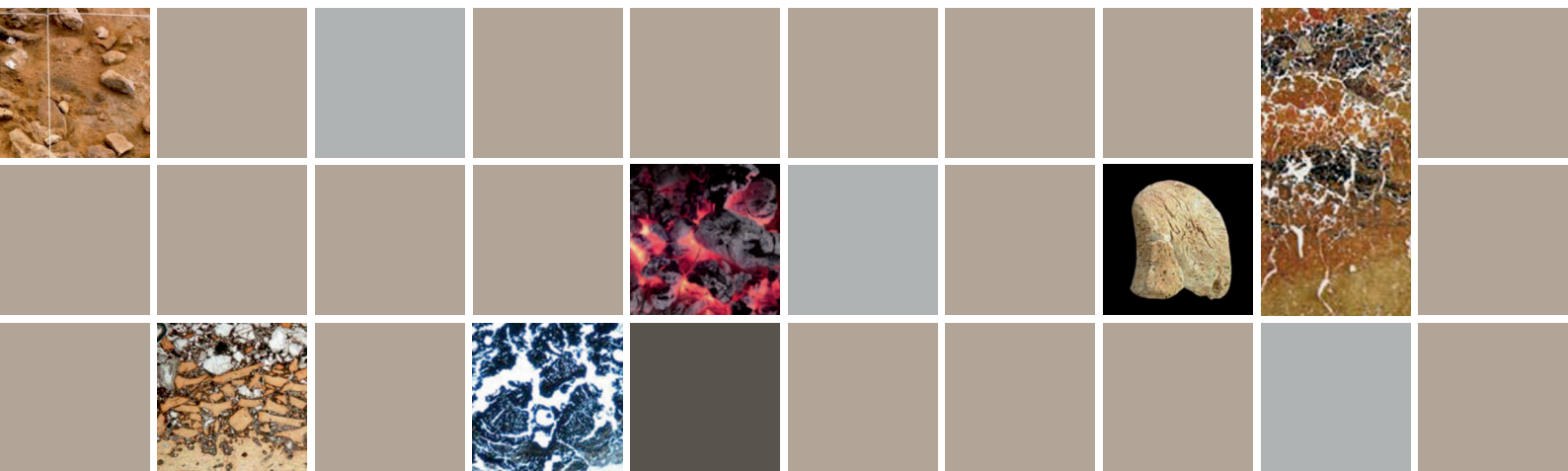


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# THE TAPHONOMY OF BURNED ORGANIC RESIDUES AND COMBUSTION FEATURES IN ARCHAEOLOGICAL CONTEXTS



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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, Néolithique, 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 5<sup>th</sup> and 4<sup>th</sup> 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 5<sup>th</sup> 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<sup>3</sup> wood-pile with, for example, 60% split oak logs, 30% hornbeam branches and 10% diverse woods, all covered by 0.5 m<sup>3</sup> 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<sup>2</sup>. 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<sup>3</sup>. 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

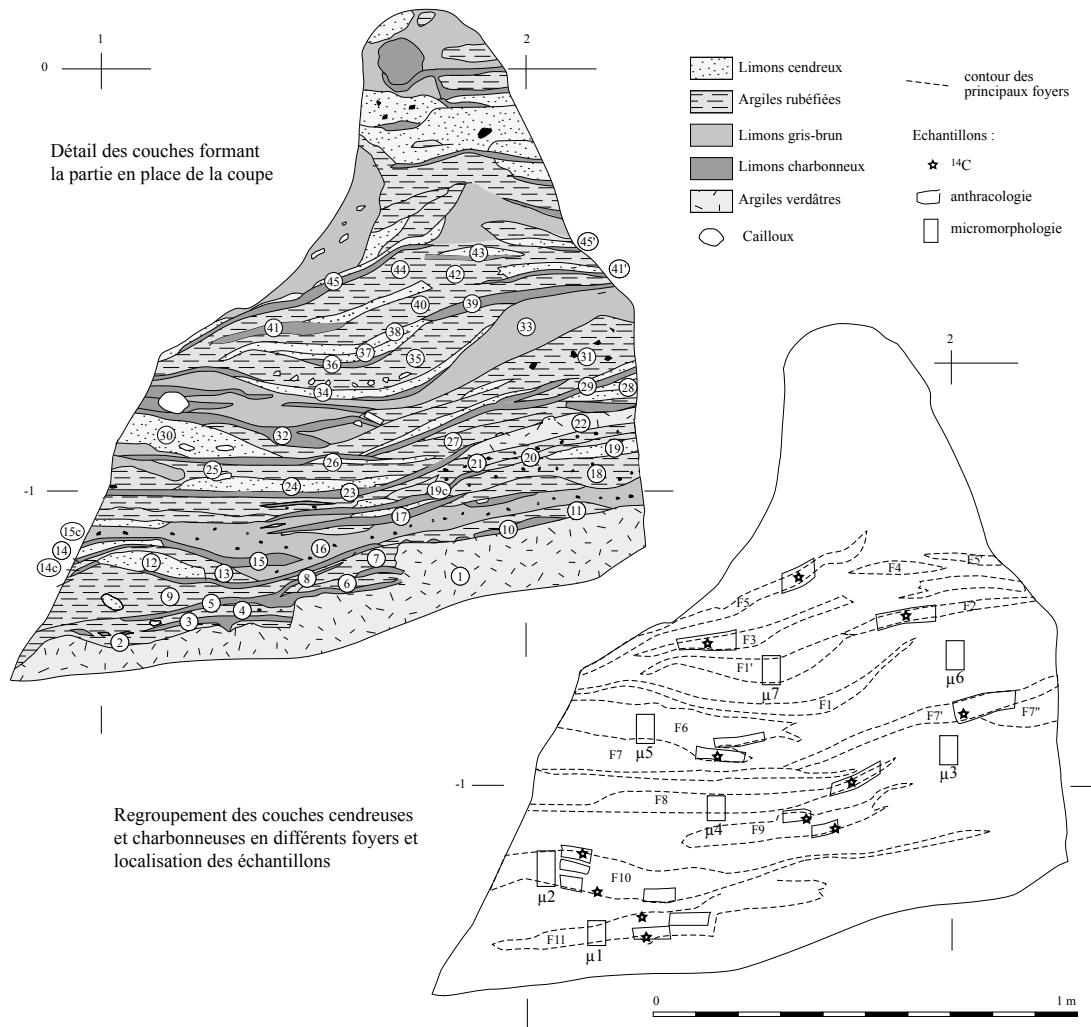
#### *Micromorphological analysis*

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.

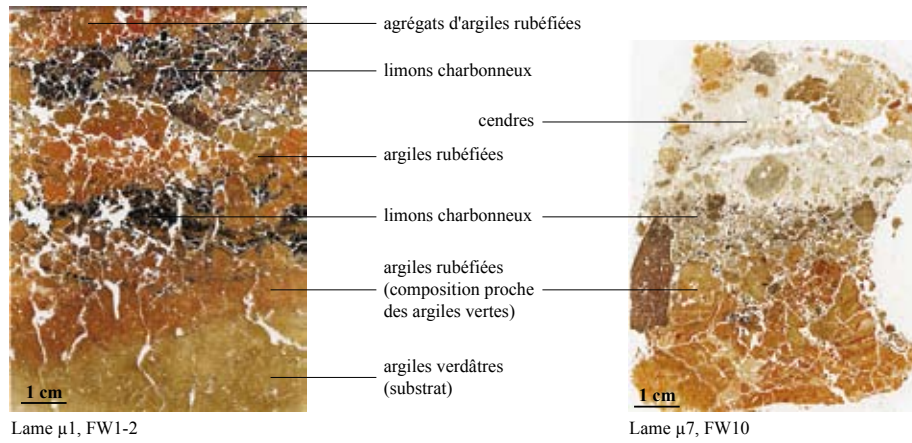
#### Secondary crystallizations

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)<sup>1</sup>. 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

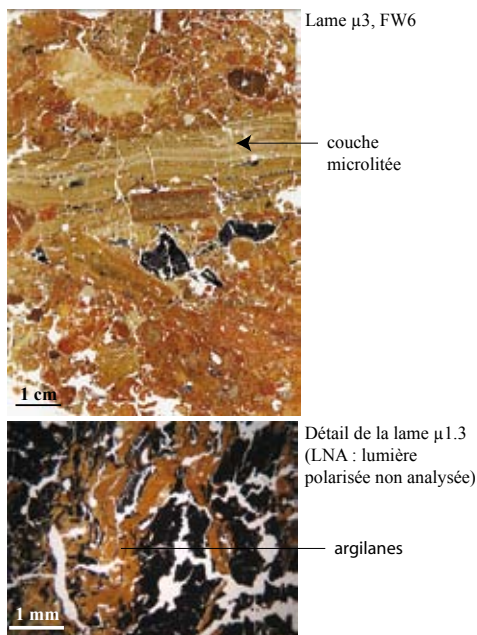
<sup>1</sup> - Only gypsum crystals were clearly identified. This determination was confirmed by J.-P. Sizun, Associate Professor, UMR 6249. The absence of halite is not surprising given the solubility of this salt and the humidity level of this context.



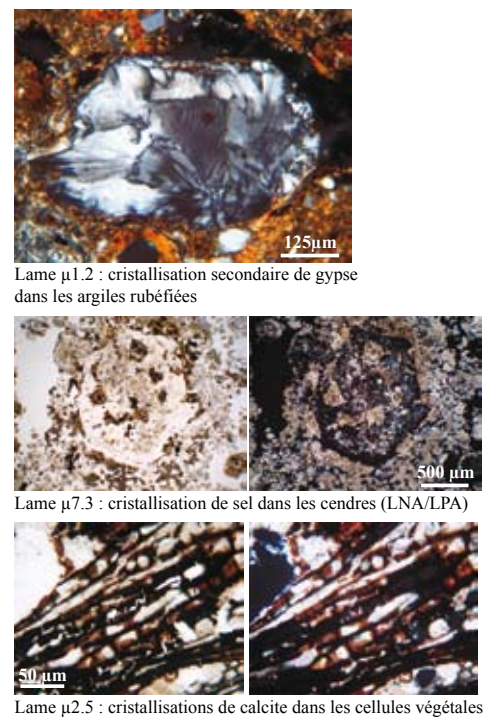
### a- Processus de combustion



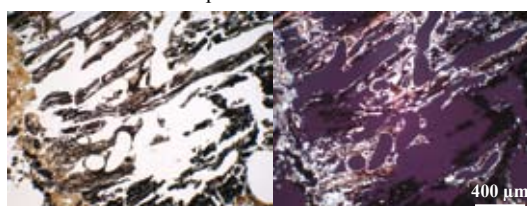
### b- Ruissellement et 'illuviation



### c- Processus de cristallisations



### d- Altération naturelle postsédimentaire



**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  $\mu 1$  and  $\mu 7$  illustrate two types of combustion feature functioning: complete combustion in  $\mu 7$ , characterized by the superposition of baked clay, few charcoals and abundant ash; interrupted combustion in  $\mu 1$ , characterized by the superposition of abundant charcoal on rubified clays; b) Runoff and illuviation processes. Thin section  $\mu 3$  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.

fireplaces was rich 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<sup>2</sup> 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,

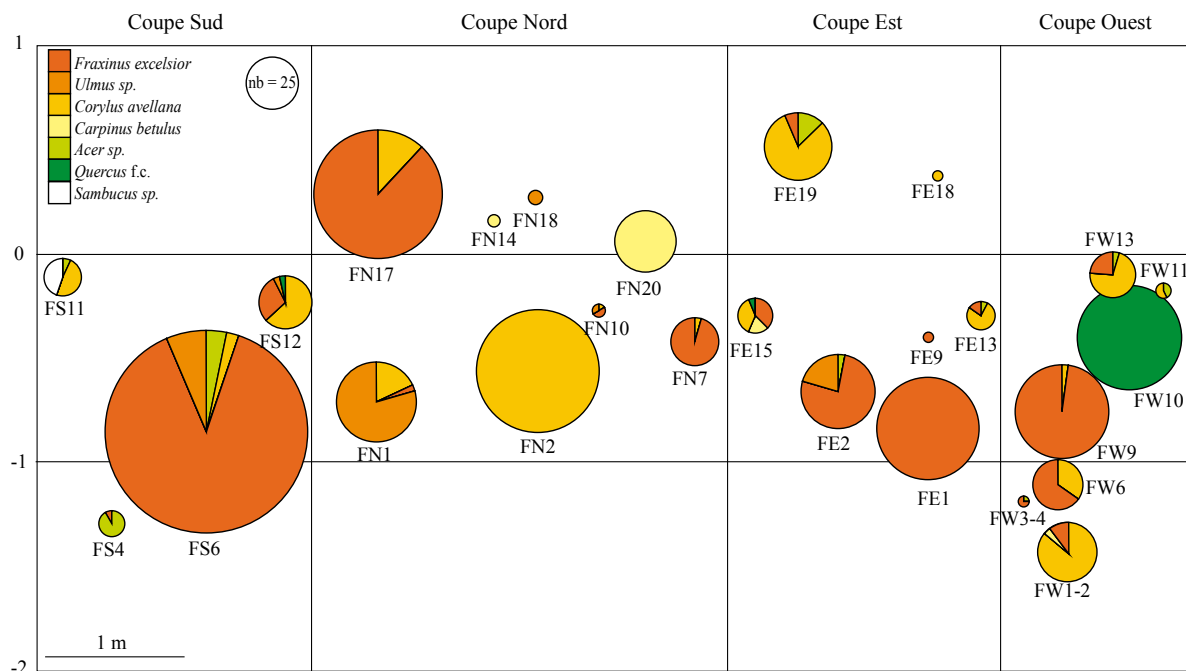
- (i) some fragments split in the direction of the fibers, which themselves were only slightly rigid (“limp”), making it impossible to obtain a transverse profile;
- (ii) before sieving, some clay or silt aggregates presented residues of carbonized ligneous structures on their surfaces, but these were too fragile to be isolated;
- (iii) some are “phantom” wood charcoal pieces, for which only the periphery remains since the interior was replaced by

<sup>2</sup> - 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	<i>Acer sp.</i>	<i>Corylus avellana</i>	<i>Carpinus</i>	<i>Corylus/Carpinus</i>	<i>Fraxinus sp.</i>	<i>Quercus sp.</i>	<i>Sambucus</i>	<i>Ulmus sp.</i>	<i>Angiospermæ</i>	TOTAL	Indéterminables	Nombre de taxons
coupe Nord	FN1		7			1			31	3	42		3
	FN2		60								60		1
	FN14			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
	FE20	6	46	3	0	91	1	0	7	38	192	112	
	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					1					0	0	0
	FS4	11									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.

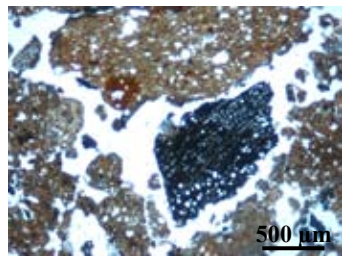
Among the forms of alteration of the wood charcoal, some fragments have a highly vitrified structure over almost their entire surface. Since pouring salt water on them causes the embers to cool rapidly and the salt acts as a dissolvent, high temperatures and thermal shock could have contributed to the 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).

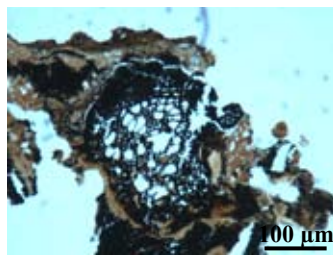
### Conclusion

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

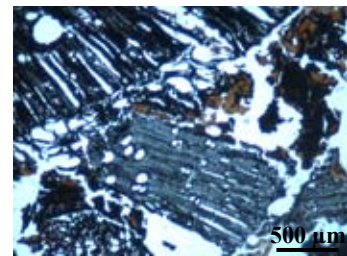
## a- Etat peu dégradé des charbons de bois



Lame μ6

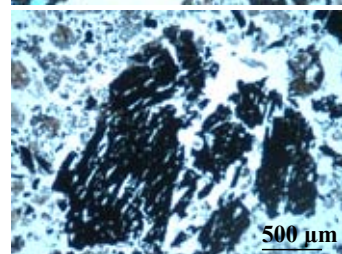
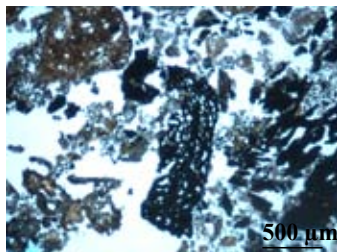


Lame μ1

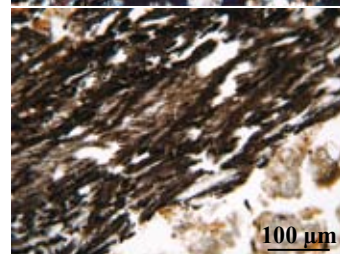
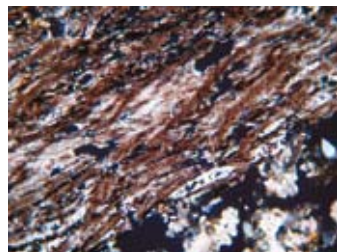


Lame μ1

## b- Etat moyennement dégradé des charbons de bois



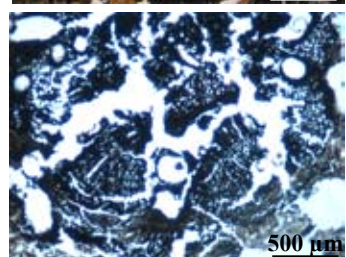
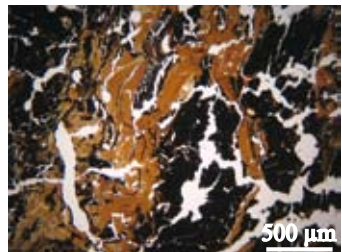
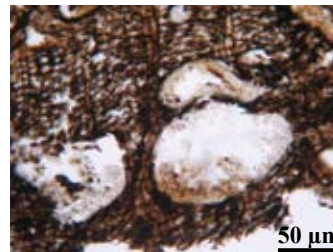
(i) : lame μ8.2



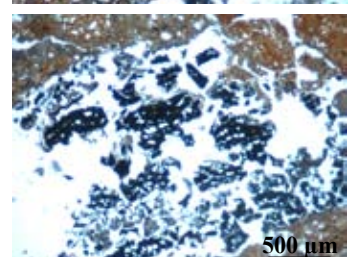
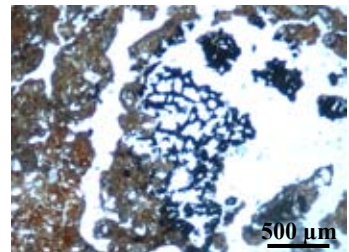
(ii) : lame μ8.5

**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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