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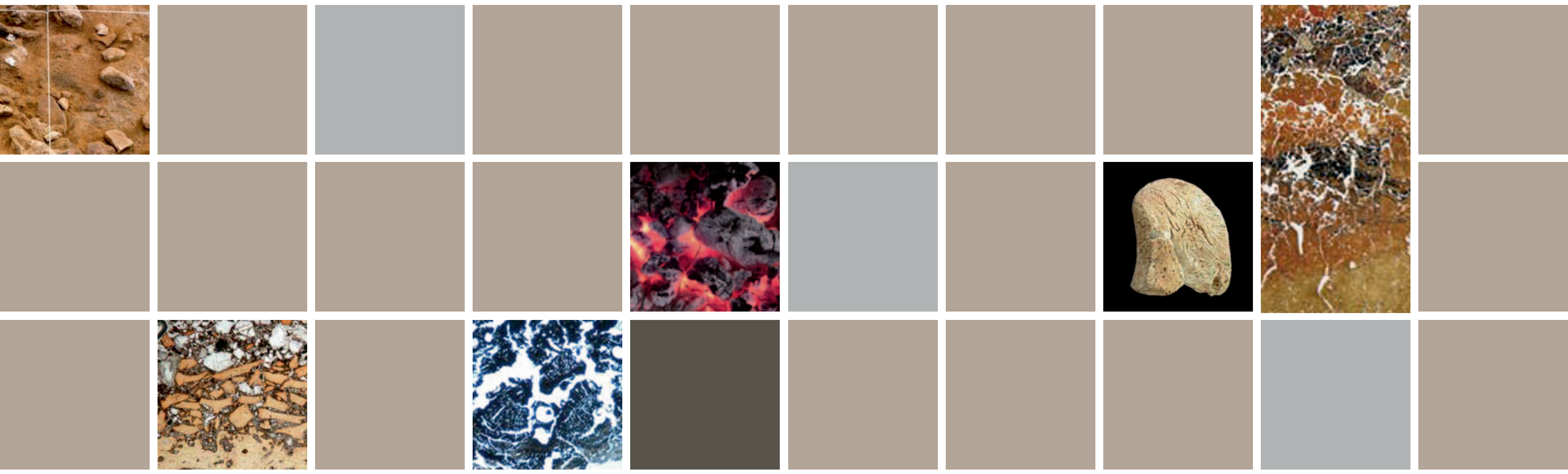
THE TAPHONOMY OF BURNED ORGANIC RESIDUES AND COMBUSTION FEATURES IN ARCHAEOLOGICAL CONTEXTS



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

Claire Delhon

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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

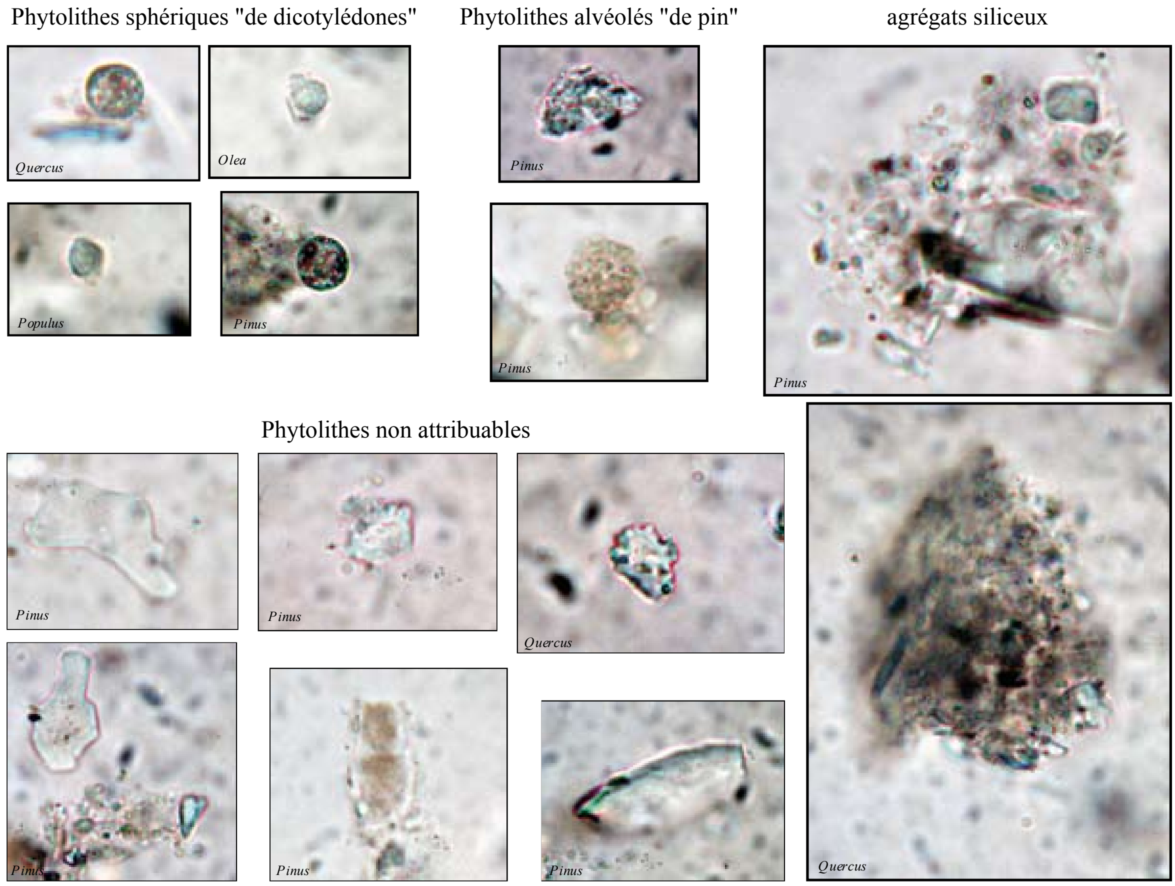

Fig. 1 - Siliceous particles observed in the wood ashes (transmission optic microscopy, magnification 1000 X) (C. Delhon).

	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)

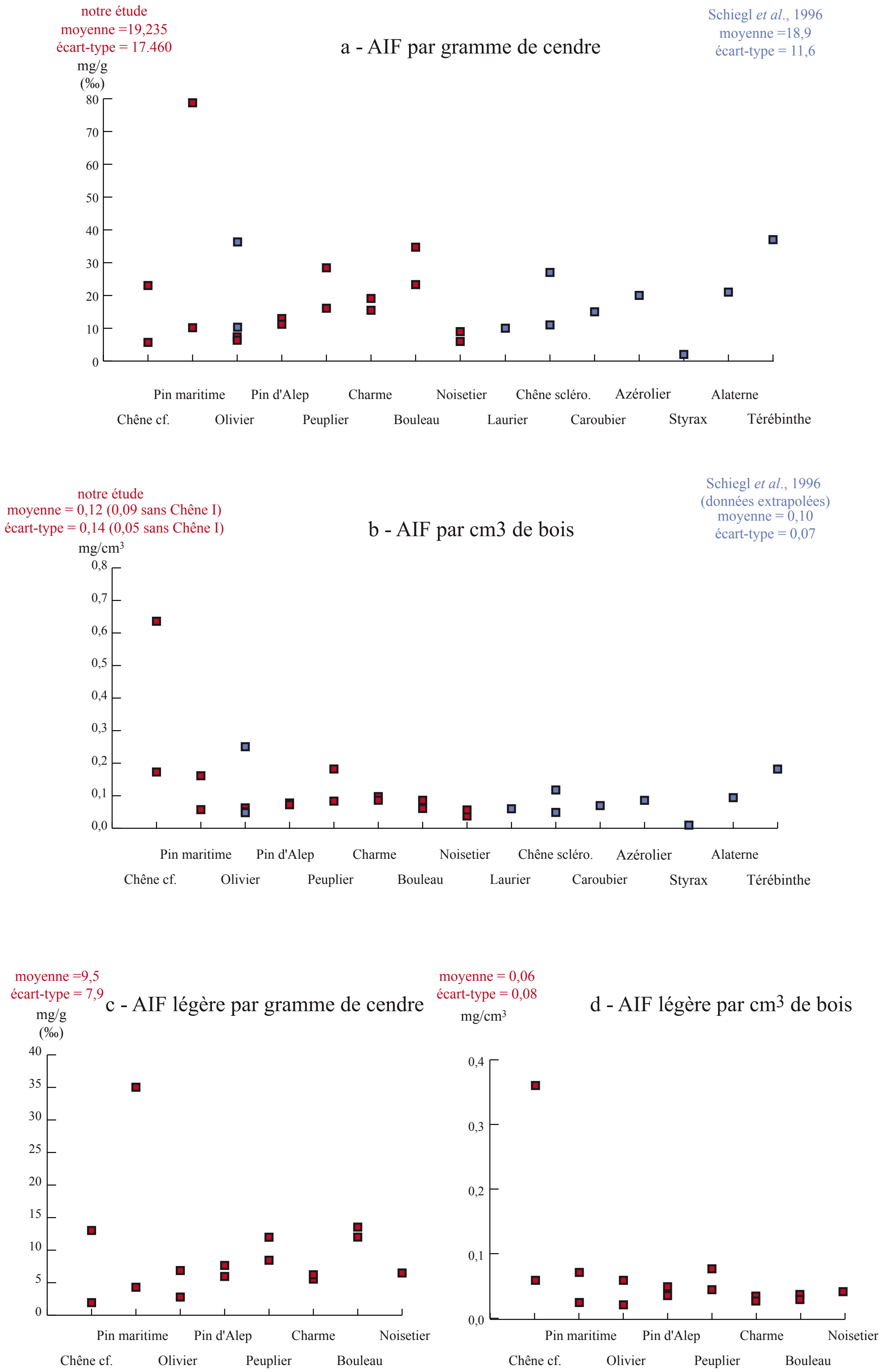

Fig. 2 - AIF (2a & 2b) light AIF (2c & 2d) content correlated with the mass of ash obtained (2a & 2c) and the volume of wood pout into the fire (2b & 2d) (C. Delhon).

	p = poids de cendres (1)	v = volume de bois (2)	proportion d'AIF dans la cendre (1)	AIF / cm³ de bois
	g	cm³	% du poids	mg/cm³
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** **6,5**	**6,8** **5,0**	X	MOYENNE ECART-TYPE
Pins	**83,1** **2,8**	**4,8** **2,0**	**12,1** **1,0**	MOYENNE ECART-TYPE
Dicotylédones	**92,6** **5,6**	**7,4** **5,6**	X	MOYENNE ECART-TYPE
Quercus & Olea	**85,3** **2.2**	**14,7** **2,2**	X	MOYENNE ECART-TYPE
Autres dicot.	**96,3** **1,0**	**3,7** **1,0**	X	MOYENNE 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,

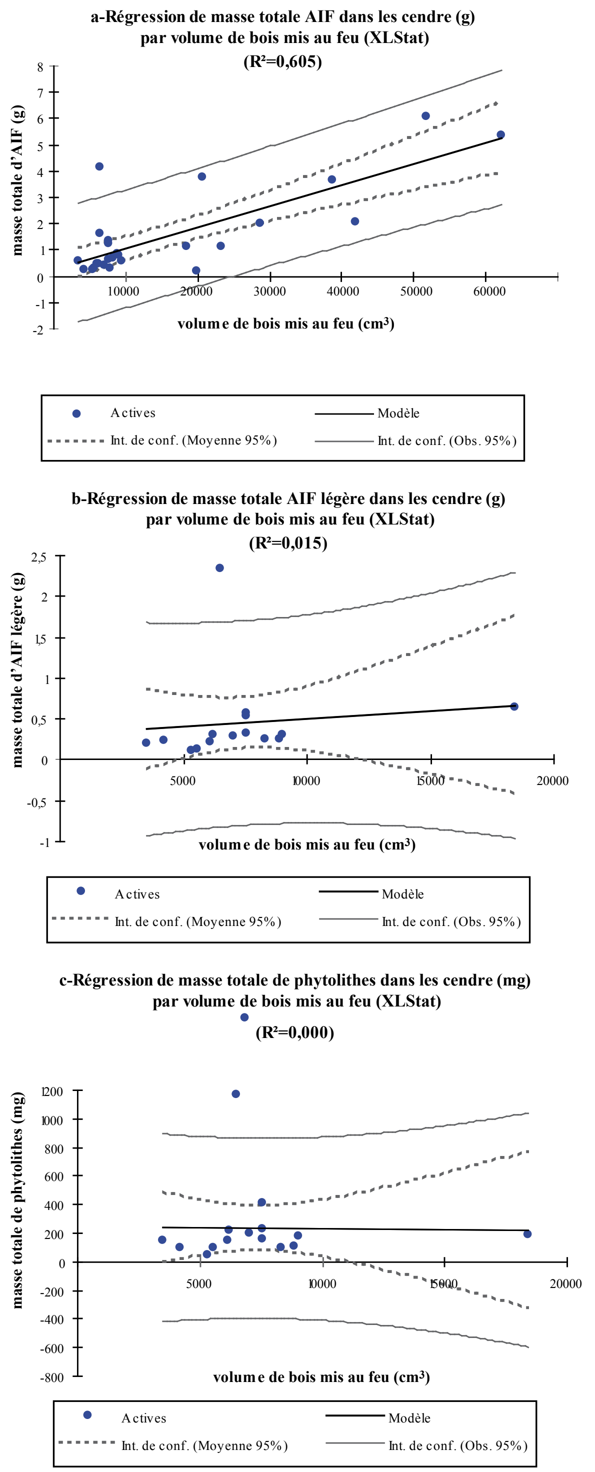

Fig. 4 - Regression curves of the residues (4a-AIF, 4b-light AIF, 4c-phytoliths) for the volume of wood put into the fire. Only the first one (total AIF mass per volume of wood put into the fire) shows a correlation between the variables (C. Delhon).

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

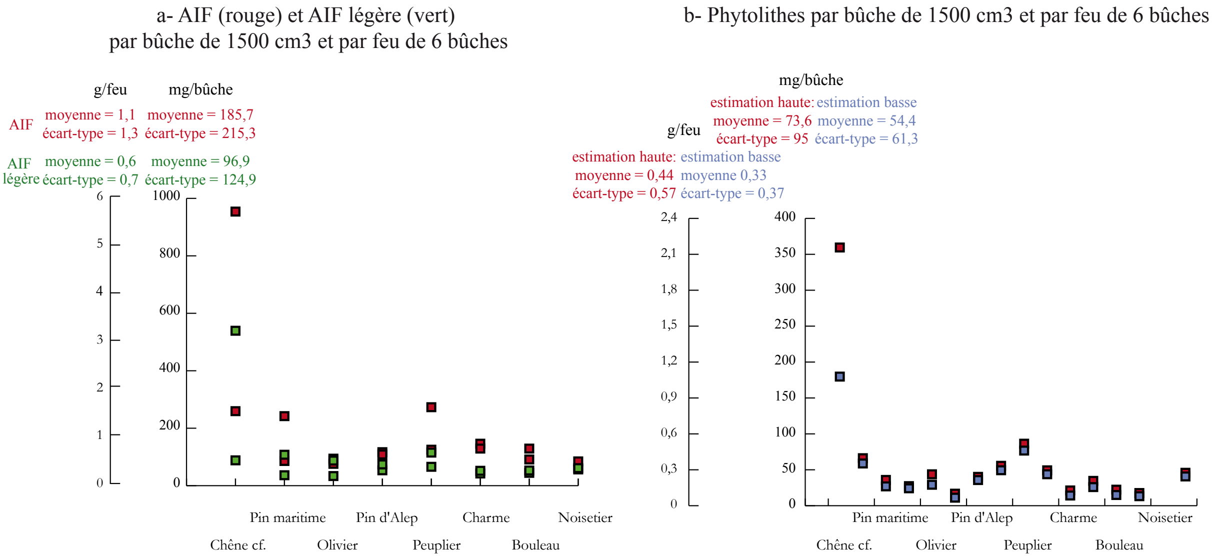

Fig. 5 - Quantity of AIF, light AIF (5a) and phytoliths (5b) per "standard" log of 1500 cm3 and per fire with 6 logs (C. Delhon).

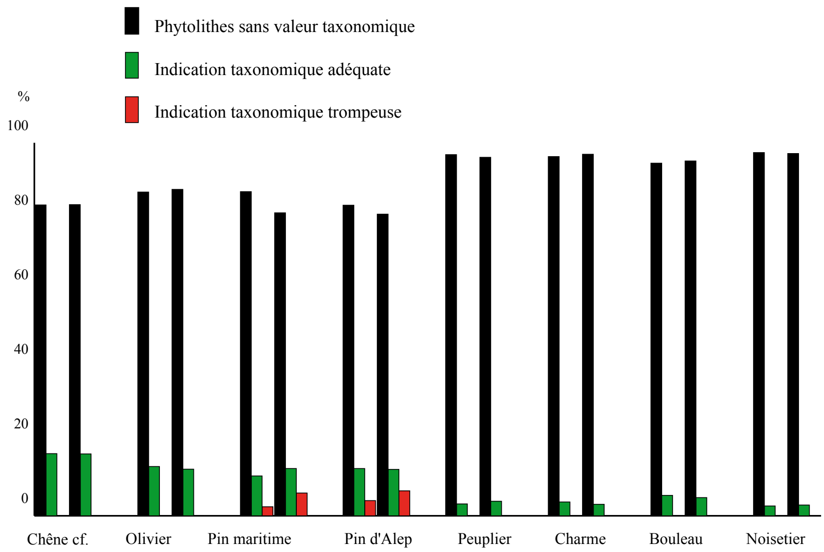

Fig. 6 - Results of the phytolithic analysis of ash: relative proportions of phytoliths to which no taxonomic value is attributed (black), phytoliths to which an adequate taxonomic value is attributed (green) and phytoliths to which an inadequate taxonomic value is attributed (red), for each studied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