archaeological Science*, 32 : 873-884.

Carcaillet C., Bouvier M., Fréchette B., Larouche A.C. & Richard P.J.H. 2001 - Comparison of pollen-slide and sieving methods in lacustrine charcoal analyses for local and regional fire history. *Holocene*, 11: 467-476.

Carcaillet C. 2007 - Charred particles analyses. *Encyclopedia of Quaternary Science* : 1582-1593.

Chabal L. 1992 - La représentativité paléo-écologique des charbons de bois archéologiques issus du bois de feu. *Bulletin de la Société Botanique de France*, 139 : 213-236.

Chabal L. 1997 - *Forêts et sociétés en Languedoc (Néolithique final, Antiquité tardive) : l'anthracologie, méthode et paléoécologie*. Editions de la maison des sciences de l'Homme, Documents d'Archéologie Française, 63 : 189 pp.

Chabal L., Fabre L., Terral J.-F. & Théry-Parisot I. 1999 - L'anthracologie. *In : La Botanique*, Bourquin-Mignot C., Brochier J.-E., Chabal L., Crozat S., Fabre L., Guibal F., Marinval P., Richard H., Terral J.F.& Théry I. (eds) : 43-104.

Clark R.B. 1984 - Effects on charcoal of pollen preparation procedures. *Pollen et Spores*, 26 : 559-576.

Clark J.S. 1988 - Particle motion and the theory of charcoal analysis : source area, transport, deposition, and sampling. *Quaternary Research*, 30 : 67-80.

Clark J.S. & Hussey T.C. 1996 - Estimating the mass flux of charcoal from sedimentary records : effects of particle size, morphology, and orientation. *Holocene*, 6 : 129-144.

Cohen-Ofri I., Weiner L., Boaretto E., Mintz G. & Weiner S. 2006 - Modern and fossil charcoal: aspects of structure and diagenesis. *Journal of Archaeological Science*, 33 : 428-439.

Cohen-Ofri I., Popovitz-Biro R. & Weiner S. 2007 - Structural characterization of modern and fossilized charcoal produced in natural fires as determined by using electron energy loss spectroscopy. *Chemistry European Journal,* 13 : 2306-2310.

Costamagno S., Griggo C. & Mourre V. 1999 - Approche expérimentale d'un problème taphonomique : utilisation de combustible osseux au Paléolithique. *Préhistoire Européenne*, 13 : 167-194.

Costamagno S., Théry-Parisot I., Brugal J.-P. & Guibert R. 2005 - Taphonomic consequences of the use of bones as fuel. Experimental data and archaeological applications. *In* : *Biosphere to Lithopshere. New studies in vertebrate taphonomy*, T. O'connor (ed). Actes du 9° Colloque de l'ICAZ, Durham, août 2002. Oxford, Oxbow Books : 51-62.

Cour P. 1974 - Nouvelles techniques de détection des flux et des retombées polliniques : étude de la sédimentation des pollens et des spores à la surface du sol. *Pollen et Spores*, XVI : 103-141.

Daniau A.L. 2008 - *Variabilité des incendies en Europe de l'Ouest au cours du dernier cycle climatique : relations avec le climat et les populations paléolithiques. Etude des microcharbons préservés dans les carottes marines*. Thèse de doctorat, Université de Bordeaux I, 238 pp.

Despriée J., Rigaud A., Vialou D. & Gageonnet R. 2004 - Le Paléolithique de la vallée moyenne de la Creuse: le coteau de « la Garenne », Grand Abri et Grotte Blanchard, Saint-Marcel (Indre). *Bulletin de l'Association pour la Sauvegarde du Site Archéologique d'Argentomagus et Amis du Musée*, numéro spécial, 119 pp.

Faegri K. & Iversen I. 1989 - *Textbook of pollen analysis*. London, John Wiley and sons, 328 pp.

Guiter F., Andrieu-Ponel V., Beaulieu de J.L., Cheddadi R., Calvez M., Ponel P., Reille M., Keller T. & Goeury C. 2003 - The last climatic cycles in Western Europe : a comparison between long continuous lacustrine sequences from France and other terrestrial records. *Quaternary International*, 111 : 59-74.

Hounslow M.W. & Chepstow-Lusty A. 2002 - Magnetic properties of charcoal rich deposits associated with a Roman bath-house, Butrint (Southern Albania). *Physics and Chemistry of the Earth*, 27 : 1333-1341.

Hounslow M.W. & Chepstow-Lusty A. 2004 - A record of soil loss from Butrint, southern Albania, using mineral magnetism indicators and charcoal (AD 450 to 1200). *Holocene*, 14 : 321-333.

McDonald G.M., Larsen C.P.S., Szeicz J.M. & Moser K.A. 1991 - The reconstruction of boreal forest fire history from lake sediments : a comparison of charcoal, pollen sedimentological, and geochemical indices. *Quaternary Science Reviews*, 10 : 53-71.

Marquer L., Nespoulet R., Chiotti L., Otto T. & Théry-Parisot I. 2008a - Etudes sur la taphonomie des charbons de bois au cours des occupations du Paléolithique supérieur de l'abri Pataud (Dordogne, France). Colloque international Q6: « *Biodiversité au Quaternaire: Climats, Environnements et Peuplements* », 166 pp.

Marquer L., Pomel S., Abichou A., Schulz E., Kaniewski D. & Van Campo E. 2008b - Late Holocene high resolution palaeoclimatic reconstruction inferred from Sebkha Mhabeul, Southeast Tunisia. *Quaternary Research*, 70 : 240-250.

Marquer L. 2009 - Apports des signaux polliniques et charbons à la reconstitution des paléoenvironnements et à la connaissance des relations homme/milieu au Pléistocène dans le bassin de la Loire moyenne (France). Thèse du Muséum national d'histoire naturelle, 233 pp.

Marquer L., Despriée J., Otto T., Migaud B. & Renault-Miskovsky J. sous presse - Apport de l'étude des microcharbons dans la compréhension de la taphonomie des macrorestes végétaux brûlés sur le site magdalénien du coteau de « la Garenne » (Indre, France). *Cahiers d'archéologie romande*.

Naughton F., Sanchez Goñi M.F., Desprat S., Turon J.L., Duprat J., Malaizé B., Joli C., Cortijo E., Drago T. & Freitas M.C. 2007 - Present-day and past (last 25000 years) marine pollen signal off western Iberia. *Micropaleontology*, 62 : 91-114.

Nichols G.J., Cripps J.A., Collinson M.E. & Scott A.C. 2000 - Experiments in waterlogging and sedimentology of charcoal: results and implications. *Paleogeography, Paleoclimatology, Paleoecology*, 164 : 43-56.

Patterson W. A., Edwards K.J. & Maguire D.J. 1987 - Microscopic charcoal as a fossil indicator of fire. *Quaternary Science Reviews*, 6 : 3-23.

Perlès C. 1977 - *Préhistoire du Feu.* Paris, Masson, 174 pp.

Reille M. & Beaulieu de J.L. 1988 - History of the Würm and Holocene vegetation in western Velay (Massif Central, France) : a comparison of pollen analysis from three corings at lac du Bouchet. *Review of Palaeobotany and Palynology*, 54 : 233-248.

Reille M. & Beaulieu de J.L. 1990 - Pollen analysis of a long upper Pleistocene continental sequence in a Velay maar (Massif Central, France). *Palaeogeography, Palaeoclimatology, Palaeoecology*, 80 : 35-48.

Rhodes A.N. 1998 - A method for the preparation and quantification of microscopic charcoal from terrestrial and lacustrine sediment cores. *Holocene*, 8 : 113-117.

Stiner M.C., Kuhn S.L., Weiner S. & Bar-Yosef O. 1995 - Differential burning, recrystallization, and fragmentation of archaeological bone. *Journal of Archaeological Science*, 22 : 223-237.

Théry-Parisot I. 1998 - *Économie du combustible et Paléoécologie en contexte glaciaire et périglaciaire, Paléolithique moyen et supérieur du sud de la France (Anthracologie, Expérimentation, Taphonomie)*. Thèse de doctorat, Université Panthéon-Sorbonne de Paris, 500 pp.

Théry-Parisot I. 2001 - *Économie des combustibles au Paléolithique, Anthracologie, Expérimentation, Taphonomie*. Editions CNRS, Dossier de Documentation Archéologique, 20, 195 pp.

Théry-Parisot I. 2002a - Fuel management (bone and wood) during the Lower Aurignacian in the Pataud Rock Shelter (Lower Palaeolithic, Les Eyzies de Tayac, Dordogne, France). Contribution of experimentation. *Journal of Archaeological Science*, 29 : 1415-1421.

Théry-Parisot I. 2002b - The gathering of firewood during Palaeolithic time. *In* : *Nouvelles approches méthodologiques, histoire de la végétation et des usages du bois depuis la Préhistoire*, Thiébault S. (ed). Actes du second colloque international d'Anthracologie, BAR international series, 1063 : 243-250.

Théry-Parisot I. & Costamagno S. 2005 - Propriétés combustibles des ossements. Données expérimentales et réflexions archéologiques sur leur emploi dans les sites paléolithiques. *Gallia Préhistoire*, 47 : 235-254.

Théry-Parisot I. & Texier P.J. 2006 - L'utilisation du bois mort dans le site moustérien de la Combette (Vaucluse). Apport d'une approche morphométrique des charbons de bois à la définition des fonctions de site, au Paléolithique. *Bulletin de la Société Préhistorique Française*, 103 : 453-463.

Théry-Parisot I., Costamagno S., Brugal J.-P., Fosse P. & Guilbert R. 2005 - The use of bone as fuel during the Palaeolithic, experimental study of bone combustible properties. *In* : *The zooarchaeology of fats, oils, milk and dairying*, J. Mulville & A.K. Outram (eds). Actes du Colloque de l'ICAZ, Durham, août 2002. Oxford, Oxbow Books : 50-59.

Thevenon F. 2003 - *Les résidus carbonés de feux dans les sédiments lacustres et océaniques intertropicaux : Implications méthodologiques, climatiques et anthropiques*. Thèse de doctorat, Université de Droit, d'Économie et des Sciences d'Aix-Marseille III, 127 pp.

Thinon M. 1992 - *L'analyse pédoanthracologique : aspects méthodologiques et applications*. Thèse de doctorat, Université de Droit d'Économie et des Sciences d'Aix-Marseille, 297 pp.

Vannière B. 2001 - *Feux, Agro-Pastoralisme et Dynamiques Environnementales en France durant l'Holocene. Analyse du signal incendie, approches sédimentologiques et études de cas en Berry, Pyrénées et Franche-Comté.* Thèse INA-PG/Université de Franche-Comté, 327 pp.

Villa P., Bon F. & Castel J.C. 2002 - Fuel, fire and fireplaces in the Palaeolithic of western Europe. *Review of Archaeology*, 23 : 33-42.

Yravedra J., Baena J., Arrizabalaga A. & Iriarte M.J. 2005 - El empleo de material óseo como combustible durante el Paleolítico Medio y Superior en el Cantábrico. Observaciones experimentales. Museo de Altamira. *Monografias*, 20 : 10-38.

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THE ALTERATION OF NEOLITHIC WOOD CHARCOAL FROM THE SALT SPRING OF POIANA SLATINEI IN LUNCA (NEAMT, ROMANIA): A NATURAL EVOLUTION OR CONSEQUENCE OF EXPLOITATION TECHNIQUES?

Alexa DUFRAISSE, Dominique SORDOILLET & Olivier WELLER

Abstract

Located in immediate proximity to a salt spring still in use, the site of *Poiana Slatinei* in Lunca (Neamt, Romania) has yielded the earliest evidence of salt production in Europe (6050-5500 B.C.). It contains several dozen combustion features that form a large stratified mound of ashes, charcoal and rubified sediment layers.

In 2004, a vast sondage allowed detailed stratigraphic analysis and recording of the Early Neolithic levels and the collection of soil, charcoal and ash samples with the goal of more precisely identifying the techniques, management and interactions with the natural environment associated with salt production at this site.

While the micromorphological study led to the proposition of interpretations concerning the functioning of the fireplaces and the modes of salt exploitation, an anthracological analysis revealed a high degree of alteration of the wood charcoal fragments, or even the absence of ligneous structures. In this paper, we discuss this atypical preservation of charred particles through an analytical summary of the sedimentary, post-sedimentary and technical processes (choice of fuel material, evaporation method) observed at Lunca, and which could have played a role in their alteration.

Keywords : salt, Néolithic, Cris, Romania, techniques, anthracology, micromorphology

Introduction

More than 20 years ago, researchers from the Piatra Neamt Museum identified the earliest traces of salt production in Europe (Dumitroaia, 1994). They are located at the foot of the Carpathian Mountains in Romanian Moldavia where the salt resources are the most abundant.

The salt spring of Poiana Slatinei in Lunca (Neamt, Romania) emerges above the Aquitanian salt deposits. The refuse from this exploitation, located around 20 meters from the current water collection zone, dates to the earliest Neolithic phase and continued to accumulate throughout the 5th and 4th millennia BC. This site is unique in Europe due to its 60 meter long and 25 meter wide mound of ashes that form a small hill rising a dozen meters above the modern spring. It contains several dozen combustion layers and the succession of combustion zones and refuse formed a nearly 3 meter high mound of ashes, charcoal and rubified sediments. No evidence of a habitat has been identified. This exploitation, beginning in the earliest Neolithic phase, was probably facilitated by the high sodium chloride content of the Lunca spring (around 150 g/l). Meanwhile, due to the lack of earthenware remains associated with the briquetage method of salt fabrication, as well as adapted analyses, we had no precise knowledge of the salt exploitation procedure or the detailed chronology of these exploitations, or, moreover, of their management or impacts on the natural environment.

Later, starting in the middle of the 5th millennium, the first earthenware molds, or *briquetages*, appear. These are attributable to the Cucuteni culture and were used for the fabrication of hard, transportable salt cakes. The most seductive hypothesis is thus to see these intentionally formed salt cakes as the object of exchanges and probably envy given the guardian posts and large habitation sites found nearby.

In 2004, a multidisciplinary, French-Romanian, project was developed under the direction of O. Weller and G. Dumitroaia (Weller *et al.*, 2007). Its objectives were to identify the ensemble of technical procedures employed, to understand how this work was managed and how it interacted with the natural environment, as well as to evaluate the socio-economic organization of these ancient European salt productions. In this paper, we focus on the Early Neolithic exploitation at Lunca (from 6050 to 5500 BC) (Weller *et al.*, 2008a). The question of exploitation techniques is approached in an original manner through combined micromorphological and anthracological analyses. More precisely, the taphonomy of the combustion residues is studied based on a micromorphological analysis of the sedimentary and post-sedimentary processes.

Exploitation techniques and experimentation

Since no briquetages or specific earthenware recipients that could have served in the evaporation of salt have been found at this site, we oriented our work from the beginning around the hypothesis of a salt exploitation method that did not employ recipients, as has already been shown elsewhere. In effect, an earlier research project concerning salt exploitation in the Franche-Comté region of France, directed by Pierre Pétrequin and Olivier Weller, led us to imagine a hypothesis based on an actualistic model in Western New Guinea. The fabrication principle, which has been abundantly described (Weller *et al.*, 1996; Pétrequin *et al.*, 2000, 2001), consists of soaking young urticaceae shoots, or split pepper plant wood, in basins installed at the location where salt water emerges. After one or two days of soaking, the vegetal materials engorged with salt by osmosis are burned on piles of hardwood. The ashes and charcoal are sorted by hand and the charcoal is rejected and thrown into the river, while the salt crystals and salty ashes are agglomerated into grey salt cakes, which are dried for a long time before being used as compensatory payments and in long distance exchanges.

Based on this ethnoarchaeological model, an experimental attempt to fabricate salt by evaporation was made in June 2000 (Dufraisse *et al.*, 2004). This hypothesis was eventually rejected, however, since the majority of plants in our temperate regions do not possess a cellular structure adapted to the penetration of salt water by osmosis, except for clematis, which has not been identified among the wood charcoal analyzed (Dufraisse, 2002).

Meanwhile, during our experiments, we observed that if we poured a brine of 30 g/l, the equivalent of sea water, onto the plant materials, white salt concretions formed on the wood charcoal and the ashes were highly charged with salt. Other experiments were conducted based on Antique texts in which certain authors (Pliny the Elder, Tacitus) allude to salt water being poured directly on incandescent logs. Realized on a 1 m^3 wood-pile with, for example, 60% split oak logs, 30% hornbeam branches and 10% diverse woods, all covered by 0.5 m^3 of split fir, these experiments permitted the evaporation of 420 liters of 30 g/l brine, which resulted in a final production of 23 kg of salt residues, including 11 kg of salt crystals, 11 kg of salted ashes and around 1 kg of residual wood charcoal, as well baked earth aggregates. This shows that it is fully possible to produce salt by evaporation by directly pouring brine on burning wood, and with a greater yield. The final product, once sorted, is a grey salt with a high level of sodium chloride and around 15% of potassium resulting from the wood combustion.

It is thus this salt fabrication hypothesis that we tested for the site of Lunca.

Stratigraphy and sampling methods

In July 2004, an ancient sondage at the top of the deposit was reopened and enlarged to 20m^2. The stratigraphy was studied and carefully recorded and numerous samples (soils, charcoal, and pollen) were directly taken by three French specialists.

The stratigraphic analysis by D. Sordoille resulted in the description of around fifty more or less lenticular levels on each of the four profiles of the sondage: greenish clays, rubified clays, charcoal or ashy silts and brown, gray or gray-brown clays. These levels were then regrouped when they appeared to belong to the same phase of functioning. On the western profile, 11 principle fireplaces were thus distinguished. At the end of this fieldwork, 12 micromorphology samples were taken from the different sedimentary ensembles considered to be representative of the accumulation (fig. 1).

At the same time, 55 charcoal samples were taken from the stratigraphy (A. Dufraisse) in accordance with the descriptions of the sedimentary profiles, in the charcoal filled black lenses and, when possible, near the micromorphology samples. The lower levels (rubified clays) and upper levels (ash) of each combustion feature were included in order to fully contain the charcoal filled lens. The volumes of sediments collected vary from one fireplace to another, between 40 and 400 cm^3. The samples, meanwhile, represent only a minor proportion of the combustion features whose dimensions were estimated at between one and two meters.

Results and first interpretations

Micromorphological analysis

The different facies of the deposit represent four broad sedimentary or post-sedimentary processes that played an important role in the creation of the archaeological accumulation.

Combustion processes

The stratigraphic analysis in the field had already led to the hypothesis of the existence of numerous combustion features, characterized by the superposition of ash and charcoal combustion residues on rubefied clays (fig. 2a). The analysis of thin sections supported and clarified this hypothesis. We first observed that the greenish clays that form the substratum of the site are the same as those present in the form of interstratified lenses in the archeological mound. These clays lenses has either a greenish tinge, like those of the substratum, or appear rubefied. In the second case, the presence of ash or charcoal residues in primary position at the surface of clay lenses confirms that combustion is the cause for the rubefaction of the clays. It is thus possible to interpret the lenses of rubefied clay as the floors of fireplaces and the pairing of ash-charcoal lenses over reddened clay as hearths.

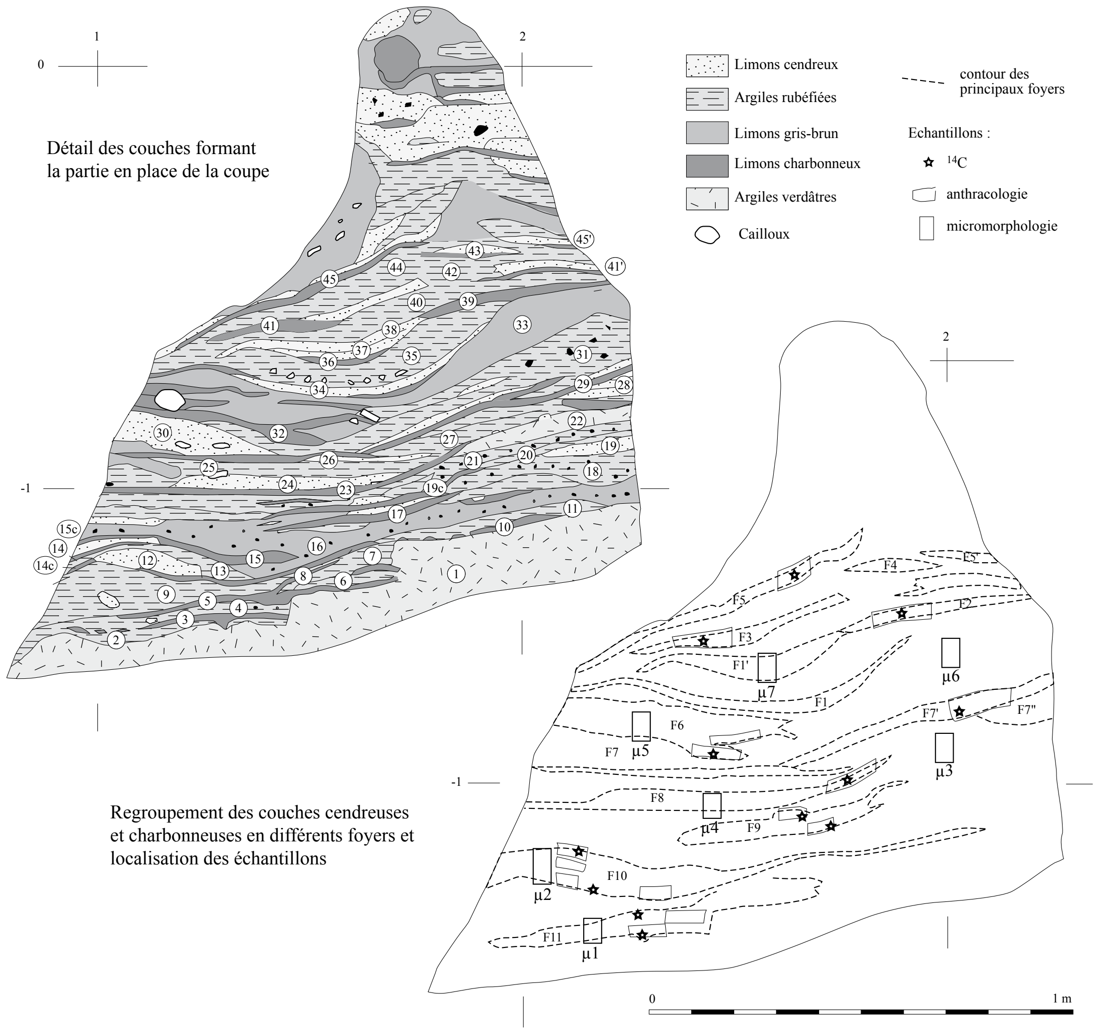


Fig. 1 : Western profile of sondage S1.02 enlarged. Stratigraphy, samples and interpretation. Drawing and CAD: D. Sordoillet.

Runoff, illuviation and argilloclastic processes

Within the archaeological mound, different sedimentary accumulations show evidence for water runoff processes on the surface or illuviation deeper within the sediments (fig. 2b). This evidence consists of micro and interstratified, sand-clay layers within the archeological levels, or thick argillans filling the empty spaces between or within charcoal fragments. We interpret these features as the consequence of the pouring of a large quantity of liquid charged with fine mineral particles on top of the hearths. The clay particles deposited in the pores of the charcoal fragments then contributed to their fragmentation by argilloclastic (clay-clastic) processes. This latter clastic process results from alternations of wetting and drying of the swelling clay.

Secondary crystallizations

Neo-formed salt crystals appear to be well preserved in the reddened clay and charcoal-rich silts, while they suffered greater alteration in the ashes (fig. 2c)[1]. We also note the presence of secondary crystallizations of calcite, in the charcoal-silts, for example, within the cellular vessels of the wood. These secondary crystallizations lead us to propose the hypothesis that the water poured on the

[1] - Only gypsum crystals were clearly identified. This determination was confirmed by J.-P. Sizun, Associate Professor, UMR 6249. The absence of halite is not surprising given the solubility of this salt and the humidity level of this context.

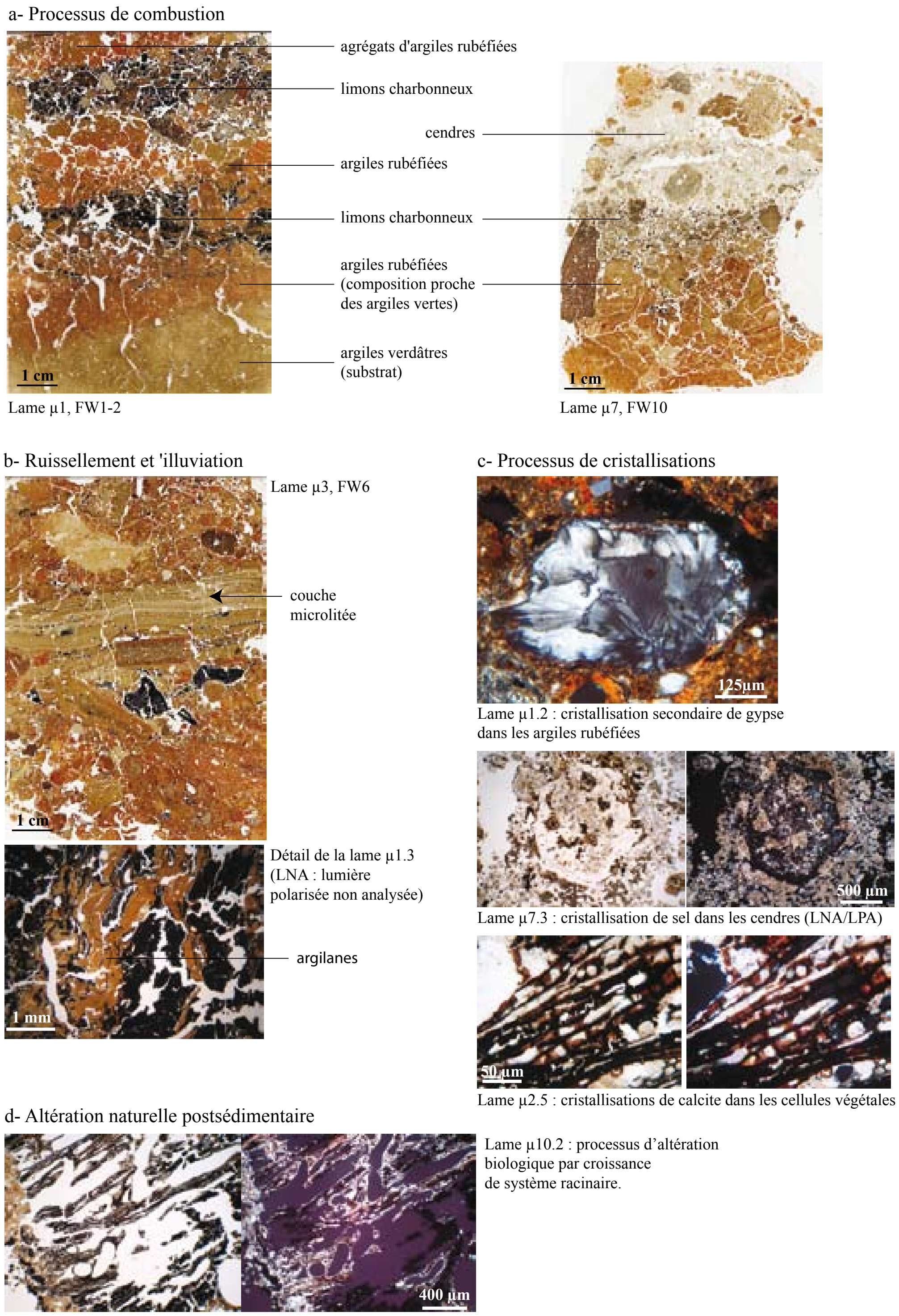


Fig. 2 : Sedimentary and post-sedimentary processes represented in the archeological deposits of the site of Lunca (Photos: D. Sordoillet; Layout: A. Dufraisse): a) Combustion process. The thin sections $\mu1$ and $\mu7$ illustrate two types of combustion feature functioning: complete combustion in $\mu7$, characterized by the superposition of baked clay, few charcoals and abundant ash; interrupted combustion in $\mu1$, characterized by the superposition of abundant charcoal on rubified clays; b) Runoff and illuviation processes. Thin section $\mu3$ shows a micro-bedded level indicating water flow and decantation processes. The close-up photo shows a charcoal accumulation strongly affected by leaching, which leaves thick yellow-orange accumulations between the vegetal residues; c) Secondary crystallization processes (gypsum, salt, calcite); d) Natural, post-sedimentary degradation.

in dissolved salts. These dissolved salts would have then crystallized as the brine evaporated when in contact with the embers.

Reworking after abandonment

The dismantling of the combustion features resulted in brown or gray-brown, heterogeneous accumulations composed of burned or unburned clay aggregates, charcoal, ash, shards and herbaceous phytoliths. These sedimentary accumulations, as well the charcoal contained within them, are often traversed by rootlets (fig. 2d). Their formation may correspond to periods of abandonment.

Anthracological analysis

Throughout the entire site, 7 taxa were identified (table 1, fig. 3). Ash (*Fraxinus excelsior*) is the most frequent, followed by hazelnut (*Corylus avellana*), oak (*Quercus* f.c.), elm (*Ulmus sp.*), hornbeam (*Carpinus betulus*) and maple (*Acer sp.*). One species, elderberry (*Sambucus sp.*), appears only once. This hierarchical order is modified, however, if we consider each fireplace independently. In this case, ash (present in 17 fireplaces, dominant in 9) and hazelnut (present in 15 fireplaces, dominant in 9) are always the most frequent. Elm, present in 5 fireplaces, including 2 in which it is almost exclusive, is the third most frequent species. Next is hornbeam (present in 4 fireplaces, dominant in 2), then maple and oak, which are represented respectively in 4 and 3 fireplaces, but compose nearly all of the contents only once. Finally, elderberry is represented in only one fireplace in which it is not the dominant species.

Though this "deposits in fireplaces" vision gives only a "snapshot" image of the fuel used at a given moment, the low number of taxa does not appear to be due to a sampling bias given the quantity of samples taken and the number of fragments (863) identified. Moreover, these species do not represent the dominant taxa in temperate forests and the local presence of an ashbush with elm[2] could not be demonstrated by the palynologist (analysis realized by E. Gauthier).

We also attempted to detect a possible change in the choice of fuel and/or environment by analyzing chronologically the specific contents of the fireplaces throughout the 2.5 meters of stratigraphy, representing five centuries of exploitation, but we observed no specific trend.

It thus appears that these data show a fuel selection that could be partly determined by an ash and elm forest environment. However, a certain number of the taxa that would potentially be included in this floristic assemblage are not represented in the anthracological spectrum.

Observations made through photonic microscopy reveal that the wood structures altered in various ways that have been identified in other contexts as deformations by radial or tangential compression, variable degrees of vitrification, frequent shrinkage cracks, the presence of crystals in the fibers (possibly salt or calcite crystals) or perforations, in principle of a biological origin (for the identification interpretation of these anatomic signatures, see for example Schweingruber 2001, Théry-Parisot, 2001, Marguerie & Hunot, 2007).

However, of the 32 fireplaces sampled in sondage S1, only 28 could be studied since the other 4 were sterile, though when they were sampled, charcoal fragments were visible with the naked eye. In addition, in the 28 remaining fireplaces, 36% of the charcoals could not be determined, which is a considerable proportion.

Among the undetermined charcoals,

(i) some fragments split in the direction of the fibers, which themselves were only slightly rigid ("limp"), making it impossible to obtain a transverse profile;

(ii) before sieving, some clay or silt aggregates presented residues of carbonized ligneous structures on their surfaces, but these were too fragile to be isolated;

(iii) some are "phantom" wood charcoal pieces, for which only the periphery remains since the interior was replaced by

[2] – The optimal growth of ash occurs in peduncle oak forests with ash et elm "*in well drained, but always cool locations, at a deep, impermeable level with no saturation and a certain limestone content: silts or alluviums of an eutrophic brown soil with common elm, hornbeam, maple, linden […]*" (Jacamon, 1996 : 310).

Coupe stratigraphique	n° de foyer/taxons	*Acer sp.*	*Corylus avellana*	*Carpinus*	*Corylus/Carpinus*	*Fraxinus sp.*	*Quercus sp.*	*Sambucus*	*Ulmus sp.*	*Angiospermae*	**TOTAL**	Indeterminables	Nombre de taxons
coupe Nord	FN1		7			1			31	3	**42**		3
	FN2		60								**60**		1
	NF14			4						3	**7**	8	2
	FN7		1			21				7	**29**		3
	FN10		1			3			2		**6**		3
	FN17inf					52				4	**56**	5	2
	FN17sup		7			2				2	**11**	15	3
	FN18								7	8	**15**	8	2
	FN20			30						1	**31**	5	2
	TOTAL	*0*	*76*	*34*	*0*	*79*	*0*	*0*	*40*	*28*	*257*	*41*	
coupe Est	FE1					50				3	**53**	16	2
	FE2					5				2	**7**	14	1
	FE9	1				26			7	7	**41**	5	4
	FE13	1	10			2				7	**20**	42	4
	FE15inf		3			4				4	**11**	15	3
	FE15sup		3	3		2	1			6	**15**	6	5
	FE18		5							3	**8**	4	1
	FE19	4	25			2				6	**37**	10	4
	TOTAL	*6*	*46*	*3*	*0*	*91*	*1*	*0*	*7*	*38*	*192*	*112*	
coupe Ouest	F2inf						51			4	**55**	0	2
	F3	3	1		3					4	**11**	14	3
	F5	1	15			5				3	**24**	12	4
	F6inf		1			42				2	**45**	10	3
	F6sup					11				12	**23**	13	2
	F7'	1									**1**	5	1
	F8		8			15				10	**33**	7	3
	F9inf										**0**	0	0
	F9sup		2								**2**	2	1
	F10inf?	1				1				1	**3**	9	3
	F10inf										**0**	0	0
	F10moy										**0**	3	0
	F10sup					2					**2**	5	1
	F11bis		1				4			5	**10**	3	3
	F11inf										**0**	0	0
	F11sup		25	1		3				3	**32**	23	3
	TOTAL	*6*	*53*	*1*	*3*	*79*	*55*	*0*	*0*	*44*	*241*	*106*	
coupe Sud	FS2										**0**		0
	FS4	11				1					**12**	0	2
	FS6	3	2			82			6		**93**	8	4
	FS7										**0**	0	0
	FS8										**0**	0	0
	FS11	2	14					13		5	**34**	17	4
	FS12		17			8	1		1	7	**34**	24	5
	FS14inf										**0**	0	0
	FS14sup										**0**	0	0
	TOTAL	*16*	*33*	*0*	*0*	*91*	*1*	*13*	*7*	*12*	*173*	*49*	
	TOTAL	**28**	**208**	**38**	**3**	**340**	**57**	**13**	**54**	**122**	**863**	**308**	

Tab. 1 : Lunca, sondage S1.02 enlarged: identification and counts of wood charcoal from the 32 fireplaces sampled in the 4 stratigraphic profiles

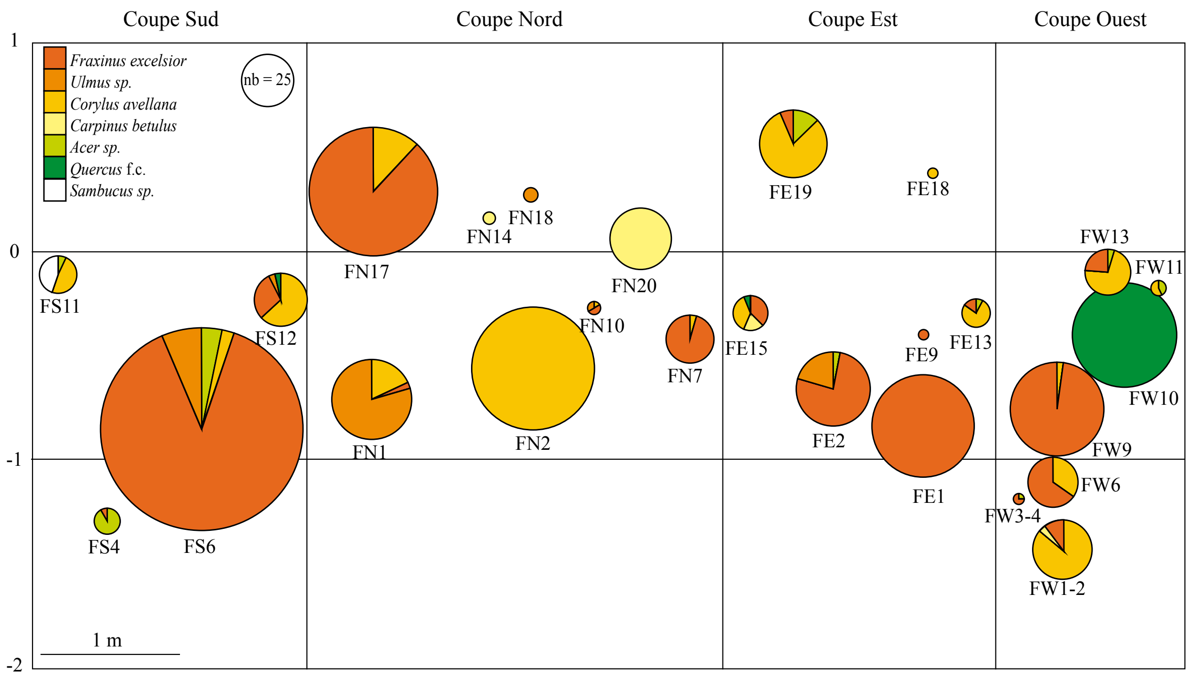


Fig. 3 : Anthracological spectra for each charcoal level repositioned in function of their stratigraphic position in the four profiles of sondage S1.02 enlarged. Drawing: A. Dufraisse.

silt or clay deposits;

(iv) others are compacted agglomerates of charcoal, silt and ash with a powdery consistency.

Several different hypotheses could explain these alterations. The first is related to freeze/thaw cycles, but this phenomenon is not supported by the micromorphological observations. The second could be associated with the evaporation techniques used because as salt crystallizes, its volume increases, which can cause wood charcoal to explode. This hypothesis is plausible if we follow the micromorphological data arguing in favor of the pouring of salt water onto a fireplace with clay-coated surface. The combination of these two principal phenomena, the explosion of charcoal and successive leaching, could be at the origin of the different forms of alteration observed.

Synthesis and discussion: a comparison of the anthracological and micromorphological data

In order to better understand the origin of this degradation of charcoal residues, we integrated micromorphological and anthracological data. After classifying the charcoals according to their degree of fragmentation and alteration, we examined the type of deposit with which they were associated (fig. 4).

It first appears that the identifiable, and thus relatively well preserved, wood charcoals are present in all of the deposit types, whether or not they were subject to leaching or secondary crystallizations (fig. 4a). Their proportion, on the other hand, is variable since they are relatively infrequent in these latter cases. This could be due to the fact that the processes of leaching and secondary crystallization often result in the explosion of charcoal when clays or dissolved salts penetrate into the wood structure.

The moderately degraded fragments, with cellular structures that are still identifiable, are generally found in the brown or gray-brown silt deposits. Their formation is probably related to refuse actions or trampling, which accentuated their fragmentation (fig. 4b-i). We thus observe partially carbonized vegetal fibers with no apparent cohesion, which could correspond to charcoals that split in the direction of their fibers. Their partial carbonization could explain this weak rigidity (fig. 4b-ii).

When the post-sedimentary characteristics attest to hydric percolations, the stage of degradation is much more advanced (fig. 4c). In thin sections, we observe charcoal masses traversed by argillans that can favor argilloclastic processes (fig. 4c-i). These deposits could correspond to the charcoal agglomerates analyzed by anthracology. Successive leaching would have thus resulted in the mechanic fragmentation of the charcoal particles subject to the alternation of soaking and drying of clays. Other process could have accentuated their fragmentation, such as the dismantling or trampling of fireplaces (fig. 4c-iii).

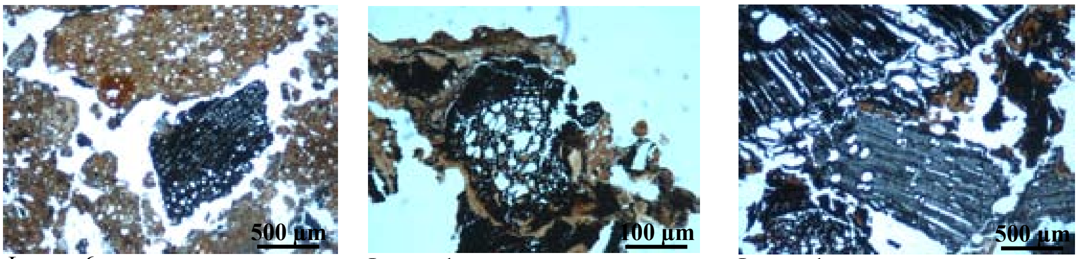
The secondary crystallizations of salt or calcite lead to a hyper-fragmentation of charcoal (fig. 4c-ii). In effect, the augmentation of volume caused by the growth of the crystals causes the charcoal to explode. However, the particularly high porosity of the species exploited favors the impregnation of this water, which increases their capacity for absorption and consequently accentuates their fragmentation at the moment of crystallization.

Among the forms of alteration of the wood charcoal, some fragments have a highly vitrified structure over almost their entire surface. Since pouring salt water on them causes the embers to cool rapidly and the salt acts as a dissolvent, high temperatures and thermal shock could have contributed to the vitrification of the wood charcoal whose degree of reflectance is partially correlated with combustion temperatures (Braadbaart & Poole, 2008).

Finally, natural post-sedimentary processes, as well as the circulation of worms and the growth of root systems, also played a significant role in the degradation of the charcoal (fig. 4c-iv) (Courty *et al.* 1989, p.111).

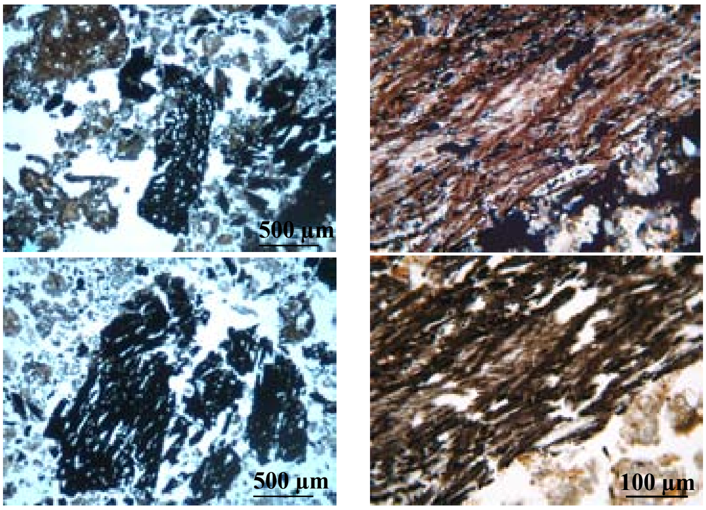
Conclusion

In response to our original question, it appears that the majority of alteration processes that affected the charcoal at Lunca were induced by exploitation techniques. Pouring a- Etat peu dégradé des charbons de bois



Lame µ6 Lame µ1 Lame µ1

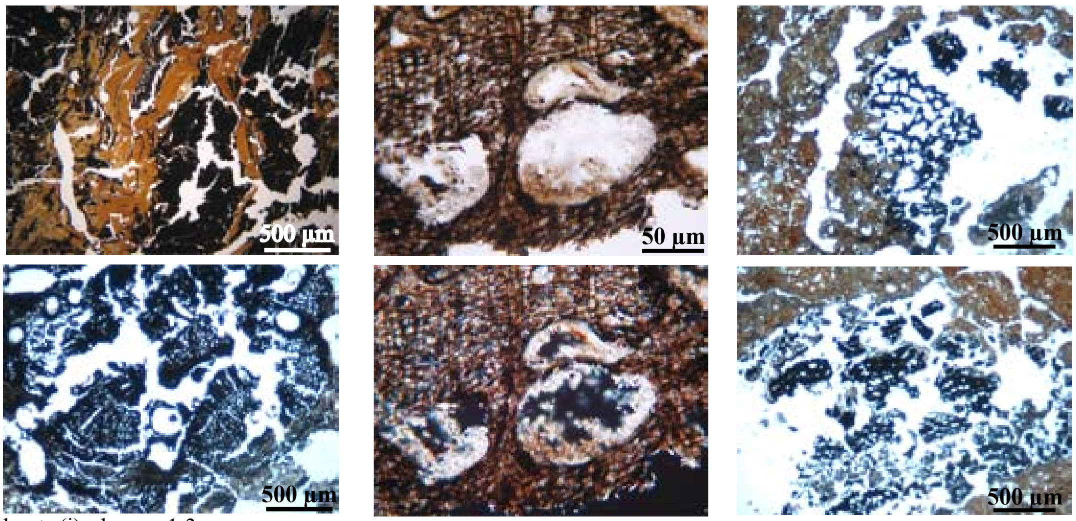
b- Etat moyennement dégradé des charbons de bois



(i) : lame µ8.2 (ii) : lame µ8.5

Fig. 4 : State of alteration of wood charcoals and associated deposit types (Photos and layout: A. Dufraisse and D. Sordoillet): a) Presence of slightly altered wood charcoal in different deposit types; b) Moderately degraded wood charcoal with (i) fragmentation of wood charcoal in accumulations of baked and ashy clay aggregates and (ii) partially carbonized vegetal fibers that could correspond to "fibrous" wood charcoal; c) Highly degraded wood charcoal traversed by argillanes (i), secondary crystallizations inside the wood cells (ii), over-fragmentation of charcoal particles associated with the dismantling of fireplace floors (iii), biological porosity in the wood charcoal (iv).

c- Etat très dégradé des charbons de bois



haut- (i) : lame µ1.3
bas- (iv) : lame µ12.2
(ii) : lame µ2.5
(iii) : lame µ6

a large quantity of brine on the embers led to leaching and fragmentation of the charcoal particles. The clays that penetrated into the combustion residues resulted in the exploding or crushing of the charcoal by argilloclastic processes. The evaporation of highly mineralized water then permitted the neo-formation of salt or calcite crystals that caused the vegetal cellular structures to break apart. Finally, anthropogenic disturbances, such as the collection of salt, also contributed to the degradation of the charcoal. In comparison to these alteration processes linked exploitation techniques, the post-sedimentary phenomena, such as biological mixing or the mechanical action of roots, appear secondary, even if they are not negligible.

These types of charcoal alteration, which concern one third of the anthracological assemblage, led us to consider the techniques of salt evaporation. The watering of a wood-pile with brine, our original hypothesis, would have caused alterations very similar to those observed for the wood charcoal. Meanwhile, the determination of nearly 800 fragments allows us to consider as well the possibility of a selection of species that may have been related to the porosity of the charcoal and thus of their absorption capacity. Another hypothesis, which does not exclude the first, is possible. The species exploited at Lunca could also have been used to feed livestock (Pétrequin *et al*. 1998; Thiébault, 2005), which would imply a complementarity between the exploitation of salt and animal husbandry, especially since today sheep-pens, salt springs and trimmed trees are still present on the Lunca landscape. Thanks to the discovery of a new salt exploitation site in 2005 (Weller *et al*., 2008b), with an 8 meter deep stratigraphy and combustion features dated to between 6000 and 3500 BC, these different hypotheses may be clarified in the future.

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References

Braadbaart F. & Poole I. 2008 - Morphological, chemical and physical changes during charcoalification of wood and its relevance to archaeological contexts. *Journal of Archaeological Science* (Online first).

Courty M.-A., Goldberg P. & Macphail R-I. 1989 - *Soils and micromorphology in archaeology*. Cambridge manuals in Archaeology, Cambridge University Press, 344 pp.

Dufraisse A. 2002 - Salt springs exploitation study in Franche-Comté (France) : contribution of charcoal. *Journal of Archaeological Science,* 29 : 667-675.

Dufraisse A., Gauthier, E., Pétrequin P. et Weller O. 2004 - L'exploitation préhistorique du sel en Franche-Comté. *In* : Approches fonctionnelles en Préhistoire. XXV^{e} Congrès Préhistorique de France, Nanterre, 24-26 novembre 2000, Paris, Société Préhistorique Française : 427-444.

Dumitroaia Gh. 1994 - Depunerile neo-eneolitice de la Lunca si Oglinzi, judetul Neamt. *Memoria Antiquitatis,* XIX : 7-82.

Jacamon M. 1996 - *Guide de dendrologie. Arbres, arbustes, arbrisseaux des forêts françaises*. Nancy, ENGREF, 349 pp.

Marguerie D. & Hunot J.-Y. 2007 - Charcoal analysis and dendrology : data from archaeological sites in North-western France. *Journal of Archaeological Science*, 34 : 1417-1433.

Pétrequin P., Arbogast, R.-M. Bourquin-Mignot, C. Lavier, C. & Viellet, A. 1998 - Demographic growth, environmental changes and technical adaptations : responses of an agricultural community from the 32nd to the 30th centuries BC. *World Archaeology,* 30 : 181-192.

Pétrequin P., Pétrequin A.-M. & Weller O. 2000 - Cuire la pierre et cuire le sel en Nouvelle-Guinée: des techniques actuelles de régulation sociale. *In* : *Arts du feu et productions artisanales,* Pétrequin, P., Fluzin, P., Thiriot, J. & Benoit, P. (eds). XXe Rencontres Internationales d'Archéologie et d'Histoire d'Antibes, Antibes, APDCA-CNRS : 545-564.

Pétrequin P., Weller O., Gauthier E., Dufraisse A. & Piningre J.-F. 2001 - Salt springs exploitation without pottery during Prehistory. From New Guinea to the French Jura. *In: Ethno-Archaeology and its transfers* (Bournemouth 1999), Beyries, S. & Pétrequin, P. (eds). Oxford, Archaeopress, British Archaeological Reports international series, 983 : 37-65.

Schweingruber F. H. 2001 - *Dendroökologische Holzanatomie. Anatomische Grunglagen der Dendrochronologie,* Bern, Stuttgart, Wien, Verlag Paul Haupt, 472 pp.

Théry-Parisot I. 2001 - *Economie des combustibles au Paléolithique. Expérimentation, taphonomie, anthracologie.* Dossier de Documentation Archéologique, Paris, CNRS-CEPAM, 195 pp.

Thiébault S. 2005 - L'apport du fourrage d'arbre dans l'élevage depuis le Néolithique. *Anthropozoologica,* 40 : 95-108.

Weller O., Pétrequin P., Pétrequin A.-M. & Couturaud, A. 1996 - Du sel pour les échanges sociaux. L'exploitation des sources salées en Nouvelle-Guinée (Irian Jaya, Indonésie). *Journal de la Société des Océanistes,* 102 : 3-30.

Weller O. & Dumitroaia Gh. 2005 - The earliest salt production in the World. A Neolithic exploitation in Lunca-Poiana Slatinei, Romania. *Antiquity,* 79.
www.antiquity.ac.uk/projgall/weller/index.html

Weller O., Dumitroaia Gh., Monah D. & Nuninger L. 2007 - L'exploitation des sources salées de Moldavie : un exemple de ressource structurante du territoire depuis le Néolithique. *In : L'exploitation du sel à travers le temps*, Monah, D., Dumitroaia, Gh., Weller, O. & Chapman, J. (eds). Colloque international de Piatra Neamt, Roumanie, octobre 2004, CMJN (*Biblioteca Memoriae Antiquitatis*, XVIII) : 99-113.

Weller O., Dumitroaia Gh., Sordoillet D., Dufraisse A. & Gauthier E. 2008a - Première exploitation de sel en Europe. Techniques et gestion de l'exploitation de la source salée de Poiana Slatinei à Lunca (Neamt, Roumanie). *In : Sel, eau et forêt. D'hier à aujourd'hui,* Weller, O., Dufraisse, A. & Pétrequin, P. (eds). Colloque d'Arc-et-Senans, octobre 2006, Besançon, Presses Universitaires de Franche-Comté, Cahiers de la MSHE Ledoux 12 (Homme et Environnement, 1) : 205-230.

Weller O., Brigand R. & Alexianu M. 2008b - Recherches systématiques autour des sources salées de Moldavie. *Memoria Antiquitatis* XXIV, 2007 : 121-190.

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CHAPTER 3

Archeozoology and taphonomy

HEATING AND DIAGENESIS-INDUCED HETEROGENEITIES IN THE CHEMICAL COMPOSITION AND STRUCTURE OF ARCHAEOLOGICAL BONES FROM THE NEOLITHIC SITE OF CHALAIN 19 (JURA, FRANCE)

Ina REICHE

Abstract

Bone materials, including numerous fragments of burned bones, are often found at archaeological sites. A comparative study of burned and unburned bone materials originating from the lacustrian site 19 of Chalain Lake, Jura, France, was conducted in order to assess their potential to yield information on past lifeways. The bones were analysed using various complementary physico-chemical methods (SEM-EDX, microPIXE/PIGE, FTIR, XRD, TEM-EDX) taking into account their chemical and structural heterogeneity. Through this "high resolution" approach, it was possible to observe the modifications of the bone mineral phase induced by heating and the processes of soil diagenesis, at the scale of the sample.

Our research has made it possible to distinguish the markers that characterise the diagenesis and heating of the specimens. The markers of heating could be used as quantitative indicators of the heating temperature reached by the bone prior to being buried. In addition, the results show different modification mechanisms depending on the possible heating of the bone prior to burying. Specifically, the concentration profiles determined on cross-sections by PIXE/PIGE make it possible to reveal an increase in exogenous chemical species at the centre of the heated sample, in contrast with the unburned bones which show higher concentrations of exogenous elements at the edge than at the centre. Thanks to the nanoscale structural analysis by transmission electron microscopy (TEM), it was possible to correlate the specific modifications of the elemental composition with the size of the apatite crystals present in the specimens.

This method can now be applied to other archaeological problems such as the investigation of various funerary customs or the taming of fire, in the knowledge that it can be applied to a small number of samples that must consequently be carefully selected from a larger corpus in order to be representative.

Keywords : archaeological bones, heating, diagenesis, Neolithic, physico-chemical microanalysis, FTIR, microPIXE-PIGE, TEM

Heating of archaeological bones and diagenesis

The discovery of an anthropic activity linked to fire or heating—utilised both to modify objects and in the activities of daily life—is of utmost interest in archaeology and prehistory because the mastery of fire represents a crucial step in human cultural and socio-economic evolution. For this reason, it is very important to find meaningful markers in the objects unearthed during archaeological excavations. Bones or objects made from bones, ivory, or Cervid antlers are among the objects most commonly found at prehistoric sites. Bone remains or worked bone objects sometimes bear signs of heating. The temperature rise during the heating of bones translates into a modification of their structure, as well as their chemical and isotopic composition. Physico-chemical methods of analysis can be utilised to detect the signs of heating within the material. However, archaeological bones are complex nanocomposite biomaterials, which are modified by diagenetic and taphonomic processes in archaeological sediments. When trying to distinguish the modifications linked to thermal treatments from those related to diagenetic processes, the analysis of archaeological bones proves to be a real challenge requiring complementary techniques both to elucidate the structure and to study the changes in chemical composition at various scales; this distinction is achievable perhaps only at the nanometre scale. Moreover, the alterations are not necessarily homogeneous within the bone specimens.

This paper thus more specifically addresses the study of the diagenesis of the mineral phase of burned archaeological bones at a small scale, utilising high resolution analytical methods in order to detect the heterogeneities induced by the different processes. It presents a physico-chemical analysis of unburned bones compared to that of burned bones from the same archaeological site, that of station 19 of Chalain Lake (3850 - 2900 B.C., Jura, France) (fig. 1).

Present state of knowledge

Brief review of the structure of bone

Biomaterials such as bone, dentine, or ivory are composite materials with a highly hierarchical structure at the macro-, micro-, and even nanoscale level. However, their physico-chemical and mechanical properties are essentially dependent on the close imbrication of the mineral and organic phases at the nanometric scale (fig. 1.)

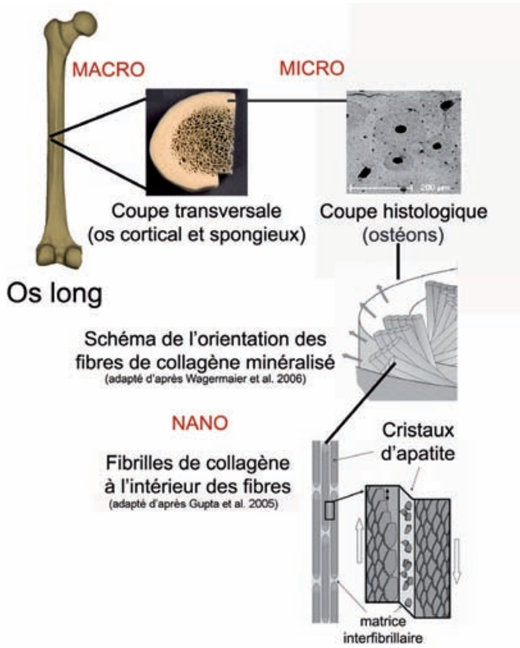


Fig. 1 - Schematic structure of a bone at various scales.

At the molecular level, the organic phase of bone material is composed of 90% type I collagen (tropocollagen) made rigid by an extremely close-packed filling of crystallites of carbonated hydroxylapatite with the general formula (carb-HAP) that constitutes the mineral phase. Collagen is a protein arranged in a triple helix in which each strand is composed of a chain of amino acids (the most frequent sequence being a glycine, proline, hydroxyproline chain.) The collagen molecules are organised hierarchically: that is, the molecules are assembled in the form of fibrils that in turn form fibres. The organic phase also contains lipids and non-collagenous proteins (like osteocalcin) that provide the interface between the collagenous organic matrix and the mineral phase. It is generally acknowledged that the apatite crystals grow inside the organic framework. These carb-HAP crystals have the shape of platelets whose dimensions are still subject to controversy. Cui *et al.* (2007) report crystals having dimensions of 50 x 28 x 2 nm^3.

Modifications to bone during heating

Numerous researches have already addressed the heat-induced modifications of bone material. They have considered modifications of the both general appearance of objects and of their structure or chemical composition at the molecular level. In general, a reference standard has been developed based on fresh cortical bone as this is the type of bone that is preserved best in archaeological contexts (Shipman *et al*., 1984; Baud and Tochon-Danguy, 1985; Stiner *et al*., 1995; Michel *et al*., 1996 ; Person *et al*., 1996; Reiche *et al*., 2007; Lebon, 2008).

Modifications to the general appearance of bone

A colour change is the most obvious modification caused by heating. When a fresh bone is heated, its colour evolves from beige-light brown, through black, to gray and finally, white. An increase in the friability and porosity of the objects occurs at the same time at this colour change. The modified bones can also assume other colours such as blue, green, red, and black. The colours, except the blue, are not necessarily due to heating of the bone, but rather to the presence of crystalline phases containing iron, manganese, or copper (Reiche & Chalmin, 2008; Shahack-Gross *et al*., 1997.) Two heat treatment stages can generally be distinguished: carbonisation and calcination. Carbonisation consists of the formation of black carbonised products at temperatures between 280 and 650°C. Calcination refers to a heat treatment at higher temperatures (650°C and higher) that yields grey or white residues. In addition, Stiner *et al*. (1995) have defined more elaborate heating stages, distinguishing a total of six heating stages ranging from non-heated (0) to calcination (6).

Changes in the structure and chemical composition

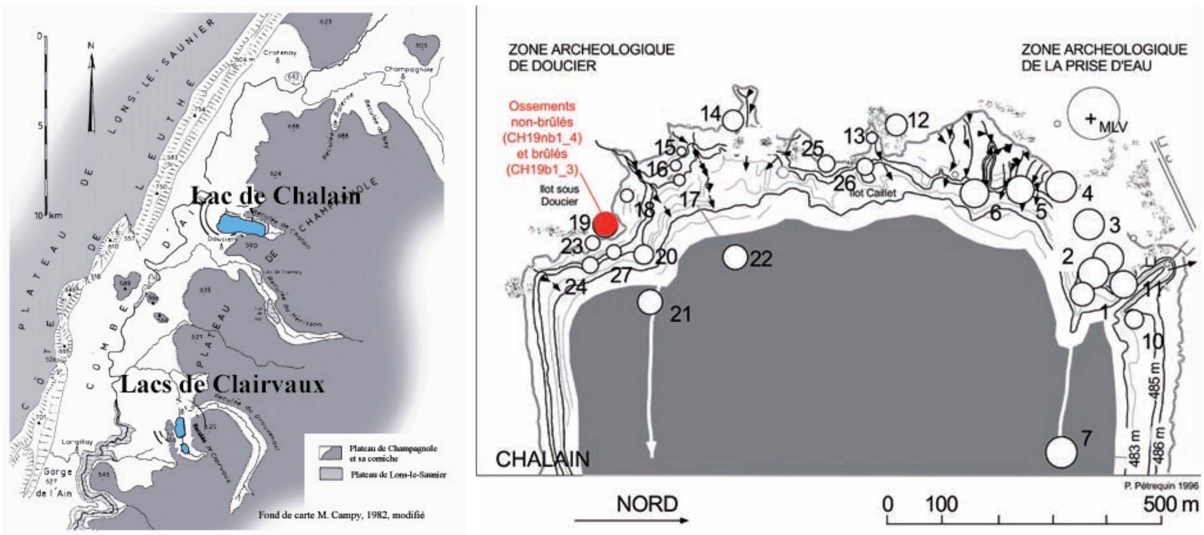
The changes on a macroscopic scale are associated with changes at the micro- or even nanoscale level in the structure and chemical composition of the material during heating. To start, the material is fissured and loses water. Starting at 170°C, the organic fraction changes and volatilises (Chadefaux & Reiche, 2009) which, from a macroscopic point of view, translates into a brownish colour. At around 400°C, a large portion of the organic matter has been carbonised. The bone then assumes a black colour. The carbonisation products are eliminated as the temperature rises to 650°C. The modifications in the mineral phase are observed starting at 500°C, but they do not result in a colour change. They consist of both the transformation of the carb-HAP and of the improvement in the crystallinity of the bone's apatite, that is to say an increase in the size and an improvement in the regularity of the crystals. The carbonates separate from the phosphates in the carb-HAP, thus yielding a purer apatite and some calcite. At around 600°C, the carbonates and hydroxyl ions are lost through the liberation of CO_2 and water, leading to the formation of ß-tri-calcium-phosphate (ß-$Ca_3(PO_4)_2$) and lime (CaO) in addition to the apatite.

Employing various investigation techniques, such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), electron paramagnetic resonance (EPR), or transmission electron microscopy (TEM), it has been possible to establish reference standards that allow the detection of modifications to bones during heating and the determination of the temperatures achieved (Person *et al*., 1996; Reiche *et al*., 2007; Piga *et al*., 2008; Lebon *et al*., 2008.) The determination of the temperature is based either on the calculation of the crystallinity index (CI or SF), or on the average size of the crystal (by XRD or TEM), or on the shift of specific peaks after the splitting of vibrational peaks in the FTIR spectrum. The establishment of this reference standard has now made it possible to estimate the approximate heating temperature reached by a bone before its burial. It should be stressed that in general these analyses approached the object as a whole and did not take into account the possible heterogeneities induced within the material by the various heating procedures. Several heating stages can sometimes be observed on the same ancient specimen. Moreover, it should be noted that the diagenetic processes in the soil can also lead to the modification of the crystallinity of ancient bones. This is why it is difficult to distinguish the changes in crystallinity due to these diagenetic processes from those linked to heating at low temperatures (less than 300°C.) Nevertheless, a study of the form and size of the crystals by TEM gives a more accurate determination of heating, at least for temperatures above 500°C, because the morphology of the crystal formed upon heating is characteristic and allows us to precisely distinguish a heat induced recrystallisation from one obtained as a result of low temperature geochemical processes. It is obvious that other parameters such as the colour of the specimen or of other burned objects found within the same archaeological context must also be considered to confirm the results obtained.

Description of the study site

Over thirty archaeological sites were discovered during the excavations and prospections carried out by P. Pétrequin and his team at Chalain Lake (fig. 2). This lake is located in the Combe d'Ain, a closed alluvial valley in the Franche-Comté region, at the foot of the folded Jura (Jura plissé). It displays long chronological sequences of littoral villages dating from approximately 3850 to 850 BC. Seven Neolithic villages in the chronological range of 3850-2650 BC have been undergoing excavation for around the pas thirty years by P. Pétrequin's team (Pétrequin &Pétrequin, 1988; Pétrequin, 1997; Pétrequin *et al*., 1998).

Chalain Lake is located at about 500 m altitude (fig. 3), which is the upper limit for the extension of cereal cultivation; it thus presents an example of a specific adaptation by Neolithic civilisations to a harsh climate. The sites corresponding to the archaeological villages of Chalain are not distributed along the entire perimeter of the lake. The majority of the villages are located on the lake's west side, which has the advantage of long-lasting sunlight and proximity to the cereal fields. The sites are separated from the lakeshores by a line of swamps and the littoral platforms are relatively wide. The occupations are concentrated in the two bays nearest to the arable fields (Pétrequin & Pétrequin, 1988; Pétrequin, 1997; Pétrequin *et al*., 1998). The Chalain sites are in their original topographical positions, with a progressive drying linked to the lowering of the lake level starting in 1904 (P. Pétrequin, pers. comm.)
These sites are marginally located relative to the epicentres of cultural developments and in terms of population density during the period under study. In fact, only two littoral zones of Neolithic sites, 12 km apart as the crow flies, are known in this region (those of Chalain Lake and those of Clairvaux Lake.) They constitute a coherent whole in terms of lifeways and methods of exploiting the environment. The amalgamation of research on the sites of Chalain



a b

Fig. 2 - a) Map of the Jura region, France with Chalain Lake and b) map of the archaeological sites of Chalain Lake, Jura, France. Reproduced with the permission of P. Pétrequin.

Fig. 3 - Photograph of Chalain Lake, I. Reiche, 1998.

(abbreviated CH) and Clairvaux is therefore justified and allows comparisons of the overall development of Neolithic communities, particularly in demographic and agricultural terms. The geographic situation is favourable because the region is well demarcated and its dendrochronological datings thus allow precise determinations of the ages of contemporary or successive villages.

Multidisciplinary research projects have been undertaken at these locations in order to take advantage of the archaeological, biological, chemical, and geological information contained in the objects found (Pétrequin, 1997.) Research on the modifications associated with heating and diagenesis of bone remains are integrated in the context of these investigations.

At Chalain, the bones are exceptionally well preserved throughout the site. According to archaeological observations (P. Pétrequin, pers. comm.), the bone fragments originating from the preparation of meals (butchering and cooking) were discarded in the dumps in front of the only entrance to the houses on pilings, built on flood prone ground (fig. 4.) This refuse (including the bone remains) therefore fell onto humid soil or into shallow water. They were quickly covered by the vegetal litter brought in by humans to stabilise and reclaim the exterior soils during low water periods. After the villages were abandoned, the lake's level rose again and lake chalk was deposited. These conditions favoured the preservation of the remains in an anoxic environment, quite submerged or below the level of the water table. This burial environment is specifically favourable to the preservation of organic materials.

Fig. 4 - Pile house built on Chalain Lake following the building methods of the era. Reproduced with the permission of P. Pétrequin.

Bone materials studied

Despite the exceptional preservation of archaeological remains at lacustrian sites, the bones found can be more or less modified depending on the conditions to which they have been subjected before or after their disposal (Pétrequin *et al*., 1998):

Type 1: a large quantity of bones was burned as a consequence of deliberately set or accidental fires at the time the littoral dwellings were abandoned or as a result of specific methods of utilisation of bone material (meat cooking, glue manufacture, etc.) These bones were found in the form of small undefined fragments.

Type 2: some unburned bones are characterised by a remarkable state of preservation free of superficial attack and with good conservation of the organic and mineral matter.

Type 3: bones that have undergone another form of degradation apparently have nearly intact organic material. Nonetheless, the bone mineral material is modified to such extent that the bones can be flexible. This is especially the case for bones found in waterlogged environments.

During the excavation campaign conducted in 1998, some fragments of burned and unburned bones from the emerged station 19 were entrusted to us for analysis. Having been preserved in similar burial conditions, the various burned and unburned specimens allow us to study the influence of heating on the diagenetic processes and therefore to assess their potential to yield information and the representivity of these two types of remains.

Four fragments of unburned bones, labelled AB_CH19nb1 - 4, corresponding to conservation type 2 and three fragments of burned bones (type 1), labelled ABB_CH19b1-3, were analysed (fig. 5, table 1.) They originate from layers H and K of station 19 of Chalain which are dated to 3040–2970 BC. These layers correspond to villages with raised floor houses with sediments of anthropic dung that contain a small percentage of calcium carbonates deposited by the lake (fig. 6).

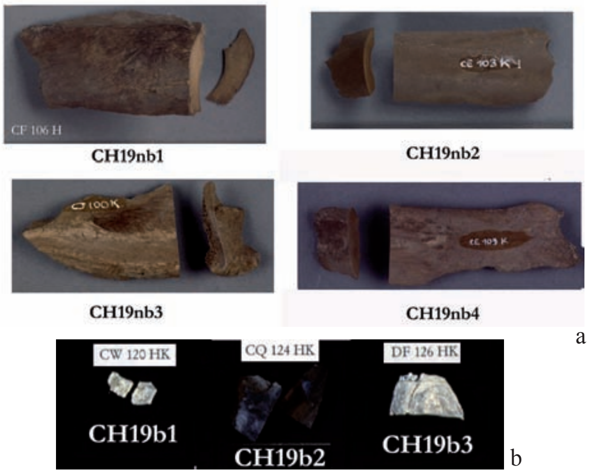


Fig. 5 - Photographs of the bones from Chalain Lake 19 studied: a) unburned specimens and b) burned specimens.

"High resolution" physico-chemical approach

The ensemble of this work led us to apply a broad range of complementary analytical methods since in order to evaluate the state of preservation of complex and very heterogeneous bone materials and reliably interpret the results obtained, it is essential to acquire quantitative information on the object itself. This information concerns its chemical composition and the spatial distribution of its constituent elements, its crystallinity, and the morphology of its crystalline phases. To this end, it is necessary to employ a large number of complementary techniques that allow the detection of the changes in the bone structure and trace elemental composition from the microscale to the nanoscale.

The data obtained on the archaeological samples are then compared to those obtained on a fresh reference bone in order to assess the state of preservation. If the archaeological bone shows characteristics similar to those of the fresh bone (made up of the same phases, homogeneous distribution of the component elements, low degree of crystallinity of the carb HAP), it is presumed to be well preserved.

Scanning electron microscopy (SEM), paired with a system of analysis by energy dispersive X-ray spectroscopy (EDX), is utilised to observe and analyse

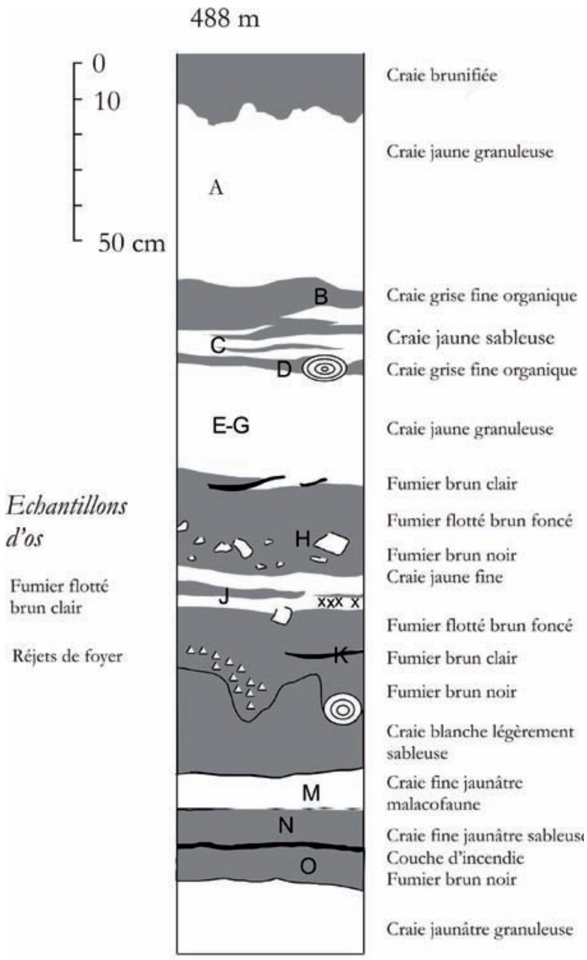


Fig. 6 - Stratigraphy of station 19 of Chalain Lake. Reproduced with the permission of P. Pétrequin.

Site and archaeological reference	Archaeological layer	laboratory reference	Nature of the sample (1)	state of conservation at the naked eye
19 : CF106 H	H	AB_CH19nb1	undetermined	unburned, brown
19 : CE103 K1	K	AB_CH19nb2	undetermined	unburned, brown
19 : CJ100 K1	K	AB_CH19nb3	left humerus of wild boar	unburned, brown
19 : CE103 K2	K	AB_CH19nb4	undetermined	unburned, brown
19 : CW120 HK	HK	ABB_CH19b1	undetermined	burned, white
19 : CQ 124 HK	HK	ABB_CH19b2	undetermined	burned, black in the centre and blueish at the border
19 : DF126 HK	HK	ABB_CH19b3	undetermined	burned, black in the core and gray at the border

(1) Rose-Marie Arbogast is acknowledged for the determination of the analysed bone samples.

Tab. 1 - Characteristics and denominations of the bone specimens chosen for this study.

the inclusions precipitated in the bone matrix during their modification. This technique makes it possible to distinguish the constituent elements of the bone material and the chemical species present in the inclusions, without, however, allowing the detection of the trace elements.

The trace element content is obtained through a very sensitive spectroscopic analysis by X-rays and gamma-rays generated by a proton micro-beam (microPIXE/PIGE) utilising the *Accélérateur Grand Louvre d'Analyse Elémentaire* [Grand Louvre Elemental Analysis Accelerator] (AGLAE) of the LC2RMF. This technique also shows the spatial distribution of the elements (major, minor, trace) through the measurement of concentration profiles.

TEM permits a direct structural analysis of monocrystals of nanometre size. This technique is used to complement XRD and FTIR methods that allow the structural and molecular analysis of the overall bulk specimens. In addition, the splitting factor SF (IR)[1], commonly utilised in archaeology to estimate the state of preservation of the samples and a possible heating, is calculated from the FTIR spectra.

The analytical conditions of the techniques are described in detail in the thesis and in the articles: I. Reiche (2000), Reiche *et al*. (1999, 2002a and b, 2003, 2007).

Results of observations and physico-chemical analyses at different scales

The unburned bones from the Neolithic village of Chalain 19

The bones from Chalain 19 are well preserved in the sense that they are composed of slightly crystallised carb-HAP and of a collagenous fraction. The SF (IR) crystallinity index of the four specimens, calculated according to the reference standard of Reiche *et al*. (2003), varies between 2.3 and 2.5, thus remaining very close to that of fresh bone (2.0) (fig. 7.) The elemental composition of the four unburned bone specimens, measured by microPIXE/PIGE, is shown in table 2. In all bones, the Ca/P ratio is higher than in the reference fresh bone; the bones also contain numerous trace elements such as iron, manganese, aluminium, silicon, sulphur, and fluorine in concentrations higher than those measured in the fresh bone.

The XRD and FTIR analyses demonstrate the presence of calcite ($CaCO_3$) and secondary phases such as boehmite (AlO(OH)). Moreover, as shown by the SEM-EDX analysis, all unburned bone samples contain localised iron sulphide based microcrystals in the pores or the fissures. These

1 - The calculation of a crystallinity index derived from the IR spectrum is commonly utilised in archaeometry for the purpose of showing the bone modifications during diagenesis (Termine and Posner 1966; Shemesh 1990; Weiner *et al*. 1993; Michel *et al*. 1996; Sillen and Parkington 1996; Wright and Schwarcz 1996). This index is based on the splitting of the peaks corresponding to the vibrations of the phosphate groups ν_4 around 560-600 cm^{-1} obtained in the absorbance mode. This index or "splitting factor" (SF) is calculated from the intensities of the absorbance peaks at 605 and 565 cm^{-1}: SF = (A(605 cm^{-1}) + A(565 cm^{-1}))/ A(base)

The SFs measured by Sillen and Parkington (1996) vary between 2.80 for an untreated modern cow bone to 5.33 for the same bone once it has been burned. *This SF index represents only an overall indication of the crystallinity*. In reality, other adsorption peaks can be superimposed on the phosphate peaks (those of sulphates, for example), but here we will utilise the SF solely to estimate the progress of crystallinity in cases that do not require a more accurate measurement by transmission electron microscopy.

	AB_CH19nb1		AB_CH19nb2	AB_CH19nb3		AB_CH19nb4		MB (modern sheep bone)	
	mean concentration (ppm)	Range (ppm)	mean concentration (ppm)	mean concentration (ppm)	Range (ppm)	mean concentration (ppm)	Range (ppm)	mean concentration (ppm)	Range (ppm)
F	2800±100	2400-3400	non analysé	2000±100	1000-4000	3600±100	2100-6300	550	0-1200
Na	7200±900	6000-8420	3000±300	2800±330	1400-3900	5700±900	4100-9050	8500	7600-9100
Mg	1500±30	900-1700	-	1000±170	650-1250	1500±320	960-2500	7800	7200-8700
Al	630±320	140-1640	-	640±120	-	850±350	200-3800	-	-
Si	-	-	1700±160	6300±220	3800-10000	2900±	180-12100	-	-
P	176000±1000	168000-182000	160900±480	152000±1000	146000-157000	178000±1400	171000-204000	179 000	178000-180000
S	4300±300	2100-18400	2300±100	8100±220	2300-32400	3900±600	2200-18200	1600	1200-1900
Cl	450±15	120-690	160±60	200±80	100-230	220±60	70-410	750	650-820
Ca	404000±2800	379000-417000	427000±1300	419000±3800	380000-431000	390500±2300	353000-405000	384000	382000-386000
Mn	220±10	170-260	360±10	280±10	200-380	370±20	300-580	-	
Fe	5600±200	4000-7300	6200±180	10700±300	5200-33800	7100±250	3100-18800	-	
Zn	220±10	160-330	260±10	300±10	200-400	160±800	90-350	130	120-140
Sr	190±10	130-230	130±10	150±10	130-170	140±10	90-190	250	180-280
Ca/P	2,3		2,7	2,8		2,2		2,1	2,1-2,2

Tab. 2 - Average elemental composition and concentration range of unburned bones from site Chalain 19 and of modern sheep bone, analysed via PIXE/PIGE. - = below the method's detection limits.

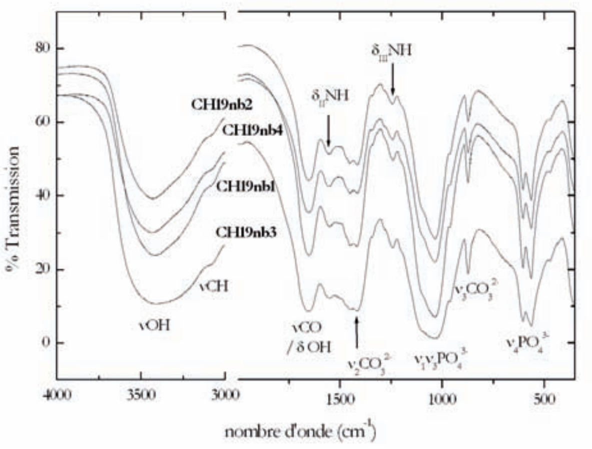


Fig. 7 - FTIR spectra of unburned bones (AB_CH19nb1_4).

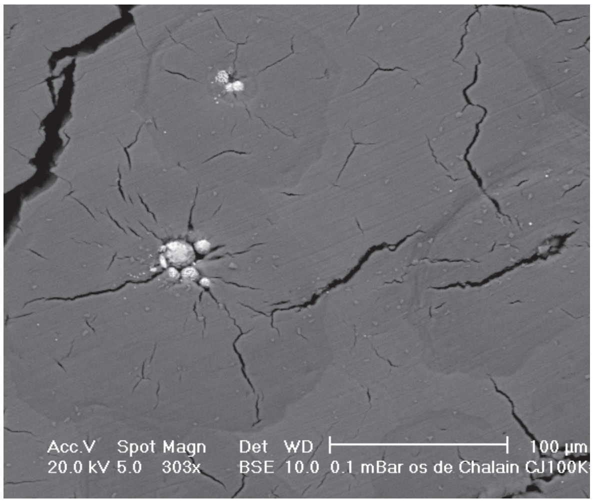


Fig. 8 - Electron micrograph (in backscattering electron mode) showing one of the numerous iron sulphide inclusions in the pores of bone AB_CH19nb3. The size of the aggregates of microcrystals is generally between 10 and 20 µm and that of the individual microcrystals is between 1 and 2 µm. The average stoichiometry of the iron sulphides in this specimen corresponds to $FeS_{\sim 2}$.

microcrystals, measuring approximately one µm and having a stoichiometry varying between $FeS_{1.5}$ and FeS_2, generally form agglomerates between 5 et 20 µm in size (fig. 8.) This observed pyrite has a particular framboidal or botryodial form. The average diameter can be set at 10 µm, which is consistent with other pyrite-based "raspberries" observed in various geochemical surroundings (Wilkin *et al.*, 1996).

The spatial distributions of the chemical elements were measured on transversal cross-sections of bone specimens. The results for the concentration profiles of the bone fragments are given for sample AB_CH19nb3 (fig. 9a-b.) Several types of concentration profiles were measured on the bone cross-sections: flat (homogeneous distribution of concentration); irregular; U-shaped (displaying an enrichment starting at the periosteum and the endosteum); reverse U-shaped (displaying lower concentrations at the periosteum and endosteum relative to the centre); decreasing from the periosteum; increasing form the periosteum.

In general, the unburned bones are characterised by a good preservation of the mineral matter, even though numerous trace elements such as fluorine, iron, manganese, aluminium, silicon, and zinc have been incorporated, and others (sodium and magnesium) leached out during diagenesis. In the latter case, the exogenous chemical species are substituted or adsorbed at the surface of the bone phases. The preservation of the organic phase in

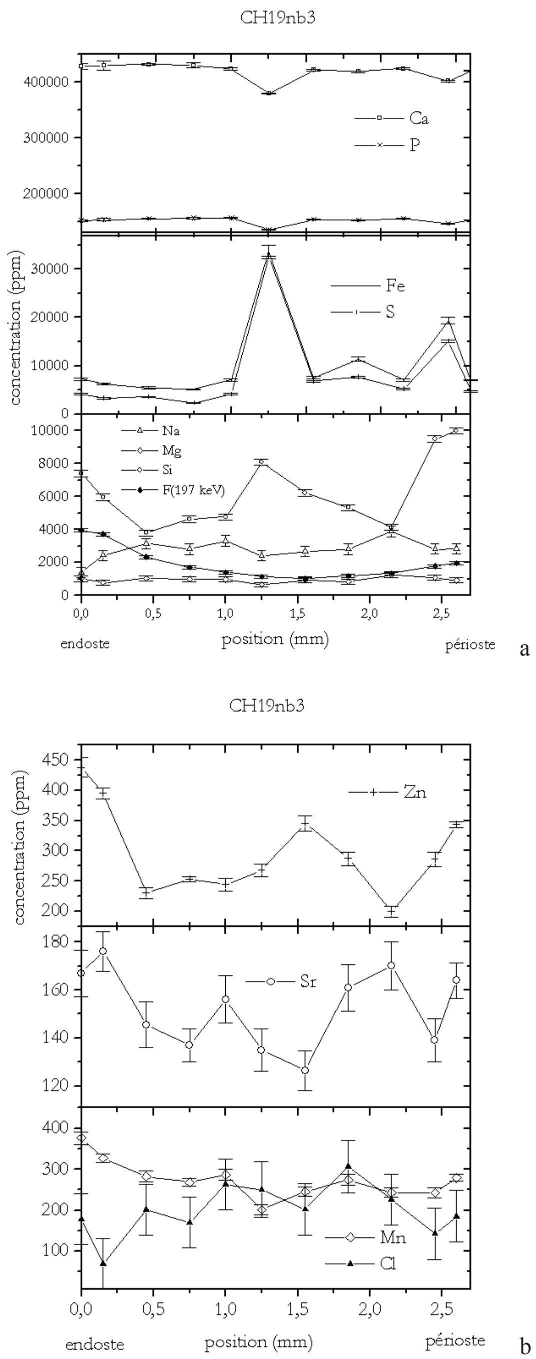


Fig. 9 - a-b : Concentration profiles of the constituent elements of an unburned bone (AB_CH19nb3) measured by microPIXE/PIGE at the micro-beam line extracted from the AGLAE accelerator.

these specimens has been evaluated through the presence of specific peaks in the FTIR spectrum (fig. 7.) On the whole, their width and ratio match those of fresh bone. The organic phase thus seems well preserved, even though a portion of the nitrogen from the collagen has likely been leached out.

The burned bones from the Neolithic village of Chalain 19

The burned bones from Chalain are made up of well crystallised HAP and contain very little organic matter according to the FTIR spectrum and the powder X-ray diffractogram (fig. 10a-b.) They are heavily modified relative to fresh bone. Unlike unburned bones, the samples display an almost complete loss of the histological structure and a high porosity as measured by SEM. The calculated crystallinity indexes are higher than that of a fresh bone (2.0) and those of unburned bones from the same site (2.3-2.5) with average values of 4.5 for CH19b1, 2.8 for CH19b2 and 3.2 for CH19b3.

In addition to their constitutive elements, the bones contain trace elements from the burial environment (tab. 2). Iron, manganese, zinc, strontium, and barium were detected at higher concentrations than those

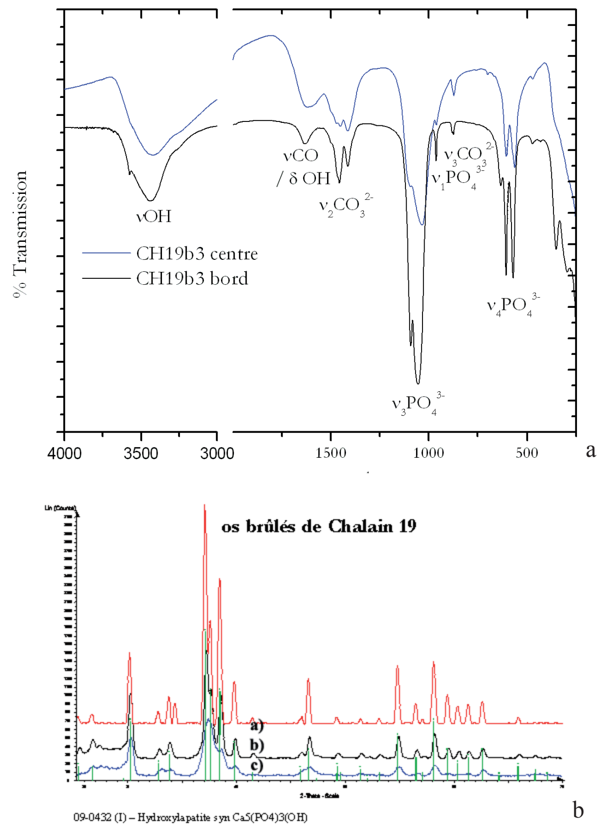
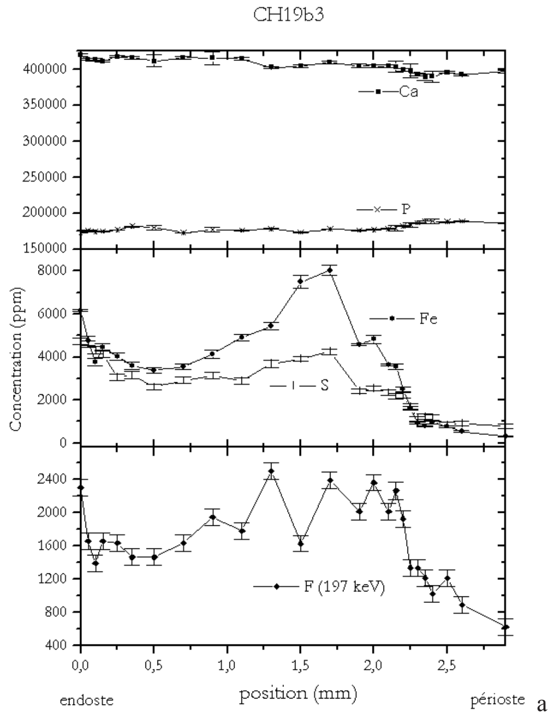
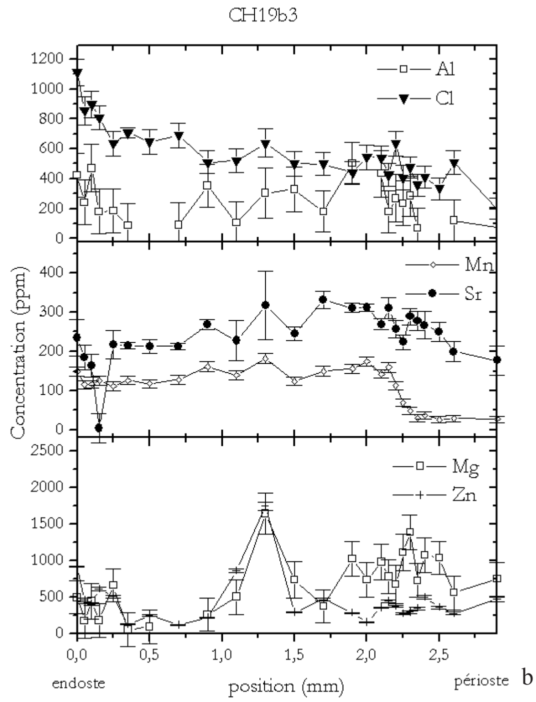


Fig. 10 - FTIR spectrum of a burned bone (edge and centre of specimen ABB_CH19b3) and b. powder X-ray diffractograms (a) ABB_CH19b2, b) ABB_CH19b3, and c) ABB_CH19b1).





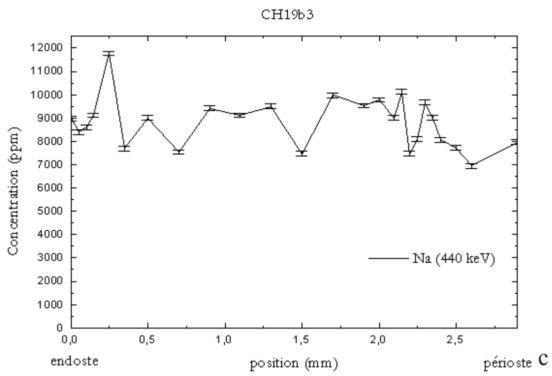


Fig. 11 - a-b-c : Concentration profiles of the constituent elements of a burned bone (ABB_CH19b3) measured by microPIXE/PIGE at the micro-beam line extracted from the AGLAE accelerator.

characterising modern bones. The bones are slightly enriched in calcium. Traces of calcium carbonate ($CaCO_3$) are moreover detected in some specimens.

The spatial distribution of the elements was studied in the case of a burned bone, ABB_CH19b3. The concentration profiles of calcium and phosphorous show a preferential loss of calcium relative to phosphorous towards the edge of the bone. A new type of elemental distribution, showing increasing concentrations from the edge towards the inside of the specimen was observed for a number of exogenous chemical elements such as fluorine, sulphur, iron, and manganese (fig. 11a-c).

Moreover, inclusions of pyrite of $FeS_{\sim 2}$ stoichiometry were detected in the fissures, equivalent to those observed in unburned bones from the same site. These inclusions are also present in the form of agglomerated microcrystals. These framboidal aggregates measure about 20 µm and the individual crystals that make them up measure from 1 to 2 µm (fig. 12.)

Since one of the burned bones more specifically showed an unusual elemental distribution of the exogenous elements having an inverse U-shape, TEM observations were carried out on a sample showing traces of homogeneous heating (ABB_CH19b1, fig.13a), as well as at the edge and at the core of a sample showing heterogeneous heating (ABB_CH19b3, fig.13b-c.) It is hoped that these phenomena of specific enrichment can be linked to a highly variable crystallinity within the bone. The electron micrographs show large size crystals with polygonal morphology in the homogeneously burned sample (ABB_CH19b1) and near the surface of the heterogeneously burned sample (ABB_CH19b3), while at the core of the latter sample, irregular or needle-shaped crystals were detected. In addition, crystalline phases rich in F, probably in CaF_2 form, were detected very locally.

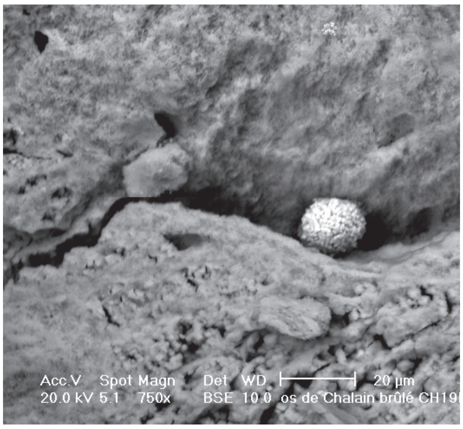


Fig. 12 - Electron micrograph (in backscattering electrons mode) showing a pyrite (FeS_2) inclusion in a fissure of the burned specimen ABB_CH19b3.

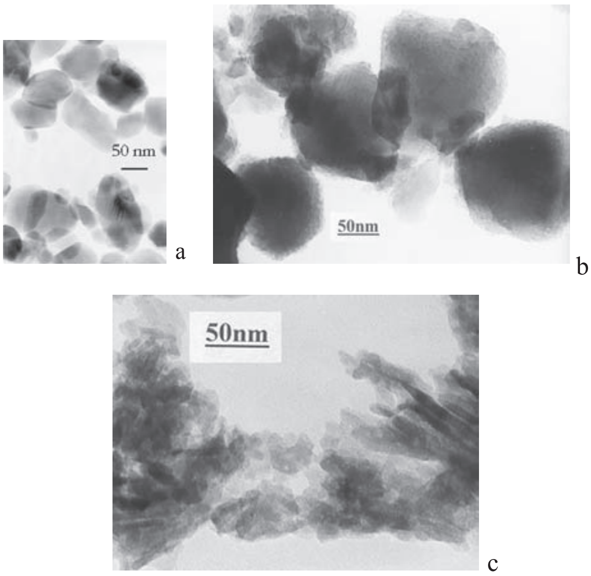


Fig. 13 - Electron micrograph a) of a burned specimen (ABB_CH19b1), b) of the edge, and c) of the core of object ABB_CH19b3.

Discussion

Comparison between unburned and burned bones from station 19 of Chalain Lake

The flood environment of Chalain 19 can be considered as hydrologically fluctuating. Periods of submersion alternated with periods of emersion, indicating a large flow of material as the authors Hedges & Millard, 1995 propose for this type of conditions. During submersion, a seasonal influx of dissolved material in the water and dissolution/hydrolysis, as well as recrystallisation/repolymerisation processes, take place; during emersion, on the other hand, leaching of elements and precipitation of secondary phases by supersaturation occur. Erosion, if it occurs, should lead to relatively porous, recrystallised, and carbonated samples because the site is found in an environment rich in lake chalk.

The signs of change in the case of unburned bones are the partial modifications of their mineral and organic phases. They display numerous exogenous chemical species adsorbed or substituted on the bone material. On the other hand, burned bones are strongly modified in their organic phase and the mineral phase also underwent transformations induced by heating prior to burial; they show a relatively “pure” recrystallised mineral phase free of preserved organic material. On the one hand, in comparison with unburned bones, the exogenous chemical species are trapped to a larger extent in the pores, created by the degradation of the organic material and the recrystallisation of the apatite during heating, in the form of various inclusions like pyrite and calcite; on the other hand, the high crystallinity of the apatite of burned bones makes them less subject to the dissolution-recrystallisation process, which limits the introduction and adsorption of exogenous elements in the bone’s apatite.

The formation of pyrite, observed in both types of burned and unburned bones, takes place directly after the disposal of the bones in the surface sediments. The archaeological layer in question was thus covered by a deposit of calcium carbonate by sedimentation. During the first years of burial, the environment must have been sufficiently reductive with an abundance of organic material rich in sulphur and iron to facilitate the formation of pyrite. As already observed in many cases (Turner-Walker, 1999), the pyrite remains stable afterwards and is found in the form of “raspberries” in these bones, even after the emersion of the site, and the subsequent oxidation, as was the case at Chalain 19 starting in 1904 due to the gradual drying associated with the lowering of the lake level. This corresponds rather well with the environmental situation determined through the study of climatic fluctuations reconstructed from the fluctuations of the lakes levels

	ABB_CH19b1 mean concentration (ppm)	*ABB_CH19b2* mean concentration (ppm)	*ABB_CH19b3* mean concentration (ppm)	concentration range (ppm)	*MB (modern sheep bone)* Concentration moyenne ppm)	concentration range (ppm)
F	510±100	4520±100	1670±100	600-2500	550	0-1200
Na	5110±600	9500±900	8800±900	6950-11800	8500	7600-9100
Mg	900±300	1600±260	-	-	7800	7200-8700
Al	-	1700±170	-	-	-	-
Si	2500±220	-	-	-	-	-
P	186000±2800	178000±2000	180000±1260	172000-189000	179 000	17,8-18,0 %
S	1280±170	1760±130	2670±180	800-4250	1600	1200-1900
Cl	1220±120	290±80	570±90	200-810	750	650-820
Ca	396000±6700	399000±5200	405300±3200	389000-417000	384000	38,2-38,6 %
Mn	20±10	200±10	110±15	25-180	-	
Fe	380±30	1120±10	3500±100	310-8020	-	
Zn	40±5	710±10	440±20	110-1740	130	120-140
Sr	120±10	200±10	240±20	10-330	250	180-280
Ca/P	2,1	2,2	2,3		2,1	

Tab. 3 - Average elemental composition of the burned bones from Chalain 19 determined by PIXE/PIGE. - = below the method's detection limits.

sample	colour	estimated temperature by the colour of the sample (°C)	estimated heating temperature by SF(IR) (°C)	estimated heating temperature by analysis of the X-ray diffractograms (°C)
ABB_CH19b1	white	700-940	800-940 (4,5 ± 0,1)	700
ABB_CH19b2	brown black blueish	400	300-400 (2,8 ± 0,1)	300
ABB_CH19b3	black gray	500-600	500-600 (3,2 ± 0,1)	550

Tab. 4 - Estimate of the heating temperature of archaeological specimens derived from powder X-ray diffractograms, from the reference standard established by infrared spectroscopy (Reiche *et al*., 2007), and from the colour of the specimens.

and the correlation of these data with those of the concentration of residual ^{14}C in the atmosphere (Arbogast *et al*., 1996). According to this work, the installation of Neolithic villages is closely tied to the climatic fluctuations; the dynamics of occupation of the shores are consequently regulated by the fluctuations in the lake level.

We can conclude that the unburned and burned bones display common characteristics that are linked to the geochemical environment in which they are buried. One example is the presence of pyrite in both cases. They also display differences in the degree of preservation that are due to heating of one type in contrast with the other. Burned bones are more porous because they have almost no organic material left; consequently numerous inclusions are trapped within their structure. Relatively few chemical species are adsorbed or substituted in the apatite crystals due to the increase of their size caused by heating. Unburned bones are less porous and still contain some organic material. The exogenous chemical species are found adsorbed or as inclusions in the still intact channels.

Estimation of the heating temperatures sustained by the burned bones before burial determined through an evaluation of the crystallinity of the bone apatite

According to our reference standard (Reiche, 2000; Reiche *et al*., 2007), the bone ABB_CH19b1 was heated to at least 700°C, ABB_CH19b2 to 300°C, and ABB_CH19b3 to 550°C. The bones therefore reached relatively high temperatures prior to burial; these temperatures match quite closely those achieved in fireplaces or in a natural fire (Bennett, 1999). The reference standard is, however, limited to temperatures above 300°C because the changes in the mineral phase alone are not detectable below this temperature (Chaudefaux & Reiche, 2009) For this reason, bone ABB_CH19b2 could also have attained its crystallinity as a result of modification processes in the soil. Nevertheless, its superficial appearance and its colour are also indicative of heating. The bones acquire a white colour staring at 700°C, confirming that only bone ABB_CH19b1 was heated to at least 700°C.

The crystallinity of the apatite of the burned bones shows that they underwent various processes before and after burial. Bone ABB_CH19b1, heated to at least 700°C, would have had to be in direct contact with the heat source, meaning it was burned directly, to reach its level of recrystallisation. For specimen ABB_CH19b2 and b3, several processes can be hypothesised. Either the bones were buried and then heated in the course of a fire, or they were thrown into a fireplace that could reach these temperatures. In effect, Bennett (1999) has shown that sand and clay layers can reach temperatures of 400-500°C at a depth of 5 cm during fires in a fireplace or in open, natural fires.

Heterogeneity of the effects of heating on bones demonstrated by the measurement of microPIXE/PIGE concentration profiles

The microPIXE/PIGE analyses show the distribution of chemical elements in bone remains at the major, minor, and trace level. Various types of distribution can thus be observed depending on the element considered and the conditions of the bone before burial (burned or unburned.) In fact, inverse U-shaped elemental distributions, that is with a concentration of the element considered higher in the core of the sample than at the outside edges, have been observed only very rarely on cross sections of ancient bones. Two phenomena can in principle lead to such a profile. In the case of a constituent element of the bone, like sodium or magnesium, their leaching during diagenesis can give rise to these profiles. With regard to exogenous elements, the explanation of the phenomenon is more complex. It would seem that generally this type of profile is only observed on burned bones that display heterogeneities of the heat-induced changes. Due to the finite rate of heat diffusion, especially in the cases of short duration heating, the heat does not homogeneously modify the structure and the chemical composition of the bone specimen. Its surface reaches higher temperatures than its core. Consequently, the loss of organic material and the heat induced recrystallisation of the bone apatite are more pronounced at the edges than at the centre of the object. The differential loss of the combustion products limits the growth of the apatite crystals. The crystals attain larger sizes towards the edge of the sample than at the centre. For this reason, the exogenous chemical species like fluorine, iron, or sulphur found in the interstitial water at the burial site can attach themselves preferentially to the smaller crystals at the centre, which offer a much larger specific surface (100-200 $m^2.g^{-1}$), than to the large crystals at the edge of the porous specimen. Moreover, the small crystallites at the centre display a higher solubility than the large crystals and this allows them to react to a larger extent by dissolution/recrystallisation with the chemical species of the surroundings. However, unburned bones could also display this type of profile for the exogenous chemical elements, especially if during the object's history, the burial or conservation conditions changed and dissolution/recrystallisation phenomena occured preferentially at the surface of the object. The observation of inverse U-shape concentration profiles is not sufficient by itself, therefore, to prove heating of the bone specimens; it can nonetheless corroborate other indications of heating such as the observation of high crystallinity.

The observation of the same trends of elemental distribution in the burned bones of another lacustrian Neolithic site corroborates these results. In the course of our research, we have had at our disposal three bone samples collected at another Neolithic site at Gletterens (Neuchâtel Lake, Switzerland) thanks to Denis Ramseyer (*Service Archéologique Cantonal* [Canton Archaeological Service], Fribourg). These bones, dated to approximately 3000 years BC, probably belong to small vertebrates. This settlement is entirely trapped in a thick fine sand layer and the remains remained in a humid layer at the level of a water table, which explains the good preservation of the specimens (Reiche *et al*., 2002).

Conclusions and archaeological prospects

This study has demonstrated the archaeological importance of the analysis of burned bones at a microscale, and even nanoscale, for understanding diagenetic processes and for the assessment of their potential to yield information. Burned bones are also well preserved in archaeological contexts, but display specific diagenetic modifications; they can thus be utilised also to infer archaeological information. The Rose-Marie Arbogast method, consisting of using the number of bones in an archaeological layer to estimate the relative roles of hunting and animal husbandry in the diets of Neolithic humans is also applicable when burned bones are utilised (Arbogast, 1997; Arbogast & Pétrequin, 1997).

Moreover, our results demonstrate that when trying to understand heat-induced transformations, it is important to take into account the heterogeneities of the structure and chemical composition caused by heating. The study, through various analyses at the core and at the edge of the specimens, makes it possible to gather information not only on the possible heating of the objects and on the temperatures reached, but also to refine the information obtained, specifically concerning the precise heating method (long or short duration, direct flame, fire, multiple combustions in a fireplace, etc.), since this approach makes it possible to determine and to precisely localise the heat-induced modifications undergone by the specimen.

It would be interesting to develop a heated bone reference standard that would take into account the various thermal treatments and which would also allow a more refined detection of the heat-induced modifications of the organic phase at lower temperatures (Chadefaux & Reiche, 2009). It would be possible to conduct a series of bone heating experiments in order to model the relationships between the various types of modifications (size and morphology of the crystals, distribution of chemical species, crystallinity index, and colour). This new reference standard could lead to a refinement of the study of various archaeological problems, such as that of funerary practices by incineration, or that of the domestication of fire.

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References

Arbogast R.-M., Magny M. & Pétrequin P. 1996 - Climat, cultures céréalières et densité de population au Néolithique : Le cas des Lacs du Jura Francais de 3500 à 2500 av. J.-C. *Archäologisches Korrespondenzblatt*, 26 : 121-144.

Arbogast R.-M. 1997 - La grande faune de Chalain 3. *In* : *Les sites littoraux néolithiques de Clairvaux-les-Lacs et de Chalain (Jura), Chalain station III. 3200-2900 av. J.-C.*, P. Pétrequin (ed). Paris, Editions de la Maison des Sciences de l'Homme, 2 : 641-691.

Arbogast R.-M. & Pétrequin P. 1997 - Approvisionnement carné et équilibres agropastoraux : l'exemple des communautés lacustres du Néolithique final de Chalain « Station 3 » (Jura, France). *Anthropozoologica* 26,25 : 391-398.

Baud C.A. & Tochon-Danguy H.J. 1985 - Les caractéristiques physico-chimiques de l'ivoire et leurs modifications par traitement thermique. *Bulletin de l'association pro Aventico,* 29 : 49-52.

Bennett J.L. 1999 - Thermal alteration of buried bone. *Journal of Archaeological Science,* 26 : 1-8.

Chadefaux C. & Reiche I. 2009 - Archaeological bone from macro- to nanoscale. Heat-induced modifications at low temperatures. *Journal of Nano Research*, 8 : 157-172.

Cui F.-Z., Li Y. & Ge J. 2007 - Self-assembly of mineralized collagen composites. *Materials Science and Engineering R: Reports,* 57 : 1-27.

Gupta H., Wagermaier W., Zickler G.A., Ra-Ben Aroush D, Funari S.S., Roschger P., Wagner D. & Fratzl P. 2005 - Nanoscale deformation mechanism in bone ». *NanoLetters,* 5 : 2108-2111.

Hedges R.E.M. & Millard A.R. 1995 - Bones and groundwater: towards the modelling of diagenetic processes. *Journal of Archaeological Science,* 22 : 155-164.

Lebon M. 2008 - *Caractérisation par spectrométrie infrarouge à transformée de Fourier des ossements chauffés en contexte archéologique - Comparaison entre référentiel moderne et matériel fossile, implications diagénétiques*, Thèse de doctorat, Muséum National d'Histoire Naturelle, 339 pp.

Michel V., Ildefonse P. & Morin G. 1996 - Chemical and structural changes in *cervus elaphus* tooth enamels during fossilization (Lazaret cave) : a combined IR and XRD rietveld analysis. *Applied Geochemistry,* 10 : 145-159.

Person A., Bocherens H., Mariotti A. & Renard M. 1996 - Diagenetic evolution and experimental heating of bone phosphate. *Palaeogeography, Palaeoclimatology, Palaeoecology*, 126 : 135-149.

Pétrequin P. & Pétrequin A.M. 1988 - *Le Néolithique des Lacs. Préhistoire des lacs de Chalain et de Clairvaux (4000-2000 av. J.-C.).* Paris, éditions Errance, 285 pp.

Pétrequin P. (dir.) 1997 - *Les sites littoraux néolihiques de Clairvaux-Les-Lacs et de Chalain (Jura) III. Chalain Station 3. 3200-2900 av. J.-C.* Paris, Edition de la Maison des Sciences de l'Homme, 2 vol.

Pétrequin P., Arbogast R.-M., Bourquin-Mignot C., Lavier C. & Viellet A. 1998 - Demographic growth, environmental changes and technical adaptations : responses of an agricultural community from the 32nd to the 30th centuries BC. *World Archaeology,* 30 : 181-192.

Reiche I., Favre-Quattropani L., Calligaro T., Salomon J., Bocherens H., Charlet L. & Menu M. 1999 - Trace element composition of archaeological bones and post-mortem alteration in the burial environment. *Nuclear Instruments and methods in Physics*, 150 : 656-662.

Reiche I. 2000 - *Processus physico-chimiques d'altération des ossements et ivoires anciens*. Thèse de doctorat, Université Pierre et Marie Curie Paris VI., 278 pp.

Reiche I., Vignaud C., Favre-Quattropani L. & Menu M. 2002a - Fluorine analysis in biogenic and geological apatite by analytical transmission electron microscopy and nuclear reaction analysis. *Journal of Trace and Microprobe Techniques,* 20 : 211-231.

Reiche I., Vignaud C. & Menu M. 2002b - Crystallinity of ancient bone and dentin. New insights by transmission electron microscopy. *Archaeometry,* 44 : 447-459.

Reiche I., Favre-Quattropani L., Vignaud C., Bocherens H., Charlet L. & Menu M. 2003 - A multi-analytical study of bone diagenesis : the Neolithic site of Bercy (Paris, France). *Measurement Science and Technology,* 14 : 1608-1619.

Reiche I., Chadefaux C, Vignaud C. & Menu M. 2007 - Les matériaux osseux archéologiques Des biomatériaux nanocomposites complexes. *L'actualité chimique,* 312-313 : 86-92.

Reiche I. & Chalmin E. 2008 - Synchrotron radiation and cultural heritage : combined XANES/XRF study at Mn K-edge of blue, grey or black coloured palaeontological and archaeological bone material. *Journal of Analytical Atomic Spectrometry,* 23 : 799-806.

Shahack-Gross R., Bar-Yosef O. & Weiner S. 1997 - Black-coloured bones in Hayonim Cave, Israel : differentiating between burning and oxide staining. *Journal of Archaeological Science,* 24 : 439-446.

Shemesh A. 1990 - Crystallinity and diagenesis of sedimentary apatites. *Geochimica et Cosmochimica Acta,* 54 : 2433-2438.

Shipman P., Foster G. & Schoeninger M. 1984 - Burnt bones and teeth : an experimental study of color, morphology, crystal structure and shrinkage. *Journal of Archaeological Science,* 11 : 307-325.

Sillen A. & Parkington J. 1996 - Diagenesis of bones from Eland's Bay Cave. *Journal of Archaeological Science*, 23 : 535-542.

Stiner M.C., Kuhn S.L. Weiner S. & Bar-Yosef O. 1995 - Differential burning, recrystallisation, and fragmentation of archaeological bone. *Journal of Archaeological Science,* 22 : 223-237.

Termine J. D. & Posner A. S. 1966 - Infrared determination of the percentage of crystallinity in apatite calcium phosphates. *Nature,* 211 : 268-270.

Turner-Walker G. 1999 - Pyrite and bone diagenesis in terrestrial sediments : Evidence from the West Runton Freshwater Bed. *Bulletin of the geological Society of Norfolk,* 48 : 3-26.

Wagermaier W., Gupta H., Gourier A., Burghammer M., Roschger P. & Fratzl P. 2006 - Spiral twisting of fiber orientation inside bone lamellae. *BioInterphases,* 1 : 1-5.

Weiner S., Goldberg P. & Bar-Yosef O. 1993 - Bone preservation in the Kebara Cave, Israel using On-Site Fourier Transform Infrared Spectrometry. *Journal of Archaeological Science,* 20 : 613-627.

Wilkin R.T., Barnes H.L. & Brantley S. L. 1996 - The size distribution of framboidal pyrite in modern sediments : An indicator of redox conditions. *Geochimica and Cosmochimica Acta,* 60 : 3897-3912.

Wright L. E. & Schwarcz H. P. 1996 - Infrared and isotopic evidence for diagenesis of bone apatite at Dos Pilas, Guatemala : Palaeodietary implications. *Journal of Archaeological Science,* 23 : 933-944.

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CHARACTERIZATION OF BURNT BONES IN ARCHAEOLOGICAL CONTEXT: A COMPARATIVE STUDY OF MODERN AND FOSSIL MATERIAL BY INFRARED SPECTROSCOPY

Matthieu LEBON

abstract

The identification of burnt bones in an archaeological context can entail characterization techniques such as infrared spectroscopy. However, it is often difficult to clearly distinguish bones burnt at low temperatures (<500oC) because the alterations that occur during heating are similar to those that occur during burial. Moreover, these analyses are generally carried out on samples reduced to powder and they do not permit us to take into account the heterogeneity of the bone material.

In order to address these various problems, we became interested in the$v_1v_3PO_4$ domain, whose study, on modern bones burnt under experimental conditions, allowed us to establish parameters that make it possible to evaluate the crystallinity of the samples (1030/1020 ratio) and to gather information on the crystal structure of the mineral phase (wavenumbers of the peaks centred near 961, 1022, 1061, and 1092 cm-1.) In particular, the wavenumbers of these various peaks have made it possible to identify bones burnt at temperatures as low as 250oC in the Magdalenian levels of the site of Bize-Tournal, while crystallinity by itself allowed only the clear identification of bones burnt above 500oC. This method can therefore contribute to an improved identification of bones burnt at low temperatures in an archaeological context. Moreover, this analytical protocol will make it possible to study the spatial variations in the composition of bone material by infrared micro-spectroscopy and thus to define and distinguish the alterations occurring during heating and during diagenesis.

Keywords : burned fossil bones, modern reference base, carbonated hydroxypatite, FTIR, FTIR imagery, $v_1v_3P0_4$ domain

Introduction

Since humans first started using fire, burnt bones have represented a large part of the burned organic residues associated with archaeological levels. This material can result from various activities such as cooking meat, utilizing bones as fuel, or certain funerary practices (Buikstra & Swegle, 1989; Théry-Parisot *et al.,* 2002; Cain, 2005.) These different activities are manifest by different heating intensities and durations whose estimation can be crucial to the identification of the origin of burned bone materials.

The changes in colour or texture undergone by the bone during heating can make it possible to identify the burnt bones in most cases, and to estimate the maximum temperatures reached. Different degrees of alteration have thus been established on the basis of the changes in the colour of bones during heating (Shipman *et al.,* 1984; Nicholson, 1995; Stiner *et al.,* 1995.) The colour of the bones begins to evolve at 200°C, with the bone assuming an increasingly dark brown colour and achieving almost complete carbonisation at nearly 350°C. Above this temperature, the carbonised organic material is gradually eliminated and the bone's colour becomes progressively lighter, becoming grey (500-650°C) and then white once the calcination is complete (700°C) (fig.1.)

Various analytical techniques have been developed to remedy this situation and to reliably characterise burnt bones. This is notably the case of x-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and electron microscopy, which have made it possible to follow the significant modifications made to the structure and composition of the bone's mineral phase during heating. Notably, an increase in crystallinity (size and degree of perfection of the crystallites) at high temperatures has been demonstrated (Shipman *et al.,* 1984; Stiner *et al.,* 1995; Person *et al.,* 1996; Reiche *et al.,* 2002; Piga *et al.,* 2008.)

The composition of bones is indeed deeply modified during heating. *In vivo* bone is a complex material made up of a mineral phase associated with an organic matrix composed mainly of collagen. The composition and structure of the mineral phase of bone are close to those of hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) but they differ due to the presence of numerous impurities. The major constituents (calcium and the phosphate and hydroxyl groups) can be replaced by minor elements (Na^+, K^+, Mg^{2+}, CO_3^{2-}, HPO_4^{2-}…) or by trace elements (Sr^{2+}, Ba^{2+}, Pb^{2+}, Zn^{2+}….) These impurities are at the origin of deformations of the crystal lattice of the mineral phase and they set a limit to the size of crystallites. For example, carbonates can represent over 5% of the total weight of the mineral phase and this phase can

Fig. 1 - Examples of various categories of heat-induced alterations as defined by Stiner *et al.*, 1995.

However, it appears that various taphonomic processes during burial can influence the appearance of bones. This is the case, for example, of iron and manganese oxides, which are responsible for a brown or black colouring of bone surfaces, making difficult the identification of burnt bones in archaeological contexts (Shahack-Gross *et al.,* 1997).

also be described as a slightly crystallised carbonate-hydroxyapatite (LeGeros, 1981).

The organic material, composed mostly of collagen, is quickly destroyed during burning and its carbonisation is complete at about 300°C. Above 450°C, the products of carbonisation of the organic material are in turn gradually eliminated and only the mineral phase persists above 600°C. The mineral phase is also affected during heating: water is gradually eliminated starting at 100°C and the proportion of carbonates decreases rapidly above 300°C. The result is a gradual decrease of the number of defects in the mineral phase, defects that become negligible near 500°C. Their disappearance induces a characteristic increase in the size of crystallites at high temperatures (Person *et al.,* 1996; Hiller *et al.,* 2003.)

Infrared spectroscopy is a technique especially suited to the characterisation of these modifications in the composition of the mineral phase during heating because it allows the study of the structure and composition of this phase, as well as those of the organic phase when it is preserved (Fig. 2.) Furthermore, this technique is used particularly in archaeology to determine the state of preservation of the mineral phase of fossil bones through an assessment of their crystallinity. This parameter can be measured by calculating the splitting factor (SF), calculated from the degree of separation of the individual absorbance peaks ν4 of the phosphate groups centred near 565 et 605 cm^{-1} (Termine and Posner, 1966 ; Weiner and Bar-Yosef, 1990.) The SF value augments with an increasing degree of crystallinity. However, the utilisation of the splitting factor has shown its limitations both in the context of the identification of burnt bones and in the assessment of the state of preservation of bones recovered in an archaeological context. It seems that the increase in crystallinity that occurs during exposure of the bones to edaphic or climatic elements is of the same magnitude as that which occurs during moderate heating (below 500°C.) Therefore, the identification of the lower heating stages of fossil bones by infrared spectroscopy remains difficult (Stiner *et al.,* 1995.) Moreover, it seems that the crystallinity cannot reflect all modifications undergone by the mineral phase during diagenesis (Trueman *et al.,* 2008.)

Finally, this method of analysis requires sampling in a powder form and therefore does not allow us to take into account the heterogeneity of the bone material. Over the past few years, it has become possible to combine infrared spectroscopy with microscopy to study the spatial distribution of the constituents of a material with a resolution of about 50 to 100 µm in the laboratory. If applied to bones, this technique will allow a better understanding of the modifications that occur in the bone tissue during heating. The technical specifications of these instruments generally do not allow the study of the spectral region between 600 and 400 cm^{-1} and therefore make it impossible to calculate the SF currently used to evaluate the crystallinity of the samples.

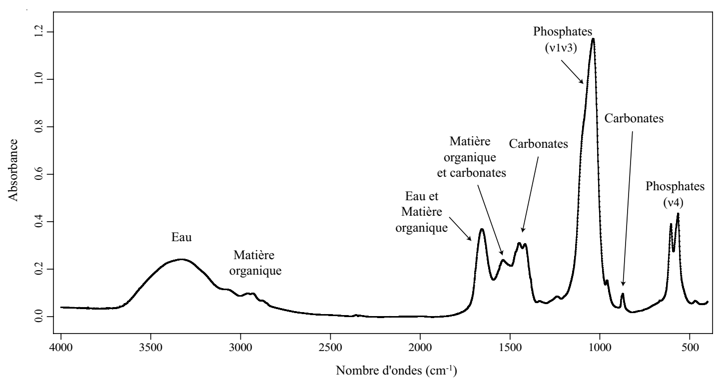

Fig. 2 - Infrared spectrum of a modern unburnt bone. The various absorption peaks make it possible to quantify the various components of the mineral and organic phases of the bone.

The goal of this work is to present the development of an infrared spectroscopic method that allows both a precise identification of bones burnt at low temperatures and the application of infrared micro-spectroscopy to the study of fossil bone material.

The ν4 phosphate domain, on which the calculation of the SF is based, is not the only phosphate domain accessible in the infrared range. The $\nu_1\nu_3PO_4$ domain, which is characterised by a large and intense band that ranges between 1200 and 900 cm^{-1}, has been the object of numerous studies in infrared micro-spectroscopy, especially in the biomedical field. Various studies have shown that this domain can be decomposed into over a dozen component peaks, and that their number and wavenumber could provide information on the composition of the bone mineral phase (Rey *et al.,* 1991; Leung *et al.,* 1990; Gadaleta *et al.,* 1996.) In particular, it has been shown that the ratio of the areas or amplitudes of the components centred near 1030 et 1020 cm^{-1} makes it possible to estimate the crystallinity of bones *in vivo* (fig. 3a) (Paschalis *et al.,* 1996 ; Boskey and Pleshko-Camacho, 2007.) These different peaks, very near each other, are difficult to observe and their identification requires the use of mathematical treatments, such as Fourier self-deconvolutions, or mathematical derivatives of the spectrum (Fig. 3b) (Rey *et al.,* 1991 ; Gadaleta *et al.,* 1996.)

The analytical protocol proposed here is based on a qualitative and quantitative study of the $\nu_1\nu_3PO_4$ domain carried out by jointly using the derivative infrared spectra and the spectral decompositions. This method was developed with an experimental reference base of modern bones heated to various temperatures and for various lengths of time in order to better understand the changes that occur in the mineral phase during heating. It was then possible to compare the results obtained to those obtained from the fossil bone materials from the Magdalenian levels of the site of Bize-Tournal (Aude).

Materials and Methods

The first stage of this work consisted of creating a reference base of modern bones composed of of ox humerus diaphyses. After removal of the bone marrow and flesh, the cortical part of the bone was ground up in order to obtain a particle size between 1.25 and 2 mm. Sub-samples of about 0.75 g were then heated for 15, 30, 45, 60, 90, and 120 minutes to temperatures between 120°C and 900°C in a muffle furnace under oxidising conditions. This reference standard after heating is shown in figure 4.

The fossil samples studied come from the Magdalenian levels of the site of Bize-Tournal (ensemble IV, level G, layer 1.) These samples, which are fairly recent (15,000 BP), display a good state of preservation, which motivated their selection for this study. The traces of carbonisation and calcination are numerous and easily identifiable by microscopy (Tavoso, 1987; Patou-Mathis *et al.,* 1999; Magniez, in press) This material can thus constitute a fossil reference base that allows testing of the method developed. In order to facilitate the comparison between the modern reference and the fossil material, the Bize-Tournal samples were classified on the basis of their colour into the seven categories of heating defined by Stiner *et al.* (1995) and shown in figure 1. For each of the 23 fossil samples selected, approximately 25 mg of material were collected for the infrared analyses. The size of these samples is small and the heating categories identified macroscopically refer only to the sampled zone.

The infrared data were obtained utilizing the KBr pellet technique. A few milligrams of each sample were ground up in acetone in order to obtain a particle size of less than 5 µm. The pellets were then prepared by mixing 2.5 ± 0.02 mg of this powder brought up to 1 g with potassium bromide (KBr.) Finally, 300 mg of

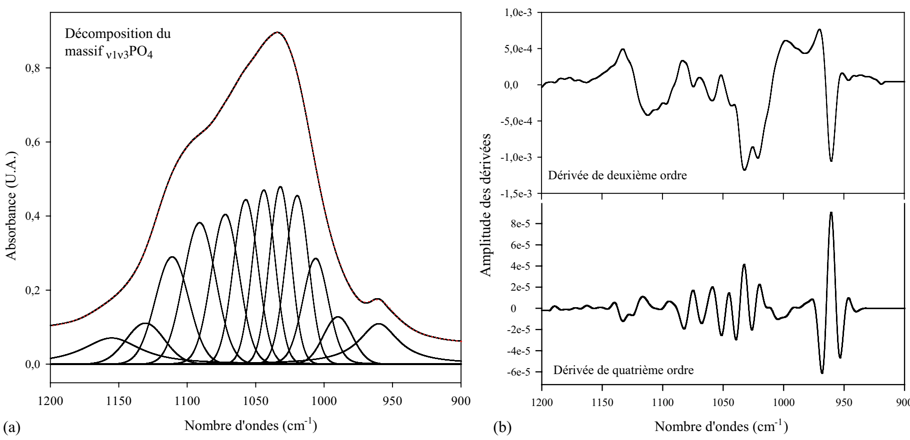

Fig. 3 - (a) Diagram of the various component peaks of the $\nu_1\nu_3PO_4$ domain identifiable from the second and fourth derivative spectra between 900 and 1200 cm^{-1} (b.)

this mixture were pressed at 11T/cm^{-1} for 1.5 minutes (Fröhlich, 1989.) The infrared spectra were recorded on a Vector 22 Bruker spectrometer by accumulating 64 scans with a resolution of 2 cm^{-1}.

The spectral range of the $\nu_1\nu_3PO_4$ domain included between 800 and 1200 cm^{-1} was selected for each specimen. The second and fourth order derivatives of this range of the infrared spectrum were calculated utilising an algorithm of the Savisky and Golay type and a smoothing of respectively 7 and 15 points. The derivatives of the infrared spectra could thus be used to identify the wavenumbers of the components of the $\nu_1\nu_3PO_4$ domain. It was then possible, knowing the wavenumber of these different components, to model this domain starting from simple components of the Gaussian type, placed at the wavenumbers identified in the derivatives, and to adjust the area of each of them by the least square method until the simulated spectrum best matched the experimental one.

It was also possible to assess the crystallinity of the bones by the splitting factor according to the model developed by Weiner and Bar-Yosef (1990) by adding the absorbances of the peaks centred near 565 cm^{-1} and 605 cm^{-1} divided by the absorbance of the baseline between them. Each of these absorbances is measured in relation to the baseline drawn between 500 and 700 cm^{-1} (fig. 5.)

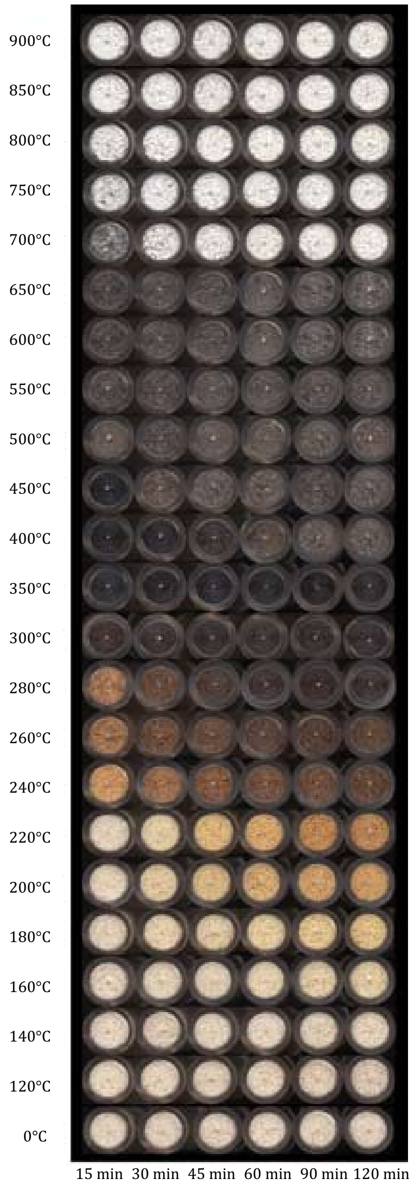

Fig. 4 - Reference base of modern bones heated to between 120 and 900oC for 15, 30, 45, 60, 90, or 120 minutes.

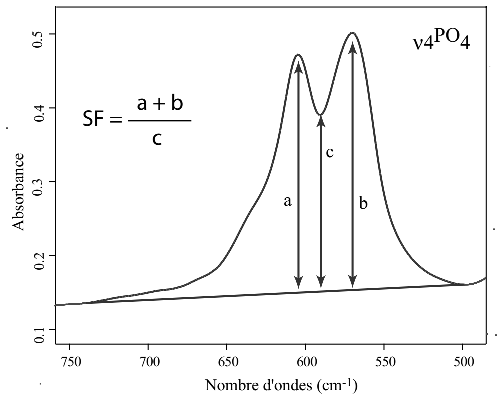

Fig. 5 - Method for measuring the splitting factor according to the model established by Weiner and Bar-Yosef (1990). The splitting factor is calculated from the sum of the absorbances of the two ν_4PO_4 peaks centred near 565 (a) and 605 cm^{-1} (b) divided by the absorbance of the baseline between them (c.)

Study of the modern reference base

Assessment of the crystallinity: 1030/1020 indexes and splitting factor

The work of Gadaleta *et al.,* (1996) and Paschalis *et al.* (1996) has shown that it is possible to assess the crystallinity of the mineral phase of the specimens by measuring the ratio between the area or amplitude of a peak assigned to the phosphates in a well crystallised pure apatite and that of a peak assigned to the phosphates in a poorly crystallised apatite. Figure 6 shows the $\nu_1\nu_3PO_4$ domain of a sample of

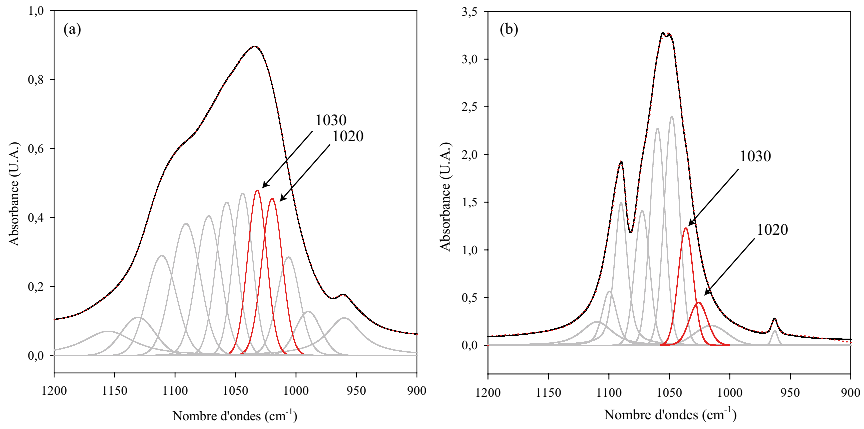

Fig. 6 - Relative absorbance of the peaks centred on average near 1030 and 1020 cm^{-1} for a modern bone: unburnt (a) and heated to 900oC (b.)

unburnt modern bone (a) and of a sample heated to 700°C during 30 minutes (b.) The peaks centred near 1030 and 1020 cm^{-1} have very similar amplitudes in the case of the unburnt samples, indicating the coexistence of a well crystallised mineral phase and of another less well crystallised one, in relatively equal proportions. The $\nu_1\nu_3PO_4$ domain of the bones burnt at high temperature, on the other hand, shows a peak at 1030 cm^{-1} largely dominant relative to that centred near 1020 cm^{-1}. The evolution of this 1030/1020 cm^{-1} ratio as a function of temperature allows the observation of a trend entirely similar to that observed for the splitting factor; these two indexes are indeed correlated ($r^2 = 0.95$; $p<0.001$; fig. 7.) It seems therefore that the 1030/1020 ratio can be used to assess the crystallinity of the burnt bones and that it provides information similar to that obtained through the splitting factor.

Changes in the general appearance of the derivatives

During heating, the $\nu_1\nu_3PO_4$ domain undergoes significant changes that translate notably in the appearance of a peak centred near 1090 cm^{-1}, which becomes clearly distinct starting at 600°C. This peak, visible only in samples heated to high temperatures, makes it possible to distinguish the unburnt samples and those burnt at low temperatures from those heated to at least 600°C (fig. 8a.).

The second and fourth derivatives show much more complex appearances that make it possible to display more subtle changes in this domain. These changes in the appearance of the derivatives allow the identification of at least three temperature ranges: 0-450°, 500-600°C, and temperatures above 600°C (fig. 8b.).

The number of peaks is similar for the unburnt samples and for those burnt up to 450°C. These peaks, identified on the basis of literature data, can be assigned at the same time to phosphate groups located on the one hand in a very pure crystallised hydroxyapatite, and on the other hand in a poorly crystallised carbonated hydroxyapatite. The coexistence of these two phases within the mineral phase is typical of the *in vivo* bones, whose mineral phase contains significant proportions

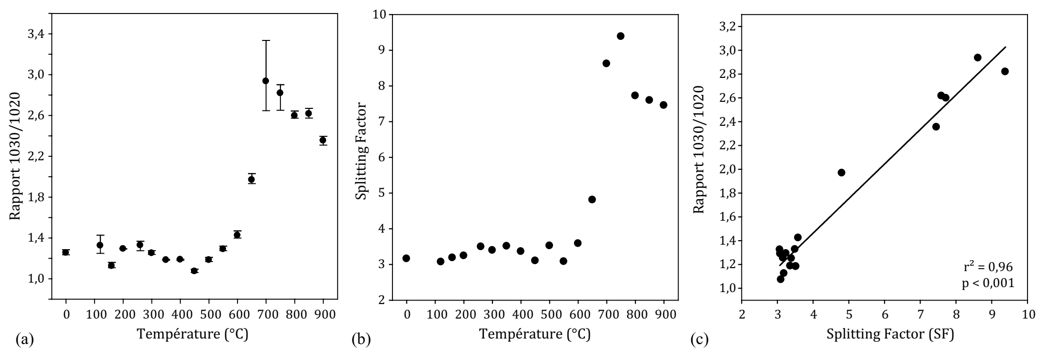

Fig. 7 - Evolution of the value of the 1030/1020 index (a) and of the splitting factor (b) as a function of the temperature for modern specimens heated to between 120 and 900oC for 30 minutes. Correlation between the 1030/1020 ratio and the splitting factor for the same specimens (c.)

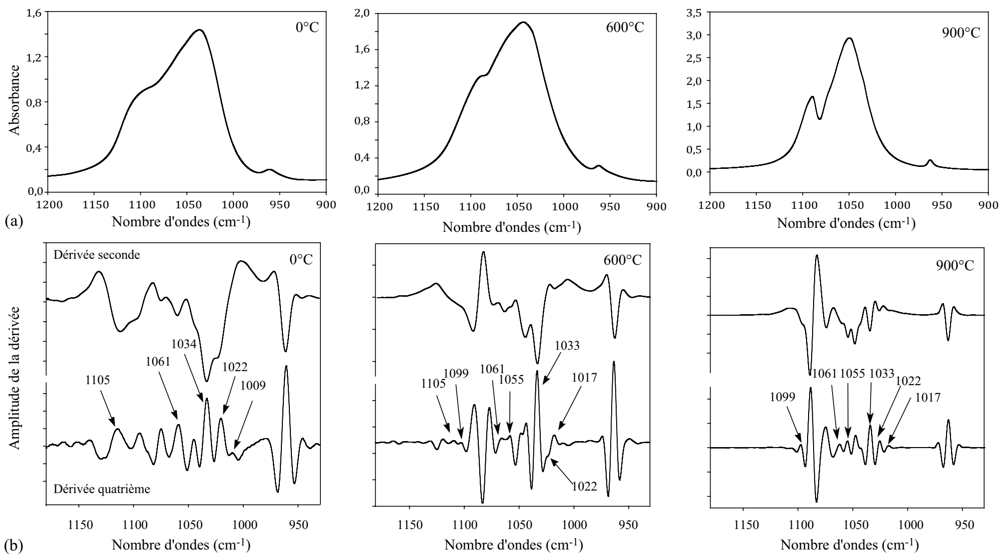

Fig. 8 - Appearance of the $\nu_1\nu_3PO_4$ domain (a) and of the second and fourth order derivatives of this domain (b) for modern specimens: unburnt, and heated to 600oC and 900oC.

of carbonate CO32-) and hydrophosphates (HPO_4^{2}) ions (Young and Holcomb, 1984; Pasteris *et al.,* 2004.).

Near 450°C, the appearance of the derivatives is heavily modified and new peaks appear near 1017, 1056, and 1098 cm^{-1}. These peaks could correspond to a new type of calcium phosphate generated at high temperatures, such as whitlockite, or they could be the result of artefacts of the derivation.

Above 600°C, the derivative spectra display a very different appearance due to the disappearance of several component peaks on the boundaries of the domain: the components centred near 1140 and 1150 disappear at around 650°C and those centred near 1114 and 1128 disappear at around 850°C. These peaks are assigned to the hydrophosphate groups present in the mineral phase of impure apatites (Rey *et al.,* 1991; Gadaleta *et al.,* 1996) and therefore their disappearance at high temperatures indicates in an improvement in the quality of the mineral phase.

Wavenumber variations of the component peaks centred near 961, 1022, 1061, and 1092 cm^{-1}

Among these different components of the $\nu_1\nu_3PO_4$ domain, four peaks display clear wavenumber variations during heating. They are the peaks centred near 961, 1022, 1061, and 1092 cm^{-1}, with a positive shift for the first three peaks and a negative one for the last (fig. 9.). Comparing these data with those from earlier studies on synthetic apatites or *in vivo* bones, it was possible to verify that the wavenumbers observed in our samples, unburnt or burnt at low temperatures, correspond to those generally observed in the literature for poorly crystallised apatites containing CO_3^{2-} and HPO_4^{2-} ions. The wavenumbers observed in our specimens heated to high temperature correspond, on the contrary, to those generally observed for pure, well crystallised apatites (Rey *et al.,* 1991; Paschalis *et al.,* 1996; Pasteris *et al.,* 2004). It seems therefore that the variation in the wavenumber of these peaks indicates an improvement of the quality of the mineral phase during heating (Lebon *et al.,* 2008.).

Study of the fossil material

Assessment of the crystallinity: 1030/1020 indexes and splitting factor

The 1030/1020 index and the splitting factor were calculated for each of the fossil specimens. The values of the splitting factor measured on the unburnt specimens are close to 3.5 (category 0; fig. 10.) For the samples of the heating categories 1 to 4, the measured

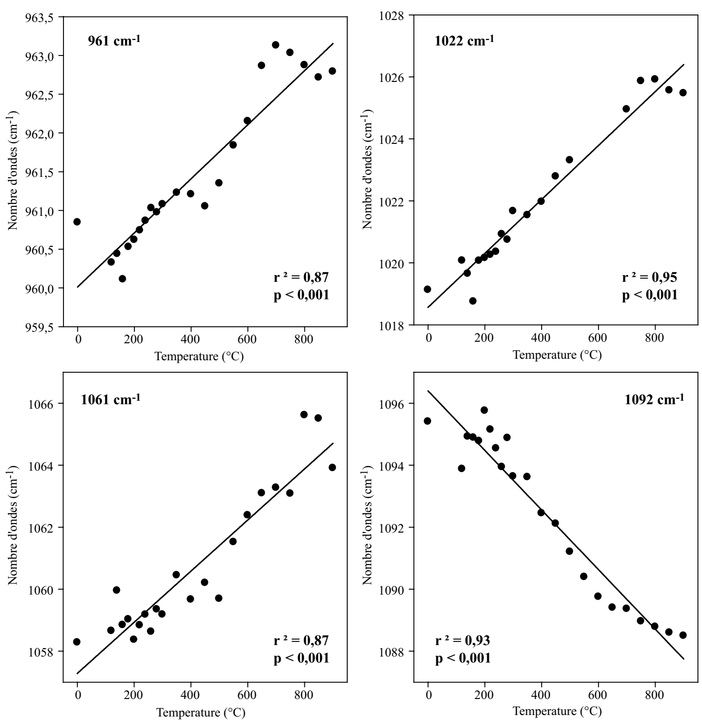

Fig. 9 - Wavenumber shift of the peaks centred near 961, 1022, 1061, and 1092 cm^{-1} as a function of temperature for modern specimens heated for 30 minutes.

values range between 3.55 and 4.14 and therefore are not clearly distinguishable from those of the unburnt specimens. Only the samples of categories 5 and 6 display SF values clearly distinct from those of the unburnt samples, with values approaching 9 for certain samples belonging to category 6.

The 1030/1020 ratio displays values close to 1.2 for the unburnt fossil samples and increases to values close to 2.4 for samples that show complete calcination (stage 6.) The trends observed for these two indexes as a function of the stage of heating are very similar and Figure 10c shows the correlation between the two indexes ($r^2 = 0{,}98$; $p<0{,}001$).

Appearance of the derivative spectra of the fossil specimens

The general appearance of the infrared spectra, and more specifically of the $\nu_1\nu_3PO_4$ domain, of these fossil specimens is quite similar to that of the modern reference specimens. Changes can however be observed in the appearance of the derivatives of the $\nu_1\nu_3PO_4$ domain, as a function of the heating degree determined for these specimens on the basis of macroscopic criteria. In particular, it seems possible to clearly distinguish, by the appearance of the derivative spectra, the unburnt fossil bones or those heated to temperatures lower than 500°C, from those that have been burnt at temperatures between 500°C and 600°C, or at temperatures higher than 600°C (figure 11.).

Wavenumber variations of the component peaks centred near 961, 1022, 1061 and 1092 cm^{-1}

It was also possible to measure the wavenumber of the four peaks centred near 961, 1022, 1061, and 1092 cm^{-1} for each of the fossil samples. The wavenumbers of these peaks as a function of the heating stage of the

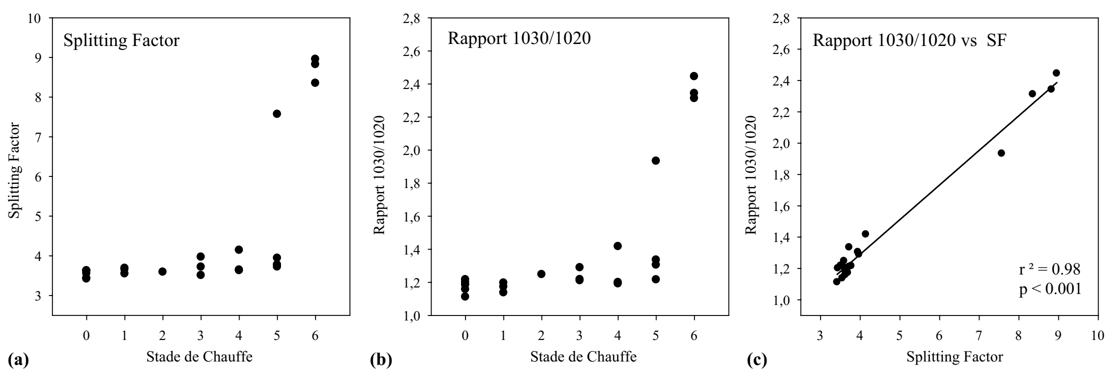

Fig. 10 - Value of the splitting factor (a) and of the 1030/1020 index (b) as a function of the temperature for modern specimens heated to between 120 and 900oC for 30 minutes. Correlation between the 1030/1020 ratio and the splitting factor for the same specimens (c.).

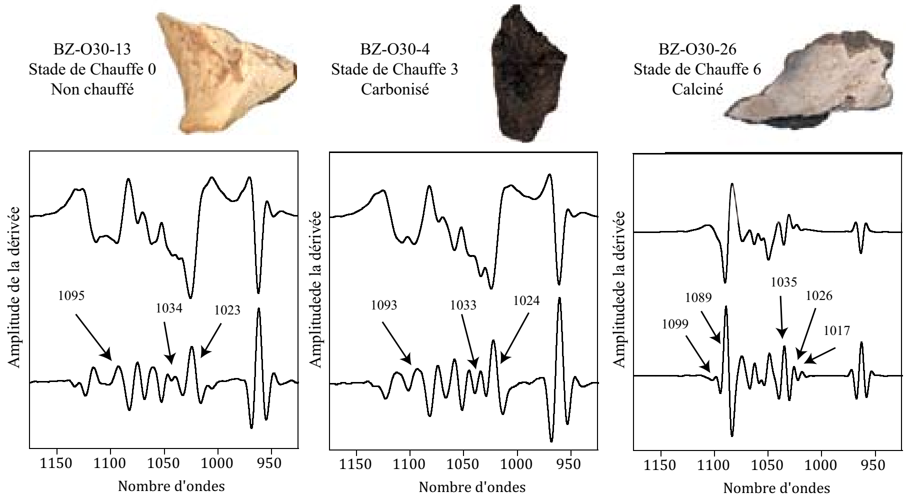

Fig. 11 - Appearance of the second and fourth order derivatives of the $\nu_1\nu_3PO_4$ domain of the specimens BZ-O30-13, BZ-O30-4, and BZ-O30-26 from the Magdalenian levels of the Bize-Tournal site.

specimen are shown in figure 12. For these specimens, the wavenumbers of the first three peaks are correlated to the heating stages to a lesser degree than is the case for the previously studied modern specimens. The peak centred at 1092 cm^{-1} does not show a significant correlation with the stage of heating of the specimen. These weak correlations between the wavenumbers of the peaks and the heating stages make it difficult to identify the burnt bones by this criterion.

Discussion

During burning, the composition of the mineral of bone phase undergoes significant changes that can be shown with characterisation techniques such as infrared spectroscopy.

The identification of burnt bones by infrared spectroscopy generally involves an assessment of the degree of crystallinity through measurement of the splitting factor. The study reported here on the $\nu_1\nu_3PO_4$ domain of a reference base of burnt modern bones and of fossil specimens from Magdalenian levels of the site of Bize-Tournal has made it possible to demonstrate the informative potential of this spectral region for the identification of burnt samples.

It is confirmed that the evolution of the composition of the mineral phase of bone during burning is manifest in significant modifications of the appearance of the $\nu_1\nu_3PO_4$ domain, identifiable especially in derivative spectra: these modifications make it possible to distinguish three ranges of heating temperatures (0-500°C, 500-600°C, >600°C.) It has also been found that the $\nu_1\nu_3PO_4$ domain allows a good assessment of the degree of crystallinity because the values of the 1030/1020 index are strongly correlated to those of the splitting factor, both for modern and fossil material.

Moreover, several components of the $\nu_1\nu_3PO_4$ domain display a wavenumber variation as a function of the heating temperature in the case of modern material. These wavenumber variations occur mainly between 120°C and 650°C, while, above this temperature, the wavenumber remains fairly stable for the four peaks. In contrast, the change of the splitting factor during heating occurs essentially between 600 and 700°C, thus indicating and increase in the size of the crystals also identifiable by electron microscopy and x-ray diffraction. In infrared spectroscopy, the wavenumber variations of the absorption peaks can be related to modifications of the molecular environment of the groups considered. Such modifications of the molecular structure at low temperatures could be revealed earlier through XRD by significant changes in the unit cell parameters (Rogers and Daniels, 2002; Etok *et al.*, 2007.) These changes, which occur mainly between 120 and 500°C, could result notably from the elimination of carbonates and water present in the mineral phase. The variations in the wavenumbers of the observed

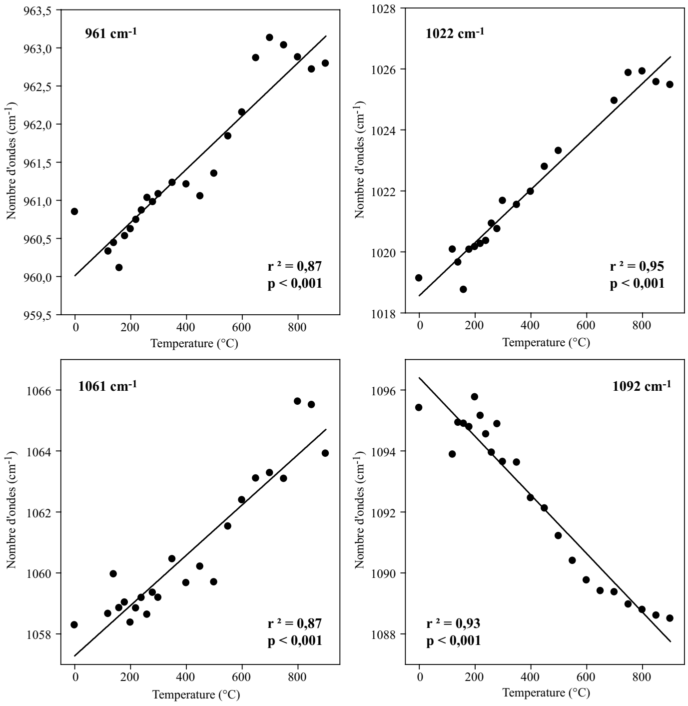

Fig. 12 - Wavenumbers of the peaks centred near 961, 1022, 1061, and 1092 cm^{-1} as a function of the burning stage for the specimens from the Magdalenian levels of the Bize-Tournal site.

peaks would thus indicate an improvement in the quality of the mineral phase during heating, while the splitting factor would mainly characterise the increase in the size of the crystallites induced at high temperatures by an improved organisation of the crystal lattice.

In the case of fossil material, the wavenumbers of the observed peaks as a function of the stage of heating display less clear trends; this limits the use of this criterion to assess the heating temperature. Such variations, indicative of the changes in the composition of the mineral phase, can result from various diagenetic processes. The utilisation of the wavenumber of these peaks could therefore contribute also to the evaluation of the degree of alteration of unburnt bones. Three of these peaks show a significant upward shift in their wavenumber as a function of the degree of heating, although the coefficients of determination are weak. These are the peaks centred on average near 961, 1022, and 1061 cm^{-1}. In order to minimise the effect of individual variations of these wavenumbers and to increase the spread between specimens belonging to different categories, the wavenumbers of these peaks were added together. This makes it possible to more clearly detect the stage of heating of the specimens (fig. 13.) In fact, the cumulative wavenumbers of the unburnt specimens (category 0) are clearly distinct from those of specimens belonging to at least stage 2. In contrast, only the specimens belonging to stages 5 and 6 are clearly distinguished from unburnt specimens when the splitting factor is used; this limits its potential for characterising the more lightly burnt bones in an archaeological context. The utilisation of the wavenumber of these peaks seems then to allow the identification of the bones burnt from stage 2, or about 240°C, and up for the Bize-Tournal material.

Another advantage of the use of the $\nu_1\nu_3PO_4$, between 1200 and 900 cm^{-1}, is that it can be studied by infrared micro-spectroscopy. In fact, the spectral range of infrared micro-spectroscopy is generally limited between 4000 and 600 cm^{-1} and therefore it does not allow measures of the splitting factor. It was possible to carry out some preliminary tests by infrared micro-spectroscopy on polished thin sections of burnt modern bone (protocol established by Miller *et al.,* 2007), in order to study the spatial distribution of the crystallinity and the relative concentration of carbonates within the mineral phase. Figure 14 shows the spatial variability of these two parameters for a fragment of ox diphysis heated to 500°C for 45 minutes: its peripheral portion shows the onset of calcination while the interior portion is still carbonised. It can be seen that the crystallinity is higher in the external zone, submitted to higher temperatures, and that it is, in contrast, lower in the interior portion where the carbonate concentration remains high. It seems therefore that exposing the bone to heat results in a gradual diffusion of the temperature within the specimen and in the establishment of a composition gradient. The use of infrared micro-spectroscopy to study these composition gradients could thus make it

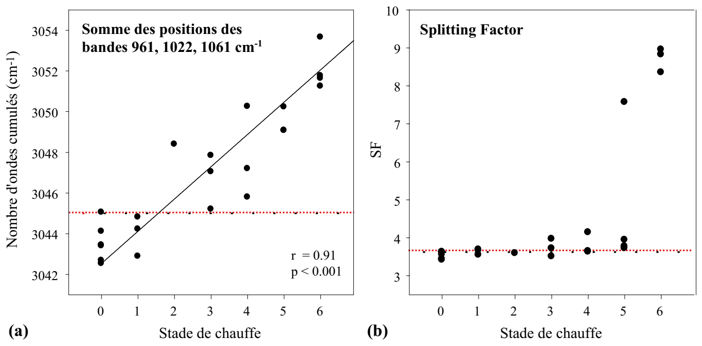

Fig. 13 - Cumulative wavenumbers of the peaks centred near 961, 1022, and 1061 cm^{-1} (a) and values of the splitting factor (b) as a function of the burning stage for the Bize-Tournal specimens. The dotted line represents the maximum value observed for the unburnt specimens (category 0.)

possible to assess, not only the heating temperature, but also the time during which the specimen was exposed to the heat. This technique can also make it possible to consider the spatial variability of the composition of unburnt bones and thus contribute to a better understanding of the heterogeneities of the alterations occurring in bones during diagenesis. It could then be possible to identify zones diagenetically less modified, at whose level the heating signal would be preserved.

Conclusion

The results obtained through this work have demonstrated the interest of the $\nu_1\nu_3$ phosphate domain in the study of the changes in bone composition during heating. The 1030/1020 index was found to be effective in the assessment of the crystallinity in a way entirely equivalent to that of the splitting factor, both for modern and fossil bones, whether burnt or not. The utilisation of this index in the context of infrared micro-spectroscopic analyses can therefore make it possible to take into account the structure and the heterogeneity of the changes undergone during both heating and diagenesis. This type of study will thus make it possible to better define the effects of heating on a

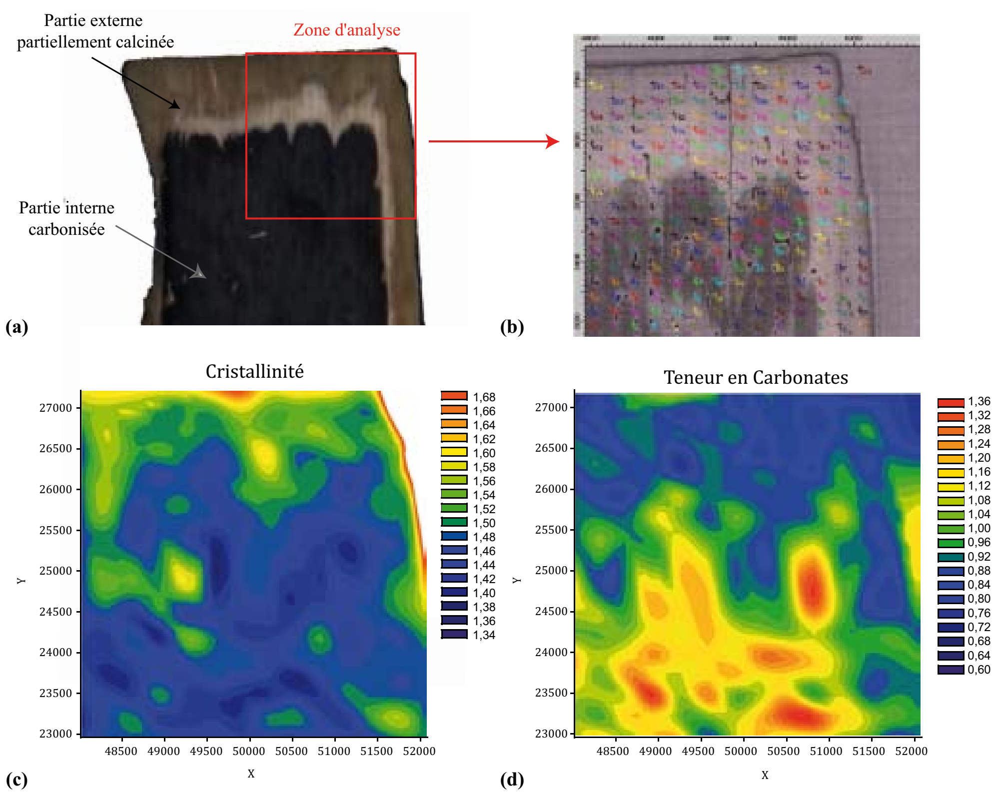

Fig. 14 - Infrared mappings carried out on polished thin sections of a fragment of compact bone heated to 500oC for 45 minutes (a.) After selecting an area of 4.5 mm^2, about 200 points of analysis were carried out (b) in order to establish a distribution of the values of crystallinity (c) and of the relative carbonate content (d) within the mineral phase.

micro-scale and to attempt to define more reliable markers of thermal treatment.

This study has also made it possible to detect wavenumber variations for certain component peaks of the $\nu_1\nu_3PO_4$ domain. These variations, which occur at a lower temperature than the crystallinity, seem to indicate an improvement in the organisation of the crystal lattice, which could be the result of the elimination of the carbonates and water present in the bone mineral phase. Such criteria represent sensitive markers of the modifications of the composition during heating. Although these markers appear to be modified during diagenesis, they nevertheless made it possible to identify bones burnt at temperatures as low as 240°C in the case of material from Magdalenian levels. The application of this analytical protocol thus makes it possible to characterise burnt bones, as well as the temperatures and durations of the heating phase; it could also contribute to a better identification of the activities at the origin of the bones in various archaeological contexts.

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References

Boskey A. & Pleshko-Camacho N. 2007 - FT-IR imaging of native and tissue-engineered bone and cartilage. *Biomaterials,* 28 : 2465-2478.

Buikstra J. & Swegle M. 1989 - Bone modification due to burning : Experimental evidence. *In : Bone modifications*, Bonnichsen R. & Sorg M.H. (eds) : 247-258.

Cain C. R. 2005 - Using burned animal bone to look at Middle Stone Age occupation and behavior. *Journal of Archaeological Science,* 32 : 873-884.

Etok S., Valsami-Jones E., Wess T., J.C. H., Maxwell C., Rogers K., Manning D., White, M. Lopez-Capel E., Collins M., Buckley M., Penkman K. & Woodgate, S. 2007 - Structural and chemical changes of thermally treated bone apatite. *Journal of Materials Science,* 72 : 9807-9816.

Fröhlich F. 1989 - Deep-sea biogenic silica : new structural and analytical data from infrared analysis-geological implications. *Terra Nova,* 1 : 267-273.

Gadaleta S., Paschalis E., Betts F., Mendelsohn R. & Boskey A. 1996 - Fourier transform infrared spectroscopy of the solution-mediated conversion of amorphous Calcium phosphates to hydroxyapatite : new correlations between X-Ray diffraction and infraredd data. *Calcified Tissue International,* 58 : 9-16.

Hiller J., Thompson T., Evison M., Chamberlain A. & Wess, T. 2003 - Bone mineral change during experimental heating : an X-Ray scattering investigation. *Biomaterials,* 24 : 5091-5097.

Lebon M., Reiche I., Fröhlich F., Bahain J.J. & Falguères C., 2008 - Archaeological burnt bones characterization : contribution of a new analytical protocol based on derivative FTIR Spectroscopy and curve fitting of the $\nu_1\nu_3PO_4$ domain. *Analytical and Bioanalytical Chemistry,* 392 : 1479-1488.

LeGeros R. 1981 - Apatites in Biological System. *Prog. Crystal Growth Charact,* 4 : 1-45.

Leung Y., Walter M. & LeGeros R. 1990 - Second derivative infrared spectra of hydroxyapatite. *Spectrochimica Acta,* 46 : 1453-1459.

Magniez P. (sous presse) - Taphonomic study of the middle and Upper Palaeolithic large mammal assemblage from Tournal Cave (Bize-Minervois, France). *Journal of Taphonomy.*

Miller L.M., Little W., Schirmer A., Sheik F., Busa B. & Judex, S. 2007 - Accretion of bone quantity and quality in the developing mouse skeleton. *Journal of Bone and Mineral Research,* 22 : 1037-1045.

Nicholson R. 1993 - A morphological investigation of burnt animal bone and an evaluation of its utility in archaeology. *Journal of Archaeological Science,* 20 : 411-428.

Paschalis E., DiCarlo E., Betts F., Sherman P., Mendelsohn R. & Boskey A. 1996 - FTIR microspectroscopic analysis of human osteonal bone. *Calcified Tissue International*, 59 : 480-487.

Pasteris J., Wopenka B., Freeman J., Rogers K., Valsami-Jones E., van der Houwen J. & Silva M. 2004 - Lack of OH in nanocristalline apatite as a function of degree of atomic order : implications for bone and biomaterials. *Biomaterials,* 25 : 229-238.

Patou-Mathis M., Bayle G. & Paletta C. 1999 - Etude archéozoologique du niveau magdalénien « ancien » de la grotte Tournal à Bize (Aude). *Préhistoire Européenne,* 14 : 115-133.

Person A., Bocherens H., Mariotti A. & Renard M. 1996 - Diagenetic evolution and experimental heating of bone phosphate. *Palaeogeography, Palaeoclimatology, Palaeoecology,* 78 : 37-54.

Piga G., Malgosa A., Thompson T. & Enzo, S. 2008 - A new calibration of the XRD technique for the study of archaeological burned human remains. *Journal of Archaeological Science,* 35 : 2171-2178.

Reiche, I., Vignaud, C. & Menu, M. 2002 - The cristallinity of ancient bone and dentine : new insights by transmission electron microscopy. *Archaeometry,* 44 : 447-459.

Rey C., Shimizu M., Collins B. & Glimcher M. 1991 - Resolution-enhanced fourier tranform infrared spectroscopy study of the environment of phosphate ion in the early deposits of a solid phase of calcium phosphate in bone and enamel and their evolution with age : investigations in the v_3PO_4 domain. *Calcified Tissue International,* 49 : 383-388.

Rogers K. & Daniels P. 2002 **-** An x-ray diffraction study of the effects of heat treatment on bone mineral microstructure. *Biomaterials,* 23 : 2577-2585.

Shahack-Gross R., Bar-Yosef O. & Weiner S. 1997 - Black-coloured bones in Hayonin Cave, Israel : Differentiating between burning and oxide staining. *Journal of Archaeological Science,* 24 : 439-446.

Shipman P., Foster G. & Shoeninger M. 1984 - Burnt bones and teeth : an experimental study of color, morphology, crystal structure and shrinkage. *Journal of Archaeological Science,* 11 : 307-325.

Stiner M., Kuhn S., Weiner S. & Bar-Yosef O. 1995 - Differential burning recrystallization and fragmentation of archaeological bone. *Journal of Archaeological Science,* 22 : 223-237.

Tavoso A. 1987 - Le remplissage de la grotte Tournal à Bize-Minervois (Aude). Girona, *Cypsela,* VI : 23-25.

Termine J. & Posner A. 1966 - Infra-red determination of the percentage of crystallinity in apatic calcium phosphates. *Nature,* 211 : 268-270.

Théry-Parisot I., Costamagno S., Brugal J.-P., Fosse P. & Guilbert R. 2005 - The use of bone as fuel during the Palaeolithic, experimental study of bone combustible properties. *In* : *The zooarchaeology of fats, oils, milk and dairying*, J. Mulville & A.K. Outram (eds). Actes du colloque de l'ICAZ, Durham, août 2002. Oxford, Oxbow Books : 50-59.

Trueman C., Privat K. & Field J. 2008 - Why do cristallinity values fail to predict the extent of diagenetic altération of bone mineral ? *Palaeogeography, Palaeoclimatology, Palaeoecology,* 266 : 160-167.

Weiner S. & Bar-Yosef O. 1990 - States of preservation of bones from prehistoric sites in the near East: a survey. *Journal of Archaeological Science,* 17 : 187-196.

Young R. & Holcomb D. 1984 - Role of acid phosphate un hydroxyapatite lattice expansion. *Calcified Tissue International,* 36 : 60-63.

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ISOTOPE GEOCHEMISTRY OF BURNED BONES: IMPLICATIONS FOR PALEODIETARY RECONSTRUCTION AND RADIOCARBON DATING

Antoine Zazzo

Abstract

Though they are frequently found at archaeological sites, burned bones have long been neglected by geochemists. After a brief review of the mineralogy and diagenesis of vertebrate skeletal tissues, all the physico-chemical changes induced by the high temperature combustion of bones are summarized. The implications of these changes for the reconstruction of diets through stable isotope ratios analysis and for the radiocarbon dating of bone remains are then discussed. It is thus shown that the high-temperature (>600ºC) re-crystallisation of the mineral fraction of bones: (1) provokes a fractionation of the isotopes that modifies the $\delta^{13}C$ of the bone and therefore makes it unsuitable for paleo-dietary reconstructions; (2) protects the bone from chemical interactions with the surrounding environment during fossilisation, thus making calcined bone a reliable material for radiocarbon dating.
The calcined bones can in turn be used to estimate the state of preservation of the unburned bones found at the same site when the collagen has not been preserved.

Key-words : bioapatite, stable isotopes, radiocarbon, diagenesis, diet

Introduction

Biogeochemistry is a powerful tool for reconstructing the diets of humans and animals in archaeological contexts. The analysis of the composition of stable isotopes of carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) in the organic fraction (collagen) of bones, and of the ratios of trace elements (strontium/calcium and barium/calcium) of the mineral fraction (hydroxylapatite, or bioapatite) in bones has provided information on the nutrition of Neanderthals and anatomically modern humans (Balter 2007; Bocherens *et al.*, 2005; Fizet *et al.*, 1995; Richards *et al.*, 2000). Because bone tissues are continuously renewed, they provide information averaged over the last years of an individual's life. Teeth, which develop during the first few years of an individual's life, offer the opportunity to document the nutritional history of animals and humans with a high temporal resolution, on a scale of months or seasons (Balasse *et al.*, 2003, 2006; Balter *et al.*, 2008; Sponheimer *et al.,* 2006.). One of the advantages of this method is that it makes it possible to calculate the proportions of the main nutritional resources independently of their preservation at the archaeological site and thus to diminish the taphonomic bias (Balter 2007.). Moreover, the organic and mineral fractions of bones and teeth can be dated by the radiocarbon method. The advantage of direct dates of human and animal remains is that they are not dependent on the dates obtained for associated materials, whose strict contemporaneity with the bone remains is not always verifiable (Sealy et Yates 1994; Zilhao 2001).

Though they are frequently found at archaeological sites, burned bones have long been neglected by geochemists. Bone collagen, which has long been the preferred support for paleo-dietary reconstructions and dating, decomposes at low temperatures and thus become *a priori* useless. The mineral fraction of bone, on the other hand, has long be considered to offer limited reliability for paleo-dietary reconstructions or radiocarbon dating due to its limited resistance to diagenetic alterations (Tamers et Pearson, 1965; Schoeninger et DeNiro, 1982.). Meanwhile, paradoxically, the ensemble of physico-chemical changes associated with the combustion of bones at high temperatures make the mineral fraction of bones a support as reliable as collagen, and a very useful one when the latter has disappeared. Following a brief review of the mineralogy of bioapatites, I will describe the combination of physico-chemical changes induced by combustion. Finally, I will discuss the implications of these changes for the reconstruction of diets and for the radiocarbon dating of bone remains.

Composition and physico-chemical properties of bioapatites

The mineral fraction of the biominerals of vertebrates is composed of a calcium phosphate with the general formula $Ca_{10}(PO_4)_6OH_2$, or in terms of crystallographic sites, $Z_{10}B_6A_2$, and crystallising in the hexagonal system. We can also speak of carbonate hydroxylapatite (or bioapatite) due to the presence of carbonate ions that are essentially substituents in the B sites of phosphates. Although the mineralised tissues of vertebrates are all composed of carbonate hydroxylapatite, each one has its own characteristics in terms of the calendar and geometry of its development during ontogenesis, as well as at the level of its crystalline structure or even its physico-chemical characteristics.

The mineralisation of the hard tissue of vertebrates is initiated by an essentially collagenous organic matrix. This interconnection between the organic and the mineral and the mechanisms operating during the mineralisation process are specific to each tissue. Bone is a mesodermic tissue composed of 65% apatite and 35 % organic matter (Posner, 1987.). Dentine, which constitutes the bulk of the thickness of the tooth is also a mesodermic tissue whose mineral fraction (70-75%) is connected to an organic matrix of collagenous proteins. Unlike bone and dentine, enamel is a highly mineralised (about 97%) ectodermic tissue whose organic phase is gradually eliminated during the maturation phase (Weinmann *et al.*, 1942.). Due to this structural difference, the porosity decreases by a factor of 40 from bone to enamel (Brudefold et Soremark, 1967; Rowles, 1967; Trautz, 1967.) While, with few exceptions, the teeth of mammals are developed during the first years of life and are not subsequently altered (Hillson, 2005), bone is a living tissue that undergoes continuous changes. Numerous ions, other than carbonate ions, can be incorporated into the bioapatites and this high chemical complexity reflects the role played by bone tissues in the regulation of an organism's needs.

Almost all the ions incorporated have an impact on the physico-chemical properties of the bioapatites (LeGeros et LeGeros, 1984.). Specifically, it seems that the differences in crystallinity between bone, enamel, and dentine can be partially explained by their different carbonate content. Bone is the tissue that has the highest carbonate content (about 6% by weight) while enamel has the lowest (3.5% by weight on average) (Elliot, 1985.) .When the divalent and planar carbonate ion replaces the trivalent and tetrahedral phosphate ion at the B sites, it modifies both the electrical neutrality and the symmetry of the crystal. The incorporation of carbonate ions into the crystalline network also induces an internal stress that has been shown by spectroscopy on synthetic apatites (Blumenthal, 1975.). This destabilization of the network causes an increase in the solubility of apatite (Gron, 1963; Okazaki, 1998.). Finally, the carbonate ion, which is smaller than the phosphate ion, is also responsible, when it is in the B position, for a large decrease of the *a* parameter of the crystal lattice and a slight decrease of the *c* parameter (Posner, 1987); it thus causes an overall decrease in the size of the crystallites. These differences then have repercussions for the solubility, size, and shape of the crystallites. The crystallites in the enamel are large (400 Å wide, 1600-10,000 Å long) and well crystallised (needle-shaped), while in the bone they are smaller (25-50 Å wide, 200-1,000 Å long) and less well crystallised (equiaxial) (Bottero, 1992.). These physico-chemical differences (in tissue porosity, size, and solubility of the crystals) are directly responsible for the quality of conservation during the diagenesis of the isotope signature acquired by the animal during its life.

Diagenesis of bioapatites

At the death of an individual, the thermodynamic equilibrium within its bone structure is disrupted. The result of the interaction between the tissue and the fluids percolating through the soil is called diagenesis. Extrinsic factors linked to the properties of the fossilisation environment, such as pH, temperature, pressure and the degree of saturation of the solution, determine the direction of the exchanges and the reaction kinetics. Intrinsic factors linked to the physico-chemical properties of the bioapatite, such as its solubility and porosity, control the intensity of the exchanges with the fluids. Since bone is the tissue with the greatest solubility and porosity, it is the most susceptible to exchanges with diagenetic fluids. The physical and chemical criteria for recognising diagenesis have generally been based on this tissue (Hedges, 2002.).

The sources of pollution are varied and concern both the organic and the mineral phase. They can be related to the addition of organic materials (humic and fulvic acids) or mineral materials (precipitation of secondary calcite) into the pores of the bones. They can also consist of isotope exchanges between the bone's carbonate and the dissolved inorganic carbon (DIC) in surface or groundwater, or of the dissolution/neo-formation of apatite when allowed by the pH conditions. Generally, these different modification mechanisms have the effect of rejuvenating bones, especially in temperate environments. The techniques developed to purify bones mainly concern the elimination of the secondary phases. To isolate the organic phase of bone, a treatment with sodium hydroxyde is typically employed, in between two immersions in hydrochloric acid to eliminate the secondary carbonates (acid-base-acid or ABA treatment.). Starting recently, this protocol is completed by an ultrafiltration stage, which allows separation of the collagen from low molecular weight organic molecules not removed during the ABA treatment (Higham *et al.*, 2006.).

To isolate the mineral phase of bone (bioapatite), a pre-treatment under vacuum with acetic acid allows the elimination of secondary calcites without altering the less soluble carbonate in bone (Balter *et al.*, 2002.). This approach is sufficient when there have not been further chemical exchanges between the bioapatite of the bone and the external environment, which is the case when the bones have been protected from an aqueous environment. The most favourable environments for dating the mineral phase of bone are thus arid and semi-arid ones, in which, moreover, collagen is very poorly conserved (Saliège *et al.*, 1995; Braemer *et al.,* 2001; Sereno *et al.*, 2008.). Temperate environments are, on the contrary, much less favourable to the preservation of the geochemical composition of bones since the isotopic exchanges between the bone and the soil's DIC are frequent and no solution exists at present to eliminate this source of pollution. In these environments, the use of unburned bones is not an option. Tooth enamel, which is more resistant to isotopic exchanges because of the larger size of its crystals and its low porosity, is widely employed in archaeological studies (Balasse *et al.*, 2003; 2006.) It must be noted, however, that its utilisation is generally limited to measuring the ratios of stable isotopes ($\delta^{13}C$, $\delta^{18}O$.) Though still rare, the radiocarbon dating of tooth enamel is becoming increasingly common due to the decreased test sample sizes made possible by the AMS technique (Surovell 2000; Munoz *et al.*, 2008; Sereno *et al.*, 2008).

Morphological, physical and chemical modifications during combustion

The heating of bioapatites causes a series of macro- and microscopic, morphological, physical, and chemical changes. The evolution of these parameters can be studied in the laboratory during combustion experiments in order to establish at what temperatures these transformations take place (fig. 1.).

The heating of bones modifies their resistance and solidity (Newesely, 1989; Stiner *et al.*, 1995; van Strydonck *et al.*, 2005.) Burned bones develop fractures and display a considerable reduction in size (Holden *et al.*, 1995; Shipman *et al.*, 1984.) In addition to these morphological changes, colour changes are also observed. (Munro *et al.*, 2007.) The bones first become brown (200°C) and then black (300-400°C) due to the combustion and then carbonisation of the organic matter (fig. 2.) These are referred to as burned or charred bones. The progressive disappearance of the organic matter is indicated by a colour approaching beige-ochre at around 500–600 °C. Above this temperature, the bone becomes white. This is referred to as cremated or calcined bone. This evolution of colour can be utilised to make a semi-quantitative estimate of the bone's exposure temperature during heating (Bonucci et Graziani, 1975; Shipman *et al.*, 1984; Taylor *et al.*, 1995).

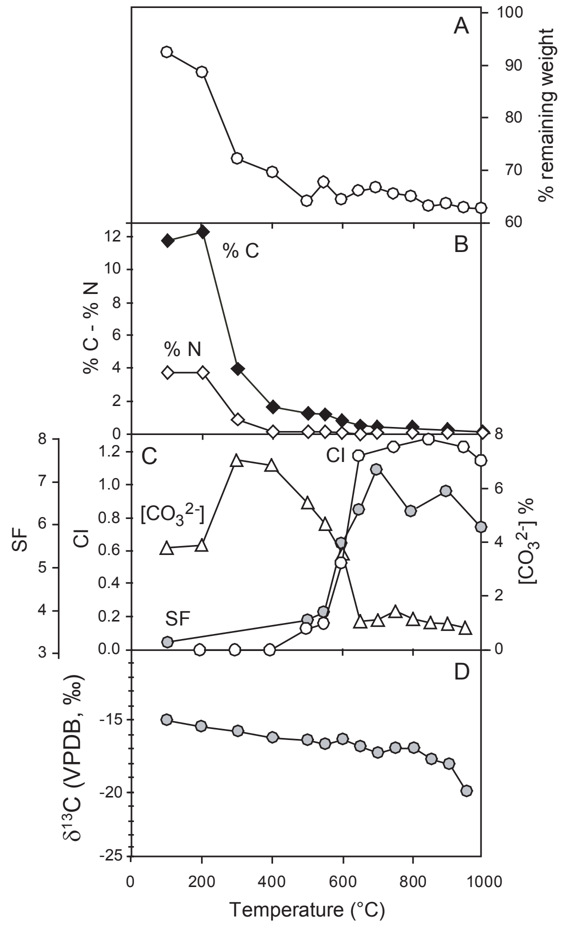

Fig. 1 - Effect of temperature on the behaviour of several physico-chemical properties of a modern bone heated to 100-1000°C. Between 100 and 400°C, heating causes a decrease in mass of about 35% (A) and in the carbon and nitrogen content (B); this is linked to the combustion of the bone's organic matter. Around 600°C, the recrystallisation of the mineral fraction of the bone causes an increase in the crystallinity index (CI) measured by X-ray diffraction, as well as in the splitting factor (SF) measured by Fourier transform infrared spectroscopy (C.) This recrystallisation is accompanied by a decrease in the bone's carbonate content (C) as well as a decrease in the $\delta^{13}C$ value (D). The detailed experimental conditions are described in Zazzo *et al.*, (2009).

The heating of bone also results in a loss of mass (fig. 1A). This loss of mass is estimated at 35-40% during experiments conducted in the laboratory (Person *et al.*, 1996; Zazzo *et al.*, 2009). At low temperatures (< 225°C), this loss is linked to a dehydration of the bone (10-15% of the weight.) Between 225 and 550°C, it is associated with the combustion of the bone's organic matter (20-25% of the weight) and is accompanied by a decrease in the carbon and nitrogen content of the bone (fig. 1B.) At higher temperatures, the loss of mass is related to the decomposition of the bone's structural carbonate in the form of CO_2 (<5% of the weight) (Haas et Banewicz, 1980; Newesely, 1989; Shipman *et al.*, 1984; Stiner *et al.*, 1995; Zazzo *et al.*, 2009).

Fig. 2 - Burned archaeological bone (In Tékébrine, Niger) viewed in cross-section. The gradient of temperature to which the bone was subjected during cremation is visible from the core of the bone (black, carbonised) to the surface (white, calcined.) Scale= 5mm.

With regard to the mineral phase of bone, the most significant physico-chemical changes occur at around 600°C (Shipman *et al.*, 1984; Stiner *et al.*, 1995.) Above this temperature, the small apatite crystals recrystalise and increase in size (Holden *et al.*, 1995; Shipman *et al.*, 1984.). This recrystallisation is accompanied by a loss in carbonate content of about 50% (fig. 1C.) This loss decreases the stress within the crystal structure and results in an increase in the crystallinity index as measured by X-ray diffraction or infrared spectroscopy (Person *et al.*, 1995; Shipman *et al.*, 1984; Munro *et al.*, 2007; Zazzo *et al.*, 2009). Calcined bone is thus very similar to tooth enamel from a crystallographic point of view.

Finally, heating causes significant changes in the isotope composition of the mineral fraction of bioapatites (van Strydonck *et al.*, 2005; Olsen *et al.*, 2008; Zazzo *et al.*, 2009). These modifications result in a decrease in the $\delta^{13}C$ values that can be as high as 15% and whose magnitude is independent of the bone's initial value (fig. 3.). The comparison of the $\delta^{13}C$ of the charred (black) and calcined (white) portions of the same bone shows that only the calcined portions undergo these modifications. This observation is confirmed by cremation experiments and shows that the decrease in the $\delta^{13}C$ values is linked to the recrystallisation of the bioapatites at around 600°C (van Strydonck *et al.*, 2005; Zazzo *et al.*, 2009). It should be noted, however, that Munro *et al.* (2008) have observed an opposite trend (increase in the $\delta^{13}C$ associated with a decrease of the $\delta^{18}O$ values), though this discrepancy is not discussed.

Implications for the reconstruction of diets and for ^{14}C dating

The modification of carbon isotope ratios renders calcined bones unsuitable for the reconstruction of diets. In fact, the decrease in the $\delta^{13}C$ is highly variable within the same archaeological site (fig. 3), making it impossible to apply a uniform correction factor to all bones in order to return to their original value. The $\delta^{13}C$ of calcined bones is easily identified as "anomalous" in temperate environments where plants utilise exclusively C_3 photosynthesis. This is not the case in tropical environments where the two types of photosynthesis (C_3 and C_4) coexist. In these environments the $\delta^{13}C$ of calcined C_4 bones becomes similar to that of the non-calcined bones of animals that have consumed C_3 plants, making all paleo-dietary inferences impossible.

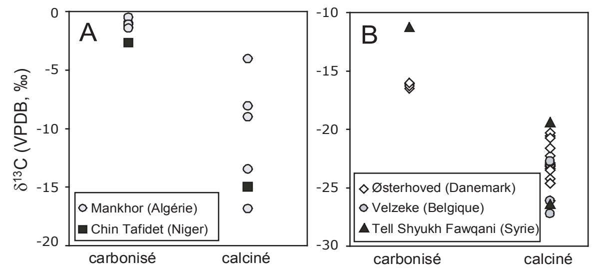

Fig. 3 - Carbon isotope composition ($\delta^{13}C$) of archaeological bones carbonised and calcined in C_4 (A) and C_3 (B) contexts. Data compiled based on Olsen *et al.*, (2008); van Strydonck *et al.*, (2005); Zazzo *et al.*, (2009).

We observed as well that the crystallographic transformations make calcined bone as resistant as tooth enamel to digenetic change. The small amount of residual carbon in calcined bones (around 0.5% by weight) is nevertheless sufficient for dating by AMS. Lanting *et al.* (2001) were the first to understand that this property of calcined bones was useful for radiocarbon dating. The dating of a large series of calcined bones (n=54) and associated charcoals, originating essentially from the Netherlands, demonstrates a perfect preservation of the bones during the last eleven millennia. In order to validate the method, it was important to demonstrate that the residual carbon indeed comes from the stock of mineral carbon originally present in the bone. In effect, during re-crystallisation, the crystal lattice must open up in order to reorganize itself and possible interactions with the external environment cannot be excluded. To test this hypothesis, Zazzo *et al.* (2009) designed several combustion experiments during which they varied various parameters: composition of the atmosphere, role of the organic matter of bone, speed and duration of heating, and temperature. They showed that the decrease of the $\delta^{13}C$ values is observed even when the bone does not contain organic matter and that it is not correlated with the concentration or with the $\delta^{13}C$ of the CO_2 present in the atmosphere during cremation. On the other hand, this decrease is only observed in the case of a rapid rise in temperature (fig. 1D.) These results support the hypothesis of a kinetic origin of the isotope fractionation, and thus validate the use of calcined bones as supports for ^{14}C dating.

Calcined bones offer another advantage: they can be used to evaluate the preservation of unburned bones present at the same site. In the absence of objective criteria of the preservation of the isotope composition in the mineral fraction of a bone, it is very difficult to establish with certainty the validity of a ^{14}C date obtained on bioapatite. This limitation has long hampered the dating of the mineral fraction of bones (Tamers et Pearson, 1965; Haynes, 1968; Hassan *et al.*, 1977; Hedges *et al.*, 1995.) Burned bones finally make it possible to surmount this difficulty. Indeed, the ^{14}C date of a burned bone can be measured independently in three different ways: on the mineral fraction of a carbonised bone (black), on the mineral fraction of a calcined bone (white), and on the organic fraction (the bone's residual collagen.) The structure of the mineral fraction of carbonised bone is very similar to that of unburned bone because it has not undergone recrystallisation. If the taphonomic conditions favour the preservation of the geochemical composition of the bioapatite, the ages derived from the carbonised and calcined bones must match. The two dates can be compared to that obtained on the carbonised bone's organic fraction.

N° target	sample reference	Site	Country	Fraction	colour	$\delta^{13}C$ (‰, VPDB)	radiocarbon age BP			Reference
SacA 11363	Bahla 2003	Bahla	Sultanate of Oman	calcined bioapatite	white	-18,2	3655	±	30	Munoz *et al.* (2008)
SacA 11362	Bahla 2003	Bahla	Sultanate of Oman	charred bioapatite	black	-18,5	3615	±	30	Munoz *et al.* (2008)
SacA 11364	Bahla 2003	Bahla	Sultanate of Oman	degraded collagen	black	-24,2	3690	±	30	Munoz *et al.* (2008)
SacA 11369	RJ1-F1	Ra's al-Jinz	Sultanate of Oman	calcined bioapatite	white	-16,6	4190	±	30	Munoz *et al* . (2008)
SacA 11370	RJ1-F1	Ra's al-Jinz	Sultanate of Oman	charred bioapatite	black	-2,5	4105	±	30	Munoz *et al.* (2008)
SacA 11371	RJ1-F1	Ra's al-Jinz	Sultanate of Oman	degraded collagen	black	-11,3	4100	±	30	Munoz *et al.* (2008)
SacA 11374	HD7-T5	Ra's al-Hadd	Sultanate of Oman	calcined bioapatite	white	-11,1	4100	±	30	Munoz *et al.* (2008)
SacA 11373	HD7-T5	Ra's al-Hadd	Sultanate of Oman	degraded collagen	black	-11,5	4045	±	30	Munoz *et al.* (2008)
Pa 1889	T1	Mankhor	Algeria	degraded collagen	black		5450	±	55	Saliège (pers. comm.)
Pa 2430	T1	Mankhor	Algeria	bioapatite	white		5255	±	100	Saliège (pers. comm.)
Pa 1891	96 B sup	Mankhor	Algeria	degraded collagen	black		5400	±	70	Saliège (pers. comm.)
Pa 1707	96 B sup	Mankhor	Algeria	bioapatite	white		5360	±	80	Saliège (pers. comm.)
Pa 1890	R2	Mankhor	Algeria	degraded collagen	black		5470	±	70	Saliège (pers. comm.)
Pa 1709	R2	Mankhor	Algeria	bioapatite	white		5365	±	80	Saliège (pers. comm.)
KIA-36268	Mdt-2107	Can Missert	Spain	calcined bioapatite	white		2745	±	25	van Strydonck *et al.* (2009)
KIA-36266	Mdt-2107	Can Missert	Spain	charred bioapatite	black		2330	±	25	van Strydonck *et al.* (2009)
KIA-36269	Mdt-2120	Can Missert	Spain	calcined bioapatite	white		2760	±	25	van Strydonck *et al* . (2009)
KIA-36267	Mdt-2120	Can Missert	Spain	charred bioapatite	black		2675	±	30	van Strydonck *et al.* (2009)
KIA-35567	MEV-3581	Can Missert	Spain	calcined bioapatite	white	-17,2	2815	±	30	van Strydonck *et al* . (2009)
KIA-36270	MEV-3579	Can Missert	Spain	charred bioapatite	black		2535	±	25	van Strydonck *et al.* (2009)
AAR-9396	na	Østerhoved	Danemark	calcined bioapatite	white	-23,2	3756	±	28	Olsen *et al.* (2008)
AAR-9390	na	Østerhoved	Danemark	charred bioapatite	black	-16,1	3614	±	27	Olsen *et al* . (2008)
AAR-8784	na	Østerhoved	Danemark	calcined bioapatite	white	-20,3	3682	±	43	Olsen *et al.* (2008)
AAR-8785	na	Østerhoved	Danemark	charred bioapatite	black	-16,0	3576	±	29	Olsen *et al* . (2008)

Tab. 1 - List of sites for which several ^{14}C ages were obtained on the different fractions of a single burned bone.

This last test allows us to evaluate the preservation of the mineral phase relative to the collagen, which is generally considered as more reliable for ^{14}C dating. If, on the other hand, the ages of the calcined and carbonised bones diverge, it is likely that the carbonised bone's mineral fraction has been altered. This means that the unburned bone was also altered and that it must not be considered reliable for ^{14}C dating. Table 1 shows a list of sites where two or even three fractions of the same bone have been dated. In the arid environment sites (Sahara, Arabian Peninsula), the age difference between the different fractions is very small, close to the experimental error. This result demonstrates the absence of significant isotope exchanges between the bioapatite and the diagenetic fluids and indicates that bioapatites, even not heated, can be dated. The situation is more complex in the temperate European sites and the carbonised bone can be rejuvenated by 100 to 400 years relative to the collagenous fraction or the calcined bone. In this case, only the calcined bioapatite (or the collagen, if preserved) will give a reliable ^{14}C age. The number of sites where this methodological work has been carried remains limited at present. It would be profitable, meanwhile, to extend this approach, supplemented by the systematic dating of tooth enamel, so as to better understand the conditions of the preservation of bioapatite geochemistry for the purpose of their radiocarbon dating.

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References

Balasse M., Smith A.B., Ambrose S.H. & Leigh, S.R. 2003 - Determining sheep birth seasonality by analysis of tooth enamel oxygen isotope ratios : the Late Stone Age site of Kasteelberg (South Africa). *Journal of Archaeological Science*, 30 : 205-215.

Balasse M., Tresset A. & Ambrose S.H. 2006 - First evidence for seaweed winter foddering in the Neolithic of Scotland. *Journal of Zoology*, 270 : 170-176.

Balter V. 2007 - Le comportement alimentaire des Néandertaliens. *In* : *Les Néandertaliens. Biologie et cultures.* Paris, Éditions du CTHS, 23 : 199-212.

Balter V, Saliège J.-F., Bocherens H & Person A. 2002 - Evidence of physico-chemical and isotopic modifications in archaeological bones during controlled acid etching. *Archaeometry,* 44 : 329-36.

Balter V., Telouk P., Reynard B., Braga J., Thackeray F. & Albarède F. 2008 - Analysis of coupled Sr/Ca and $^{87}Sr/^{86}Sr$ variations in enamel using laser-ablation tandem quadrupole-multicollector ICPMS. *Geochimica et Cosmochimica Acta*, 72 : 3980–3990.

Blumenthal N.C., Betts F. & Posner A.S. 1975 - Effect of carbonate and biological macromolecules on formation and properties of hydroxyapatite. *Calcified Tissue Research*, 18 : 81-90.

Bocherens H., Drucker D., Billiou D., Patou-Mathis, M. & Vandermeersch B. 2005 - Isotopic evidence for diet and subsistence pattern of the Saint-Césaire I Neanderthal : review and use of a multi-sources mixing model. *Journal of Human Evolution*, 40 : 497-505.

Bonucci E. & Graziani G. 1975 - *Comparative thermogravimetric, Xray diffraction and electron microscope investigations of burnt bone from recent, ancient and prehistoric age*. Atti Accademia Nazionale dei Lincei. Classe di Scienze, Fisiche, Matematiche e Naturali Rendiconti : 517–532.

Bottero M.J., Yvon J. & Vadot J. 1992 - Multimethod analysis of apatites in sound human tooth enamel. *European Journal of Mineralogy*, 4 : 1347-1357.

Braemer F., Steimer-Herbert T., Buchet L., Saliège J-F. & Guy H. 2001 - Le bronze ancien du Ramlat as-Sabatayn (Yémen). Deux nécropoles de la première moitié du III[e] millénaire à la bordure du désert : Jebel Jidran et Jebel Ruwaiq. *Paléorient,* 27 : 21-44.

Brudefold F. & Soremark R. 1967 - Chemistry of the mineral phase of enamel- Crystalline organization of dental mineral. *In : Structural and Chemical Organization of Teeth*, Miles A.E.D. (ed). London, Academic Press : 247-277.

Elliot J.C., Holcomb D.W. & Young R.A. 1985 - Infrared determination of the degree of substitution of hydroxyl by carbonate ions in human dental enamel. *Calcified Tissue International*, 37 : 372-375.

Fizet M., Mariotti A., Bocherens H., Lange-Badré B., Vandermeersch B., Borel J.P. & Bellon G., 1995 - Effect of diet, physiology and climate on carbon and nitrogen stable isotopes of collagen in a Late Pleistocene anthropic palaeoecosystem : Marillac, Charente, France. *Journal of Archaeological Science*, 22 : 67-79.

Gron P., Spinelli M., Trautz O. & Brudevold F. 1963 - The effect of carbonate on the solublity of hydroxyapatite. *Archive of Oral Biology*, 8 : 251-263.

Haas H. & Banewicz J. 1980 - Radiocarbon dating of bone apatite using thermal release of CO_2. *Radiocarbon*, 22 : 537-544.

Haynes C.V. Jr. 1968 - Radiocarbon : analysis of inorganic carbon of fossil bone and enamel. *Science,* 161 : 687-688.

Hassan A.A. Termine J.-D. Haynes C.V. Jr. 1977 - Mineralogical studies on bone apatite and their implications for radiocarbon dating. *Radiocarbon,* 19 : 364–74.

Hedges R.E.M. 2002 - Bone diagenesis: an overview of processes. *Archaeometry*, 44 : 319–28.

Hedges R.E.M., Lee-Thorp J.A. & Tuross N.C. 1995 **-** Is tooth enamel carbonate a suitable material for radiocarbon dating ? *Radiocarbon,* 37 : 285–290.

Higham T.F.G., Jacobi R.M. & Bronk Ramsey C. 2006 - AMS radiocarbon dating of ancient bone using ultrafiltration. *Radiocarbon*, 48 : 179-95.

Hillson S. 2005 - *Teeth*. Cambridge, University Press, 373 pp.

Holden, J.L. Phakey P.P. & Clement J.G. 1995 - Scanning electron microscope observations of heat-treated human bone. *Forensic Science International*, 74 : 29-45.

Lanting J.N., Aerts-Bijma, A.T. & van der Plicht J. 2001 - Dating cremated bone. *Radiocarbon,* 43 : 249–254.

LeGeros R.Z. & LeGeros J.P. 1984 - Phosphate minerals in human tissues. *In* : *Phosphate minerals,* Nriagu J.O. & Moore P.B. (eds). Berlin, Springer-Verlag : 352-385.

Munoz O., Zazzo A., Bortolini E., Seguin G., Saliège J.-F. & Cleuziou S. 2008 - Reconstructing the diet of the ancient fishermen of Ra's al-Hadd and Ra's al-Jinz (Sultanate of Oman) using radiocarbon dates. *Les Déserts d'Afrique et d'Arabie : Environnement, climat et impact sur les populations*. Colloque de l'Académie des sciences, Institut de France, Paris.

Munro L.E., Longstaffe J. & White C.D. 2007 - Burning and boiling of modern deer bone : Effects on crystallinity and oxygen isotope composition of bioapatite phosphate. *Palaeogeography, Palaeoclimatology, Palaeoecology,* 249 : 90-102.

Munro L.E., Longstaffe J. & White C.D. 2008 - Effects of heating on the carbon and oxygen-isotope compositions of structural carbonate in bioapatite from modern deer bone. *Palaeogeography, Palaeoclimatology, Palaeoecology,* 266 : 142-150.

Newesely H. 1989 - Fossil bone apatite. *Applied Geochemistry*, 4 : 233-245.

Olsen J., Heinemeier J., Bennike P., Krause C., Hornstrup K.M. & Thrane H. 2008 - Characterisation and blind testing of radiocarbon dating of cremated bone. *Journal of Archaeological Science,* 35 : 791-800.

Okazaki M., Matsumoto T., Taira M., Takahashi J. & LeGeros R.Z. 1998 - CO3-apatite preparations with solubility gradient: potential degradable biomaterials. XI^e International Symposium on Ceramics in Medicine, New York.

Person A, Bocherens H, Saliège JF, Zeitoun V & Gérard M. 1995 - Early diagenetic evolution of bone phosphate: an X-ray diffractometry analysis. *Journal of Archaeological Science,* 22 : 211-221.

Posner A. 1987 - Bone mineral and the mineralisation process. *In : Bone and Mineral Research*, Peck W.A. (ed). Elsevier : 65-116.

Rowles S.L. 1967 - Chemistry of mineral phase of dentine. *In: Structural and Chemical organization of Teeth,* Miles A.E.D. (ed). London, Academic Press : 201-245.

Richards M.P., Pettitt P.B., Trinkaus E., Smith F.H., Paunovic, M. & Karavanic, I. 2000. Neanderthal diet at Vindija and Neanderthal predation: the evidence from stable isotopes. *Proceedings of the National Academy of Sciences*, 98 : 7663-7666.

Saliège J.F., Person A. & Paris F. 1995 - Preservation of $^{12}C/^{13}C$ original ratio and ^{14}C dating of the mineral fraction of human bones from Saharan tombs, Niger. *Journal of Archaeological Science*, 22 : 301-312.

Sealy J. & Yates R. 1994 - The chronology of the introduction of pastoralism to the Cape, South Africa. *Antiquity,* 68 : 58-68.

Sereno P.C., Garcea E.A.A., Jousse H., Stojanowski C.M., Saliège J.-F., *et al.* 2008 - Lakeside Cemeteries in the Sahara : 5000 Years of Holocene Population and Environmental Change. *PLoS ONE*, 3 : 2995.

Shipman P., Foster G. & Schoeninger M. 1984 - Burnt bones and teeth : an experimental study of color, morphology, crystal structure and shrinkage. *Journal of Archaeological Science,* 11 : 307-325.

Schoeninger M.J. & DeNiro M.J. 1982 - Carbon isotope ratios of apatite from bone cannot be used to reconstruct diets of animals. *Nature,* 297 : 577-78.

Sponheimer M. Passey B.H., de Ruiter D.J., Guatelli-Steinberg D. Cerling T.E. & Lee-Thorp J.A. 2006 - Isotopic Evidence for Dietary Variability in the Early Hominin. *Paranthropus robustus*. *Science*, 314 : 980-982.

Stiner M.C., Kuhn S.L., Weiner S. & Bar-Yosef O., 1995 - Differential burning, recrystallization, and fragmentation of archaeological bone. *Journal of Archaeological Science*, 22 : 223-237.

Surovell T.A. 2000. Radiocarbon dating of bone apatite by step heating. *Geoarchaeology,* 15 : 591-608.

Tamers M.A. & Pearson F.J. 1965 - Validity of radiocarbon dates on bone. *Nature,* 208 : 1053-1055.

Taylor R.E., Hare P.E. & White T.D. 1995 - Geochemical criteria for thermal alteration of bone. *Journal of Archaeological Science,* 22 : 115-119.

Trautz O.R. 1967 - Crystalline organization of dental mineral. *In* : *Structural and Chemical organization of teeth,* Miles A.E.D. (ed). London, Academic Press : 165-200.

Van Strydonck M., Boudin M., Hoefkens M. & De Mulder G. 2005 - ^{14}C dating of cremated bones, why does it work? *Lunula,* 13 : 3-10.

Van Strydonck M., Boudin M. & De Mulder G. 2009 - ^{14}C dating of cremated bones: the issue of sample contamination. *Radiocarbon,* 51 : 553-568.

Weinmann J.P., Wessinger G.D. & Reed G. 1942 - Correlation of chemical and histological investigation on developing enamel. *Journal of Dental Research,* 21 : 171-182.

Zazzo A., Saliège J.-F., Person A. & Boucher H. (2009) - Radiocarbon dating of cremated bones : where does the carbon come from? *Radiocarbon*, 51 : 601-611.

Zilhao J. 2001 - Radiocarbon evidence for maritime pioneer colonization at the origins of farming in west Mediterranean Europe. *Proceedings of the National Academy of Sciences,* 98 : 14180-14185.

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TAPHONOMIC IMPACT OF PROLONGED COMBUSTION ON BONES USED AS FUEL

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Abstract

The combustion of bones results in numerous processes whose impact on the representivity of fossil bone assemblages is increasingly well known due to the multiple experimental approaches developed over the last ten years. Recent experiments conducted with outdoor hearths have shown the consequences of prolonged combustion on bone combustion residues.

The average loss of bone mass after combustion is 65%. The weight of the fine fraction (ashes and fragments less than 2 cm) corresponds to more than one quarter of the residual mass of the remains collected, while the mass of calcined (i.e. white) bone represents an average of 77.2% of the residues. Finally, the residual bone mass is not correlated with the duration of use of a hearth, but with the manner in which it is maintained. These experiments thus clearly document the significant role of fire maintenance methods on the nature and form of bone residues.

Keywords : experimentation, burned bones, bone fuel, hearth, taphonomy

Introduction

The hypothesis that bone was used as fuel in hearths has been proposed for several Paleolithic sites. Until recently, these interpretations were most often based on the abundance of burned bones found in association, or not, with combustion features, regardless of the diversity of possible origins for bone combustion: intentional discard of bone waste in hearths (Spennemann & Colley, 1989; Cain, 2005), alimentary cooking (Gifford-Gonzalez, 1989; Pearce & Luff, 1994; Wandsnider, 1997; Costamagno & Fano Martínez, 2005), ritual combustion (Tchesnokov, 1995; Vaté & Beyries, 2007), accidental combustion after burying (David, 1990; Stiner *et al.*, 1995; Bennett, 1999; Cain, 2005) and natural fires (Bellomo & Harris, 1990; Bellomo, 1993). A series of laboratory experiments (tab. 1) enabled the definition of the combustible properties of bones (Théry-Parisot & Costamagno, 2005; Théry-Parisot *et al.*, 2005), as well as a more precise characterization of the bone remains originating from this type of hearth (Théry-Parisot *et al.*, 2004; Costamagno *et al.*, 2005). Based on these results, a statistical model of the origin of burned osseous assemblages was proposed (Costamagno *et al.*, 2009). Despite these advances, a certain number of questions persist concerning both the post-depositional processes that can modify burned osseous assemblages and the identification of burned osseous residues originating from other combustion contexts (Costamagno *et al.*, 2009). For example, with the exception of sites in acid contexts Gilchrist & Mytum, 1986; Costamagno *in* Bordes & Lenoble, 2000; Airvaux *et al.*, 2003), the quantity of intensively burned bones (gray or white) is generally low in archaeological sites, which strongly contrasts the quantities obtained through the experimental use of bone as fuel. According to different studies, calcined (i.e. burned white) bones are more reactive to mechanical constraints than bones that are simply carbonized or *a fortiori* non-burned (Stiner *et al.*, 1995; Thiébaut *et al.*, in press), which could explain the recorded distortions. A prolonged exposition to atmospheric agents could result in a similar phenomenon (Gerbe, 2004, Gerbe this volume). Experiments in progress by two of us, conducted as part of the Gavarnie research program (directed by P. Bertran) and taphonomy workshops (directed by M.-P. Coumont), will contribute new elements concerning the biases introduced by other taphonomic processes in the context of burned osseous remains (human trampling, gelifraction and dissolution).

In parallel to these experiments concerning the action of taphonomic processes after combustion, new experiments related to the combustion of bone materials have been undertaken. This project is conducted in conjunction with the study of the open-air Aurignacian site of Régismont-le-Haut (Poilhes, Hérault), which has yielded around a dozen combustion features, including several in association with wood charcoal and burned bone (Maurin & Ambert, 1979; Bon, 2002; Bon, Mensan and collaborators, 2007). In this paper, we present the results of a first series of experiments concerning the prolonged maintenance of hearths in which bone is used as fuel. These experiments, realized in August 2006 in close proximity to the site, were conducted with three objectives:

1 – to obtain a better understanding of the functioning of the hearths at Régismont-le-Haut and to determine the type(s) of combustible materials used by its Aurignacian occupants;

Experimental protocol	
	laboratory experiments bone (cow humerus) as only fuel used no fuel added
Variables tested	
	Bone dessication (dry (non humid)/fresh) Bone fragmentation (whole/fractured) Bone tissue (compact/spongy)

Tab. 1 - Experimental protocol and variables tested in the first experimental series realized in the laboratory.

[1] - *e.g.* Abri Pataud (Théry-Parisot, 2002), Cuzoul de Vers (Castel, 2003), Esquilleu (Yravedra *et al.*, 2005), Le Flageolet I (Bombail, 1987), Hohle Fels (Schiegl *et al.*, 2003), Labeko Koba (Yravedra *et al.*, 2005), Pech de l'Azé I (Rendu, 2007), Le Placard (Costamagno *et al.*, 1998), Saint-Germain-la-Rivière (Costamagno *et al.*, 1998), Brassempouy (Letourneux, 2003) and several layers gravettians of central Europe (Soffer, 1985).

2 – to evaluate the constraints related to the behaviour of a fire fuelled with osseous materials;
3 – to evaluate the impact of prolonged combustion on osseous residues.
In this paper, we focus on the latter objective.

Experimental methods and procedures

The experiments were conducted outside, in the open-air. The hearths (8) were installed on a horizontal ground surface in shallow pits 5 cm deep and 50 cm in diameter (fig. 1). The bones used as fuel were principally salted pork bones and a few fresh cow bones (tab. 2). The bones were meticulously defleshed and then fractured with quartzite cobbles on limestone anvils (fig. 2) comparable to those found on the site (Bon, Mensan and collaborators, 2006). The marrow was removed from the bones before they were burned. The wood necessary to start the fire represents 5% of the weight of the bone used during ignition (fig. 3). The hearths were then exclusively fuelled with bone (fig. 4), except when the temperature dropped too low for the combustion to continue. In this case, a quantity of bone identical to the initial one was used. At the moment of ignition, and at each reloading with combustible materials, the hour and exact nature of the bones (taxon and anatomical portion) was recorded, along with their total weight and number of fragments.

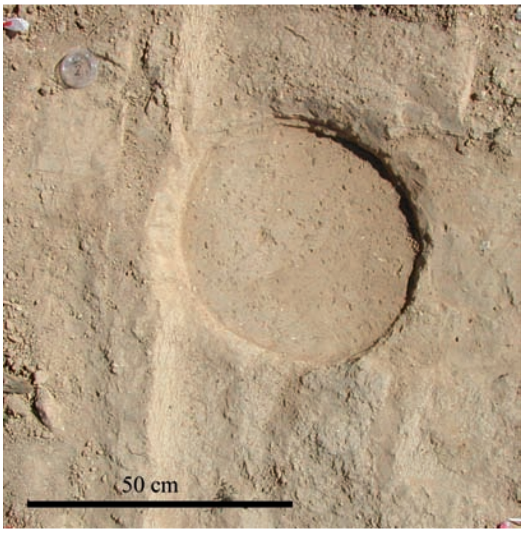


Fig. 1 - Hearth pit.

Fig. 2 - Bone defleshing and fracturation.

Fig. 3 - Combustibles before the fire was lit.

Fig. 4 - Feeding the fire with bones.

	Series 1				Series 2			
	Hearth 1	Hearth 2	Hearth 3	Hearth 4	Hearth 1	Hearth 2	Hearth 3	Hearth 4
Pork								
Pelvis	1	2	5	3	2	2	1	1
Proximal femur	30	32	32	31	15	16	17	16
Distal femur	33	30	32	33	18	15	15	14
Proximal tibia	35	33	33	32	15	15	15	15
Patella	17	16	18	18	5	5	6	5
Tarsal massif	1	1	1	1	2	2	1	2
Cow								
Cervical							1	
Thoracic						1		1
Rib	4	6	6	4		1		1
Scapula	1	1	1	1	1	1		
Proximal humerus				1		1		
Distal humerus							1	
Proximal radius	1							
Carpal massif			1					
Pelvis							1	1
Proximal femur					1			
Distal femur		1					1	
Proximal tibia					1			1
Tarsal massif						1		
Bone mass	**17040**	**17603**	**17214**	**18235**	**9781**	**10327**	**10143**	**9503**
Average maximal temperatures (°C)	**-**	**560,8**	**681,6**	**-**	**503,5**	**569,3**	**692,7**	**-**
Maintenance method	**rapid**	**rapid**	**rapid**	**rapid**	**slow**	**slow**	**slow**	**slow**

Tab. 2 - Experimental parameters: skeletal elements used, bone mass employed, fireplace maintenance methods, average maximal temperatures recorded by the three sensors in the same fireplace (the sensors in fireplace 1 of the first series did not function).

Fig. 5 - Bone residues after combustion.

The experiments were realized following two distinct protocols with the purpose of varying the combustion durations and rate of maintenance of the hearths. In the first series, the fires were fed with no particular precaution, which we designate as a “rapid feeding”. In the second series the fires were fed with objective of economizing the bone fuel and prolonging the combustion duration as long as possible: we designate this as “slow feeding”. Each series was replicated four times. In the first series, an average of 16,326 grams of bone was burned versus 9,334 grams in the second series (tab. 2). After they were completely cooled, the bone residues were collected (fig. 5), with the exception of those of the fourth hearth of each series, which were left *in situ* in order to record their evolution over several years.

Several factors were recorded during these experiments, starting with temperature. To record the variability of thermal fluxes, the first three hearths in the two series were equipped with three sensors that simultaneously recorded the temperatures every two minutes during the full duration of the experiments. The data were recorded and transmitted into computer format by infrared. For each experiment, three curves that express the temperatures according to time were thus recorded. The second factor relates to kinetics by distinguishing between combustion with flame emission and pyrolysis without flames or calcination (the process that follows the extinction of the flames until the end of calcination). In practice, we considered that the combustion was finished when the average temperature of the hearth recorded by the sensors was below 100° C. In the experiments of the first series, the hearths were fed in two stages: in the morning for an average duration of two hours, then four hours later for a duration of two and a half hours. For the flame duration, we added the two recorded durations. For the calcination duration, we took into account only the first combustion phase. For this reason, the calcination durations of the fires of the first experimental series are not exploitable for taphonomic analyses as the osseous residues result from the two successive combustion phases.

The bone residues were then sorted by 10 mm size classes. The fragments over 20 mm were also sorted according to their tissue type (spongy, compact or compact + spongy) and combustion intensity (non burned, partially burned, mostly black, mostly gray or mostly white) (fig. 6). The bones of each category were then weighed. The fine fraction, after sieving the residues through 0.5 and 0.2 mm screens, were systematically weighed for each experiment (fig. 7 and 8).

The results of the experiments realized at Régismont-le-Haut were compared to those obtained in laboratory experiments (Théry *et al.*, 2004; Costamagno *et al.*, 2005; Théry *et al.*, 2005) according to the following criteria:

- combustion kinetics;
- loss of material, recorded through the percentage of residual mass, meaning the percentage of bone mass collected after combustion relative to that of the bone mass put into the fire;

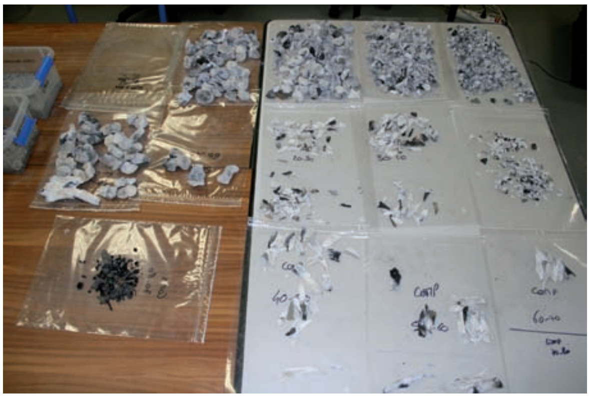


Fig. 6 - Sorting of bone residues.

Fig. 7 - Fragments less than 2 cm

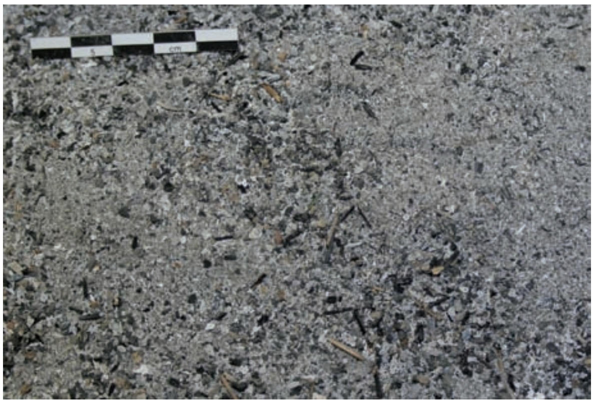


Fig. 8 - Bone ash.

- intensity of fragmentation, estimated based on the relative abundance of bone fragments by 10 mm size class;
- combustion intensity, estimated based on the relative abundance of calcined bones, meaning those that are mostly white (fig. 9).

Fig. 9 - Calcined bones

Results

Preliminary observations

The first experiments conducted in the laboratory showed that the fresh or dry state of bone strongly influences the degree of fragmentation of bone residues; the high fragmentation of fresh bone relative to dry bone is probably due to the high pressure created by water evaporation (Théry *et al.*, 2004; Costamagno *et al.*, 2005).

In the experiments conducted at Régismont-le-Haut, the use of salted pork bones, which are relatively dry, could thus explain the presence of numerous articular extremities and whole epiphyses among the bone residues (fig. 10). For comparisons, we thus favoured laboratory experiments in which the bones were burned after desiccation and fracturation.

Fig. 10 - Whole epiphyses of pork long bones.

Meanwhile, some effects were observed only on the cow bones, and thus exclusively on fresh and humid bones. On certain spongy fragments, we observe the formation of compact surface scales that develop in a concentric manner over the entire surface (fig. 11). As they detach, they progressively expose the spongy tissue. The bones affected can thus eventually take the form of a spongy ball with no *compacta*. Such objects have been observed in many faunal assemblages.

Numerous laboratory experiments have shown that the maximal temperatures attained in the hearths depends on the mass of the combustible material and not on the material itself (Théry-Parisot & Costamagno, 2005).

The hearths in the new experiments conducted in the open-air attained maximal temperatures between 561 and 692°C. The temperatures of these fires thus appear inferior to those recorded for the fires in the laboratory (605–805°C), though the mass of combustible material was greater. The differences observed can be easily explained by the impact of atmospheric agents on the combustion temperatures during the open-air experiments. In addition, the intra-series variability is greater than the inter-series variability (tab. 2). For this reason, the results obtained are difficult to exploit for questions concerning temperatures.

Fig. 11 - Concentric surface scales that gradually expose the *spongiosa*.

Parameters that influence the kinetics of combustion

The first laboratory experiments showed that the flame duration is determined by the combined effects of humidity, the density of the bone tissues and their fragmentation, while the initial mass of the combustible material put into the fire explains only 24% of the variability (Théry-Parisot & Costamagno, 2005).

For an equivalent fuel mass, the flame duration appears to be longer in the open-air hearths than in the laboratory hearths (in which no bone materials were added) (fig. 10). In the latter, the average flame duration is 112 minutes for the hearths with proximal extremities versus 165 for those with distal extremities, as opposed to 232 minutes for series 1 at Régismont-le-Haut and 356 for series 2. A variance analysis shows that the average duration of the flames in series 1 differs significantly from that of series 2 (fig. 12). The hearths of series 1 (rapid feeding) require 54% more bone than the hearths of series 2 (slow feeding) to produce flames for an identical amount of time. In terms of bone fuel consumption, the flame durations in series 1 generally follow the same tendency as those observed in the laboratory experiments, in which no fuel was added to the fires (fig. 13). The hearths of series 2, on the other hand, show a real economy of bone fuel when the fires are progressively fed. This is not the case for

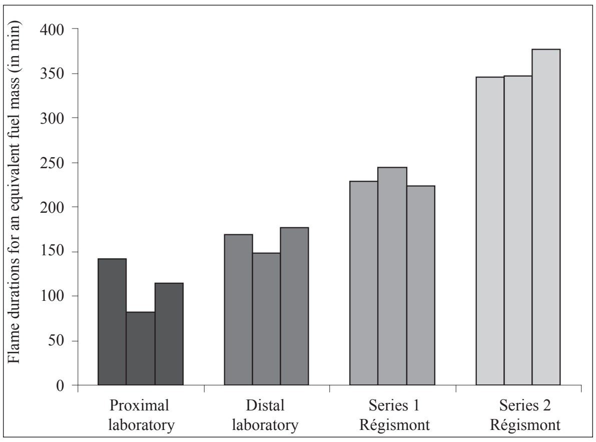


Fig. 12 - Flame duration for an equivalent fuel mass.

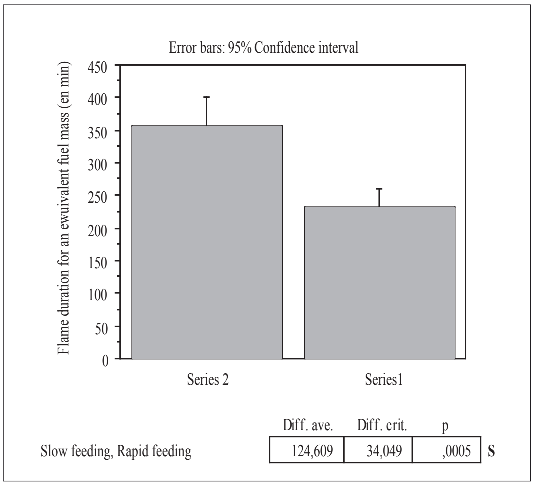


Fig. 13 - Effect of maintenance methods on flame duration (Fisher's exact test)

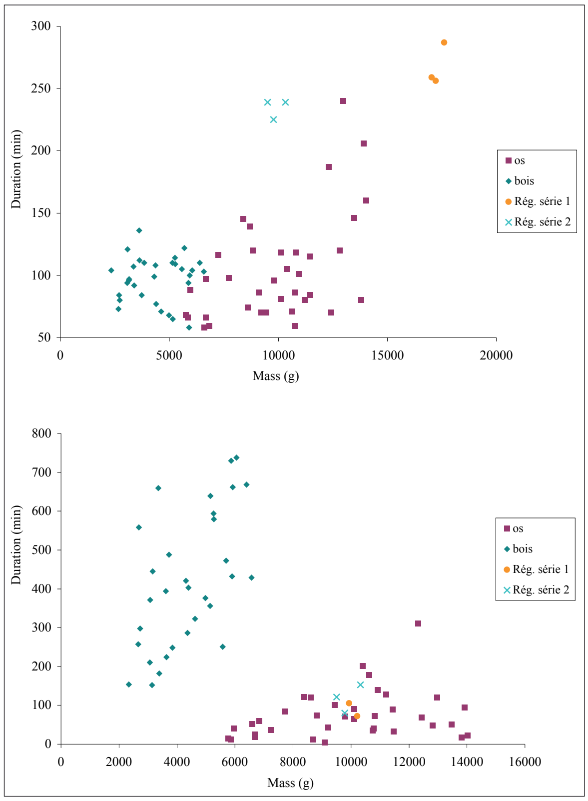


Fig. 14 - Combustion duration in function of the fuel mass used: a- flame duration, b- calcination duration.

the calcination durations. Regardless of the maintenance methods, they remain low and are comparable to those recorded in the laboratory experiments (fig. 14).

Loss of material

The laboratory experiments, conducted under controlled conditions, showed that the reduction of mass is strongly correlated with the bone density: the higher the density, the higher the percentage of residual mass. On the other hand, the state of the bone before combustion (fresh/dry, whole/fragmented) has no effect on this variable (Théry *et al.*, 2004 ; Costamagno *et al.*, 2005).

In the experimental series realized at Régismont-le-Haut, the percentage of resdidual mass varies between 33.2 and 36.6% (fig. 15). These values, which are statistically comparable to those recorded for the distal extremities of humeri, could be explained by the simultaneous combustion of spongy and compact portions (fig. 16). If we consider the ensemble of experiments, it appears that flame duration is not a significant factor in the loss of mass, as is shown by the Spearman's rank correlation coefficient, which is not statistically significant (r_s = - 0,215). Even if we distinguish the laboratory experiments (proximal series and distal series) and those at Régismont-le-Haut, the correlation coefficients remain low and non significant, respectively - 0,288: ddl 13; - 0,350: ddl 13 et - 0,551: ddl 6 (fig. 15).

Concerning the relationship between the loss of mass and calcination duration, in previous experiments we have shown the existence of three distinct groups: fires in which diaphyses without marrow were burned, fires in which distal extremities were burned, and fires with proximal extremities (fig. 17). In these latter, the loss of mass is strongly correlated with the duration of calcination (r_s = - 0,790, p < 0,01): the longer the calcination duration, the greater the amount of material lost, which is not the case for combustions

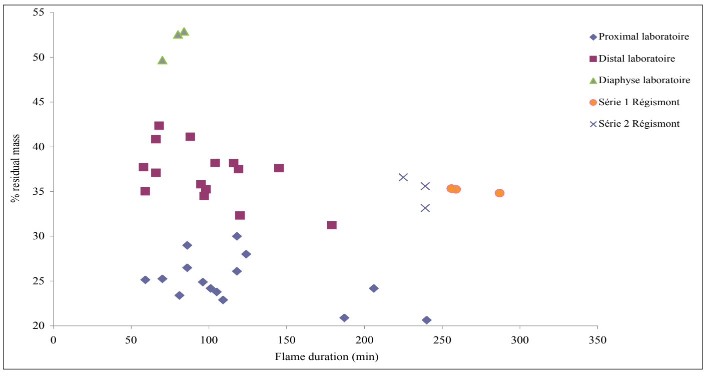
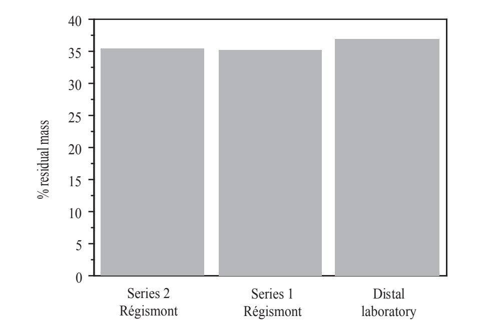


Fig. 15 - Percentage of residual bone mass in function of flame



	Diff. ave.	Diff. crit.	p
Series 1, Series 2	-,012	4,794	,9960
Series 2, Distal laboratory	-1,859	3,713	,3067
Series 1, Distal laboratory	-1,848	3,713	,3096

Fig. 16 - Effect of maintenance methods on the percentage of residual bone mass (Fisher's exact test).

realized with distal extremities (r_s = - 0,358). If we make the same comparison with series 2 of Régismont-le-Haut (the data from series 1 is not exploitable in this context (*cf. supra*) the same type of relationship appears; once again, the longer

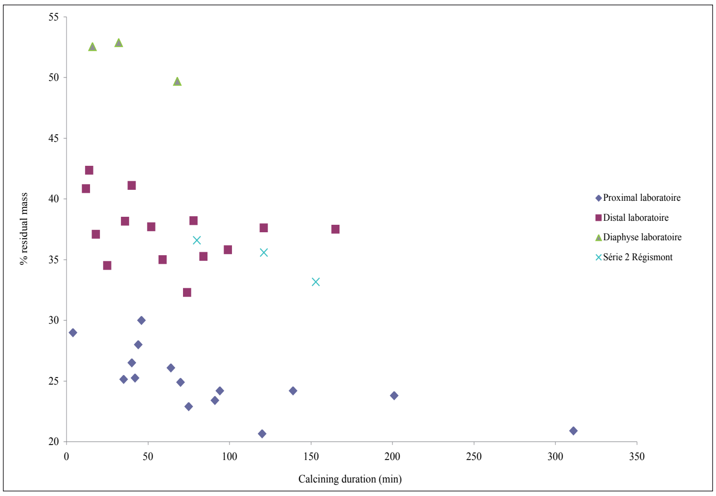
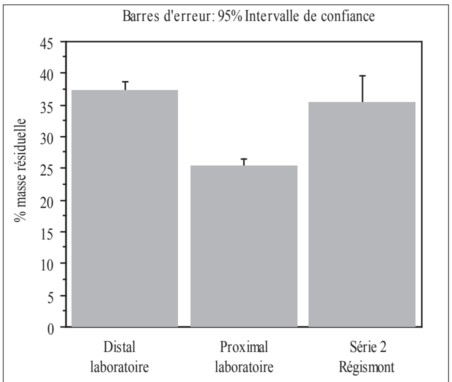


Fig. 17 - Percentage of residual bone mass in function of calcination duration.

the calcination duration, the greater the loss of material. Additional data will be necessary to determine if this relationship is statistically significant. In comparison to the hearths fueled by the proximal extremities of humeri, for the same calcination duration, the loss of mass in the hearths at Régismont-le-Haut is clearly lower. It thus appears that the proximal extremities of humeri, and probably the portions with a low density, behave in a very specific way in fire, as is shown by a variance analysis (fig. 18).



	Diff. moy.	Diff. crit.	Valeur p	
Distal, Proximal	11,978	2,106	<,0001	S
Distal, Série 2 Régismont	1,859	3,648	,3062	
Proximal, Série 2 Régismont	-10,119	3,648	<,0001	S

Fig. 18 - Effect of bone density on the percentage of residual bone mass (Fisher's exact test).

Intensity of fragmentation

The state of bone before combustion (dry/fresh, whole/fragmented) plays a role in the degree of fragmentation of bone residues (Théry *et al.*, 2004; Costamagno *et al.*, 2005). The bones dried before burning fragment six times less than humid bones, while the fractured bones re-fracture very little, the average size of the combustion residues being greater. Therefore, for the comparisons, only the laboratory experiments in which the humeri were dry and fractured were used.

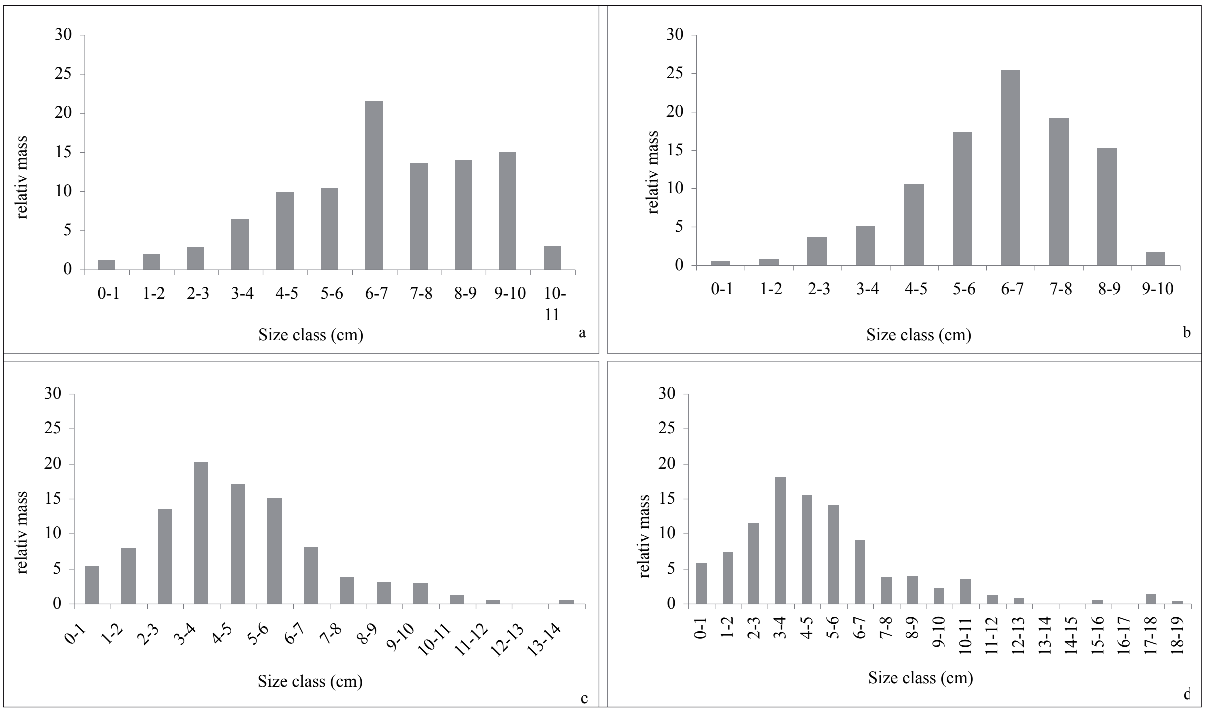


Fig. 19 - Distribution of bone residues according to their size: a- laboratory series, dry and fragmented proximal extremities; b- laboratory series, dry and fragmented distal extremities; c- Régismont, series 1; d- Régismont, series 2.

In the experiments realized at Régismont-le-Haut, the 3-4 cm fragment class is the best represented in all series, while in the laboratory experiments, the 6-7 cm class is always dominant (fig. 19). Is this difference due to a more intensive fragmentation of bones in the series with a prolonged combustion? It is difficult to respond to this question since the bones used were not of the same size (tables 1 and 2). For the small fragments, the question of the size of bones put into the fire is not relevant. The fragments less than 2 cm appear clearly more abundant in the experimental series of Régismont-le-Haut, (average 13.3%) than in the laboratory series (2.3%) (fig. 19). As we could expect, a prolonged combustion results in a more intensive fragmentation of residues, which is represented by a significant increase in the quantity of small fragments (lower than 2 cm). At the same time, in the prolonged combustions, a notable part of the bone residues is reduced to the state of ashes (between 12 and 14% of the residual mass. The values recorded for the fine fraction (% of ash and fragments smaller than 2 cm) are comparable from one series to the other, as is shown by a variance analysis (fig. 20).

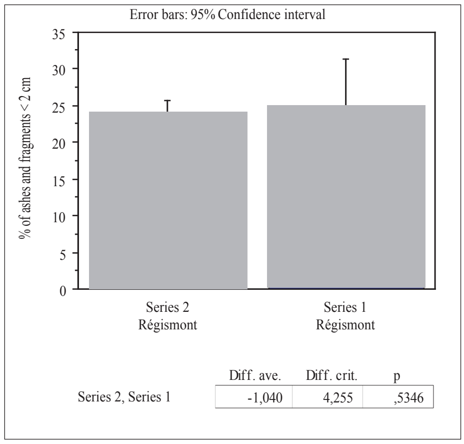


Fig. 20 - Effect of maintenance methods on the relative mass of the fine fraction (Fisher's exact test).

Intensity of combustion

The calcined remains in the prolonged combustion hearths represent an average of 77.2% of the total weight of the osseous residues versus 32% in the laboratory experiments (fig. 21).

In the laboratory experiments, we showed that the percentage of calcined bones was not correlated with the flame duration. The differences recorded are related to the density of the spongy tissue and the degree fragmentation of the bones before combustion (Théry-Parisot *et al.*, 2004). In the prolonged combustions, the hearths were initiated and then secondarily fed with the same types of combustible materials. In these experiments, in which the type of fuel does not play a role, the percentage of calcined bones seems logically correlated with the flame duration: the longer the hearth functions, the more intensively the bones are burned (fig. 21). The variance analysis shows a highly significant difference between the two experimental series at Régismont-le-Haut in terms of the proportion of calcined bones: the rapidly fed hearths (series 1) have an average of 15% more calcined bones than the slowly fed hearths (series 2) (fig. 22). Therefore, in addition to the flame duration, the methods of maintaining the hearths also play a notable role in the combustion intensity of osseous residues. In the rapidly fed hearths, the greater intensity of the flames accentuates the combustion process.

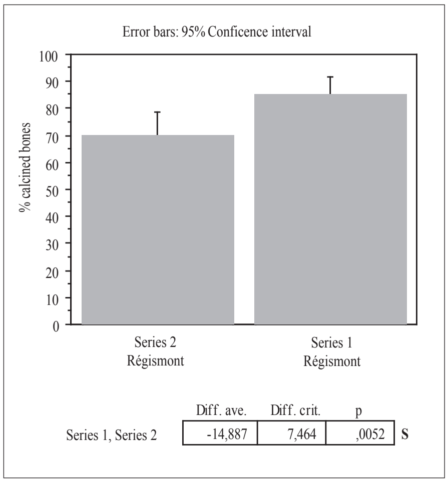


Fig. 22 - Effect of maintenance modalities on the relative mass of calcined remains (Fisher's exact test).

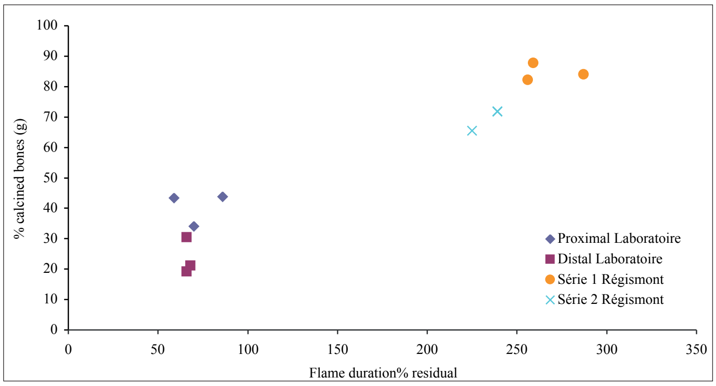


Fig. 21 - Relative mass of calcined bones in function of flame duration.

Concerning the tissue type, the spongy portions in both experimental series are more frequently calcined than the fragments of compact tissue or compact tissue with spongy tissue (fig. 24). Carbonized spongy remains are very poorly represented. The spongy tissues appear, moreover, to be more intensively fragmented than the compact tissues, which themselves are smaller in size than the fragments of *compacta* with *spongiosa* (fig. 25). The fragments smaller

Multi-criteria interrelations

A confrontation of the criteria "fragment size class" and "combustion intensity" indicates that the calcined remains are more intensively fragmented once the combustion is completed than the mostly black or mostly gray remains, regardless of the series considered. Measured by weight, the fragments over 6 cm are clearly dominant among the black and gray bones, while for calcined bones, the 3-4 cm class dominates, the distribution by size class being more homogeneous (fig. 23).

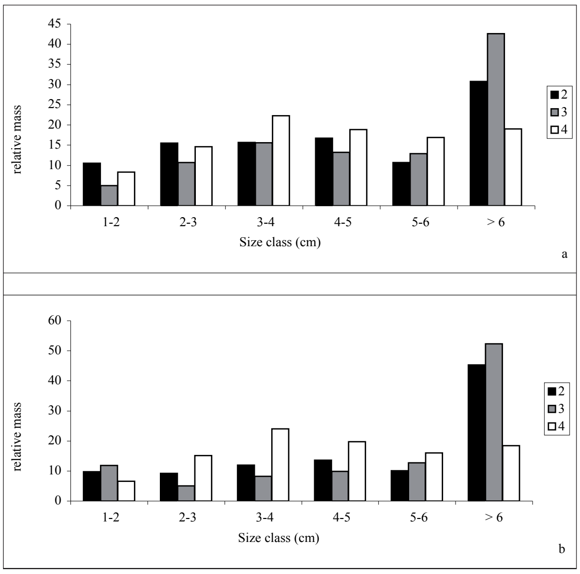


Fig. 23 - Relative size of bone fragments according to combustion intensity: a- Régismont, series 1; Régismont, series 2 (2: mostly black bones, 3: mostly gray bones, 4: mostly white bones).

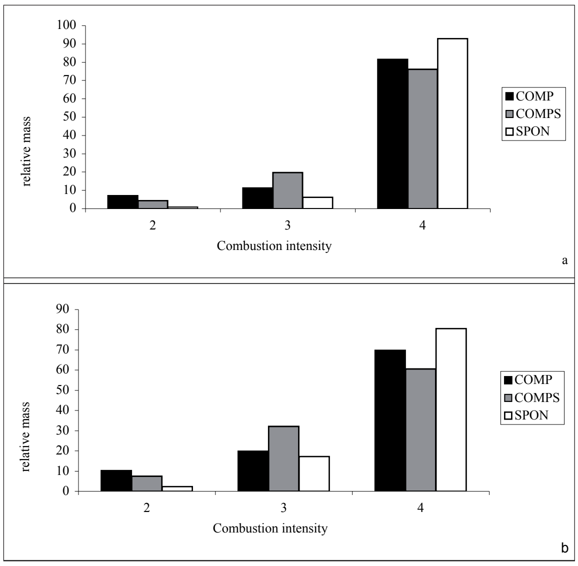


Fig. 24 - Relative combustion intensity of bone fragments according to bone tissue type: a- Régismont, series 1; b- Régismont, series 2 (COMP: compact tissue fragment, COMPS: compact tissue with spongy tissue fragment, SPON: spongy tissue fragment).

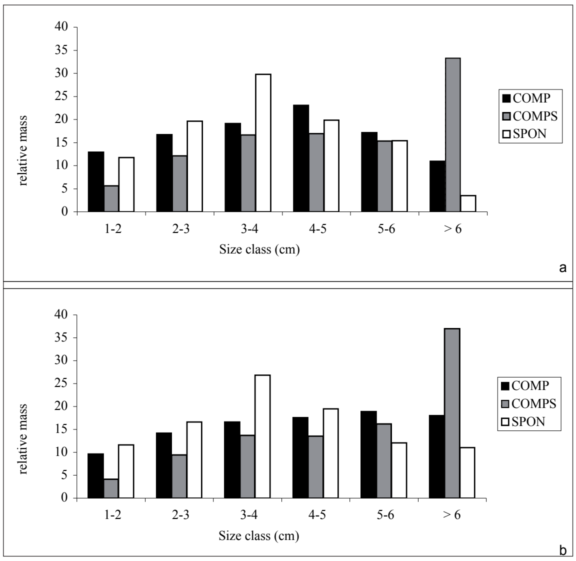


Fig. 25 - Relative size of bone fragments according to bone tissue type: a- Régismont, series 1; b- Régismont, series 2 (COMP: compact tissue fragment, COMPS: compact tissue with spongy tissue fragment, SPON: spongy tissue fragment).

than 4 cm thus represent an average in the two series of 58.1% of the residual mass of spongy tissues versus 44.6% of the compact tissue and 30.8% of the fragments of compact tissue with spongy tissue. Since the initial size of the fragments of each tissue type was not recorded before combustion, a potential bias related to the type of material used cannot be excluded. In series 1, relative to series 2, we observe a greater fragmentation of osseous remains regardless of the tissue type in relation to the flame intensity (remains less than 4 cm: spongy – series 1: 61.2%; series 2: 55.1%; compact – series 1: 48.8%; series 2: 40.4%; compact + spongy – series 1: 34.4%; series 2: 27.2%).

Conclusions

The experiments conducted at Régismont-le-Haut provide information complementary to that obtained in laboratory experiments and contribute to a better characterization of burned osseous residues associated with the use of bone as fuel.

It appears that in a hearth maintained with bone fuel, the duration of the flames is not correlated with the mass of combustible material burned. In addition to the factors identified in laboratory experiments (tissue type, degree of fragmentation), the methods of hearth maintenance play a determinant role. If we vary only this parameter, for the production of an equivalent flame duration, the slow feeding method allows an economy of one third of the fuel necessary for the rapid feeding method. Therefore, even if we could identify the impact of the different taphonomic agents that can modify burned bones, it is impossible based on the mass of bone fuel recorded in an archaeological assemblage to estimate the use duration of hearths at a given site.

These new experiments confirm that flame duration has very little influence on the loss of bone residue mass. It is the bone density, and thus probably the quantity of fat (Lyman, 1985), that plays a determinant role. In the experiments realized at Régismont-le-Haut, the loss of bone mass is on average 65%. A prolonged combustion also results in a more intensive fragmentation and combustion of osseous residues: fragments less than 2 cm long are six times more abundant in the experiments conducted at Régismont-le-Haut than in the laboratory experiments, and the calcined pieces two times more abundant. The fine fraction (ash and fragments less than 2 cm) corresponds to more than one quarter of the residual bone mass in the two open-air series, while the calcined pieces represent more than three quarters of this mass.

Contrary to the degree of fragmentation, the intensity of calcination depends largely on the methods of maintaining the hearths: the residues from rapidly fed hearths (series 1 at Régismont-le-Haut) are more intensively burned than those of slowly fed hearths (series 2 at Régismont-le-Haut). It would be interesting, through the maintenance of hearths over several days, to determine if the fine fraction and calcined piece proportions increase progressively according to the duration of use of the hearths.

Among the osseous residues produced by the combustions realized at Régismont-le-Haut, calcined pieces are smaller in size than the black and gray fragments. They are also more numerous regardless of the bone tissue type (60.5 to 92.9% of the residual mass), though in both experimental series, the spongy fragments are always more intensively burned than compact bone or compact bone with spongy tissue fragments.

What conclusions can be drawn from these observations? The experiments conducted at Régismont-le-Haut show that a fire fuelled with bone for a few hours produces a significant quantity of ash (approximately 15% of the residual mass), as well as a multitude of bone fragments less than 2 cm long (13% of the residual mass). Since small fragments can be further reduced in size by diverse taphonomic agents, the use of bone as fuel can considerably diminish the proportion of faunal remains at a site relative to lithic remains, which can be problematic for interpretations of site function and animal exploitation. Various studies have moreover shown that certain taphonomic processes, such as trampling (Stiner *et al.*, 1995; Thiébaut *et al.*, in press) or weathering (Gerbe, this volume), result in a preferential destruction of spongy and calcined bones, which correspond more or less to the osseous residues collected when bone is used as fuel. In rock shelters and caves, where occupations can be repeated over time, human trampling can have significant repercussions for the preservation of burned bone residues and thus mask a potential use of bone as fuel. The same is true for the action of atmospheric agents at open-air sites.

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References

Airvaux J., Berthet A.-L. & Castel J.-C. 2003 - Le gisement aurignacien de Chez-Pinaud 2 à Jonzac, Charente-maritime. *Préhistoire du Sud-Ouest*, 10 : 25-76.

Bellomo R.V. 1993 - A methodological approach for identifying archaeological evidence of fire resulting from human activities. *Journal of Archaeological Science*, 20 : 525-553.

Bellomo R.V. & Harris J.W.K. 1990 - Preliminary reports of actualistic studies of fire within Virunga National Park, Zaïre : toward an understanding of archaeological occurence. *In* : *Evolution of environments and Hominidae in the African Western Rift Valley*, Boaz N.T. (ed). Martinsville, VA, Virginia Museum of Natural History : 317-338.

Bennett J.L. 1999 - Thermal alteration of buried bone. *Journal of Archaeological Science*, 26 : 1-8.

Bombail C. 1987 - *Les structures de combustion des niveaux aurignaciens du Flageolet I*. Mémoire de Maîtrise, Université de Neuchâtel.

Bon F., Mensan R. avec la collaboration de **Costamagno S., Araujo Igreja M., Gardère Ph., Kuntz D., Ménard C., Sellami F. & Szmidt C.** 2006 - *Régismont-le-Haut (Poilhes, Hérault). Campagne de fouilles d'un habitat de plein air aurignacien*. Rapport d'opération de fouille programmée, 68 pp., 9 fig.

Bon F., Mensan R. avec la collaboration de **Araujo Igreja M., Costamagno S., Gardère P., Ménard C., Sellami F., Szmidt C. & Théry-Parisot I.** 2007 - Le site de plein air de Régismont-le-Haut : une halte aurignacienne dans les plaines du Languedoc. *In* : *Qui est l'Aurignacien ?* Aurignac, Editions Musée-forum : 53-71.

Bon F., 2002 - *L'Aurignacien entre Mer et Océan. Réflexion sur l'unité des phases anciennes de l'Aurignacien dans le sud de la France*. Mémoire de la Société Préhistorique Française, XXIX, 253 pp.

Bordes J.-G. & Lenoble A. 2000 - *Caminade (Sarlat, Dordogne)*. Document final de Synthèse, Service régional de l'Archéologie d'Aquitaine, Bordeaux.

Cain C.R. 2005 - Using burned animal bone to look at Middle Stone Age occupation and behavior. *Journal of Archaeological Science*, 32 : 873-884.

Castel J.-C. 2003 - Economie de chasse et d'exploitation de l'animal au Cuzoul de Vers (Lot) au Solutréen et au Badegoulien. *Bulletin de la Société Préhistorique Française*, 100 : 41-65.

Costamagno S. & Fano Martínez M.A. 2005 - Pratiques cynégétiques et exploitation des ressources animales dans les niveaux du Magdalénien supérieur-final de El Horno (Ramales, Cantabrie, Espagne). *Paleo*, 17 : 31-56.

Costamagno S., Griggo C. & Mourre V. 1998 - Approche expérimentale d'un problème taphonomique : utilisation de combustible osseux au Paléolithique. *Préhistoire Européenne*, 13 : 167-194.

Costamagno S., Théry-Parisot I., Brugal J.-P. & Guibert R. 2005 - Taphonomic consequences of the use of bones as fuel. Experimental data and archaeological applications. *In* : *Biosphere to Lithopshere. New studies in vertebrate taphonomy*, O'connor T. (ed). Proceedings of ICAZ, Durham, 2002. Oxford, Oxbow Books : 51-62.

Costamagno I. Théry-Parisot J-C Castel & J-P Brugal 2009 - Combustible ou non ? Analyse multifactorielle et modèles explicatifs sur des ossements brûlés paléolithiques. *In* : *Gestion des combustibles au Paléolithique et au Mésolithique : nouveaux outils, nouvelles interprétations*, Théry-Parisot I., Costamagno S., Henry A. (eds). Proceedings of workshop 21. XV congress IUSPP, Lisbonne, 2006. BAR International Series 1914, Oxford, Archaeopress : 65-84.

David B. 1990 - How was this bone burnt ? *In* : *Problem solving in taphonomy*, Solomon S., Davidson I. & Watson B. (eds). Tempus : 65-79.

Gerbe M. 2004 - *Etude taphonomique d'ossements brûlés*. Mémoire de D.E.A., Université de Provence, 93 pp.

Gifford-Gonzalez D. 1989 - Ethnographic analogues for interpreting modified bones : some cases from East Africa. *In* : *Bone modification*, R. Bonnichsen & M. Sorg (eds). Orono, Maine, Center for the Study of First Americans : 179-246.

Gilchrist R. & Mytum C. 1986 - Experimental archaeology and burnt animal bone from archaeological sites. *Circaea*, 4 : 29-38.

Letourneux C. 2003 - *Devine qui est venu dîner à Brassempouy ? Approche taphonomique pour une interprétation archéozoologique des vestiges osseux de l'Aurignacien ancien de la grotte des Hyènes (Brassempouy, Landes)*. Thèse de doctorat, Université de Paris I Panthéon-Sorbonne, 603 pp.

Lyman R.L. 1985 - Bone frequencies, differential transport, and the MGUI. *Journal of Archaeological Science*, 12 : 221-236.

Maurin G. & Ambert P. 1979 - Une structure aurignacienne à Régismont-le-Haut. *Les amis de Nissan-lez-Ensérune*, 40 : 6-9.

Pearce J. & Luff R. 1994 - The taphonomy of cooked bones. *In* : *Whither environmental archaeology ?*, Luff R. & Rowley-Conwy P. (eds). Oxford, Oxbow Monograph : 51-56.

Rendu W. 2007 - *Planification des activités de subsistance au sein du territoire des derniers moustériens*. Thèse de doctorat, Université Bordeaux I, 363 pp.

Schiegl S., Goldberg P., Pfretzschner H.U. & Conard N.J. 2003 - Paleolithic burnt bone horizons from the Swabian Jura : Distinguishing between in situ fireplaces and dumping areas. *Geoarchaeology an International Journal*, 18 : 541-565.

Soffer O. 1985 - *The Upper Paleolithic of the Central Russian Plain*. Orlando, Academic Press, 539 pp.

Spennemann D.H.R. & Colley S.M. 1989 - Fire in a pit : the effects of burning of faunal remains. *Archaeozoologia*, 3 : 51-64.

Stiner M.C., Kuhn S.L., Weiner S. & Bar-Yosef O. 1995 - Differential burning, recrystallization, and fragmentation of archaeological bones. *Journal of Archaeological Science*, 22 : 223-237.

Tchesnokov Y. 1995 - La culture traditionnelle des éleveurs de rennes du Nord-Est de la Sibérie : problèmes et perspectives de développement. *In* : *Peuples des Grands Nords. Traditions et transitions*, Charrin A.-V.,Lacroix J.-M. & Therrien M. (eds). Paris, Presses de la Sorbonne Nouvelle, Institut National des Langues et civilisations Orientales : 305-314.

Théry-Parisot I. 2002 - Fuel management (bone and wood) during the Lower Aurignacian in the Pataud Rock Shelter (Lower Palaeolithic, Les Eyzies-de-Tayac, Dordogne, France). Contribution of experimentation. *Journal of Archaeological Science*, 29 : 1415-1421.

Théry-Parisot I., Brugal J.-P., Costamagno S. & Guilbert R. 2004 - Conséquences taphonomiques de l'utilisation des ossements comme combustible. Approche expérimentale. *Les Nouvelles de l'Archéologie*, 95 : 19-22.

Théry-Parisot I. & Costamagno S. 2005 - Propriétés combustibles des ossements. Données expérimentales et réflexions archéologiques sur leur emploi dans les sites paléolithiques. *Gallia Préhistoire*, 47 : 235-254.

Théry-Parisot I., Costamagno S., Brugal J.-P., Fosse P. & Guilbert R. 2005 - The use of bone as fuel during the Palaeolithic, experimental study of bone combustible properties. *In* : *The zooarchaeology of fats, oils, milk and dairying*, Mulville J. & Outram A.K. (eds). Proceedings of ICAZ, Durham, 2002. Oxford, Oxbow Books : 50-59.

Thiébaut C., Costamagno S., Coumont M.-P., Mourre V., Provenzano N. & Thery-Parisot I. sous presse - Approche expérimentale des conséquences du piétinement des grands herbivores sur les vestiges archéologiques. *Paleo*, numéro spécial.

Vaté V. & Beyries S. 2007 - Une ethnographie du feu chez les éleveurs de rennes du Nord-Est sibérien. *In* : *Les civilisations du renne d'hier et d'aujourd'hui. Approches ethnohistoriques, archéologiques et anthropologiques*, Beyries S. & Vaté V. (eds). XXVII[e] rencontres internationales d'archéologie et d'histoire d'Antibes. Antibes, APDCA-CNRS : 393-420.

Wandsnider L. 1997 - The roasted and the boiled : food composition and heat treatment with special emphasis on pit-hearth cooking. *Journal of Anthropological Archaeology*, 16 : 1-48.

Yravedra I., Baena J., Arrizabalaga A. & Iriarte M.J. 2005 - El empleo de material óseo como combustible durante el Paleolítico Medio y Superior en el Cantábrico.

Observaciones experimentales. *Museo de Altamira, Monografias*, 20 : 10-38.

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Article translated by Magen O' Farrell

THE ACTION OF *WEATHERING* ON BURNED BONE: AN EXPERIMENTAL APPROACH

Magali GERBE

Abstract

The alteration of bones following exposure to weathering is today well known, but it is possible that burned bones do not suffer the same type of changes since their physicochemical properties are modified by combustion. A series of experiments has been carried out with the aim of better understanding these reactions. These experiments form part of a more widespread attempt to better understand the impact of taphonomic agents on burned bones in order to estimate the distortions between bone material originating from experimental combustion and the fossil material.

Five experimental series resulting from the combustion of fresh cow humeri were exposed for eighteen months to weathering in a Mediterranean context. Several criteria were observed in order to highlight the impact of this exposure on the burned bones: degree of fragmentation, loss of bone mass, alteration of bone surface, influence of the degree of combustion (carbonised vs charred) and of the bone tissue (spongy vs compact) on the preservation of the material.

The results of these experiments mainly show a high fragmentation of the material (the small burned bones being in the majority), associated with a reduction in bone mass. In addition, spongy and charred bones have an increased sensitivity to the action of weathering, leading to their destruction. A preferential preservation of charred compact bone is thus expected.

Keywords : burned bones, bone fuel, experimentation, taphonomy, weathering

Introduction

Various studies have been conducted in order to understand the presence of burned bones discovered on archaeological sites. The proportion of these bones, whether or not associated with structures and other combustion materials (hearth, charcoals, ash, etc.), is sometimes very large. The main objective of these investigations was to determine the origin and nature of bone combustion: natural fire and accidental combustion *versus* anthropogenic origin (Spennemann & Colley, 1989; David, 1990; Stiner *et al.*, 1995; Bennett, 1999; Costamagno *et al.*, in press); and in the latter case, ignition origin: use of the bone as fuel (Costamagno *et al.*, in press), camp maintenance (Walters, 1988; Cain, 2005), culinary remains (Vigne *et al.*, 1981; Vigne, 1983; Walters, 1988; Laroulandie, 2001). These studies tend to focus on two complementary aspects:

1) the combustible properties of bone according to their tissue (spongy *vs* compact) and their state of freshness and fragmentation (Gilchrist & Mytum, 1986; Guillon, 1986; Buikstra & Sweggle, 1989; Spennemann & Colley, 1989; Fernandez Jalvo & Perales Piquer, 1990; Costamagno *et al*., 1999, 2005; Théry-Parisot, 2002; Théry-Parisot *et al*., 2004, 2005; Théry-Parisot & Costamagno, 2005; Mentzer, in press);

2) the modifications in colour and structure of the bone tissue according to the combustion temperature (Herrmann, 1977; Perinet, 1982; Shipman *et al*., 1984; Brain & Sillen, 1988; Susini, 1988; Spennemann & Colley, 1989; Brain, 1993; Nicholson, 1993; Sillen & Hoering, 1993; Stiner *et al*., 1995; Taylor *et al*., 1995; Trellisó Carreño, 2001; Joly & March, 2003; Pastó, 2003; Hanson & Cain, 2007).

The work conducted in connection with intentional combustion has highlighted, on one hand, more efficient combustion of the extremities of fresh long bones; on the other, high fragmentation of the bones, producing a majority of remains of small size (less than 2 cm), essentially charred (Costamagno *et al*., 2005; Théry-Parisot *et al*., 2004, 2005; Théry-Parisot & Costamagno, 2005).

In spite of these advances, several questions remain. In the majority of sites where burned bones are present in large numbers, the remains are mainly carbonised (black or brown) and not charred (white or grey). The hypothesis of greater fragility of charred bones as a result of the taphonomic filter is generally suggested. But combustion strongly modifies the physico-chemical properties of bones, so the behaviour of unburned bone when exposed to pre- and post-burial agents is thus not directly transferable to burned bone.

The few studies available on the subject highlight the different properties of burned bones when exposed to the various taphonomic agents. For some authors, burned bones are more vulnerable than unburned bones when subject to mechanical stresses (David, 1990; Stiner *et al.,* 1995), while for others, only burned bones would be preserved in poor preservation conditions, such as acidic soils (Gilchrist & Mytum, 1986). In recent years, studies on the taphonomy of burned bones have been carried out in order to better understand the response of burned bones to different taphonomic agents. The first approach to consider is the possible staining of bones after burial, when they are in contact with certain minerals in the soil (Franchet, 1933). The best known of these is manganese (Brain & Sillen, 1988; Shahack-Gross *et al*., 1997) which colours bones black, thus imitating a carbonised appearance. The impact of trampling (mechanical action) on burned bones has also been studied. High fragmentation of this category of remains and a greater sensitivity of charred bones relative to carbonised bones can be observed (Stiner *et al*., 1995). A study of trampling by modern bison of burned bones confirms these remarks, to which must be added the higher destruction rate of spongy tissue compared to compact tissue (Thiébaut *et al*., in press).

These studies concern post-burial processes, but burned bones are also subject to pre-burial agents. The work presented here fits into this context, with the aim of better understanding the impact of weathering on the differential preservation of burned bones: is loss of mass important? Are the bones more fragmented? What is the influence of the initial degree of bone fragmentation, the bone tissue (spongy *vs* compact) and the combustion intensity in the preservation, and thus the representation, of burned bones?

Materials and methods

The experimental material originates from combustions realised with adult cow humeri, following various protocols (*cf.* Théry-Parisot *et al.*, 2004, 2005 for a detailed description). The bone residues resulting from five experimental series were exposed to weathering:

- complete fresh proximal extremities
- fractured fresh proximal extremities
- complete fresh distal extremities
- fractured fresh distal extremities
- fresh whole humeri

Each series is comprised of a number of humeri (or portions of humeri), and thus of a bone mass, known before its deposit in the hearth (table 1). After each combustion, all of the ash was sieved in order to recover all bone remains.

Série	Nombre d'os	Poids
Extrémité proximale complète	10	11010
Extrémité proximale fracturée	10	6776
Extrémité distale complète	10	8400
Extrémité distale fracturée	10	5373
Humérus entier	20	11183

Tab. 1 : Description of the series, before combustion.

Before exposure to weathering, all of the burned bone remains were sorted, weighed and counted by size-class of one centimetre, tissue type, and combustion intensity. The 0-1 cm size-class was only weighed as a whole. Combustion intensity (Nicholson, 1993) relates to bone colour, five stages having been noted, from the most intensely burned to unburned: white, grey, black, brown, unburned. (Stiner *et al.*, 1995, modified by Castel, 1999 and Costamagno, 1999). The colour present over the majority of the bone fragment is taken into account, with no distinction being made between external and internal surfaces. For the bone tissues, three types were selected: compact, spongy and compact-spongy tissue.
Each series was then deposited separately in perforated wooden boxes, the bases of which were covered with a fine-mesh grill in order to prevent any loss of material, and placed on the roof of the CEPAM-UMR 6130 building (Valbonne, Alpes-Maritimes). The bones were exposed to the elements for eighteen months: from June 10, 2002 to November 6, 2003. The burned bones were subject to a Mediterranean-type climate (contrasting temperatures and irregular precipitation), constituting a transition between the temperate and tropical climates. It should be noted that summer 2003 was an abnormally dry period (a four month heat wave) during which precipitation was zero (fig. 1).

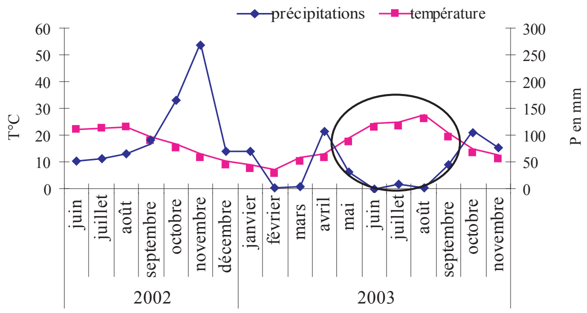

Fig. 1 : Ombrothermal diagram, Sophia-Antipolis site (data Météo France) black oval = dry period.

After exposure, the bones were sorted, weighed and counted according to the initial criteria. Observations of the surfaces (fissuring) were also made to allow comparison with the data resulting from work on the weathering of unburned bones (Behrensmeyer, 1978).
In order to evaluate the degree of fragmentation of the experimental material, an index was calculated which consisted of dividing the post-exposure number of remains (NR) by the pre-exposure NR. When the value is 1, fragmentation is zero; the higher the value, the greater the fragmentation.
Finally, a statistical analysis was employed to determin whether the differences observed between the series were significant or not. The presence of values that are too low (less than five) in the contingency tables makes it impossible perform a Chi-square analysis (Everitt, 1996). Similarly, the values that are too high for certain categories do not allow the application of an unmodified Fisher's Exact Test (F) and for this reason, a Monte Carlo was first performed. This latter produces a random sampling which then permits the application of Fisher's Exact Test, allowing data homogeneity to be tested (Everitt, 1996). The adjusted

values for each cell of the contingency table are observed; those for which the values are higher than two are significantly different (for $P < 0.05$) according to Fisher's Exact Test.

Results

The results are presented according to three analysis criteria: the state of the surfaces; the post-depositional fragmentation of the material and the bone mass. For the two latter issues, the influence of the bone tissue, combustion intensity and fragmentation intensity are taken into account in order to explain the evolution of the experimental series before and after exposure to weathering.

Surface fissuring

The action of atmospheric agents (in relation with the climate *s.l.*) on bones, more commonly known as *weathering,* has been defined by A.K. Behrensmeyer as: "*the process by which the original microscopic organic and inorganic components of a bone are separated from each other and destroyed by physical and chemical agents operating on the bone* in situ*, either on the surface or within the soil zone"* (Behrensmeyer, 1978, 153). In other words, this process is the structural response of skeletal elements to a set of complex actions governed by the climate, involving both exposure to wind, sun, rain, snow, etc., and interactions with the local sedimentary context (Lyman & Fox, 1989).

This response is mainly illustrated by fissuring, desquamation and other cracking of the bone surface, which progressively results in a near-total alteration and destruction of the bones (six stages of alteration have been given by Behrensemeyer, *op.cit.*, in a tropical context). However, this observation criterion proves to be inoperative in the case of burned bones since the bone surfaces fissure during combustion (Shipman *et al.*, 1984; Fernandez Jalvo & Perales Piquer, 1990; Stiner *et al*., 1995; Taylor *et al*., 1995; Hanson & Cain, 2007). It thus becomes impossible to distinguish macroscopically between fissures due to combustion from those resulting from exposure to weathering. This criterion is thus abandoned in assessing the effect of weathering on burned bones.

Post-depositional fragmentation

The fragmentation of the burned bone residues is highlighted by the fragmentation index (table 2) since the number of remains is doubled for the extremities, both complete and fractured. However, the whole humerus shows a small increase (index = 1.04) that can be related to the presence of the diaphysis, i.e., of *compacta*, an anatomical part absent in the other experimental series. Figure 2 illustrates this difference since the series relating to the whole humerus is the only one having a large number of compact bone remains both before exposure (n = 224) and after exposure (n = 556).

Série expérimentale	Indice de fragmentation
Extrémité proximale complète	2,33
Extrémité proximale fracturée	2,52
Extrémité distale complète	2,01
Extrémité distale fracturée	2, 07
Humérus entier	1, 04

Tab. 2 : Fragmentation index by experimental series.

The fragmentation index is shown to be correlated wih a reduction in size of the burned bones, the majority of these being fragmented into remains lower than 4 cm in size (fig. 3), the number of remains increasing by up to 50% for the small size-classes. However, not all of the series react in the same way. There is a clear non-homogeneity between the series (table 3), particularly in the distribution of the remains of small size.

Therefore, the proximal extremities show an increased number of remains in all size-classes, while for the distal extremities a reduction in remains between 6 and 10 cm is observed (fig. 3). This difference is due to the fact that the extremities that remained whole after combustion and fragments larger than 12 cm are not present before exposure, for the distal extremities. Regarding the whole humerus, the general trend in the number of remains

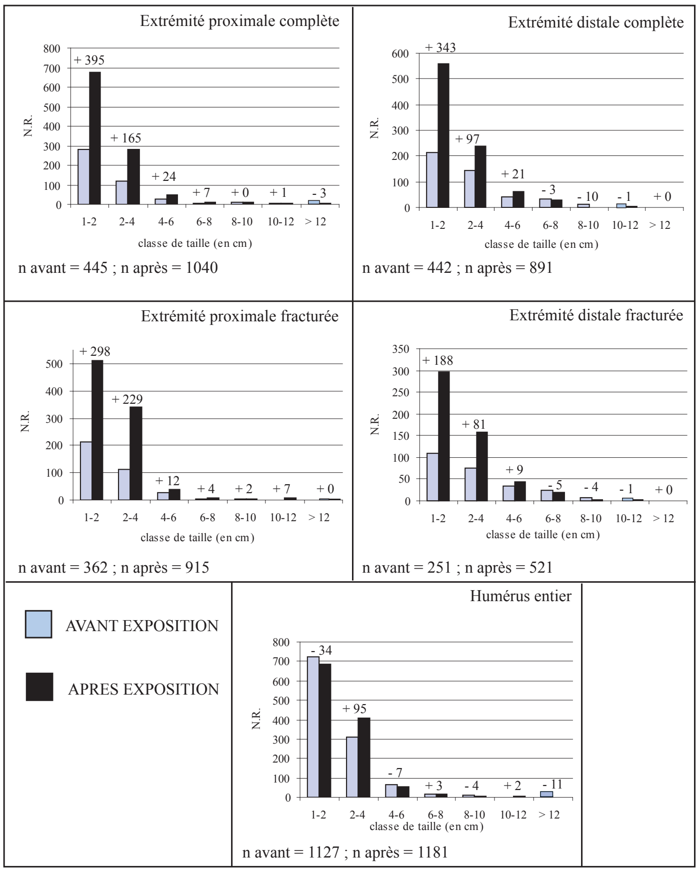

Fig. 2 : Distribution of burned bones according to bone tissue. N.R. = Number of Remains. % = percentage difference before/after exposure.

by size-class differs from the other series: contrary to these latter, few small fragments are generated after exposure to weathering. Once again, the greater presence of *compacta* and the quantity of charred bones explains this dissimilarity. Burned spongy bone crumbles more easily than compact bone (Gerbe, 2004) and charred bones appear to be more sensitive to fragmentation.

Regarding the first point, it appears that spongy bone tends to fragment and disappear upon exposure to weathering (fig. 2). Two tendencies can be observed: a reduction in the number of remains for the whole humeri and distal extremities, and an increase in the number of remains for the proximal extremities. The divergence between the proximal and distal extremities results from a higher proportion of *spongiosa* in the proximal extremity of the humerus, the distal extremity comprising slightly more *compacta*, which explains why the number of remains of burned spongy bone would be

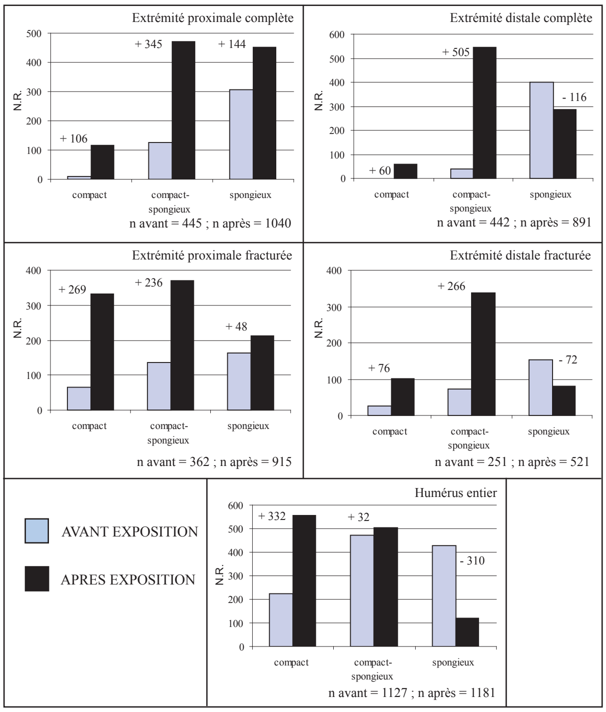

Fig. 3 : Fragmentation of the bone residues before and after exposure. N.R.: Number of Remains. % = percentage difference before/after exposure.

Classe taille (en cm)		ext. px. co	ext. px. fr	ext. ds. co	ext. ds. fr	hum. entier
1-2	décompte	678	512	557	297	688
	valeur ajustée	**3.8**	**2.8**	1.7	1.5	1.5
2-3	décompte	204	270	164	109	318
	valeur ajustée	**3.3**	**4.9**	**3.9**	1.4	**3.3**
3-4	décompte	78	71	75	48	88
	valeur ajustée	0.6	0.2	0.6	1.2	0.7
4-5	décompte	37	23	40	27	38
	valeur ajustée	0.1	**2.0**	1.5	**2.0**	0.9
5-6	décompte	12	17	22	17	19
	valeur ajustée	**2.0**	0.1	1.4	**2.4**	0.9
6-7	décompte	6	6	19	14	11
	valeur ajustée	**2.2**	1.8	**2.7**	**3.2**	1.1
7-8	décompte	6	3	9	5	7
	valeur ajustée	0.4	1.4	1.4	0.9	0.3
8-9	décompte	5	3	1	2	5
	valeur ajustée	0.8	0.1	1.3	0.1	0.5
9-10	décompte	5	2	1	1	2
	valeur ajustée	1.8	0.2	0.9	0.2	0.6
10-11	décompte	5	4	1	1	2
	valeur ajustée	1.3	1.0	1.1	0.4	0.9
11-12	décompte	1	3	2	0	1
	valeur ajustée	0.5	1.5	0.6	1.0	0.7
12-13	décompte	2	1	0	0	2
	valeur ajustée	0.9	0.0	1.1	0.8	0.7
13-14	décompte	1	0	0	0	0
	valeur ajustée	1.8	0.5	0.5	0.4	0.6

Tab. 3 : Contingency table of the distribution of bone fragments by size-class and by experimental series, with note of adjusted values.
ext.: extremity; px.: proximal; ds.: distal; co.: complete; fr.: fractured; hum.: humerus

increased for the proximal extremities. Regarding whole bone, the presence of *compacta* in the diaphysis weights the proportion of spongy bone, which is consequently under-represented. The pronounced non-homogeneity of the data (table 4) can be explained by these two trends, nearly all of the values being significantly different. The distribution of bone tissue according to size-class (fig. 4) confirms these dissimilarities, the majority of fragments of *spongiosa* being contained in the category 0-2 cm, while the "compact-spongy" and "compact" fragments have a less grouped distribution, with more remains in the size-classes above 4 cm, particularly for compact-spongy bone. Fragmentation thus has a significant impact on the spongy fragments in reducing their size, a reduction that can continue until the fragments crumble completely and thus eventually disappear.

Classe taille (en cm)		ext. px. co	ext. px. fr	ext. ds. co	ext. ds. fr	hum. entier
compact	décompte	116	333	60	102	556
	valeur ajustée	**12.2**	**8.3**	**14.4**	**3.4**	**19.6**
compact-sponieux	décompte	472	371	546	338	505
	valeur ajustée	**2.7**	**5.8**	**8.1**	**7.7**	**5.0**
spongieux	décompte	452	211	285	81	120
	valeur ajustée	**15.4**	1.7	**5.2**	**5.4**	**13.9**

Tab. 4 : Contingency table of the distribution of bone fragments by histological tissue and by experimental series, with note of adjusted values.
ext.: extremity; px.: proximal; ds.: distal; co.: complete; fr.: fractured; hum.: humerus

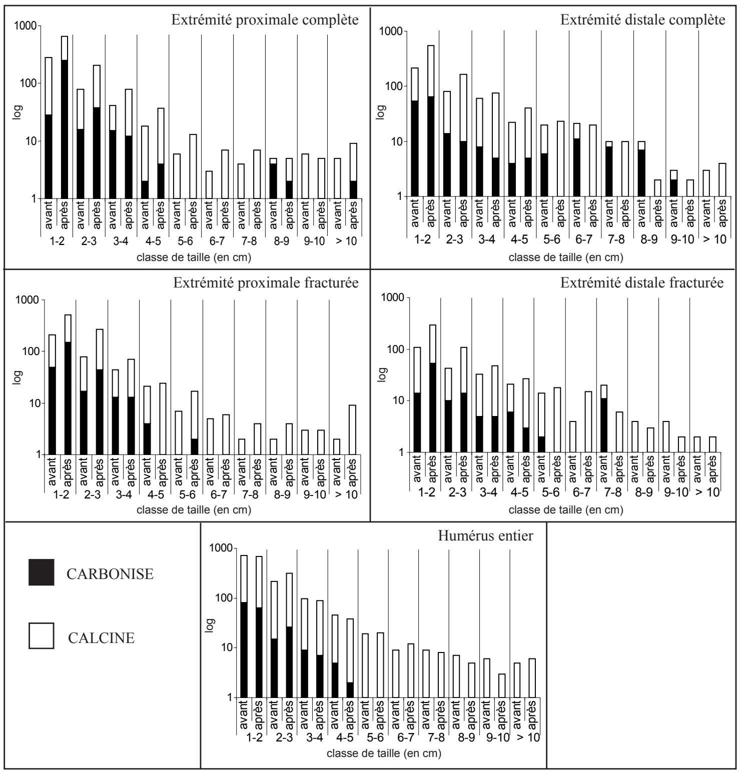

Fig. 4 : Distribution of burned bones according to bone tissue and size-class. N.R.: Number of Remains.

In addition, fragmentation also affects the most intensely burned bones. Due to their low numbers, the unburned and slightly burned fragments (brown) generally have very little influence on the homogeneity of the series, unlike the more intensely burned fragments (black to white) for which various values are significantly different (table 5). These dissimilarities arise from the differential distribution of the charred remains (white and grey) relative to the carbonised remains (black and brown). These latter fall mainly in the size-classes from 1 to 4 cm (or even 1 to 2 cm), while the charred bones have a wider distribution (fig. 5), including the presence of large fragments (greater than 10 cm). It should therefore be considered that this greater distribution is the origin of the greater quantity of charred remains, since burned bones tend to fragment into smaller remains (*cf. supra*). Moreover, only the charred bones show an increase in numbers for all of the experimental series (fig. 6). However, the whole humeri differ once again from the other series with a smaller increase in this category of bones, particularly for the white fragments. This can be explained by the disappearance of the white spongy residues, which, for the whole humerus, is the highest of all of the experimental series (fig. 7). Combustion intensity thus proves to be a factor in the fragmentation of burned bones, with charred bones being more subject to this phenomenon, all the more so as they are composed of *spongiosa*.

Finally, the presence of unburned or brown coloured remains after exposure, even if these latter were non-existent before exposure, is explained by the fragmentation of partially burned bones (fig. 6 and 7).

Classe taille (en cm)		ext. px. co	ext. px. fr	ext. ds. co	ext. ds. fr	hum. entier
non brûlé	décompte	18	0	4	0	1
	valeur ajustée	**6.3**	**2.4**	0.3	1.7	**2.4**
marron	décompte	98	127	66	44	62
	valeur ajustée	0.9	**6.2**	1.6	0.2	**4.9**
noir	décompte	214	82	19	102	37
	valeur ajustée	**13.0**	1.2	**8.7**	**7.8**	**9.1**
gris	décompte	295	334	318	98	350
	valeur ajustée	1.8	**4.3**	**3.6**	**6.2**	0.9
blanc	décompte	415	372	484	277	731
	valeur ajustée	**7.5**	**6.4**	**2.8**	1.5	**9.4**

Tab. 5 : Contingency table of the distribution of bone fragments by combustion intensity and by experimental series, with note of adjusted values.
ext.: extremity; px.: proximal; ds.: distal; co.: complete; fr.: fractured; hum.: humerus

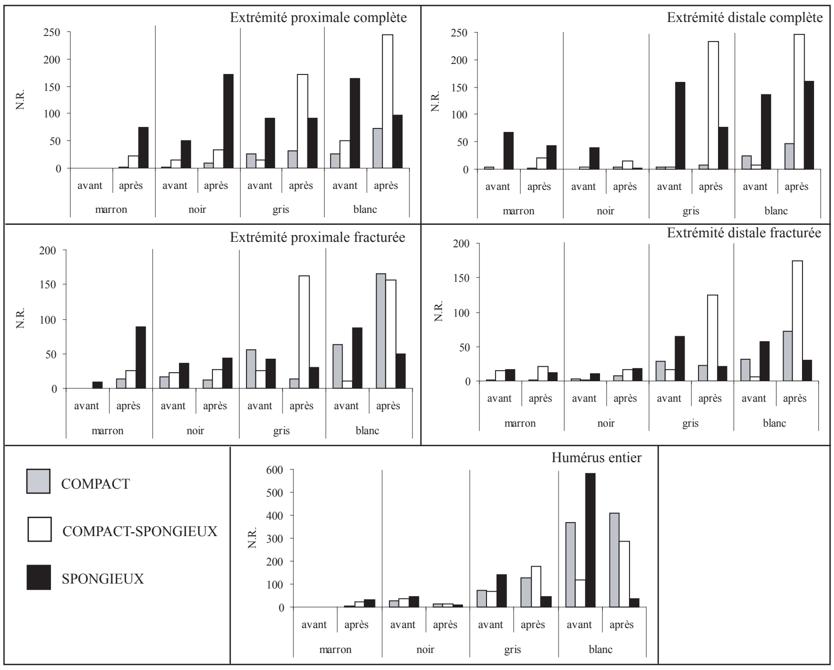

Fig. 5 : Distribution of burned bones according to combustion intensity and size-class. N.R.: Number of Remains. charred = white and grey; carbonised = black and brown the unburned bones are excluded from the count

Residual mass after exposure

The observed fragmentation and disappearance of burned residues after exposure should also be expressed in the residual bone mass. In order to address this question, the distribution of mass according to size-class, combustion intensity and bone tissue was examined. For the two latter criteria, the whole extremities before exposure and the fragments larger than 12 cm[1] after exposure are removed from the count in order to harmonise the data to be compared.

A reduction in bone mass is noted after exposure to weathering for all of the experimental series (table 6). This deficit is quite small for the whole extremities used (2 to 4%) and higher (8 to 10%) for the extremities that were fractured in advance and for the whole humerus (10%). This weight loss can mainly be explained by the distribution of the burned remains. The increase in weight of the small size-classes is due to the fragmentation of the remains of larger size (*cf. supra*), particularly the extremities (fig. 8). The series concerning the proximal extremity and whole humerus show a net reduction in mass for the 12-14 cm size-class and an increase in mass for the smaller size-classes. For the distal extremities, we observe a reduction in mass up to 6 cm, then an increase for the smaller categories. The bone mass loss thus results from the intense fragmentation of the material. If an exposure time to weathering of more than a year and a half is considered, it is imaginable that the bone mass would continue to decrease, and that the mass of burned bone then measured would no longer be representative of the initial bone mass of the deposited bones.

The hypothesis of increased fragility of the spongy bones is confirmed, these latter being the only bone

Série expérimentale	Avant exposition	Après exposition	Différence en gramme
Extrémité proximale complète	2275 g	2188 g	- 87 g
Extrémité proximale fracturée	2117 g	1840 g	- 277 g
Extrémité distale complète	2093 g	1929 g	- 164 g
Extrémité distale fracturée	1889 g	1560 g	- 329 g
Humérus entier	3921 g	3251 g	- 670 g

Tab. 6 : Comparison of bone mass before and after exposure by experimental series.

[1] - fragments larger than 12 cm after exposure result from the fragmentation of the pre-exposure extremities.

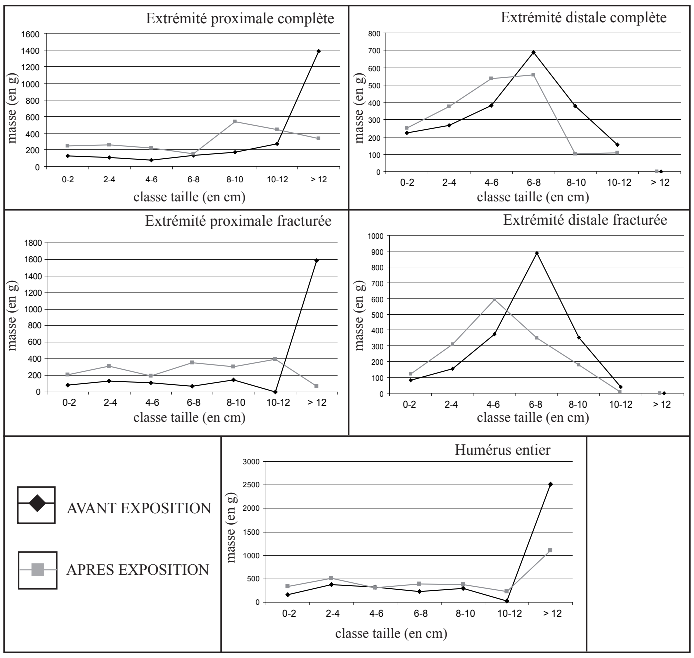

Fig. 6 : Distribution of bone residues according to their degree of combustion. N.R.: Number of Remains
% = percentage difference before/after exposure

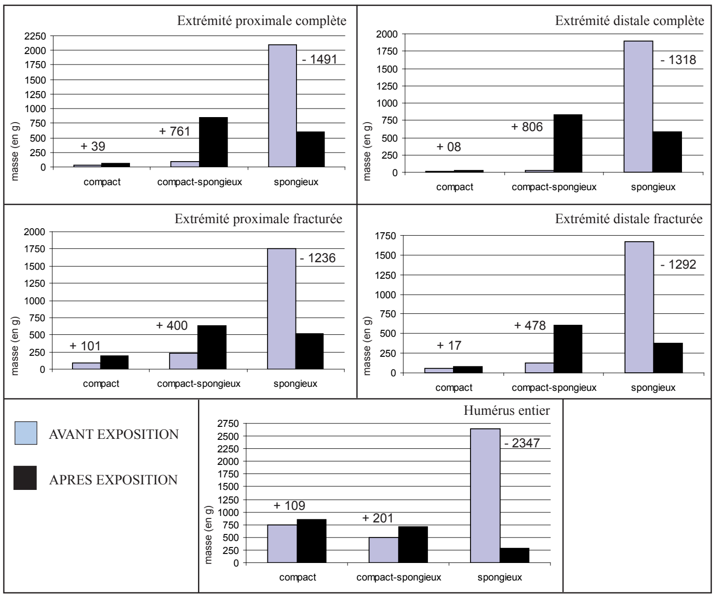

Fig. 7 : Distribution of burned bones according to their tissue and combustion intensity. N.R.: Number of Remains.

tissue to experience a mass reduction after exposure. This weight loss is less pronounced for the proximal extremities compared to the distal extremities, due to the higher proportion of *spongiosa* in the former. The whole humerus undergoes the smallest decrease, but the presence of the diaphysis (and thus of compact bone) explains this difference.

In addition, the fragility of the most intensely burned bones (white) is confirmed, since a weight loss for each of the series is noted for this bone category.

There is therefore a correlation between the loss of bone mass and the fragmentation of burned bones.

Conclusions

Differences have been observed between each of the experimental series. The discrepancies that exist between proximal and distal extremities can be explained by the difference in density of *spongiosa*, which is greater in the proximal extremity of the humerus (Lyman, 1994). Moreover, the experimental series concerning the whole humerus always differs from the others, this peculiarity seeming to result from the presence of the diaphysis (= compact bone). Better characterisation of the differences in reactivity between compact and spongy tissue is therefore necessary and will be feasible in light of other experiments, currently in progress, concerning the exposure of burned bones to weathering (Gavarnie, Hautes-Pyrénées, dir. S. Costamagno; Marvejols, Lozère, dir. M. Gerbe).

Despite these differences, general trends emerge for the whole set of experimental series and allow us to emphasize the influence of weathering on the differential preservation of burned bones. The main impact of this latter consists of fragmentation of the material associated with a bone mass reduction. The burned residues fragment into smaller pieces, fragments of less than 2 cm being quantitatively dominant. Moreover, the burned spongy bones are made fragile by exposure to weathering, which has the effect of fragmenting them intensely, or even destroying them. The charred bones, particularly those that are white, react in the same manner. Additionally, when all analysis criteria are cross-analysed, we can see that the majority of small-sized burned bones are charred and spongy.

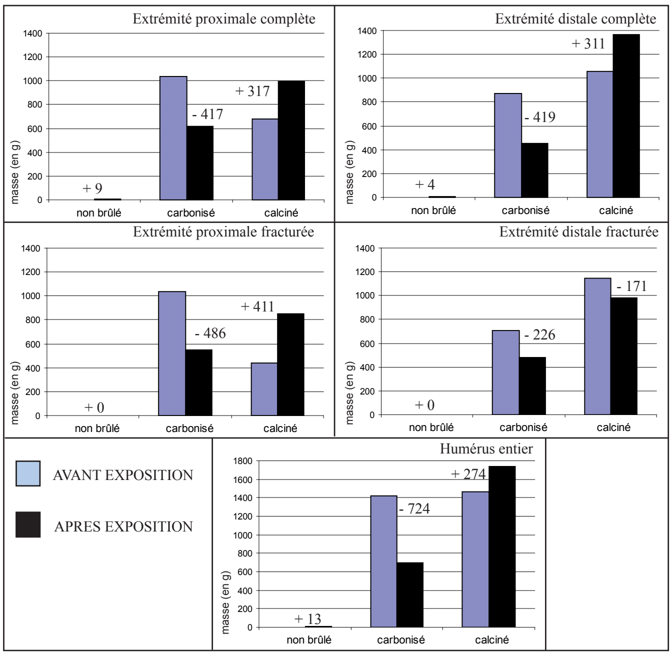

Fig. 8 : Distribution of the bone mass according to size-class. ext.: extremity

In the experiment presented here, the bones were exposed for a year and a half. If they had been exposed for a longer period, one could imagine that the alterations and destructions observed would be accentuated and that, ultimately, the charred bone would disappear like the spongy bone. On the basis of these observations, the premise of preferential preservation of compact and charred burned bone may be advanced for archaeological sites at which the action of weathering is attested.

In the light of experiments already performed, the large quantity of burned bone, mostly less than 2 cm in size, observed in archaeological assemblages (Villa *et al*., 2002; Costamagno *et al*., in press) may result from combustion itself (Théry-Parisot *et al.*, 2004, 2005; Mentzer, in press) and/or from the impact of weathering and/or of trampling (Stiner *et al*., 1995; Thiébaut *et al*., in press).

Finally, in some archaeological assemblages for which the use of bone as fuel is advanced, the hypothesis according to which the rarity of charred remains might be linked to a taphonomic bias (Théry-Parisot *et al*., 2004, 2005; Costamagno *et al*., 2005) is supported by the current study. In addition, given that burned spongy fragments disappear more rapidly than diaphysis fragments, intense weathering may mask the main criterion used to determine the use of bones as fuel, which is the over-representation of burned spongy portions relative to compact bone fragments (Costamagno *et al*., in press). It is thus essential to take into account the taphonomic actions suffered by archaeological remains, as well as their intensity, when formulating hypotheses concerning the origin of burned bones.

In order to explain the diversity observed within various archaeological assemblages, further experiments involving other taphonomic agents are thus necessary.

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References

Behrensmeyer A.K. 1978 - Taphonomic and ecologic information from bone weathering. *Paleobiology*, 4 : 150-162.

Bennett J.L. 1999 - Thermal alteration of buried bone. *Journal of archaeological Science*, 26 : 1-8.

Brain C.K. 1993 - The occurence of burnt bones at Swartkrans and their implications for the control of fire by early hominids. *In* : *Swartkrans - a cave's chronicle of early man*, Brain C.K. (ed.). Pretoria, Transvaal museum : 229-242.

Brain C.K. & Sillen A. 1988 - Evidence from the Swartkrans cave for the earliest use of fire. *Nature*, 336 : 464-466.

Buikstra J.E. & Swegle M. 1989 - Bone modification due to burning : experimental evidence. *In* : *Bone modification*, Bonnichsen R. & Sorg M.H. (eds). Peopling of the americas publications, Orono, center for the study of first americans, university of Maine : 247-258.

Cain C.R. 2005 - Using burned animal bone to look at Middle Stone Age occupation and behavior. *Journal of Archaeological Science*, 32 : 873-884.

Castel J.-C. 1999 - *Comportements de subsistance au Solutréen et au Badegoulien d'après les faunes de Combe-Saunière (Dordogne) et du Cuzoul de Vers (Lot)*. Thèse de Doctorat, Bordeaux I, 619 pp.

Costamagno S. 1999 - *Stratégies de chasse et fonction des sites au Magdalénien dans le Sud de la France*. Thèse de Doctorat, Bordeaux I, 495 pp.

Costamagno S., Griggo C. & Mourre V. 1999 - Approche expérimentale d'un problème taphonomique : utilisation de combustible osseux au Paléolithique. *Préhistoire européenne*, 13 : 167-194.

Costamagno S., Théry-Parisot I., Brugal J.-P. & Guilbert R. 2005 - Taphonomic consequences of the use of bones as fuel. Experimental data and archaeological applications. *In* : *Biosphere to lithosphere. New studies in vertebrate taphonomy*, O'Connor T. (ed). Actes du 9° colloque de l'ICAZ, Oxford, Durham, août 2002, Oxbow books : 51-62

Costamagno S., Théry-Parisot I., Castel J.-C. & Brugal J.-P. 2009 - Combustible ou non ? Analyse multifactorielle et modèles explicatifs sur des ossements brûlés paléolithiques. *In* : *Gestion des combustibles et statut des sites au Paléolithique et au Mésolithique*, Théry-Parisot I., Costamagno S. & Henry A. (eds). Oxford, BAR international series : 47-60.

David B. 1990 - How was this bone burnt ? *In* : *Problem solving in taphonomy : archaeological and paleontological studies from Europe, Africa and Oceania*, Davidson I. & Watson D. (eds.). St Lucia, Queensland, Tempus anthropology museum : 65-79.

Everitt B.S. 1992 (2° édition) - *The analysis of contingency tables*. Boca Raton (Floride), Chapman et Hall/CRC, 164 pp.

Fernandez Jalvo Y. & Perales Piquer C. 1990 - Analisis macroscopico du huesos quemados experimentalmente. *Reunion de tafonomia y focilisacion* : 105-114.

Franchet L. 1933 - *La coloration des os dans le sol. Le bouillage des cadavres au Moyen-Age. L'incinération et ses phénomènes*. Paris, la revue politique et littéraire et la revue scientifique, 77 pp.

Gerbe M. 2004 - *Etude taphonomique d'ossements brûlés*. Mémoire de D.E.A., Université de Provence, 109 pp.

Gilchrist R. & Mytum C. 1986 - Experimental archaeology and burnt animal bone from archaeological sites. *Circaea*, 4 : 29-38.

Grupe G. & Hummel S. 1991 - Trace element studies on experimentally cremated bone. I. Alteration of the chemical composition at high temperatures. *Journal of Archaeological Science*, 18 : 177-186.

Guillon F. 1986 - Brûlés frais ou brûlés secs ? *In* : *Anthropologie physique et archéologie*, Duday H. & Masset C. (eds). Actes du Colloque de Toulouse, novembre 1982. Paris, CNRS : 191-194.

Hanson M. & Cain C.R. 2007 - Examining histology to identify burned bone. *Journal of Archaeological Science*, 34 : 1902-1913.

Herrmann B. 1977 - On histological investigations of cremated human remains. *Journal of human evolution*, 6 : 101-103.

Joly D. & March R. 2003 - Etude des ossements brûlés : essai de corrélation de méthodes pour la détermination des températures. *In* : *Le feu domestique et ses structures au Néolithique et aux Ages des métaux*, Frère-Sautot M.-C. (eds). Actes du colloque de Bourg-en-Bresse et Beaune : 299-310.

Laroulandie V. 2001 - Les traces liées à la boucherie, à la cuisson et à la consommation d'oiseaux. Apport de l'expérimentation. *In* : *Préhistoire et approche expérimentale*, Bourguignon L., Ortega I. & Frère-Sautot C. (eds). Montagnac, Monique Mergoil : 97-108.

Lyman R.L. 1994 - *Vertebrate taphonomy*. Cambridge manuals in archaeology, Cambridge, Cambridge University Press, 524 pp.

Lyman R.L. & Fox G.L. 1989 - A critical evaluation of bone weathering as an indication of bone assemblage formation. *Journal of archaeological Science*, 16 : 293-317.

Mentzer S.M. 2009 - Bone as fuel source : the effects of initial fragment size distribution. *In* : *Gestion des combustibles et statut des sites au Paléolithique et au Mésolithique*, I. Théry-Parisot, S. Costamagno & A. Henry (eds.). Oxford, BAR international series : 37-45.

Nicholson R.A. 1993 - A morphological investigation of burnt animal bone and an evaluation of its utility in archaeology. *Journal of archaeological Science*, 20 : 411-428.

Pasto I. 2003 - *Cremos* : l'action du feu sur les restes osseux des archéologiques. *In* : *Le feu domestique et ses structures au Néolithique et aux Ages des métaux*, Frère-Sautot M.-C. (ed.). Actes du colloque de Bour-en-Bresse et Beaune : 261-266.

Perinet G. 1982 - Détermination des températures de cuisson des ossements humains. *Histoire et archéologie, les dossiers*, 66 : 96-97.

Shahack-Gross R., Bar-Yosef O. & Weiner S. 1997 - Black-coloured bones in Hayonim cave, Isarel : differentiating between burning and oxide staining. *Journal of archaeological Science*, 24 : 439-446.

Shipman P., Foster G. & Schoeninger M.J. 1984 - Burnt bones and teeth : an experimental study of color, morphology, crystal structure and shrinkage. *Journal of archaeological Science*, 11 : 307-325.

Sillen A. & Hoering T. 1993 - Chemical characterization of burnt bones from Swartkrans. *In* : *Swartkrans : a cave's chronicle of early man*, Brain C.K. (ed). Pretoria, Transvaal museum : 243-249.

Spennemann D.H.R. & Colley S.M. 1989 - Fire in a pit : the effect of the burning on faunal remains. *Archaeozoologia*, 3 : 51-64.

Stiner M.C., Kuhn S.L., Weiner S. & Bar-Yosef O. 1995 - Differential burning, recrystallization, and fragmentation of archaeologiacal bone. *Journal of archaeological Science*, 22 : 223-237.

Susini A. 1988 - *Etudes des caractéristiques biophysiques des tissus calcifiés humains (os, émail, dentine) soumis à des traitements thermiques. Applications anthropologiques et médicales*. Université de Genève, thèse n°2320 pp.

Taylor R.E., Hare P.E. & White T.D. 1995 - Geochemical criteria for thermal alteration of bone. *Journal of archaeological Science*, 22 : 115-119.

Théry-Parisot I. 2002 - Fuel management (bone and wood) during the Lower Aurignacian in the Pataud rock shelter (Lower Palaeolithic, Les Eyzies de Tayac, Dordogne, France) : contribution of experimentation. *Journal of Archaeological Science*, 29 : 1415-1421.

Théry-Parisot I., Brugal J.-P., Costamagno S. & Guilbert R. 2004 - Conséquences taphonomiques de l'utilisation des ossements comme combustible. Approche expérimentale. *Les Nouvelles de l'Archéologie*, 95 : 19-22.

Théry-Parisot I., Costamagno S., Brugal J.-P., Fosse P. & Guilbert R. 2005 - The use of bone as fuel during the Paleolithic, experimental study of bone combustible properties. *In* : *The zooarchaeology of fats, oils, milk and dairying*, Mulville J. & Outram A.K. (eds). Actes du 9° colloque international de l'ICAZ, Durham, août 2002. Oxford, Oxbow books : 50-59.

Théry-Parisot I. & Costamagno S. 2005 - Propriétés combustibles des ossements. Données expérimentales et réflexions archéologiques sur leur emploi dans les sites paléolithiques. *Gallia préhistoire*, 47 : 235-254.

Thiébaut C., Costamagno S., Coumont M.-P., Mourre V., Provenzano N. & Théry-Parisot I. sous presse - Approche expérimentale des conséquences du piétinement des grands herbivores sur les vestiges lithiques et osseux. *Supplément à Paleo*.

Trellisó Carreño L. 2001 - La acción del fuego sobre el cuerpo humano : la antropología física y el análisis de las cremaciones antiguas. *Cypsela*, 13 : 87-98.

Vigne J.-D. 1983 - *Les mammifères terrestres non volants du Post-glaciaire de Corse et leurs rapports avec l'homme : étude paléo-ethno-zoologique fondée sur les ossements*. Thèse de 3ème Cycle de Paléontologie des Vertébrés, Université Pierre et Marie Curie, 501 pp.

Vigne J.-D., Marinval-Vigne M.-C., Lanfranchi (de) F. & Weiss M.C. 1981 - Consommation du « lapin-rat » (*Prolagus sardus* Wagner) au Néolithique ancien méditerranéen Abri d'Araguina-Sennola (Bonifaccio, Corse). *Bulletin de la Société préhistorique française*, 78 : 222-224.

Villa P., Bon F. & Castel J.-C. 2002 - Fuel, fire, and fireplaces in the Paleolithic of Western Europe. *The review of archaeology*, 23 : 33-42.

Walters J. 1988 - Fire and bones : patterns of discard. *In* : *Archaeology with ethnography : an australian perspective*, Meehan B. & Jones R. (eds). Canberra, Australian National University : 215-221.

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REFLECTIONS ON THE POTENTIAL FOR PRESERVATION OF BURNED BONE BASED ON THE MATERIAL FROM SAINT-ANTOINE (VITROLLES, HAUTES-ALPES)

Maryline RILLARDON & Jean-Pierre BRACCO

« Hommage to Jean Gagnepain »

Abstract

The faunal assemblage from the open-air site of Saint-Antoine at Vitrolles (Hautes-Alpes, Epigravettian) has yielded an atypical composition of burned bones (NRT = 23%), being composed mainly of charred compact bones. While in an experimental context a high proportion of charred bones is typical of the use of skeletal remains as fuel, their representation in archaeological contexts is generally low, particularly in comparison to that of the less intensely burned elements. Contrary to this general principle, the Saint-Antoine deposit shows a strong representation of charred bones in a difficult taphonomic context characterised by an acid sediment and by the intensive action of different taphonomic phenomena (weathering, sediment compaction, dissolution). The high representation of charred bones seems to result from a combination of various factors, including the purpose of the combustion (camp maintenance and/or use as fuel) and their intense fragmentation, together with a higher preservation potential for burned bones (compact and spongy), including charred bones, compared to unburned bone elements when they are buried in acid sediments.

Keywords : burned bones, combustion, taphonomy, Epigravettian

Introduction

In archaeological assemblages, charred bone is generally scarce compared to less burned bone. Only experimental contexts in which the bones are deliberately burned deliver such proportions (Théry-parisot *et al.*, 2004, 2005; Costamagno *et al.*, 2005, 2009). This under-representation in archaeological contexts is generally interpreted as the result of their greater vulnerability to taphonomic phenomena (trampling, burying). This fragility is due in part to their loss of structural coherence which renders them mechanically more friable (Stiner *et al.*, 1995).

In locus 2 of Saint-Antoine (Hautes-Alpes, France), attributed to the Recent Epigravettian, these remains, thus ostensibly fragile, have been found in an unusual taphonomic context. Buried in a sediment with an acid pH (limestone-rich marl) unconducive to preservation, the unburned bones were preserved only in a restricted area of the deposit, constituted of a very dense concentration of bones and lithic remains which, after archaeological analyses, have been attributed to a waste disposal area (midden type). The density of the bones in this zone seems to have permitted their preservation by protecting them from the aggressive surrounding environment.

In order to better understand and to attempt to explain the origin of this stock of charred bones, different hypotheses have been considered: does the existence of a midden explain the good preservation of the charred bones? Did the differential fragmentation of the burned bones as a function of the intensity of their combustion influence the over-representation of some of these elements? Apart from the depositional context, can the origin of the combustion (natural/anthropogenic) and its purpose (camp maintenance, cooking) explain in part this high rate of charred bones?

Presentation

The site

The Upper Paleolithic open-air site of Saint-Antoine is located in the commune of Vitrolles in the French department of the Hautes-Alpes (photo 1 and 2). It is situated on the right bank of the Durance river at an altitude of 575 metres above sea-level, on the southern flank of a marlaceous knoll. Discovered in 1982 by Alain Muret, an initial excavation campaign (1988-1990) concentrated on a zone since named locus 1 (Muret *et al.* 1991). In 1996, in the context of preventive operation linked to the construction of the A51 autoroute, this locus 1 was the subject of renewed excavations under the direction of J. Gagnepain and J.-P. Bracco. This second campaign permitted the completion of the excavation of locus 1 and the uncovering of two new loci (locus 2 and 3) (Gagnepain *et al.,* 1997*,* 1999) (fig. 1). Only locus 2 was excavated, locus 3 not being threatened by the construction.

Ph. 1 - Aerial view of the geographical context of the Saint-Antoine deposit (C. Hussy, SRA PACA).

Ph. 2 - Aerial view of the Saint-Antoine deposit during excavation (C. Hussy, SRA PACA).

The characteristics of the lithic tools from loci 1 and 2 allow their attribution to the Recent Epigravettian of the Italian series, and more precisely to its phase 3 defined by C. Montoya (Bracco *et al.,* 1997; Montoya and Bracco, 2005; Montoya and Peresani, 2005). This chrono-cultural attribution is confirmed by two radiocarbon dates (locus 2, level B) which situate the occupation at the end of the recent Allerød-Dryas period: Ly 1525 (OXA): 11180 ± 60 BP (burned bone); Ly 1526 (OXA): 10825 ± 55 BP (charcoal) (Montoya & Bracco, 2005).

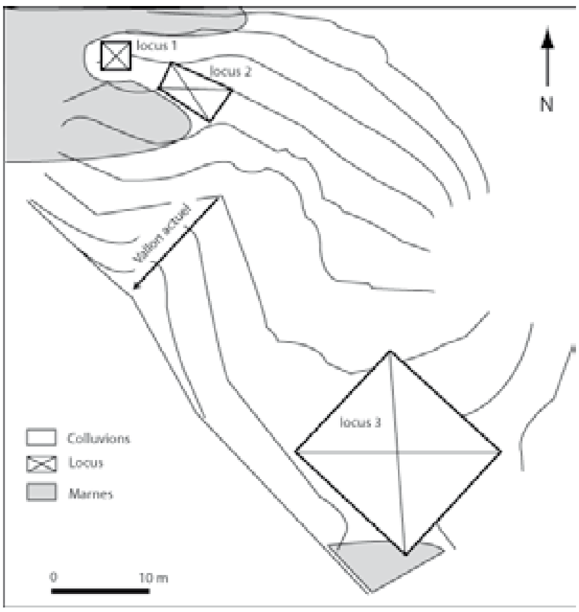


Fig. 1 - General plan of the Saint-Antoine site, with localisation of loci 1, 2, and 3 (Gagnepain *et al.*, 1999).

Locus 2

Only locus 2 has yielded faunal remains. The total archaeological surface of this locus is 150 m^2, of which 120 m^2 have been excavated to a depth of 60 to 80 cm. 94 m^2 have been manually excavated, i.e., 78%, corresponding to the areas richest in remains. The rest of the surface was explored with the help of a mechanical excavator (Gagnepain *et al.*, 1999).

Three levels were discovered in a detrital sediment composed of Jurassic marls (from top to bottom: A, B and C) (fig. 2). The examination of all data from both the excavation and the analyses shows that levels A and B were deposited separately. In contrast, level C, poorer and only present in certain areas of the archaeological surface, may correspond to a vertical slip of the base of level B (Bracco 2004, Gagnepain *et al.*, 1997, 1999). The two main archaeological levels (A and B) are separated across a large part of the excavation by a quasi-sterile layer. The absence of sedimentary distinction between these two layers, together with a limited duration of excavation, means that the attribution of the archaeological material to one or other of these two levels has not yet been carried out. This disadvantage is limited by the fact that level A is poor in remains of all types (lithic industry, bone remains, charcoals). It has nonetheless produced a hearth composed of stones laid on edge which delimit a half-circle in which has been found a reddened area and some coniferous-type charcoals (Canals i Salomo *in* Gagnepain *et al.*, 1997). While no hearth was discovered in the lower level (B), this level, otherwise very rich in lithic remains and charcoal, has also yielded a large quantity of faunal remains distributed in midden form. Measuring on average 50 cm thick, this level may correspond either to a single archaeological level expanded by post-depositional processes or to a multi-stratified accumulation of several archaeological levels (Gagnepain *et al.*, 1999; Bracco 2004).

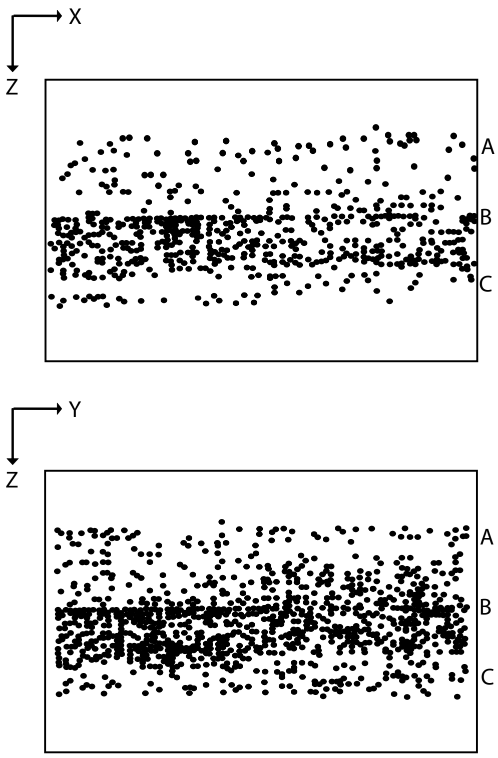


Fig. 2 - Locus 2. Stratigraphic projection of the lithic artefacts from square O20 and illustration of levels A, B and C (Gagnepain *et al.*, 1999).

The level B bone midden (locus 2)

The accumulation of faunal remains (photo 3) extends over an area 4 m long by 2.50 m wide and approximately 50 cm thick, spreading towards the south-west of the deposit, perpendicularly to the slope of the paleovalley (fig. 3) (Canal i Salomo, *in* Gagnepain *et al.*, 1997). This accumulation may be considered as a midden-type structure given the weak dispersion of the elements around a central core: “the layer of remains is organised in concentric rings with decreasing density towards the edge from an area of maximum density at the central bone midden” (Canal i Salomo, *in* Gagnepain *et al.*, 1997, 257) and its perpendicular orientation to the axis of the paleovalley which excludes deposition by sedimentary phenomena *lato sensu*.

Ph. 3 - View of the locus 2 bone midden during excavation.

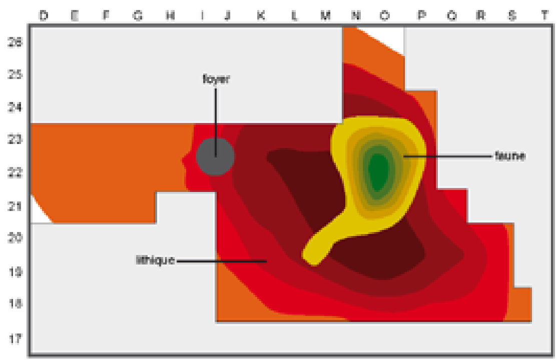


Fig. 3 - Locus 2. Planimetric projection of all lithic artefacts and relative positions of the bone midden and the hearth in the layer of remains (Gagnepain *et al.*, 1999).

The sediment is composed of partly decarbonated Jurassic marls (Muret *et al.*, 1991). The high limestone content of the marl (from 35 to 65%) gives this sediment an acid pH, unconducive to the preservation of the bones. However, the spatial concentration of the bones has led to the release of a large quantity of calcium carbonate. The current hypothesis is that this partially dissolved calcium carbonate protected the midden from the aggressive external environment, thus permitting preservation of the bone and dental elements (Bez, 1997 : *in* Gagnepain *et al.*, 1997). The faunal remains scattered outside the midden have in fact almost disappeared, only surviving as ghosts irrecoverable by excavation.

The bones present a relatively poor state of preservation, characterised by the strong action of the different pre- and post-burial taphonomic phenomena such as weathering, sediment compaction and dissolution. In general, the cortical surface is rarely preserved and the bones are very friable (photos 4, 5, and 6).

The faunal assemblage (Rillardon, 2003) is characterised by a very high predominance of red deer, which represents, depending on the counting unit employed, between 82 and 92% of the material identified (tab. 1). The study of the stages of dental eruption in this taxon indicate slaughter between midsummer and late autumn (July-December).

Ph. 4 - Bone demonstrating an advanced stage of alteration (scale: 1 cm) (Photo M. Rillardon).

The traceological data from the lithic industry (Philibert *in* Gagnepain *et al.*, 1997), reflect the

Ph. 5 - Red deer radius presenting concretion and vermiculations (scale: 1 cm) (Photo M. Rillardon).

Ph. 6 - Red deer mandible presenting a heavily altered bone surface (scale: 1 cm) (Photo M. Rillardon).

execution of various activities such as hunting, butchery and the different phases of working with hides (scraping, tanning).

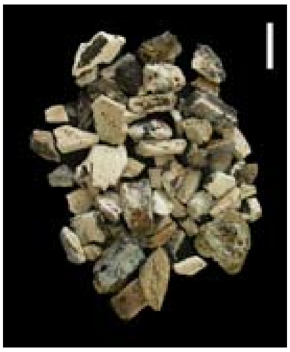
The predominance in the lithic material of elements relating to projectiles (armatures) and hide working (scrapers), and the specialisation of the cynegetic acquisition, together with the execution of operations for transforming hides into leather, reflect seasonal human occupation linked to the acquisition and treatment of red deer carcasses (Gagnepain *et al.*, 1997; Bracco 2004).

	NR	%	NMI	%
Cerf	421	92,1	19	82,6
Aurochs	35	7,7	3	13
Chevreuil	1	0,2	1	4,4
Total NRDt	**457**	**100**	**23**	**100**
NRDa	32			
cf. gd herb.	28			
cf. moy. herb.	176			
ND	16555			
Total	**17248**			

Tab. 1 - Faunal composition of locus 2 (Rillardon 2003).

The burned remains

3877 burned faunal remains were counted, i.e., 23% of the total number of remains. These are principally indeterminate remains (photo 7). Only four elements have been identified anatomically and taxonomically; all attributed to red deer (a large sesamoid, two phalange II bones - one vestigial - and a radius distal extremity) (photo 8). The burned bones are spread over nearly the whole of the excavated surface, contrary to the unburned bone remains.



Ph. 7 - Indeterminate burned bones (scale: 1 cm) (Photo M. Rillardon).

Ph. 8 - Charred Red deer sesamoid (scale: 1 cm) (Photo M. Rillardon).

The different histological categories are represented (compact, compact/spongy, spongy, dental) (tab. 2). The fragments of compact bone represent 90% of the burned material. The other categories are less common and are represented in relatively similar proportions; between 3 and 4%.

	NR	%
Compact	3497	90,2
Compact/Spongieux	163	4,2
Spongieux	120	3,1
Dents	97	2,5
Total	**3877**	**100**

Tab. 2 - Histological composition of the burned bones from Saint-Antoine.

The burned remains are very small in size (fig. 4). They all measure less than 3 cm and 91% do not exceed one

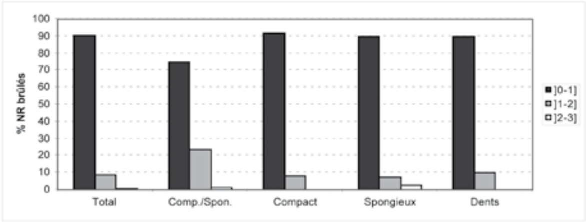


Fig. 4 - Size (in cm) of the faunal remains as a function of histology (NR = 3877).

centimetre. This characteristic is found in the different histological categories (92% for compact bone, 90% for spongy bone and dental fragments, and 75% for compact/spongy bone).

The colour code used is that established by M.-C. Stiner *et al.* (1995) and modified by S. Costamagno *et al.* (1999), to which we have added the blue shade, which appears, as does the grey shade, between the stage of carbonisation and that of charring (Hermann, 1977; Shipman *et al.*, 1984). The colouration of the burned remains is variable (fig. 5), varying from slightly burned (brown) to charred (white). However, the charred remains are predominant, representing 75% of the total of burned remains, for all histological categories with the exception of the dental fragments carbonised to 85%. This difference is probably explained by the different thermal behaviour of these two materials (Susini, 1988).

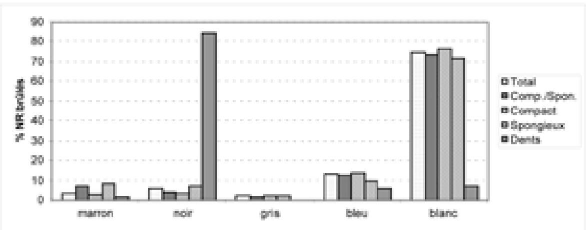


Fig. 5 - Proportion of the different colourations of burned faunal remains as a function of histology (NR = 3877).

Regarding the skeletal elements of the red deer, the three elements of the bottom of the hoof (large sesamoid and phalanges II) are charred while the radius distal extremity is blue in colour.

The combustion index (Costamagno *et al.*, 2009), is 0.86 (tab. 3). It is nearer to the index obtained in experimental contexts (average = 0.77) than that of several archaeological series of the French Palaeolithic (average = 0.5) (Costamagno *et al.*, 2009).

In conclusion, for the Saint-Antoine deposit, the burned bones are primarily composed of charred compact bone of small size.

Codes couleurs	Description	Nombre d'os	Coefficient	Indice de combustion
0	Non brûlés	7242	0	
1	Os partiellement brûlés	140	140	
2	Os carbonisés (majoritairement noir)	146	292	
3	Os majoritairement gris	98	294	
4	Os calcinés (majoritairement blanc)	2884	11536	
		10510	12262	0,86

Tab. 3 - Combustion index (according to Costamagno *et al.*, 2009) of the burned faunal remains from Saint-Antoine. (The blue shade and the dental material have been excluded from the calculations).

Hypothesis 1: The midden as a protective structure

The first hypothesis envisages the fact that the liberation of calcium carbonate, which permitted the preservation of the unburned bones in the interior of the midden, promoted the preservation of the charred bones by creating a favourable burial context. To test this hypothesis, a spatial analysis of the burned remains (bone and charcoal) according to their density per 0.25 m² (fig. 6 and 7) was conducted.

The burned bones and charcoal are present over nearly the whole of the excavated surface, although two areas of concentration appear, which are common to these two categories of remains: one situated in I-J 22 and one in N 20a which is spatially less extensive. Thus the finding that much of the charcoal and burned bones, whatever their histology and stage of combustion, were preserved outside the midden, indicates that the presence of the midden cannot explain the strong representation of charred bones.

The analysis of the spatial distribution of the burned remains shows the preservation of the burned bones in an area of the deposit where the unburned bones were not preserved. This indicates, in the present case, a greater potential for the preservation of burned compared to unburned bone. This fact is contrary to numerous experimental data which demonstrate that burned bones are more fragile and friable than unburned bones due to changes in diagenesis experienced by the former (Stiner *et al.*, 1995; David, 1990; Walters 1988). However,

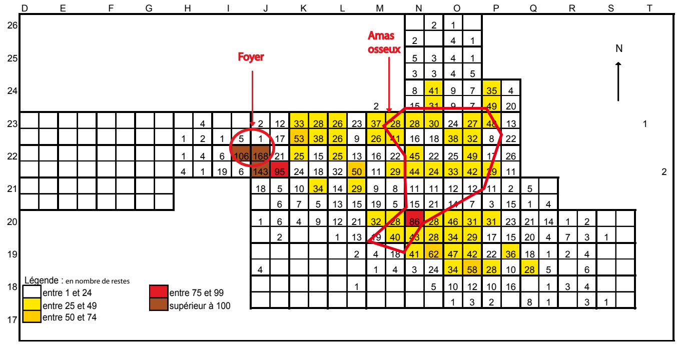


Fig. 6 - Spatial distribution of the burned bones (NR = 3836) as a function of their localisation by 0.25 m².

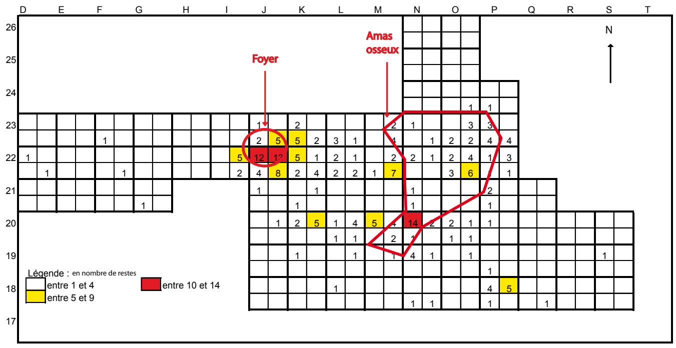


Fig. 7 - Spatial distribution of the charcoals (NR = 236) as a function of their localisation by 0.25 m².

Indeed, at Saint-Antoine we note a higher fragmentation of charred bone compared to less intensely burned bone (fig. 8). However, the fact that the fragments measuring less than one centimetre are in the majority in all colourations indicates that the phenomenon of fragmentation as a function of the intensity of combustion did not play a fundamental role in the over-representation of the burned bones.

The relatively high total percentage (23%) of burned bones found in the deposit is certainly largely influenced by the high fragmentation of these elements. The original percentage of burned compared to unburned bone was probably much lower.

Hypothesis 3: Origin of combustion

Finally, it is necessary to estimate whether the origin of combustion (natural *vs* anthropogenic), and its purpose in the case of an anthropogenic combustion, influenced the strong representation of charred bones.

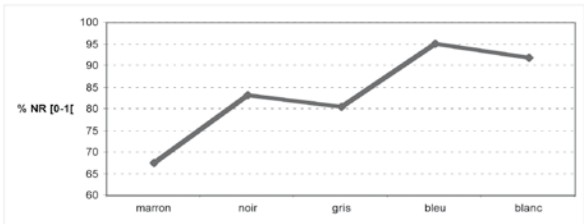


Fig. 8 - Percentage of burned bones measuring less than one centimetre as a function of different stages of combustion (NR = 3523).

there do exist other archaeological deposits which demonstrate better preservation of burned bone than of unburned bone, such as the Iron Age hillfort of Castell Henllys (Wales) (Gilchrist & Mytum, 1986) and the megalithic monument of Castelluccio (Vigne, 1983; David, 2001). The common feature of these deposits is their preservation in acid soils (clay, gravels, marl). It appears thus that the experimental results do not apply to all sedimentary contexts. In acid soils the burned bones would have a higher potential for preservation than unburned bones.

Hypothesis 2: High fragmentation

The second hypothesis tested in order to attempt to explain the over-representation of charred bones is that of a higher fragmentation of these elements. Several experiments highlight the great friability of charred bones as a result of different taphonomic phenomena such as trampling, sediment compaction (Stiner *et al.*, 1995) and weathering (Gerbe, 2004).

Natural *vs* Anthropogenic

The spatial analysis of the burned remains (fig. 6 and 7) shows the superposition in squares I-J 22-23, of the hearth from the higher level (A) and of the main area of concentration of the burned elements in level B. It is thus necessary to verify whether these organic residues are linked to the combustion activity in the hearth. The analysis of the stratigraphic distribution of the burned remains (fig. 9 and 10) highlights the presence of about ten centimetres of sediment poor in remains between the

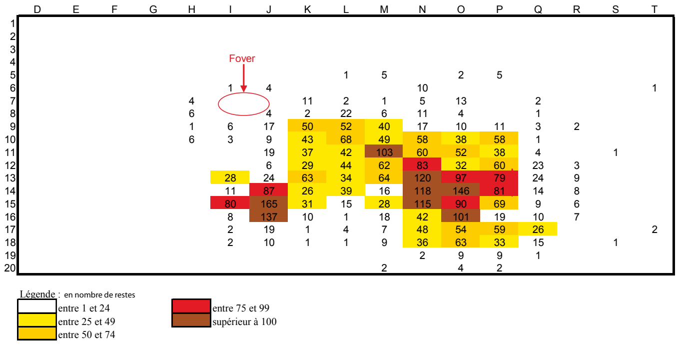


Fig. 9 - Stratigraphic distribution of the burned bones (NR = 3836) as a function of their localisation by 0.25 m².

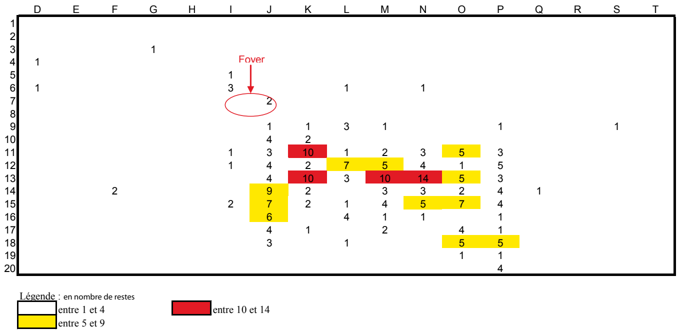


Fig. 10 - Stratigraphic distribution of the charcoals (NR = 236) as a function of their localisation by 0.25 m².

hearth and the burned elements. While the archaeological material may have undergone some vertical movements, these latter were only small-scale (Gagnepain *et al.*, 1997). This stratigraphic separation thus indicates the independence of these two combustion indicators. Indeed, as regards bones buried beneath a hearth, several experiments (Stiner *et al.*, 1995; Bennett 1999) have shown on one hand that the thermal stigmata were primarily present on the bones buried in the first ten centimetres, and on the other hand that these bones never achieved the final stage of charring (white colouration) even in the case of a long combustion.

Regarding other possible causes of natural origin, the intensity of the combustion (maximal degree of heat achieved) is not coherent with the data obtained from natural fires. These latter generate temperatures significantly lower than those created during an anthropogenic fire, rarely leading to bone charring (David, 1990; Bellomo, 1993). In addition, the fact that only 23% of the bone elements discovered possess traces of combustion allows the exclusion of the hypothesis of a natural fire.

Thus, the stratigraphic position of the burned remains and their combustion intensity allow us to reject the hypothesis of combustion linked to the hearth of the higher level or combustion linked to a natural fire. It thus appears that the combustion from which the burned remains issue is of anthropogenic origin.

Cooking *vs* waste *vs* fuel

The deliberate burning of bone may result from different activities such as the cooking of anatomical portions (Vigne *et al.*, 1981), use as fuel (Beyries, 2002; Théry-Parisot, 2002; Villa *et al.*, 2002; Castel, 2003; Théry-Parisot & Costamagno, 2005; Théry-parisot *et al.*, 2004, 2005; Costamagno *et al.*, 1999, 2005, 2009), the burning of waste during camp maintenance (Cain, 2005) or for ritual purposes, in particular to favour the reincarnation and renewal of the herd (Vaté & Beyries, 2007).

The distinction between these different purposes may be clarified through the use of various indices proposed by S. Costamagno *et al.*, (2009). At Saint-Antoine (tab. 4), the high percentage of fragmentation (99% of remains smaller than 2 cm) together with the high percentage of at least carbonised bones (86%) seems to indicate deliberate combustion of skeletal elements. However, the percentage of spongy parts burned (7%), the most combustible parts of the bone (Costamagno *et al.*, 1999), is low. Experimental data (Gerbe, 2004; Thiébault *et al.*, in press) indicate the much greater sensitivity of burned spongy bone to taphonomic processes both pre- and post-burial (trampling, weathering, etc.,) compared

Indices	Formule de calcul	Résultat Saint-Antoine
Pourcentages d'os spongieux brûlés	(SPON2 + SPON3 + SPON4)/ (NR2 + NR3 + NR4 X 100	6,8%
Pourcentages d'os brûlés inférieurs à 2 cm	NR brûlés < 2cm/ NRT brûlés X 100	98,8%
Pourcentage d'os brûlés au moins carbonisés	(NR2 + NR3 + NR4)/ NRT brûlés X 100	86,4%

Tab. 4 - Calculation of the different indices (according to Costamagno *et al.*, 2009, SPON = spongy portions (ribs, vertebrae, rib bones, scapular glenoid cavity, hip bone, articular extremities of long bones, carpals, tarsals, sesamoids, fragments of indeterminate spongy tissue); NRT = total number of remains; the index corresponds to the colour codes from table 3) (For the initial calculation, the compact/spongy elements were distributed in each of these two categories).

to fragments of diaphyses. While at Saint-Antoine the only anatomically and taxonomically identifiable burned remains are bone extremities or charred spongy bone from red deer, thus indicating the potential for preservation of these elements in the interior of this deposit, it is not possible to totally exclude the possibility of differential preservation of the burned spongy parts.

Thus, the burned remains discovered at Saint-Antoine are indicators of an intentional combustion of anthropogenic origin. However, due to a potential taphonomic bias concerning the spongy parts, it is difficult to determine whether the purpose of combustion was the camp maintenance and/or the use of bone as fuel during particular activities (drying of hides, etc.).

The absence of clear structure prevents determination of whether the concentrations of burned remains found at Saint-Antoine are indicators of a combustion zone[1] in the real sense of the word or of a waste zone[2] from a hearth located in an unexcavated or non-preserved part of the site. However, the analysis of lithic remains deposited at the heart of this midden of burned bones, and in particular the presence of numerous cores in the phase of exhaustion and cessation of lamellar production (Montoya 2004), may indicate a secondary accumulation in a waste area.

Conclusion

The burned bones from locus 2 of Saint-Antoine are primarily composed of charred compact bones preserved in an excessively unfavourable sedimentary context. The strong representation of these remains seems to result from the conjunction of different factors:

- The purpose of the combustion (maintenance of camp and/or use as fuel).

- The intense fragmentation of the charred bones.

- A greater preservation potential of the burned bones (compact and spongy), including charred bones, in comparison to unburned bone elements when they are buried in acid sediments.

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References

Bellomo R. -V. 1993 - A methodological approach for identifying archaeological evidence of fire resulting from human activities. *Journal of Archaeology Science,* 20 : 525-553.

Bennett J. -L. 1999 - Thermal alteration of buried bone. *Journal of Archaeology Science,* 26 : 1-9.

Beyries S. 2002 - Le travail du cuir chez les Tchouktches et les Athapaskans, *In* : *Le travail du cuir de la Préhistoire à nos jours,* Audouin-Rouzeau F. & Beyries S. (eds). XXII[e] Rencontres Internationales d'Archéologie et d'Histoire d'Antibes, APCDA-CNRS : 143-157.

Bracco J. -P. 2004 - Fonction et fonctionnement du gisement épigravettien de Saint-Antoine à Vitrolles (Hautes-Alpes) : données et propositions, *In* : *Approches fonctionnelles en Préhistoire,* Bodu P. & Constantin Cl. (eds). Paris, Société préhistorique française : 335-340.

Bracco J. -P, Gagnepain J., Stouvenot Ch., Bidart P. & Vigier S. 1997 - L'industrie lithique épigravettienne de Saint-Antoine - locus 2 (Vitrolles, Hautes-Alpes) : première analyse. *Paléo*, 9 : 221-243.

[1] - Combustion zones*: «Presumed site of fire(s) for which the perimeter may be defined with the help of various indices particular to combustion (reddening of the sediments, ash deposit, etc.)»* (Gascó, 2003, 109).

[2] - Waste zones: «*These are comprised of waste issued from hearths. They generally come from a maintenance operation of the hearth (cleaning). Generally these are more or less dense scatters or layers*» (Vicherd, 2003, 16)

Cain C. -R. 2005 - Using burned animal bone to look at Middle Stone Age occupation and behaviour. *Journal of Archaeology Science*, 32 : 873-884.

Castel J.-Ch. 2003 - Economie de chasse et d'exploitation de l'animal au Cuzoul-de-Vers (Lot) au Solutréen et au Badegoulien. *Bulletin de la Société préhistorique française*, 100 : 41-66

Costamagno S., Griggo Ch. & Mourre V. 1999 - Approche expérimentale d'un problème taphonomique : utilisation de combustible osseux au Paléolithique. *Préhistoire Européenne*, 13 : 167-194.

Costamagno S., Théry-Parisot I., Brugal J.-P. & Guibert R. 2005 - Taphonomic consequences of the use of bones as fuel. Experimental data and archaeological applications. *In : Biosphere to Lithopshere. New studies in vertebrate taphonomy*, T. O'connor (ed). Actes du 9° Colloque de l'ICAZ, Durham, août 2002. Oxford, Oxbow Books : 51-62.

Costamagno I. Théry-Parisot J-C Castel & J-P Brugal 2009 - Combustible ou non ? Analyse multifactorielle et modèles explicatifs sur des ossements brûlés paléolithiques. *In : Gestion des combustibles au Paléolithique et au Mésolithique : nouveaux outils, nouvelles interprétations*, Théry-Parisot I., Costamagno S., Henry A. (eds). Proceedings of workshop 21. XV[e] Congrès de l'UISPP, Lisbonne, 4-9 septembre 2006. BAR International Series 1914, Oxford, Archaeopress : 47-60.

David B. 1990 - How was this bone burnt ? *In : Problem solving in taphonomy,* Solomon S.- I., Davidson B. & Watson (eds). Tempus, 2 : 65-79.

David H. 2001 – *Paléoanthropologie et pratiques funéraires en Corse, du Mésolithique à l'Age du fer*. Oxford, British Archaeological Reports, 148 pp.

Gagnepain J., Bracco J. -P., Bidart P. & Vigier S. 1997 - *Saint-Antoine à Vitrolles (Hautes-Alpes) un site de plein air du Paléolithique supérieur final*. Document final de synthèse de fouilles de sauvetage, Aix-en-Provence, Ministère de la culture, S.R.A, P.A.C.A, A.F.A.N., Escota, 1 vol., 374 pp.

Gagnepain J., Bracco J. -P., Bertran P., Bez J.- F., Bidart P., Canals i Salomo A., Jorda Ch., Jorda M., Philibert S., Stouvenot Ch. & Vigier S. 1999 - Saint-Antoine à Vitrolles, locus 2 (Hautes-Alpes) : premiers résultats des fouilles de sauvetage urgent (1995 - 1996) d'un gisement épigravettien. *Bulletin de la Société préhistorique française*, 96 : 191-202.

Gascó J. 2003 - Contribution pour une proposition de vocabulaire des structures de combustion, *In : Le feu domestique et ses structures au Néolithique et aux Ages des métaux,* Frère-Sautot M.-Ch. (ed), Bourg-en-Bresse et Beaune, Editions Monique Mergoil, 9 : 109-112.

Gerbe M. 2004 - *Etude taphonomique d'ossements brûlés*. Mémoire de DEA, Université d'Aix-Marseille I, 109 pp.

Gilchrist R. & Mytum C. 1986 - Experimental archaeology and burnt animal bone from archaeological sites, *Circaea*, 4 : 29-38.

Hermann B. 1977 - On histological investigations of cremated human remains. *Journal of Human Evolution,* 6 : 101-103.

Montoya C. & Bracco J. -P. 2005 - L'industrie lithique du site épigravettien de Saint-Antoine à Vitrolles (Hautes-Alpes). *In* : *D'un Monde à l'Autre. Les systèmes techniques pendant le Tardiglaciaire autour de la Méditerranée occidentale,* Bracco J.-P. & Montoya C. (eds). Paris, Société Préhistorique française : 81-94.

Montoya C. & Peresani M. 2005 – Nouveaux éléments de diachronie dans l'Epigravettien récent des Préalpes de la Vénétie. *In : D'un Monde à l'Autre. Les systèmes techniques pendant le Tardiglaciaire autour de la Méditerranée occidentale*. Bracco J.-P. & Montoya C. (eds). Paris, Société Préhistorique française : 123-138.

Muret A., D'Anna A., Jaubert J. & Jorda M. 1991 - Un gisement tardiglaciaire de plein air dans les Alpes du Sud : Saint-Antoine (Vitrolles, Hautes-Alpes). *Bulletin de la Société préhistorique française*, 88 : 49-57.

Rillardon M. 2003 - *Le gisement épigravettien de Saint-Antoine (Vitrolles, Hautes-Alpes : locus 2. Etude archéozoologique*, Mémoire de maîtrise, Université d'Aix-Marseille I, 76 pp.

Shipman P., Foster G. & Scoeninger M. 1984 - Burnt bones and teeth : an experimental study of color, morphology, crystal structure and shrinkage. *Journal of Archaeology Science*, 11: 307-325.

Stiner M. -C., Kuhn S. -L., Weiner S. & Bar-Yosef O. 1995 - Differential burning, recrystallization, and fragmentation of archaeological bone. *Journal of Archaeology Science*, 22 : 223-237.

Susini A. 1988 - *Etudes des caractéristiques biophysiques des tissus calcifiés humains (os, émail, dentine) soumis à des traitements thermiques. Applications anthropologiques et médicales*. Thèse de doctorat, Université de Genève, 1 vol., 225 pp.

Théry-Parisot I. 2002 – Fuel management (bone and wood) during the Lower Aurignacien in the Pataud rock shelter (Lower Palaeolithic, Les Eyzies de Tayac, Dordogne, France). Contribution of experimentation. *Journal of Archaeological Science*, 29 : 1415-1421.

Théry-Parisot I., Brugal J.-P., Costamagno S. & Guilbert R. 2004 - Conséquences taphonomiques de l'utilisation des ossements comme combustible. Approche expérimentale. *Les nouvelles de l'Archéologie* : 19-22.

Théry-Parisot I. & Costamagno S. 2005 - Propriétés combustibles des ossements. Données expérimentales et réflexions archéologiques sur leur emploi dans les sites paléolithiques. *Gallia-Préhistoire*, 47 : 235-254.

Théry-Parisot I., Costamagno S., Brugal J.-P., Fosse P. & Guilbert R. 2005 - The use of bone as fuel during the Palaeolithic, experimental study of bone combustible properties. *In* : *The zooarchaeology of fats, oils, milk and dairying*, Mulville J. & Outram A.K. (eds). Actes du Colloque de l'ICAZ, Durham, août 2002. Oxford, Oxbow Books : 50-59.

Thiébaut C., Costamagno S., Coumont M.-P., Mourre V., Provenzano N. & Thery-Parisot I. sous presse - Approche expérimentale des conséquences du piétinement des grands herbivores sur les vestiges archéologiques. *Paleo*, numéro spécial.

Vaté V. & Beyries S. 2007 - Une ethnographie du feu chez les éleveurs de rennes du Nord-Est sibérien. *In* : *Les civilisations du renne d'hier et d'aujourd'hui. Approches ethnohistoriques, archéologiques et anthropologiques*, Beyries S. & Vaté V. (eds). XXVII[e] rencontres internationales d'archéologie et d'histoire d'Antibes. Antibes, APDCA-CNRS : 393-420.

Vicherd G. 2003 - Petit rappels sur le feu et ses effets... *In* : *Le feu domestique et ses structures au Néolithique et aux Ages des métaux*, Frère-Sautot M. -Ch. (ed). Bourg-en-Bresse et Beaune, Editions Monique Mergoil : 15-17.

Villa P., Bon F. & Castel J.-C. 2002 - Fuel, fire and fireplace in the Palaeolithic of Western Europe. *The Review of Archaeology*, 23 : 33-42.

Vigne J. -D. 1983 – *Les mammifères terrestres non volants du Post-glaciaire de Corse et leurs rapports avec l'homme : étude paléo-ethno-zoologique fondée sur les ossements*. Thèse de doctorat, Université Pierre et Mare Curie, 501 pp.

Vigne J. -D., Marinval-Vigne M. -C., Lanfranchi (de) F. & Weiss M.-C. 1981 - Consommation du « lapin-rat » (*Prolagus sardus* Wagner) au Néolithique ancien méditerranéen Abri d'Araguina-Sennola (Bonifaccio, Corse). *Bulletin de la Société préhistorique française*, 78 : 22-224.

Walters J. 1988 - Fire and bones : patterns of discard, *In* : *Archaeology with Ethnography : An Australian Perspective*, Meehan B. & Jones R. (eds). Cambera, Australian National University : 215-221.

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TAPHONOMIC IMPLICATIONS OF THE USE OF BONE AS FUEL

Eugène MORIN

Abstract

This paper explores the effects of the use of bone as fuel on archaeological skeletal part representation. Faunal data from the Paleolithic site of Saint-Césaire show that this activity may present an archaeological signature similar to that of differential preservation. The bones most frequently burned at Saint-Césaire are also those that are the least dense and that contain the most grease. The analysis of faunal remains from Saint-Césaire also suggests that spongy bone fragments from small-bodied and large-bodied taxa are subject to differential identification.

Keywords : archaeology, fauna, burning, burned bone, bone identification

Introduction

Despite some early innovative research, particularly the work of Brain (1969), taphonomic analysis of faunal assemblages did not become common practice in archaeology until the end of the 1970s. Since that time, taphonomic studies have highlighted the difficulties related to the interpretation of anatomical profiles (Binford & Bertram, 1977; Binford, 1978, 1981; Poplin, 1978; Lyman, 1984; Grayson, 1989; Marean & Kim, 1998; Bartram & Marean, 1999; Outram, 2001; Pickering *et al*., 2003; Lam & Pearson, 2005; Novecosky & Popkin, 2005; Faith *et al*., 2007). The consensus that has emerged from these studies is that numerous geological, biological, and cultural factors contribute to faunal assemblage variability. Among the causes proposed to explain this variability, differential preservation generally plays a central role.

The analysis of differential preservation, which is defined as the selective destruction of faunal remains by meteorological and postdepositional phenomena, has greatly evolved over the years. Today, methods relying on quantitative techniques have replaced the subjective evaluations carried out in the past. The latter approaches have, among others, highlighted problems of equifinality in the identification of differential preservation in archaeological contexts (Grayson, 1989). However, the intentional use of bone as fuel has largely been ignored in these discussions, despite the fact that burning may entail the destruction of bone portions or even entire elements. In this article, the factors regulating the selection of bone elements for combustion will be examined, as well as the consequences of this practice for the interpretation of anatomical profiles.

Density and differential preservation

Bone density is a determining factor in the preservation of faunal remains (Lam & Pearson, 2005). But what is really meant by the term 'bone density'? Living bone is a solid composite material composed of an organic part dominated by a protein, collagen, and of a mineral part consisting of hydroxyapatite crystals (Boskey, 2006). The first component lends flexibility to the bone, while the second provides rigidity (Seeman, 2006). According to Boskey (2006), the general characteristics of bone differ little between spongy and compact bone, as both types of bone structure are composed of a solid mineral matrix composed of small canals, spaces (*lacunae*), and bone cells. The difference between compact and spongy bone lies in the organization of the bony matrix: spongy bone is composed of thin interconnected spicules, while compact bone is organized in Haversian systems (Boskey, 2006). This structural variability results in differences in "bulk density" (Lyman, 1994), or more exactly, differences in bone porosity, a term that more accurately captures the structural changes encountered within a single bone. A bone with low bulk density is therefore porous, and these pores are generally filled with grease (Brink, 1997). The presence of a large number of pores increases the surface area to volume ratio of the bone, which may contribute to the *post mortem* fragility of bone. In archaeological contexts, the rarity or absence of spongy bone may be indicative of differential preservation of elements.

However, there are factors besides differential preservation that may modify anatomical profiles as a function of bone density. These factors include the destruction of spongy bone by carnivores (e.g. Binford, 1981; Blumenschine, 1986; Munson, 2000), the production of bone grease (Brink, 1997; Munro & Bar-Oz, 2005), and a practice that is becoming rare – selective discard during archaeological excavation, generally carried out at the expense of long bone diaphyses (Turner, 1989; Marean & Kim, 1998; Grayson & Delpech, 2008). With the exception of selective discard, which decreases correlations with density, these factors are important because their anatomical signatures may be very similar to those of differential preservation. Is this also the case for intentional burning?

The relationship between bone porosity and combustibility

A growing number of studies suggest a non-random use of bone as fuel during the Late Pleistocene in France (Castel, 1999; Costamagno, 1999; Costamagno *et al*., 1999, 2005; Morin, 2004; Villa *et al*., 2004; Théry-Parisot *et al*., 2005). Is it possible to reconstruct the decision-making process regulating the selection of bones for burning during the Paleolithic? If yes, can we determine if these decisions were mediated by bone porosity? An examination of the relationship between bone porosity and combustibility permits a response to these questions.

If certain parameters (e.g., degree of fragmentation, age and condition of the animal, humidity level) are held constant, the combustibility of bone should be correlated with the quantity of lipids the bone contains, as lipid molecules are highly flammable. Experimental data support this hypothesis and show that spongy epiphyses generally burn much better than compact diaphyses (Costamagno *et al*., 1999, 2005). However, it remains unclear whether this general relationship holds true when examined in detail. Is bone porosity in fact a good indicator of grease content?

In order to address this problem, the density of reindeer bones (*Rangifer tarandus*, Lam *et al*., 1999) was compared with the percentage of lipids in bison bones (*Bison bison*, Brink, 1997; Emerson, 1990). The use of two different species in this comparison, which was necessary due to a lack of data, limits the scope of the results obtained here. Nevertheless, given that bone porosity seems to vary little between artiodactyl species (Lam *et al*., 1999), it is not unreasonable to suggest that the skeletal fat content of reindeer and bison varies in similar ways.

Figure 1 shows a very strong negative correlation between reindeer long bone density and the percentage of fat in bison long bones (Spearman's rho = -0.87, p = <0.001). The correlation decreases, but remains high, when the carpals, tarsals, and scapula are included (Spearman's rho = -0.60, p = <0.01; Emerson 1990: 390, table 5.39, mass in grams, average of four individuals, phalanges excluded, see above for this exclusion; long bone values as calculated by Emerson). This weaker correlation can probably be explained by the absence of diaphyses in Emerson's analysis. It appears that among artiodactyls, bone density is largely correlated with fat content, but it remains to be seen if porosity can be used to predict the use of bone as fuel in archaeological contexts.

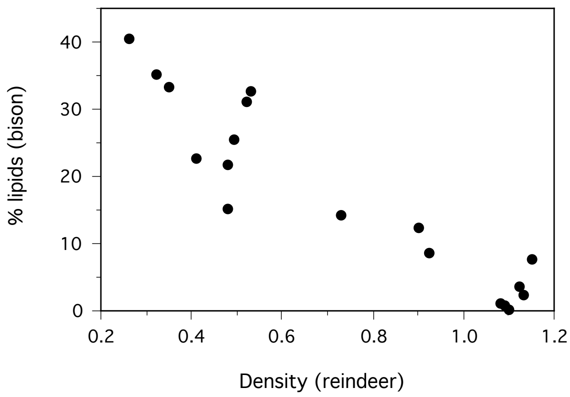

Fig. 1 - Relationship between the average percentage of fat in the long bones of three plains bison (Brink, 1997: 262, Table 1) and the average density of bones from four reindeer (Lam *et al.*, 1999: 351-353, Table 1).

Archaeological application: The case of Saint-Césaire

Located a few kilometers from the village of Saintes in Charente-Maritime, Saint-Césaire is an important site containing an archaeological sequence chronologically spanning the end of the Mousterian through the Evolved Aurignacian (Lévêque *et al*., 1993). This sequence also contains a Châtelperronian occupation (Tab. 1). Reindeer, bison, and horse are the best represented species in the Mousterian and Châtelperronian levels, while reindeer alone dominate the Aurignacian layers. The site lends itself well to the study of human behaviors, given that the impact of carnivores on the faunal assemblages is minimal (Morin, 2004, 2008).

Burned remains are abundant in the Saint-Césaire sequence, representing over 21% of the remains studied, excluding the low density layer EJO inf (Morin, 2004). As at many archaeological sites, the high degree of fragmentation of burned remains made the identification of burned specimens very difficult. As a result, these remains were rarely identified taxonomically. However, burned specimens constitute a relatively large portion of the Early Aurignacian assemblage (EJF, NISP = 4102), a rich layer in which 82% of identified remains, of which 3% are burned,

Level	Cultural attribution	TL dates (in 1000s of years)	Fauna
EJJ	Evolved Aurignacian		Dominated by reindeer
EJM	Evolved Aurignacian		Dominated by reindeer
EJF	Early Aurignacian		Dominated by reindeer
EJO sup	Proto-Aurignacian	30,8-34,0	Dominated by reindeer
EJO inf	Low density		Mixed: reindeer, bison, horse
EJOP sup	Châtelperronian	33,7-38,2	Mixed: reindeer, bison, horse
EJOP inf	Mousterian		Mixed: reindeer, bison, horse
EGPF	Denticulate Mousterian	33,5-47,1	Mixed: reindeer, bison, horse
EGP	Denticulate Mousterian	36,8-39,7	To be determined
EGF	Denticulate Mousterian	42,4 ± 4,3	To be determined
EGC sup	Mousterian		To be determined
EGC	Mousterian of Acheulean Tradition		To be determined
EGC inf	Mousterian		To be determined
EGB sup	Mousterian of Acheulean Tradition		To be determined
EGB inf	Unknown		To be determined

Tab. 1 - The Saint-Césaire sequence. Thermoluminescence dates from Mercier *et al.*, 1991. For levels where several dates are available, only the spread of dates is indicated.

are attributed to reindeer. Faunal remains from the Early Aurignacian layer also show systematic fracturing of long bones for marrow extraction.

Reindeer remains from EJF are characterized by significant variations in the frequency of burning of different skeletal parts (Tab. 2). The percentages of burned remains are particularly high among the vertebrae, pelvis, long bone extremities, carpals and tarsals. The degree of carbonization of the remains (Fig. 2) is not consistent with superficial burning during cooking or roasting of fleshed elements (Speth & Clark, 2006). This observation raises the question of whether the pattern of burning in the EJF assemblage is concordant with an intentional combustion of fat-rich, and, hence, highly porous bones.

The question of whether highly porous bones were intentionally used as fuel can be examined by looking at the relationship between the density of skeletal parts and the frequency with which they are burned. In order to avoid the biasing effects of small sample sizes, bone elements or bone portions with a frequency of less than five identified specimens were eliminated from the analysis. Because the Nunamiut avoided burning phalanges, due to the unpleasant odor these elements produce during combustion (Binford, 1978: 153), correlations were calculated twice, once including and once excluding phalanges. The exclusion of phalanges from the sample tends to increases the strength of the correlations, but does not modify the significance of the results.

In the Early Aurignacian layer of Saint-Césaire, the least dense bones of reindeer are the most often burned. The correlation between these variables is strong, negative, and statistically significant, regardless of whether phalanges are included (Spearman's rho, r_s = -0.52, p = <0.01) or not (r_s = -0.61, p = <0.01). When the minimum sample size is increased from five to ten specimens, the correlations are strengthened (r_s = -0.73, p = <0.01 with phalanges, r_s = -0.61, p = <0.01 phalanges excluded). These results demonstrate that combustion is strongly correlated with bone density in this level, particularly for larger samples

Anatomic element	Density	Burned (n)	Total (n)	%burned
vertebrae[a]	0,44	39 (8)[b]	73 (26)[b]	53,4 (30,8)
ribs (RI3)	0,96	9	427	2,1
scapula (SP1)	1,01	0	20	0,0
humerus P (HU1)	0,26	0	1	0,0
humerus SH (HU3)	1,12	1	102	1,0
humerus D (HU5)	0,48	5	5	100,0
radius P (RA1)	0,53	14	29	48,3
radius SH (RA3)	1,09	1	205	0,5
radius D (RA5)	0,49	1	4	25,0
ulna P (UL1)	0,49	2	3	66,7
ulna SH (UL2)	0,84	4	31	12,9
scaphoid (scaphoid)	0,70	2	9	22,2
lunatum (lunate)	0,67	1	8	12,5
capitatum (magnum)	0,69	0	7	0,0
unciform (UNC)	0,72	0	1	0,0
metacarpal P (MC1)	0,92	0	16	0,0
metacarpal SH (MC3)	1,10	0	102	0,0
metacarpal D (MC5)	0,48	0	7	0,0
pelvis (AC1)	0,64	10	23	43,5
femur P (FE2)	0,52	4	10	40,0
femur SH (FE4)	1,15	1	93	1,1
femur D (FE6)	0,32	4	11	36,4
patella (PA1)	0,57	1	6	16,7
tibia P (TI1)	0,35	8	11	72,7
tibia SH (TI3)	1,13	11	373	2,9
tibia D (TI5)	0,73	3	8	37,5
malleolar (fibula)	0,68	4	5	80,0
talus (AS1)	0,68	12	18	66,7
calcaneus (CA2)	0,94	5	11	45,5
cubo-navicular (NC1)	0,56	5	8	62,5
greater cuneiform (cuneiform)	0,71	1	3	33,3
metatarsal P (MR1)	0,90	1	32	3,1
metatarsal SH (MR3)	1,08	9	806	1,1
metatarsal D (MR5)	0,41	0	3	0,0
phalanx 1 (P1-2)	0,92	1	40	2,5
phalanx 2 (P2-2)	0,72	2	23	8,7
phalanx 3 (P3-1)	0,48	0	12	0,0
Total/Percentage	-	161 (130)	2546 (2499)	6,3 (5,2)

[a]For vertebrae, counts include remains attributed to the same body size class as reindeer but not identified to the species level, in order to compensate for the poor identifiability of this part of the skeleton.
[b]Numbers in parentheses indicate the number of fragments confidently assigned to reindeer.

Abbreviations : P = proximal, SH = diaphysis, D = distal.

Tab. 2 - Density and percentage of burned reindeer bones in the Early Aurignacian level at Saint-Césaire. Density values are from Lam *et al.* (1999: 352-353, Table 1). The density scan sites are identified in the first column. The density of vertebrae corresponds to the average of the following scan sites: AT1, AX2, CE1, TH1, LU1. Elements for which density values are not available, as well as cranial elements, are not included in the table.

Fig. 2 - Examples of burned and unburned radio-ulnae from Saint-Césaire. Burned specimens, indicated by arrows, are all completely carbonized epiphyseal fragments.

(Fig. 3). Given the close link that seems to exist between bone density and fat content, it seems reasonable to conclude that the selection of bones for fuel during the Early Aurignacian occupation at Saint-Césaire was essentially determined by fat content. In order to determine whether this conclusion holds for all the levels at Saint-Césaire, it is necessary to consider not only the relationship between bone density and the frequency of burning, but also an additional complicating factor: the relationship between species body size and the identifiability of burned spongy bone.

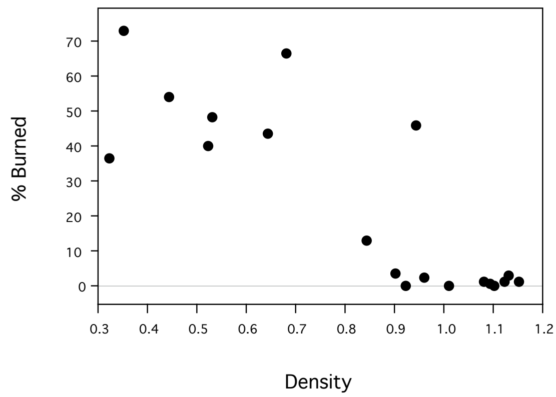

Fig. 3 - Correlation between the percentage of burned reindeer bones in the Early Aurignacian level of Saint-Césaire and bone density (Lam *et al.*, 1999). Data from Table 2. Phalanges are excluded from the analysis, and only elements with frequencies greater than or equal to 10 are included in the graph.

An allometric effect: the impact of body size on the identification of burned bones

In the Early Aurignacian layer at Saint-Césaire, 4.6% (141/3079) of postcranial remains identified to the species level are burned, all taxa included. In contrast, no burned postcranial element (0/483) was determined to the species level in the Châtelperronian level, where bison and horse are strongly represented. This highly significant difference (arcsine transformation, Sokal & Rohlf, 1969: 607-610; $t_s = 8.83$, $p < 0.0001$) is all the more surprising considering that the percentage of burned bones (Fig. 4a) and the anatomical profiles of the two levels are generally comparable, in spite of minor variations (Morin, 2004). Given this similarity, how can the low rate of identification of burned postcranial remains in the Châtelperronian level be explained?

The last two graphs in Figure 4 suggest a link between body size of the taxa dominating an assemblage and the percentage of burned postcranial remains identified to species. At Saint-Césaire, only small percentages of burned bones were identified to species in the lower levels (the Mousterian and Châtelperronian) where bison and horse are abundant. In contrast, the identification rates of burned remains are much higher in the upper (Aurignacian) levels, which are dominated by the smaller-bodied reindeer. Given the similarities in the anatomical profiles between these two sets of layers, it seems unlikely that this co-variation is purely accidental. The low identification rate of burned bones in the lower layers may signal an allometric relationship in which remains of large-bodied species are less frequently identified when burned than the remains of small-bodied species. The explanation proposed here for this phenomenon is that it is caused by a difference between small- and large-bodied taxa in the ratio of the surface area of burned fragments relative to the total surface area of the whole bone portion or element. This hypothesis requires some additional comments.

At Saint-Césaire, the average size of unidentified fragments of burned spongy bone varies less between levels than might be expected based on taxonomic composition. If this general impression is accurate, unidentified burned spongy bones of bison and horse would be *proportionally* more fragmented than those of reindeer. Consequently, as illustrated in Figure 5, spongy bone fragments of large taxa possibly sample a smaller fraction of the complete skeletal element than fragmentary remains of reindeer. This relative difference in size reduces the probability of identification of larger taxa. The next step consists of determining the reasons for this differential fragmentation. It is possible that the distributions of bone fragment size for small- and large-bodied species, which differed subsequent to marrow cracking, gradually converged due to the cumulative effects of combustion and post-depositional breakage (Fig. 6). The increasing overlap between the curves of fragment size distribution may be explained by the greater fragility of spongy bone fragments

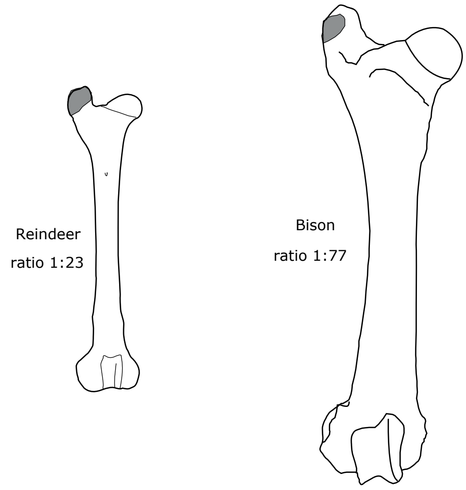

Fig. 5 - Fraction represented by an archaeological burned bone fragment of reindeer and bison relative to a complete bone. In this example, the fragment of bison is three times smaller, relative to the complete bone, than the reindeer fragment.

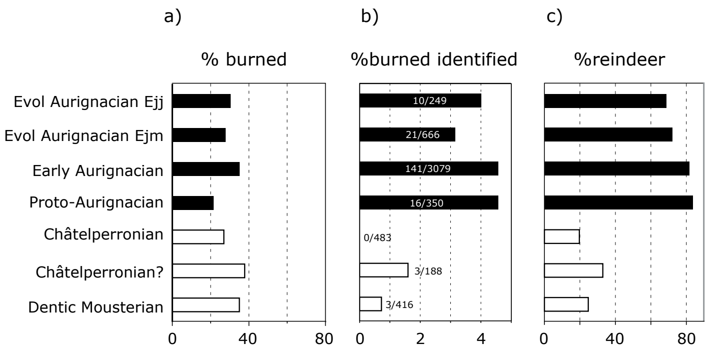

Fig. 4 - Percentage of (a) burned bones and (b) identified burned bones compared to (c) the relative abundance of reindeer in the different levels of Saint-Césaire. White bars indicate levels dominated by bison, horse, and reindeer, while black bars indicate levels dominated by reindeer alone. The raw data for graphs (a) and (c) are from Morin (2004: 141-142, 191).

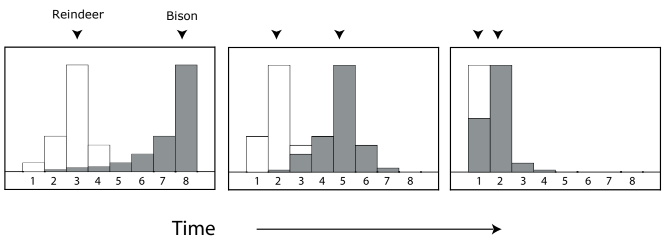

Fig. 6 - Theoretical temporal progression of the size of burned spongy bone fragments of reindeer and bison. In this example, the average size of burned bison fragments decreases more rapidly than that of reindeer due to the cumulative effects of combustion and post-depositional breakage. The arrows above the graphs indicate the average fragment size for each distribution. Numbers under the columns indicate hypothetical size classes of bone fragments.

belonging to large ungulates. Experimental work will be necessary to test these hypotheses.

Discussion and conclusion

For at least two reasons, differential preservation cannot be identified solely on the basis of a statistical correlation between anatomical profiles and bone density. The first reason is that such a correlation, *by comparing an archaeological anatomical profile to that of a living animal,* presupposes that whole carcasses were initially deposited at the site. This assumption is particularly dubious when assemblages consist of large ungulates, given the logistical constraints linked to the transport of these animals (O'Connell *et al*., 1990; Lyman, 1994; Lam & Pearson, 2005). The second, more clearly defined, problem is one of equifinality: an underrepresentation of spongy bone may have several causes, including carnivore attrition, bone grease production, or as we have seen here, the use of bone as fuel.

At Saint-Césaire, anatomical profiles appear to reflect the effects of combustion, which makes the examination of differential preservation difficult. The same pattern also seems to characterize assemblages from Grotte du Renne at Arcy-sur-Cure (David & Poulain, 2002), Cuzoul du Vers, Combe-Saunière (Castel, 1999), and Saint-Germain-la-Rivière (Costamagno, 1999). Potential solutions to this problem involve considering only unburned elements, or considering only *unburned bones of similar density*, an approach that significantly reduces the problem of differential preservation (see also Marean & Cleghorn, 2002). This second option was the one adopted in the analysis of the Saint-Césaire material (Morin, 2004: 302–304).

An additional problem raised in this article concerns the impact of species body size on the identification of fragments. At Saint-Césaire, burned spongy bone fragments of bison and horse are less frequently identified than those of reindeer. This differential identification can be attributed to two factors. First, spongy bone fragments of large ungulates are possibly more affected by combustion and post-depositional breakage than bone fragments of smaller ungulates. Secondly, the fragment area/element area ratio appears to be smaller for bison and horse than for reindeer, which renders identification of the larger species more difficult. Additional studies will show whether these conclusions hold for faunal assemblages from other sites.

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References

Bartram L. E. & Marean C. W. 1999 - Explaining the « Klasies Pattern » : Kua ethnoarchaeology, the Die Kelders Middle Stone Age archaeofauna, long bone fragmentation and carnivore ravaging. *Journal of Archaeological Science*, 26 : 9-29.

Binford L. R. 1978 - *Nunamiut ethnoarchaeology*. New York, Academic Press, 509 pp.

Binford L. R. 1981 - *Bones. Ancient men and modern myths*. New York, Academic Press, 320 pp.

Binford L. R. & Bertram J. B. 1977 - Bone frequencies and attritional processes. *In* : *For theory building in archaeology*, Binford L. R. (ed). New York, Academic Press : 77-153.

Blumenschine R. J. 1986 - *Early hominid scavenging opportunities : implications of carcass availability in the Serengeti and Ngorongoro ecosystems*. BAR international series, 163 pp.

Boskey A. H. 2006 - Mineralization, structure, and function of bone. *In* : *Dynamics of bone and cartilage metabolism. Principles and clinical applications*, Seibel M. J., Robins S. P. & Bilezikian J. P. (eds). Toronto, Academic Press : 201-212.

Brain C. K. 1969 - The contribution of Namib desert Hottentots to an understanding of Australopithecine bone accumulations. *Scientific Papers of the Namib Desert Research Station*, 39 : 13-22.

Brink J. W. 1997 - Fat content in leg bones of *Bison bison*, and applications to archaeology. *Journal of Archaeological Science*, 24 : 259-274.

Castel J. -C. 1999 - *Comportements de subsistance au Solutréen et au Badegoulien d'après les faunes de Combe-Saunière (Dordogne) et du Cuzoul de Vers (Lot)*. Thèse de doctorat, Université de Bordeaux I, 619 pp.

Costamagno S. 1999 - *Stratégies de chasse et fonction des sites au Magdalénien dans le sud de la France*. Thèse de doctorat, Université de Bordeaux I, 2 vol., 760 pp.

Costamagno S., Griggo C. & Mourre V. 1999 - Approche expérimentale d'un problème taphonomique : utilisation de combustible osseux au Paléolithique. *Préhistoire Européenne*, 13 : 167-194.

Costamagno S., Théry-Parisot I, Brugal J. - P. & Guibert R. 2005 - Taphonomic consequences of the use of bones as fuel : experimental data and archaeological applications. *In : Biosphere to lithosphere : new studies in vertebrate taphonomy*, O'Connor T. (ed.). IX^{ème} conférence de l'ICAZ, Oxbow Books : 51-62.

David F. & Poulain T. 2002 - Les mammifères (herbivores, carnivores, petits mammifères). *In : L'Aurignacien de la Grotte du Renne. Les Fouilles d'André Leroi-Gourhan à Arcy-sur-Cure (Yonne)*, Schmider B. (ed). Gallia Préhistoire 34, Paris, CNRS Éditions : 49-95.

Emerson A. 1990 - *Implications of variability in the economic anatomy of Bison bison*. Thèse de doctorat, Washington State University, 2 vol., 937 pp.

Faith J. T., Marean C. W. & Behrensmeyer A. K. 2007 - Carnivore competition, bone destruction, and bone density. *Journal of Archaeological Science*, 34 : 2025-2034.

Grayson D. K. 1989 - Bone transport, bone destruction, and reverse utility curves. *Journal of Archaeological Science*, 16 : 643-652.

Grayson D. K. & Delpech F. 2008 - The large mammals of Roc de Combe (Lot, France) : The Châtelperronian and Aurignacian assemblages. *Journal of Anthropological Archaeology*, 27 : 338-362.

Lam Y. M., Chen M. X. & Pearson O. M. 1999 - Intertaxonomic variability in patterns on bone density and the differential representation of bovid, cervid, and equid elements in the archaeological record. *American Antiquity*, 64 : 343-362.

Lam Y. M. & Pearson O. M. 2005 - Bone density studies and the interpretation of the faunal record. *Evolutionary Anthropology*, 14 : 99-108.

Lévêque F., Backer A. M. & Guilbaud M. 1993 - *Context of a late Neandertal : implications of multidisciplinary research for the transition to Upper Paleolithic adaptations at Saint-Césaire*. Monographs in World Archaeology 16. Madison, Prehistory Press, 131 pp.

Lyman R. L. 1984 - Bone density and differential survivorship of fossil classes. *Journal of Anthropological Archaeology*, 3 : 259-299.

Lyman R. L. 1994 - *Vertebrate taphonomy*. New York, Academic Press, 524 pp.

Marean C. W. & Cleghorn N. 2003 - Large mammal skeletal element transport : applying foraging theory in a complex taphonomic system. *Journal of Taphonomy*, 1 : 15-42.

Marean C. W. & Kim S. Y. 1998 - Mousterian large-mammal remains from Kobeh Cave : behavioral implications for Neanderthals and early modern humans. *Current Anthropology*, 39 : S79-S113.

Mercier N., Valladas H., Joron J.-L., Reyss J.-L., Lévêque F., & Vandermeersch B. 1991 - Thermoluminescence dating of the late Neanderthal remains from Saint-Césaire. *Nature*, 351 : 737-739.

Morin E. 2004 - *Late Pleistocene population interaction in Western Europe and modern human origins : New insights based* *on the faunal remains from Saint-Césaire, southwestern France*. Thèse de doctorat, University of Michigan, 450 pp.

Morin E. 2008 - Evidence for declines in human population densities during the early Upper Paleolithic in western Europe. *PNAS*, 105 : 48-53.

Munro N. D. & Bar-Oz G. 2005 - Gazelle bone fat processing in the Levantine Epipalaeolithic. *Journal of Archaeological Science*, 32 : 223-239.

Munson P. J. 2000 - Age-correlated differential destruction of bones and its effect on archaeological mortality profiles of domestic sheep and goats. *Journal of Archaeological Science*, 27 : 391-407.

Novecosky B. J. & Popkin P. R. W. 2005 - Canidae volume bone mineral density values : an application to sites in western Canada. *Journal of Archaeological Science*, 32 : 1677-1690.

O'Connell J.F., Hawkes K. & Blurton Jones N. 1990 - Reanalysis of large mammal body part transport among the Hadza. *Journal of Archaeological Science*, 17 : 301-316.

Outram A. K. 2001 - The scapula representation could be the key : A further contribution to the « Klasies Pattern » debate. *Journal of Archaeological Science*, 28 : 1259-1263.

Pickering T. R., Marean C. W. & Domínguez-Rodrigo M. 2003 - Importance of limb bone shaft fragments in zooarchaeology : A response to « On in situ attrition and vertebrate body part profiles » M.C. Stiner. *Journal of Archaeological Science*, 30 : 1469-1482.

Poplin, F. 1978 - Essai d'ostéologie quantitative sur l'estimation du nombre d'individus. *Kölner Jahrbuch für Vor- und Frühgeschichte*, 16, 153-164.

Seeman E. 2006 - Bone structure and strength. *In* : *Dynamics of bone and cartilage metabolism. Principles and clinical applications*, Seibel M. J., Robins S. P. & Bilezikian J. P. (eds). Toronto, Academic Press : 213-220.

Sokal R. R. & Rohlf F. J. 1995 - *Biometry : The principles and practice of statistics in biological research*, 3ème édition. San Francisco, W.H. Freeman, 887 pp.

Speth, J. D. & Clark J. L. 2006 - Hunting and overhunting in the Levantine Late Middle Paleolithic. *Before Farming*, 3 : 1-42.

Théry-Parisot I., Costamagno S., Brugal J. -P., Fosse P. & Guilbert. R. 2005 - The use of bone as fuel during the Paleolithic. *In : Biosphere to lithosphere : new studies in vertebrate taphonomy*, O'Connor T. (ed). 9ème conférence de l'ICAZ, Oxbow Books : 50-59.

Turner A. 1989 - Sample selection, schlepp effects and scavenging : The implications of partial recovery for interpretations of the terrestrial mammal assemblage from Klasies River Mouth. *Journal of Archaeological Science*, 16 : 1-11.

Villa P., Bon F. & Castel J.-C. 2002 - Fuel, fire and fireplaces in the Palaeolithic of Western Europe. *The Review of Archaeology*, 23 : 33-42.

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