



Iron Age combustion structures in the north-eastern Iberian Peninsula: an interdisciplinary experimental study

Maria Carme Belarte¹ · María Pastor Quiles² · Marta Mateu³ · Marta Portillo⁴ · Carme Saorin⁵ · Alessandra Pecci⁶ · Silvia Vila⁷ · Ada Gomar⁴

Received: 1 December 2022 / Accepted: 14 April 2023
© The Author(s) 2023

Abstract

The aim of this research is to investigate the efficiency of combustion structures, the fuels used, the structure maintenance and the range of related domestic activities. An experimental programme was carried out in which replicas of archaeological Iron Age combustion structures were put to work. Based on the available archaeobotanical records, various fuel types (wood, grasses, palm leaves and animal dung) were used. Variables such as increased heating and times were measured in order to determine whether variations occurred depending on the fuels used, the type of structure, the location, or the weather conditions. The possibility of their use for cooking was also tested. A combination of methodologies was applied for integrated analyses: anthracology, phytoliths, calcitic microfossils, Fourier transform infrared spectrometry, micromorphology and chemical analysis of hearth surfaces. Observations and data recorded during the experimental tasks, together with the results of the interdisciplinary analyses, contribute to a better understanding of the Iron Age archaeological combustion structures.

Keywords Iron Age · Western Mediterranean · Archaeological science · Experimentation · Hearths

✉ María Pastor Quiles
m.pastor@ua.es

Maria Carme Belarte
cbelarte@icac.cat

Marta Mateu
mmateu@icac.cat

Marta Portillo
mportillo@imf.csic.es

Carme Saorin
csaorin@ub.edu

Alessandra Pecci
alessandrapecci@ub.edu

Silvia Vila
svila10@gmail.com

Ada Gomar
agomar@imf.csic.es

² University of Alicante and ICAC (Catalan Institute of Classical Archaeology), Tarragona, Spain

³ ICAC (Catalan Institute of Classical Archaeology), 2021 BP 00103, Tarragona, Spain

⁴ Department of Archaeology and Anthropology, Archaeology of Social Dynamics (SGR-Cat 2021-501), Institució Milà i Fontanals (IMF), Spanish National Research Council (CSIC), Barcelona, Spain

⁵ Department of History and Archaeology, GRAP (Grup de Recerca en Arqueologia Protohistòrica), PGC2018-099579-B-I00, IAUB (Institut d'Arqueologia de La Universitat de Barcelona), Barcelona, Spain

⁶ ERAAUB, Department of History and Archaeology, INSA-UB, IAUB (Institut d'Arqueologia de La Universitat de Barcelona), Barcelona, Spain

⁷ University of Lleida, Lleida, Spain

¹ ICREA (Catalan Institution for Research and Advanced Studies) and ICAC (Catalan Institute of Classical Archaeology), Tarragona, Spain



Fig. 1 **a** Map of the study area with the location of the Ciutadella Ibèrica de Calafell archaeological site; **b** one of the streets of the reconstructed site, which is also an experimental archaeology centre

Introduction

This paper addresses experimental archaeology related to the fire structures built and used during the first millennium BC in the north-eastern Iberian Peninsula. The Iron Age settlement pattern in the study area is characterised by agglomerated sites. The houses were arranged sharing party walls built with stone and earth and roofs that consisted of a layer of earth on a bed of branches supported by wooden beams (Fig. 1b) (Belarte 2008). Combustion structures are well preserved at many archaeological sites from this period (Fig. 2) (Belarte et al. 2016), especially compared to those from previous chronologies. These structures show a diversity of morphological characteristics built with different materials and techniques (Belarte 2021; Belarte et al. 2022). In the framework of the project “Transdisciplinary and experimental study of firing structures in the Western Mediterranean during protohistory (first millennium BC) (TRANSCOMB)”, we are conducting interdisciplinary analyses to gain a better understanding of these structures (Belarte et al. 2023).

In this field of research, certain questions have arisen and remain open. Do the different traits of the combustion structures, especially the preparation layers, result in longer firing times and/or higher temperatures? Therefore, could their use have been intended for that purpose? Regarding fuels, were the different types and plant species used for



Fig. 2 Example of an Iron Age hearth, Barranc de Gàfols (Ginestar, Tarragona), sixth century BC

Table 1 Experimental combustion structures

Combustion structure	Location	Shape	Preparation layer	Measures	Features
Hearth 1	Indoors	Quadrangular	Mudbrick infill	116×120 cm	Earthen border of 20×11 cm. Mudbrick infill
Hearth 2	Indoors	Quadrangular	Pottery sherds	50×50 cm	Excavated in a shallow pit 6–7 cm in depth. Earthen border with vegetal temper
Hearth 3	Indoors	Oval	Pebbles	63×57 cm	Excavated in a shallow pit 8 cm in depth
Hearth 4	Outdoors	Oval	None	80×60 cm	Directly on the ground, an earthen layer of 1.5 cm
Hearth 5	Outdoors	Rectangular	None	72×52 cm	Excavated in a shallow pit 7 to 15 cm in depth
Hearth 6	Indoors	Quadrangular	Pebbles and pottery sherds	63×51 cm	Earthen border 6–8×9 cm
Oven	Outdoors	Oval ground plan, vaulted	None	150×60 cm	Mudbrick structure, covered with mud mixed with vegetal temper

different objectives? And how was smoke removed in the case of indoor fires? Regarding the specific use of hearths and ovens, is it possible to confirm their domestic or culinary functions?

To answer these questions, we have implemented an interdisciplinary methodology, which also has the objective of testing the suitability and limits of the different techniques.

An important part of the methodology consists of an experimental archaeology programme. It has been carried out at the Iberian Citadel of Calafell (Tarragona, Spain), an Iron Age site dating from the sixth to the first centuries BC (Pou et al. 2001) that has been reconstructed and is open to visitors. It is also an experimental archaeology centre (Fig. 1).

The experimental programme includes the following stages: the design of combustion structures of diverse types (with different building materials and techniques), building them in different locations (interior/exterior), firing and data collection, sampling and analyses and comparison with the archaeological data.

We selected two experimental structures, one indoors (Hearth 2) and one outdoors (Hearth 4), to present and discuss the results obtained from the interdisciplinary analyses. Our aim was to obtain a better understanding of the data recovered from the archaeological structures.

Materials and methods

Experimental combustions and fuels

This experimentation was carried out in three different periods between 2021 and 2022 (June 2021, November 2021 and May 2022) to compare the combustion performance in different weather and temperature conditions, and to evaluate how these affected other variables, particularly fuel performance.

The experiment involved the construction of six open combustion structures (hearths)—four in enclosed spaces and two in open-air—and one oven (Table 1 and Fig. 3).¹

The structures were used combining and alternating different fuel sources, including Aleppo pine wood and cones (*Pinus halepensis*), oak wood (both evergreen and deciduous (*Quercus* sp.)), barley straw and weeds (mainly *Hordeum vulgare*), dwarf palm leaves (*Chamaerops humilis*) and livestock dung (sheep and cow). Foddering and grazing regimes correspond to sheep grazers within pasture grounds, as well as to cattle grazing in the farm pasture fields and stabled cows foddered with straw and supplementary purchased feed.² The choice of fuels was based on archaeobotanical records acquired through the study of archaeological combustion structures in the study context, the north-eastern Iberian Peninsula (wood charcoal, Piqué et al. 2011; plant and faecal microfossils, Belarte et al. 2023).

We performed a total of 35 combustions. Table 2 summarises the fuel type and weight before and after the combustions, the duration of the combustions and the temperatures.

During some of the combustions, we carried out cooking experiments by boiling water in hand-made replicas of Iron Age pottery and adding different products (legumes and animal bones). The residues of these products were expected to be detected through chemical analyses, as will be addressed below.

When putting the structures to work, the temperatures reached and their progression in time were systematically measured using K-type thermocouple pyrometers³ that

¹ A seventh fireplace (Hearth 7) was later built on the same place as Hearth 2, removing the preparation layer and building a new earthen surface. For a more detailed information on fuel weights, see supplementary materials in <https://doi.org/10.34810/data581>

² See supplementary materials in <https://doi.org/10.34810/data581>

³ Two Data Logger Hibok 18 pyrometers and one TM-934S pyrometer.



Fig. 3 Experimental combustion structures, during construction and in use: **a** Hearth 1; **b** Hearth 2; **c** Hearth 3; **d** Hearth 4; **e** Hearth 5; **f** Hearth 6; **g** Oven

automatically record the temperatures at predetermined regular intervals. We also used infrared pyrometers⁴ to measure the flames and embers, as well as other elements, including the oven walls, ceramic pots, lids and contents, observing and recording the temperatures reached and their behaviour.

Experimental fire installations were sampled for interdisciplinary studies. Before they were first lit, samples of the earth layers were taken for different analyses (microfossil and FTIR), following the protocols explained in the “Analyses applied to experimental structures” section (Fig. 4). These served as controls for comparative purposes for the reference standards obtained in the current experimental study.

After each combustion, the fuel remains (both ashes and charcoal, in addition to partially burned plant and dung

remains), were collected for integrated macrobotanical and microfossil analyses (Fig. 5a–b) (see the “Anthracology” and “Phytoliths and calcitic microremains” sections). All the fuel remains resulting from combustion were weighed. After several combustions, blocks for micromorphology study were taken (Fig. 5d), and a systematic sampling was carried out for chemical analyses (Fig. 5c).

Analyses applied to experimental structures

Anthracology

All the fuel remains from each hearth were collected (after five combustions in Hearth 2 and seven in Hearth 4) (Table 2).

These remains were analysed in order to identify the genus and, if possible, species of woody plants (see Kabukcu and

⁴ PCE-889B.

Table 2 Summary of experimental combustions. Hearths 2 and 4 are analysed in this paper

Fire structure	Comb. no	Fuel	Fuel weight (g)	Combustion duration	Highest temperature on combustion surface/in the flames	Weight of ash + charcoal remains (ChR) (g)	Date
Hearth 1	1st	Wood, palm leaves, straw, grasses	12.481	8 h	598 °C/756 °C	294 + 1.532	16/06/2021
	2nd	Oak wood, pine cones	11.380	More than 7 h	684 °C/785 °C	No records	18/06/2021
Hearth 2	1st	Oak wood, branches, palm leaves, pine cones	6.923	More than 9 h	523 °C/805 °C	374 + ChR	15/06/2021
	2nd	Cow dung, pine branches, straw, oak wood	10.004	More than 9 h 30 min	379 °C/372 °C	305	16/06/2021
	3rd	Cow dung, pine branches, barley straw	2.550	5 h 15 min	330 °C/756 °C	762	10/11/2021
	4th	Oak wood, pine and oak branches, pine cones, straw	8.585	More than 18 h	717 °C/780 °C	317	11/11/2021
Hearth 3	5th	Cow dung, straw	1.030	6 h	477 °C/788 °C	534	12/11/2021
	1st	Oak wood, pine branches, pine cones, palm leaves	7.806	More than 8 h 30 min	450 °C/779 °C	374 + ChR	16/06/2021
Hearth 4	2nd	Sheep dung, pine branches, straw, palm leaves	3.845	More than 8 h 30 min	295 °C/706 °C	1.474	17/06/2021
	1st	Oak wood, pine branches, straw	10.505	6 h	572 °C/754 °C	360 + ChR	14/06/2021
	2nd	Oak wood, pine branches, pine cones, straw	5.984	1 h 15 min	754 °C/805 °C	140 + 970	15/06/2021
	3rd	Sheep dung, pine branches, straw	2.632	2 h 15 min	522 °C/660 °C	211 + ChR	16/06/2021
	4th	Oak wood, pine branches, pine cones, straw	12.285	More than 12 h	759 °C/817 °C	211	17/06/2021
	5th	Oak wood	No records	No records	No records	No records	17/06/2021
	6th	Cow dung, pine branches, pine cones, straw	1.780	5 h 30 min?	404 °C/656 °C	603	09/11/2021
	7th	Oak wood, branches, straw	2.175	More than 6 h 30 min	579 °C/745 °C	126 + 280	12/11/2021
	8th	Sheep dung, barley straw, small pine branches, pine cones, grass	2.449	More than 6 h	550 °C/670 °C	278 + 467	09/05/2022
9th	Oak wood, palm leaves, barley straw, wood chips	2.652	More than 10 h	607 °C/629 °C	147	11/05/22	

Table 2 (continued)

Fire structure	Comb. no	Fuel	Fuel weight (g)	Combustion duration	Highest temperature on combustion surface/in the flames	Weight of ash + charcoal remains (ChR) (g)	Date
Hearth 5	1st	Oak wood, palm leaves, grass	5.908	2 h 15 min	550 °C/686 °C	385 + ChR	14/06/2021
	2nd	Oak wood, pine cones, palm leaves, grass	7.403	2 h 40 min	759 °C/758 °C	575 + ChR	15/06/2021
	3rd	Cow dung, pine wood, pine cones, grass	2.160	5 h 45 min	458 °C / 725 °C	484 + ChR	16/06/2021
	4th	Oak wood, palm leaves, straw	2.476	More than 12 h	139 °C / 507 °C	720 (ash + oak fragm.)	09/11/2021
	5th	Sheep dung, wood chips, straw	2.052	More than 5 h	595 °C/755 °C	282	19/11/2021
	6th	Oak wood, palm leaves, barley straw	2.582	More than 8 h	773 °C/651 °C	48 + 363	09/05/2022
	7th	Sheep dung, barley, small pine branches, pine cones	1.649	8 h 30 min	548 °C/670 °C	671	11/05/2022
Hearth 6	1st	Embers, wood	211 + 2 wood fragm	More than 3 h	437 °C/828 °C	455 + ChR	17/06/2021
Hearth 7	1st	Sheep dung, barley, small pine branches	2.657	12 h	428 °C/716 °C	785	10/05/2022
Hearth 7	2nd	Oak wood, barley and weed, small pine branches, wood chips	2.590	9 h	571 °C/756 °C	120	12/05/2022
Oven	1st	Oak wood, branches, pine cones	7.788	7 h	887 °C/856 °C	No records	17/06/2021
	2nd	Oak wood, branches, pine cones	16.653	7 h 30 min	490 °C/85 °C	No records	18/06/2021
	3rd	Oak wood, pine cones, pine branches, straw	7.765	More than 12 h	761 °C/800 °C	660 + ChR	10/11/2021
	4th	Sheep dung, branches, pine cones, straw	4.479	8 h	326 °C/684 °C	664 + 2.857	11/11/2021
	5th	Oak wood, pine cones, pine small branches, straw	9.359	More than 13 h	807 °C/825 °C	636	10/05/2022
	6th	Oak wood, pine cones, pine small branches, straw, grass	2.732	9 h	544 °C/718 °C	252	12/05/2022

Chabal 2021). The methodology applied included several stages. Firstly, after each combustion, the sample was washed with water, using a > 1-mm sieve. Subsequently, the charcoal

fragments were sorted and identified, and each was individualised and stored with its numbering to facilitate future consultation.

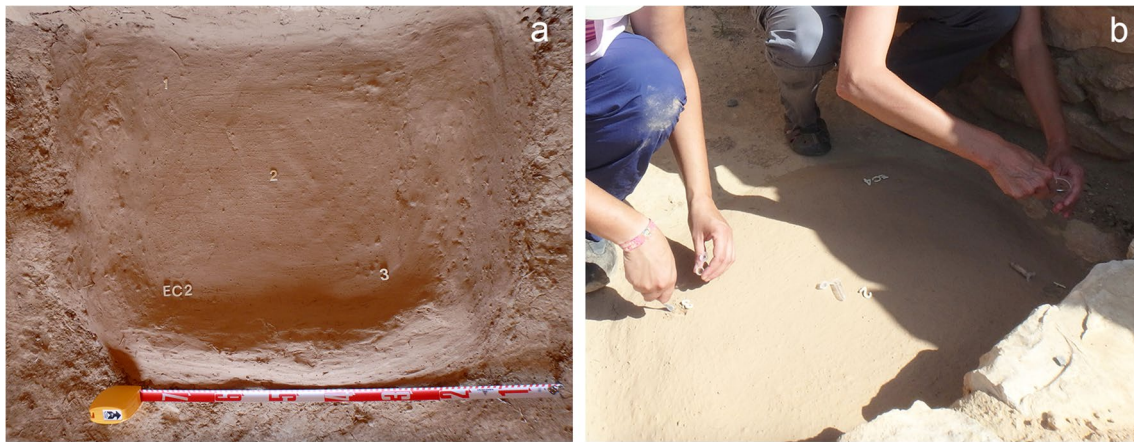


Fig. 4 a Location of samples in Hearth 2; b location of samples in Hearth 4. Both taken for microfossil and FTIR analyses



Fig. 5 a Sampling Hearth 2 after combustion for microfossil and FTIR analyses; b sampling fuel remains from Hearth 4 for macrobotanical analysis; c sampling of Hearth 2 for spot tests; d taking two blocks of sediment from Hearth 4 for micromorphology

A minimum analysis of 25 charcoal fragments per combustion was established, which is considered sufficient to presuppose the occurrence of most or all species per stratigraphic unit (Thompson 1984; Castelleti and Zimmerman

1985; Pearsall 1988; Piqué 1996; Hastorf et al. 2005; Vila Moreiras 2018). Several specific charcoal fragments could not be taxonomically determined and were therefore included in the “indeterminable” category. Alterations to

charred wood are often the cause of a high deformation of the anatomy and are vitrified (Courty et al. 2020; McParland et al. 2010), which prevents taxonomic determination.

It is worth mentioning that several elements not intentionally included during the experiments, such as olive pits, were identified through sorting. To verify whether they came from dung used as fuel, a sample of unburned dung was washed with water, also using a > 1-mm sieve, and the result was positive.

Phytoliths and calcitic microremains

Ten samples from Hearth 2 (combustions 1 and 2, codified as H2-C1 and H2-C2) and Hearth 4 (combustions 1 and 3, H4-C1 and H4-C3) were selected for integrated phytolith and calcitic microfossil analysis. Additionally, two unburned sediment samples collected from the hearth surfaces before the combustion experiments were included for comparative purposes as controls (codified as C0 samples).

Phytolith analyses followed the methods of Katz et al. (2010). A weighed aliquot of around 40 mg of dried sediment was treated with 50 µl of a volume solution of 6N HCl. Phytoliths were concentrated with 450 µl 2.4 g/ml of sodium polytungstate solution [Na₆(H₂W₁₂O₄₀)]. Aliquots of 50 µl of material were mounted on microscope slides. A minimum of 200 phytoliths with recognisable morphologies was examined at ×200 and ×400 using a Leica DMEP optical microscope. Phytoliths displaying pitted surfaces that were unidentifiable due to weathering and alteration by dissolution were listed as weathered morphotypes, whereas “melted” refers to those phytoliths that were deformed and displayed alterations due to heating. The latter ranged from slightly bubbled surfaces to completely opaque morphologies that it was no longer possible to assign to specific morphotypes. Both partially weathered and melted phytoliths that were still recognisable were then attributed to specific morphotypes (Portillo et al. 2021a). Morphological identification was based on modern Mediterranean region plant reference collections (Albert and Weiner 2001; Albert et al. 2008, 2016; Portillo et al. 2014; Tsartsidou et al. 2007) and standard literature (Brown 1984; Mulholland and Rapp 1992; Piperno 2006; Rosen 1992; Twiss 1992; Twiss et al. 1969). Where appropriate, the terms used to describe phytolith morphologies followed the standards of the International Code for Phytolith Nomenclature, ICPN 2.0 (Neumann et al. 2019).

The methods used for calcitic microfossil analyses, including both dung spherulites and wood ash pseudomorphs, were similar to those developed by Canti (1999). Approximately 1 mg of dried sediment was mounted on a microscope slide with Entellan New from Merck. Slides were examined at ×200 and ×400 magnifications under an optical microscope with crossed polarised light (XPL)

for spherulites, whereas pseudomorphs were examined in plane polarised light (PPL). Similarly to phytoliths, both calcitic microfossil numbers were related to the initial sample weight. Samples were also compared to dung spherulites and wood ash pseudomorphs from modern reference collections and ethnoarchaeological datasets from Mediterranean areas that followed a similar quantitative approach (Tsartsidou et al. 2008; Portillo et al. 2012, 2014, 2017, 2020, 2021a, b, c; Portillo and Matthews 2020; Portillo and García-Suárez 2021).

FTIR (Fourier transform infrared spectroscopy)

For FTIR analyses, three specific points were selected in each combustion structure: two in opposite corners and one in the middle (Figs. 4 and 5a). Samples from these three locations were collected repeatedly. For Hearth 2, three samplings were carried out: the first before the combustions (numbered using the acronym EXP) and later after combustions 2 and 5 (C2 and C5). In the case of Hearth 4, the same previous sampling (EXP) was carried out and subsequently after combustions 4 and 9 (C4 and C9). For both hearths, only samples corresponding to the central part of the hearth (No. 2) were analysed to ensure that they had been subjected to combustion processes.

FTIR is a technique based on how electromagnetic radiation from the middle infrared range (number of waves between 4000 and 400 cm⁻¹) interacts with materials. Berna et al. (2007) used it successfully with archaeological materials, revealing the presence or absence of thermal alterations (see also Saorin 2018).

For each sample, a few milligrams of sediment (less than 0.5 g) were homogenised, powdered with an agate mortar, mixed with a few milligrams of KBr and pressed into a 7 mm pellet using a manual hydraulic press (Specac). Infrared spectra were collected at 32 scans and a resolution of 4 cm⁻¹ with an iS5 from Thermo Fisher Scientific. The software used for the interpretation of the spectra was OMNIC and the mineral database used is available at the Kimmel Centre for Archaeological Science (Weizmann Institute of Science). The thermal alteration of the clays was identified following Berna et al. (2007). When clay is exposed to high temperatures, it is altered and becomes more disordered. The infrared spectra of clays exposed to high temperatures change depending on the type of clay, the temperature and the duration of exposure. FTIR identification of clays is only possible if the clay sample being analysed contains a single clay mineral. Several different clay minerals are often mixed together, and, when this is the case, the presence of clay can be identified by FTIR, although it is very difficult to identify the different types of clays in the sample (Weiner 2010).

Micromorphology

We took undisturbed and oriented samples from each combustion structure after they had cooled down (two from Hearth 4 and one from Hearth 2) (Fig. 5d).

The soils and sediments were studied in thin sections (on a micro-scale) in order to provide information on their in situ composition, formation processes and possible use (Mateu et al. 2019). The thin sections were prepared in the Institute of Geology laboratory at the University of Barcelona following standard protocols. The samples were consolidated with polyester resin by total impregnation through capillarity, then sliced into 30- μ m-thick sections and placed on slides (Loaiza and Poch 2015: 19–20; Verrecchia and Trombino 2021). The thin sections were observed at the Archaeometric Studies Unit of the Catalan Institute of Classical Archaeology (UEA—ICAC) under a petrographic optical microscope ($\times 20$ to $\times 200$ magnifications), in Plane Polarised Light (PPL), between crossed polarisers (XPL) or oblique incident light (OIL). The descriptions and interpretations were based on criteria from Bullock et al. (1985), Courty et al. (1989) and Stoops (2003), and the features used to infer the technological characteristics were those proposed by Friesem et al. (2017), Cammas (2018) and Mateu and Daneels (2020).

Spot tests

Twenty samples were recovered from the Hearth 2 surface and 24 from the Hearth 4 surface for chemical analysis aimed at identifying the chemical residues by applying spot tests (see below) (Barba 2007, Pecci 2021).

On site, almost 5 g of sediment was taken after having mechanically cleaned the surface. Before the experiment, four samples were recovered on the two hearths. After the experiment, samples were recovered on a 20 \times 20 cm grid on the two hearths analysed (Figs. 4 and 5). Sampling on Hearth 2 was carried out after five combustion processes. The fuel used was of plant origin (in every case oak and, depending on the combustion, palm leaves, grass and pine), as well as dung. During three of the combustions (nos. 1, 2 and 4), pork bones were boiled for 6 h on the hearth; during combustion 3, a cooking pot full of water was placed next to the fire. Although it did not contain any food, it had cracked and the water could have facilitated the absorption of residues. Hearth 4 was sampled after five combustion processes. The combustion material was of plant origin (oak and pine wood and grass) and in only one of the combustions, dung. A cooking pot with lentils in water was placed on the hearth during the first combustion.

In the laboratory, the sediment was homogenised, powdered and subsampled again for the different tests. Analyses were carried out using spot tests published by Barba et al. (1991) and more recently by Barba (2007) and Pecci et al.

(2017) in order to identify the presence of phosphates, fatty acids, protein residues and carbohydrates (Barba 2007). In particular, the phosphate spot test was developed by soil scientists to detect phosphorous compounds by making the sample react with a mild acid solution. The extracted phosphates react with molybdates to produce a yellow colour indicating phosphomolybdates, which are difficult to see. For a more visible reaction, ascorbic acid is added to produce blue complex compounds that can easily be seen on white filter paper. Finally, a solution of sodium citrate is added to fix the reaction at a given time. The intensity of the blue is directly related to the amount of phosphates present in the original sample. In practical terms, the test consists of placing 0.05 g of powdered sample in the middle of a Whatman 42 ashless filter, placing two drops of Solution A (a 5 g solution of ammonium molybdate dissolved in 35 ml of hydrochloric acid and 65 ml of distilled water), waiting 30 s, and adding two drops of Solution B (obtained by dissolving 0.5 g of ascorbic acid in 100 ml of distilled water). Two minutes after the reaction begins, several drops of Solution C (one part sodium citrate to two parts distilled water) are added, covering the whole sample, to fix the reaction. The scale used is 0–6. The presence of phosphates in the samples indicates organic material that may be related to food preparation and consumption, or even ritual activities. To determine whether protein residues are present, the amino groups contained within the proteins are decomposed by a strong alkaline reaction with calcium oxide in a hot water solution to produce ammonia (NH_3). pH indicator paper changes colour in contact with NH_3 vapours. The darker the colour, the more “protein residues” are present in the sample. In practice, the test consists of placing 0.1 g of powdered sample in a test tube, adding 0.1 g of calcium oxide and 1 ml of distilled water and placing two wet pH strips on the rim of the test tube (after cleaning it). The test tube is heated for 1 min until the pH stripes change colour and the pH value is identified. The scale used is that of the pH values observed. Values of 7 and 7.5 indicate no presence of protein residues. Protein residues are present in materials such as blood or meat and are usually found in kitchens, butchering places and ritual areas.

Carbohydrate residues produce coloured compounds by reacting with phenols in an acidic medium. The carbohydrates are hydrolysed to furfural or hydroxymethylfurfural by the action of concentrated sulphuric acid. Subsequently, the coloured compounds are generated in a condensation reaction with resorcinol. The amount of carbohydrates contained in the sample is related to the intensity of the reddish colour generated and is assigned a value ranging from 0 to 4. For the test, 0.2 g of the sample is placed in a test tube and 2 ml of Reagent A is added (0.7 g of resorcinol in 500 ml of distilled water). Two millilitres of concentrated sulphuric acid is poured into the test tube. After 1 min, the

reaction stabilises and the colour can be compared to the colour chart.

Fatty acids indicate the presence of animal or plant origin oils, fats and resins. They reflect activities such as cooking and food storage, incense/copal burning and butchering. To test for fatty acids, lipids are extracted using a non-polar solvent such as chloroform and heating. The reaction with ammonium hydroxide produces a solution of ammonium soaps through the saponification of fatty acids. Bubbles of oxygen, when in contact with hydrogen peroxide, produce a dense foam that is directly related to the presence of fatty acids in the original sample. In practical terms, the test consists of placing 0.1 g of powdered sample in a test tube, adding 2 ml of chloroform, heating for 1 min and leaving to stand (the chloroform is reduced to half). The supernatant is placed on a watch glass, two drops of ammonia solution are added and, after 1 min, two drops of hydrogen peroxide (30%) and the formation of the foam is observed. The scale used is 0–3.

Spot tests are used in our research to identify the presence of residues. They do not provide information on the quantity or exact origin of the compounds (i.e. whether they are fatty acids derived from animal products or plant oil). However, they have been useful in identifying chemical

enrichment patterns and activity areas and interpreting the use of spaces (Barba 2007; Barba et al. 1996, 201; Middleton et al. 2010; Pecci 2009, 2013; Pecci et al. 2013, 2017; Pecci and D'Andria 2014).

The results from analyses of the samples recovered on a grid were inserted in a GIS database and interpolated using the IDW method in order to produce distribution maps for each test. The results are discussed on the basis of the information known about the building techniques, fuel and use of hearths.

Results

Experimental combustions and fuels

Regardless of the building technique and materials used, the fuel chosen and the location (indoors/outdoors), all the experimental combustion structures turned out to be functionally efficient, maintaining the necessary heat to boil water (and any food in it) in a ceramic pot for at least 6 h (Figs. 6 and 7).

More fuel had to be added in some cases to heat the water to over 100 °C. Once it was boiling, the temperature

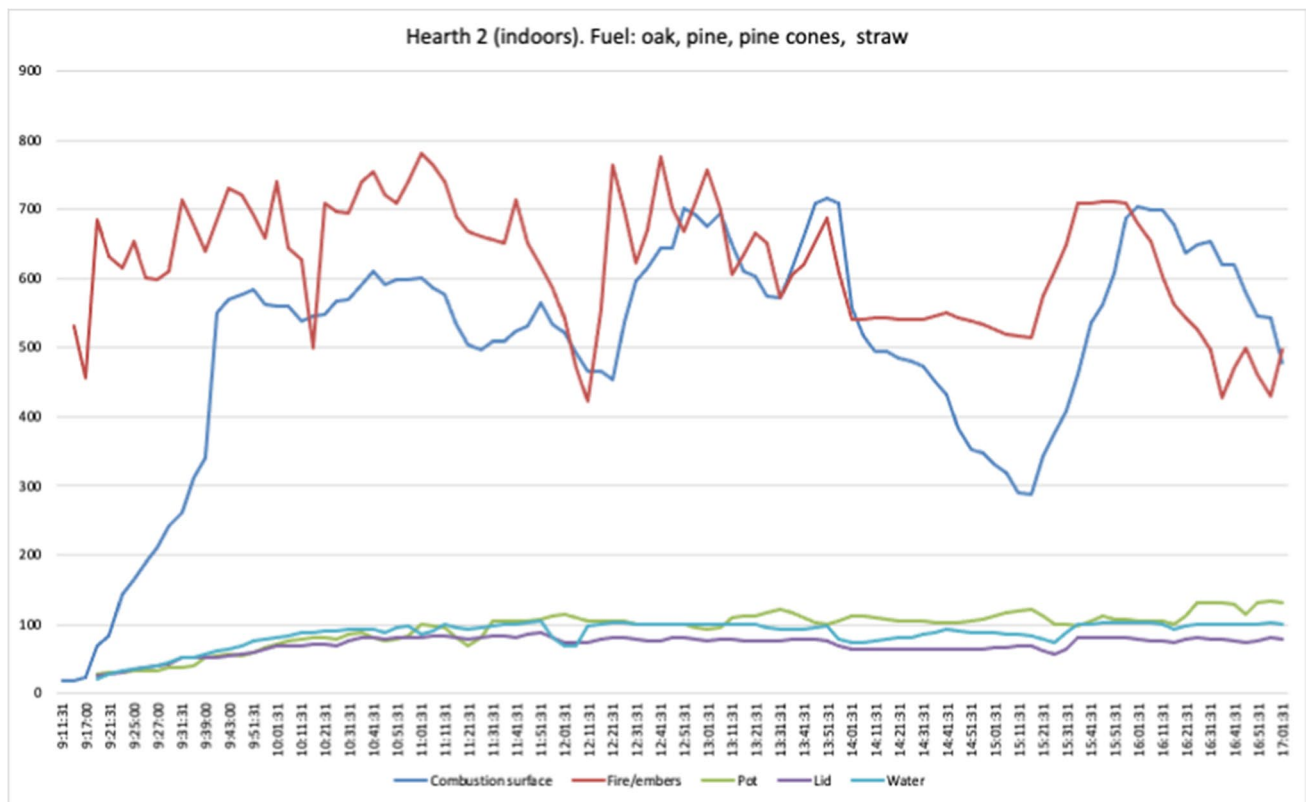


Fig. 6 Duration and temperature records of the fourth combustion of Hearth 2 in November 2021, using wood as the main fuel and boiling pork bones for more than 6 h. Colour code: dark blue, combustion surface; red, fire/embers; green, pot; violet, lid; light blue, water

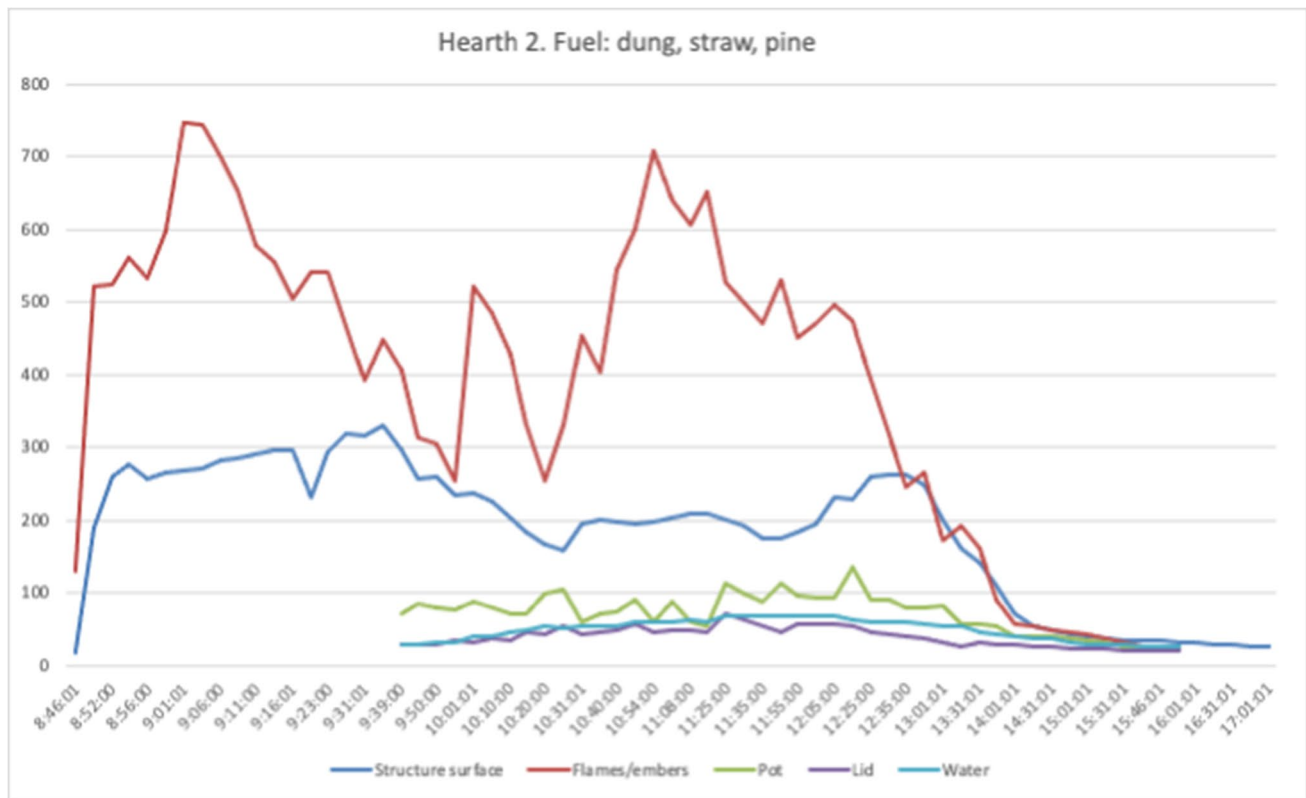


Fig. 7 Duration and temperature records from the third combustion of Hearth 2 in November 2021, using dung as the main fuel and boiling pork bones for more than 6 h. Colour code: dark blue, combustion surface; red, fire/embers; green, pot; violet, lid; light blue, water

remained stable for hours, although fuel additions were needed on occasion. All these additions were systematically recorded in terms of the time taken and the weight of the fuel, and we were able to observe that the needs varied in each combustion (see [supplementary materials](#)).

Depending on the hearth, the total amount of fuel required to boil water varied from 7 to 12 kg using combinations of different fuel types.

The temperatures of the combustion surfaces were higher when wood was used as fuel (Belarte et al. 2022). Some variation in the combustion properties could have been introduced due to the varying percentages of moisture in the fuels used in our experiments. During the experimental studies in November 2021, we were able to compare the performance of the same hearth (an indoor structure) using wood (Fig. 6) and dung (Fig. 7). Figure 6 shows a complete combustion in which we boiled pork bones for 6 h using wood as fuel. Figure 7, which shows the performance of the same hearth using dung, reflects a very different situation, with a less stable fire. Most of the time, the fire surface (dark blue line) was below 300 °C (much lower than the 600 °C or even 700 °C in Fig. 6), and we did not succeed in keeping water boiling for long enough to cook bones. Although the dung

was apparently dry, it absorbs humidity, and with a high RH, it was difficult to reach the same temperatures as with wood. This issue has been noted by other researchers who have experimented with dung as a fuel in humid climates (Budka et al. 2019).

Thermo-alteration of the firing surfaces was visible after the very first combustion, with the earthen surfaces changing colour from brown to black and darkening tones in irregular spots. For example, in Hearth 4, we see a combination of colours depending on the position in the combustion surface, after nine combustions: pale brown (10YR 6/3 in the Munsell colour system), dark grey (10YR 4/1) or grey (10YR 5/1). In addition, the upper opening of the oven blackened after the first use, probably due to the proximity of the flames, while the colour of the oven walls was not transformed in the same way. In the case of Hearth 2, built indoors by a wall under a small window and surrounded by a vegetal-tempered earthen border, an ascending black spot formed on the wall during the second combustion, in the direction where most of the smoke went out through the window above, although this was probably due to the close contact of a burning log. The area of the wall left between the spot and the window darkened slowly but progressively



Fig. 8 Darkening of the wall next to Hearth 2 during its second combustion

with each subsequent combustion (Fig. 8). Our observations regarding the colour transformation of the surfaces in contact with fire can help in interpreting archaeological remains

and structures and avoid assuming that heavily blackened surfaces must be the result of successive combustions or structures used over a long period of time.

Regarding the fuels used, straw was the first material to be combusted, rapidly within the first minute, followed by palm leaves and branches, something in which size would play an important part. Therefore, it is less efficient to place these smaller materials at the top of the fuel pile prepared for burning. Logs are logically the last to burn, on many occasions being burnt only partially, which allows them to be reused for the same or other purposes.

Anthracology

Counting both hearths, a total of 263 fragments was analysed, 100 from Hearth 2 (collected after five combustions) and 163 from Hearth 4 (collected after seven combustions) (Fig. 9). The most popular species in the first case was evergreen *Quercus* with 41%, followed by deciduous *Quercus* (oak) (28%) and *Pinus halepensis* (22%), the rest accounting for less than 7%. Those in Hearth 4 were deciduous *Quercus* (48%) and evergreen *Quercus* (33%), while the rest accounted for less than 7% of each species.

A difference was observed between the two structures. In Hearth 4, the number of taxa was much higher (seven), whereas in Hearth 2, there were only four. This could be related to the use of dung as a fuel, as some of the species determined in the anthracological study (e.g. *Arbutus unedo* and *Rhamnus/Phillyrea*) were not used intentionally in our experiment. One possibility is that these wood species came from the dung used as fuel. We verified the presence of some of the unexpected fuels by washing a sample of fresh dung

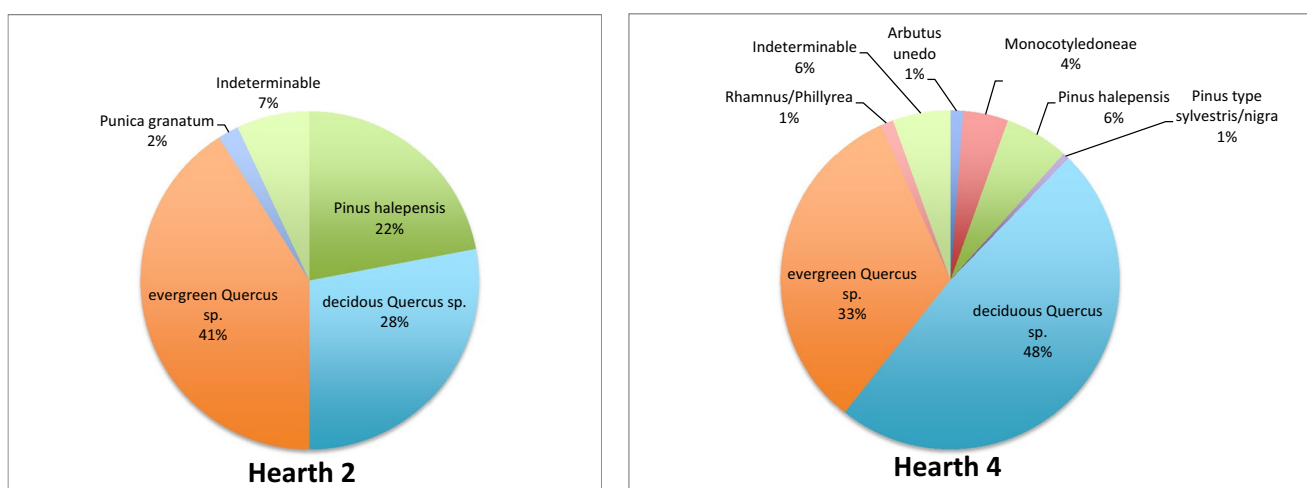


Fig. 9 Wood species determined in experimental Hearths 2 and 4

Table 3 Description of samples and main quantitative phytolith, ash pseudomorph and dung spherulite results obtained from the Hearth 2 (H2) and Hearth 4 (H4) experimental samples

Hearth-combustion-sample <i>n</i>	N. phytoliths 1 g of sediment (million)	Multi-celled phytoliths (%)	Phytoliths melting (%)	Phytoliths weathering (%)	Ratio grass inflorescence/leaves-stems	N. ash pseudo-morphs 1 g of sediment (m)	N. dung spherulite 1 g of sediment (m)
H2-C0-1	0.22	1.5	0	7.6	0.31	0.19	0
H2-C1-1	1.03	5.9	0	0.5	0.19	3.60	0
H2-C1-2	0.32	18	0	6	0.06	7.66	0
H2-C1-4	0.24	6.3	0	3.2	0.54	0.02	0
H2-C2-1	0.92	2.6	1.7	0	0.66	8.43	0.14
H2-C2-2	0.30	11.6	0.3	5.1	1.13	4.04	0
H4-C0-1	0.29	0	0	6.6	0.63	2.70	0
H4-C1-1	0.19	16.4	0	3.6	0.29	7.46	0
H4-C1-2	0.21	3.3	1.6	6.6	0.32	7.14	0
H4-C1-4	0.17	2.1	0	0	0.73	0.04	0
H4-C3-1	0.26	57.4	1.5	7.4	2.19	0.92	0.02
H4-C3-2	0.34	44.3	0	1.1	3.93	0.11	0.22

and, as mentioned above, *Arbutus unedo* and olive pits were identified.

Phytoliths and calcitic microremains

Phytolith amounts range between 0.17 and 1.03 million per 1 g of sediment (Table 3). The richest samples by far were noted in Hearth 2 (Samples H2-C1-1 and H2-C2-1, *ca.* 1 m phytoliths per 1 g/sediment). The low proportions of weathered phytoliths (below 8%), together with the presence of multicellular phytoliths (anatomically connected) in most of the samples (up to 57% in Sample H4-C3-1, belonging to fuel dominated by ovicaprine dung), point towards generally good preservation conditions of the phytolith record. In addition to weathering, phytoliths from combustion assemblages were partially affected by melting due to heating, although many of these were recognisable and morphologically identified (Fig. 11a). Melted phytoliths were completely absent in the unburned assemblages collected from the hearth surfaces prior to the experimental combustion work.

Grasses predominated in most of the samples, with around 60–94% of all the counted phytoliths and an average of *ca.* 80%, whereas wood, bark and dicotyledonous leaf morphotypes (e.g. spheroids, tracheids and blocky morphologies) represented an average close to 15% of the assemblages (Fig. 10A). The lowest dicotyledonous proportions were noted in samples belonging to cow dung-dominated fuels (Samples H2-C2-1 and H2-C2-2, Fig. 10A). Diagnostic spheroid echinate phytoliths produced by the leaves of the Arecaceae family (dwarf palm, *Chamaerops humilis*, Fig. 11b), were only noted in one of the samples, although to a much lower extent (H2-C1-1, *ca.* 1% of all the counted morphotypes, Fig. 10a). Grass short cells were by far the

most dominant in both hearths, regardless of the type of fuel, with an average of around 44% of all the counted grass morphotypes (Fig. 10B). Short cell morphologies comprised mostly rondels, although bilobates and polylobates were also noted (Fig. 11c). Grass inflorescences were represented mainly by decorated elongate dentate (echinate) and dendritics, in addition to elongate baculates and epidermal cells including papillates (Fig. 11a). Epidermal appendages produced by grass leaves and culms, including acute bulbosus (trichomes), bulliforms and stomata, were also common in all the samples in variable amounts (Figs. 10b and 11c). Lastly, the dung-dominated samples showed the largest ratios in terms of inflorescences/leaves and stems of grasses, pointing towards an important input of the floral part of grasses in the fuel related to livestock diet (Samples H2-C2-2, H4-C3-1 and H4-C3-2, ratios ranging from 1.1 to 3.9, Table 3).

In addition, calcitic wood ash pseudomorphs, calcite pseudomorphs after the heating (burning) of calcium oxalate crystals to at least 450 °C, primarily originating from wood and dicotyledonous leaves, were also noted in variable amounts (between 0.04 and 8.4 million per gram of sediment, Table 3). Most of these calcitic microfossils displayed characteristic regular-shaped rhomboid morphologies directly comparable to Aleppo pine (*Pinus halepensis*, Fig. 11d) and oak (*Quercus* sp., Fig. 11e), according to our modern reference collection (Portillo et al. 2017, 2021c). Certain morphologies noted in the fuel assemblages dominated by ovicaprine dung (Samples H4-C3-1 and H4-C3-2) compared favourably to other taxa, such as the olive tree (*Olea europaea*) (see Fig. 8c in Portillo et al. 2017). This is consistent with the above-reported macrobotanical records (see the “Anthracology” section). Furthermore, calcitic dung

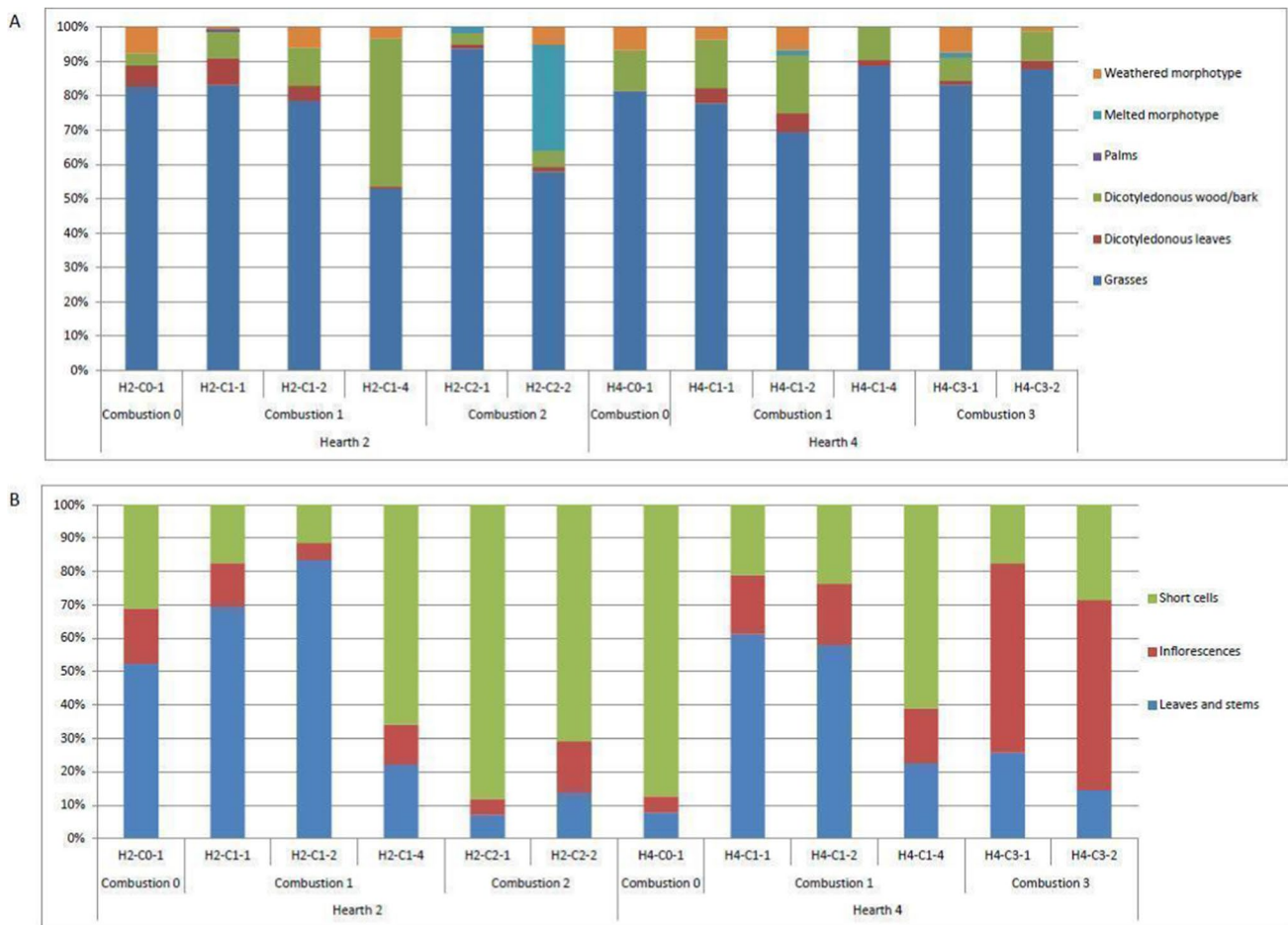


Fig. 10 **A** Relative abundance of phytoliths from grasses, dicotyledonous leaves, dicotyledonous wood/bark, palms, melted and weathered morphotypes. **B** Anatomical origin of the grass phytoliths identified in the samples

spherulites were also noted among these fuel assemblages dominated by sheep dung, as well as in one of the fuel samples based on cow dung (H2-C2-1, although in relatively lower amounts, ranging from between 0.02 and 0.2 m/1 g of sediment, Table 3, Fig. 11f).

Micromorphology

The raw material used to build the combustion surfaces was a sandy silt soil known as “terra rossa”. We did not add any vegetal temper. In Hearth 2, the combustion surface was built on top of a preparation layer of pottery sherds. In contrast, Hearth 4 was built directly on the ground without a preparation layer (Table 1 and Fig. 3). For this study, we were interested in seeing what changes were documented in this material once the combustion surfaces had been built and used and how they were affected by different combustions and temperatures.

We took two samples from Hearth 4, one from inside the hearth and the other from outside. The thin section of the

external sample (Fig. 12b) shows the difference between the combustion surface layer—with a brown colour—and the ground. The one from the interior (Fig. 12c), in contrast, no longer showed much difference between the combustion surface and the floor. The combustion affected the microstructure, and its colour changed to reddish and dark brown. The thin section of the combustion surface from Hearth 2 had a homogeneous dark brown colour (Fig. 12a).

In some cases, on the combustion surfaces, we find what appear to be layers and fissures. It seems that hearths with more combustion episodes have more fissures, as in the example of Hearth 2, which had fewer burns and did not reach a temperature as high as the inner part of Hearth 4 (Fig. 12a and c).

No ash was documented in these thin sections, either in the upper part or in the fissures. There were only a few charcoal fragments on the surface in Hearth 4 (Fig. 13a–b). We detected a few charred vegetal remains that in a micro-morphological observation could be mistaken for vegetal temper (Mateu et al. 2022); however, in this case, they must

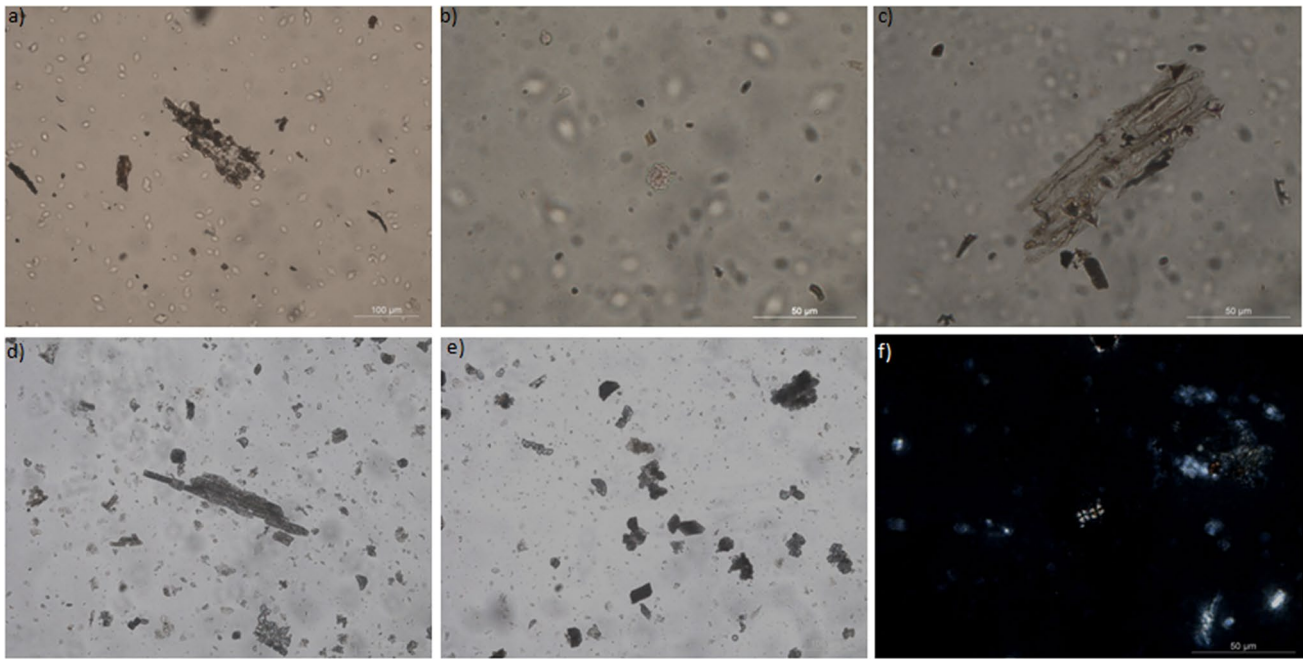


Fig. 11 Photomicrographs of phytoliths and calcitic microfossils identified in experimental samples (at 200× or 630×). **a** Partially melted multicellular dendritic phytoliths (Sample H2-C1-2); **b** spheroid echinate (H2-C1-2); **c** multicellular elongate phytoliths with

short cell rondels and stomata (H4-C1-1); **d** calcitic wood ash pseudomorphs comparing favourably to *Pinus* sp. (H4-C1-2); **e** calcitic wood ash pseudomorphs resembling *Quercus* sp. (H4-C1-2); **f** dung spherulites (H4-C3-2)

already have been included in the sediment used to build the structures (Fig. 13c–d).

FTIR (Fourier transform infrared spectroscopy)

In Hearth 2, the main composition was calcite, clay and quartz in different proportions (Table 4). The experimental sample before combustion had a higher proportion of clay (peaks 3431 and 1030) that decreased with the successive combustions (Fig. 14 above).

Hearth 4 was mainly composed of calcite, clay and quartz (Table 4). In the sample taken before combustion, H4-EXP-2, it can be seen that it did not undergo thermal alteration. In contrast, in Sample H4-C4-2 (after four combustions), the clay peaks disappeared, both on the left side of the graph, where Peak 3434 disappears completely, and on the right side where Peak 1030 had decreased (Fig. 14 below). Sample H2-C9-2 (after nine combustions) showed thermal alteration, although this was not as evident as in the sample taken after four combustions. This is probably due to repairs carried out between combustions.

Spot tests

In both hearths, spot tests carried out on a few samples taken before their use allowed chemical residues (mainly phosphates) and carbohydrates to be identified. This is

probably related to the specific raw materials used to build the hearths. Before the hearth was used, no great differences can be observed among the samples of the same structure. After use, it is possible to observe chemical concentrations in both hearths. This is evident mainly in Hearth 4, where no cooking processes involving products of animal origin were undertaken (a possible source of fatty acids and protein residues in the floor, Pecci et al. 2017). The data obtained suggest that what we are probably “seeing” in the chemical residue distribution maps is the result of the use of specific materials to make the fire that enriched the surfaces (dung and wood, such as pine, that may have been rich in resin). In the future, we will plan the experiment sampling on a grid of the whole hearth before the burning and before cooking the animal bones in pots. As such, we will be able to confirm this hypothesis. At present, we can only suggest that both the hearth building and its use enrich the surfaces below them. In particular, the high phosphate values can be related both to the use of organic material for the preparation of the hearth, the fuel (especially dung) and the use of the hearth itself to cook food. The protein residue test reacts to nitrogen-rich compounds; therefore, concentrations can be related either to cooking animal products (we cooked pork) or the use of dung as fuel. Previous analyses carried out during an ethnoarchaeological project on ovens used for pottery production in Mexico, in which dung was used as fuel, revealed the same pattern (Pecci et al. 2011). The carbohydrate test also reacts

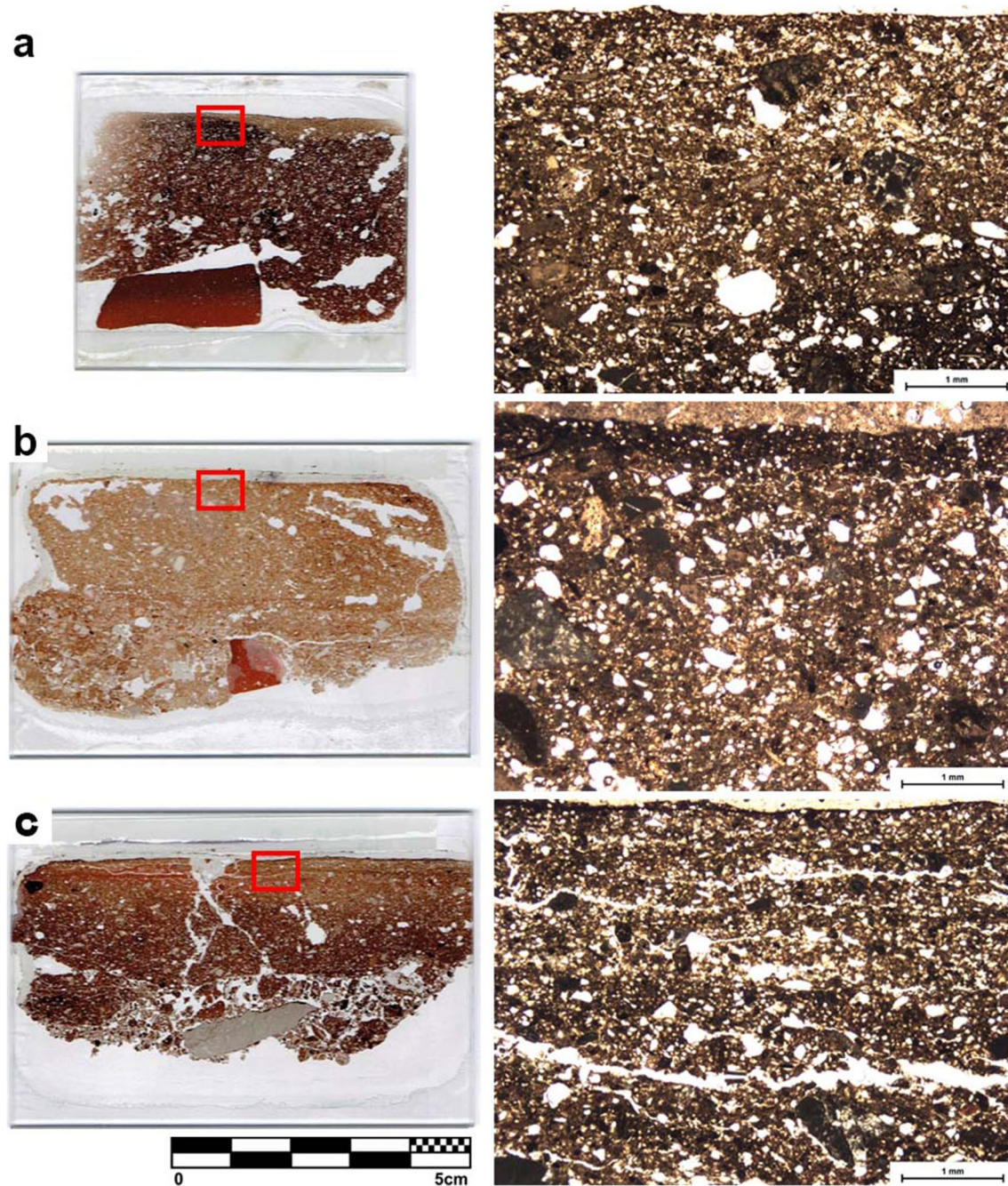


Fig. 12 Micromorphology. Thin sections and microphotographs of the parts of them are marked with the red box (all PPL). **a** Hearth 2; **b** Hearth 4, less combusted; **c** Hearth 4, more combusted

to the presence of straw. Enrichment is therefore possibly linked to the use of plant material both for the building of the hearths and as a fuel. Fatty acids can be related on one side to the presence of resin from the pine used as fuel and the cooking of pork bones. In fact, the spot test is not able to differentiate between animal and plant-origin fats and reacts both to animal fats and resin/copal (Barba et al. 1996; Pecci 2003). The pot was moved around in different positions during the cooking processes (Fig. 15). Another observation is

that after the hearths were used, the chemical concentrations were located mainly towards their exterior, possibly because the intense heat in the middle burned the organic material. Although we are aware that a more intense sampling (on a grid) should have been carried out before starting the burning and that another should have been undertaken before beginning to cook the food, we believe that the data obtained are relevant for future research. This is because the information obtained indicates that, at an archaeological level, the use of

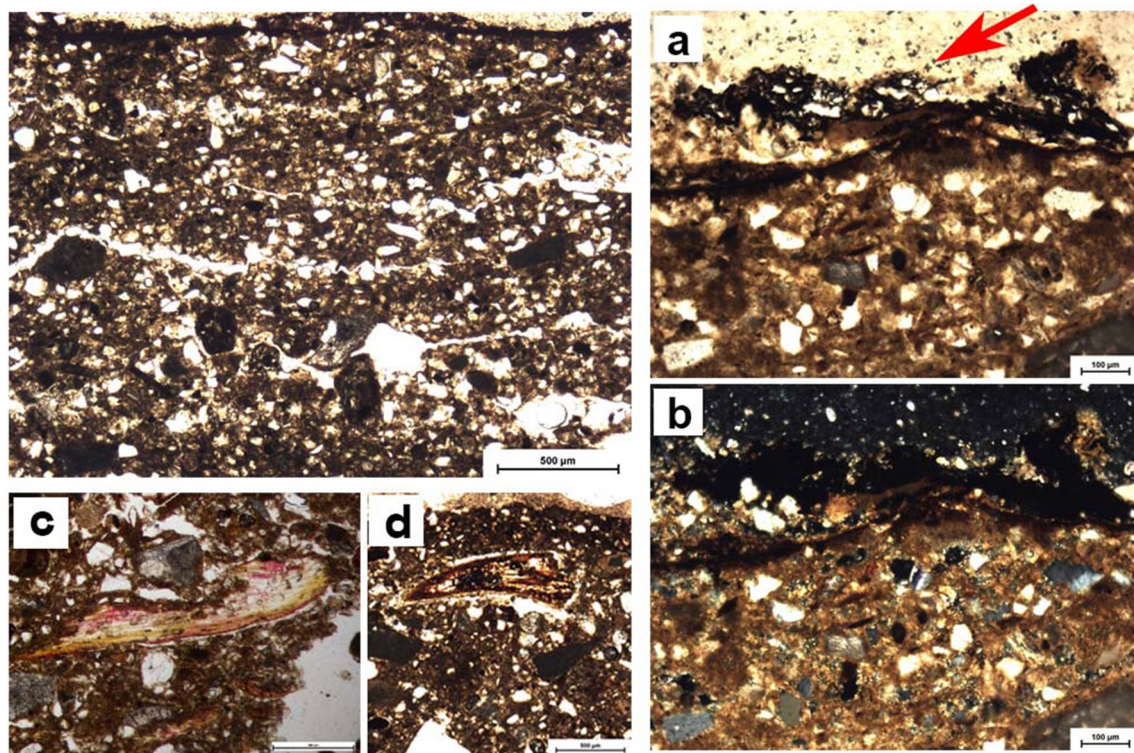


Fig. 13 Micromorphology. Hearth 4 microphotograph (PPL) with details of vegetal components; **a** and **b** small charcoal fragments on top of the hearth (PPL and XPL); **c** and **d** vegetal components accidentally included in the mass (PPL)

Table 4 FTIR results. Minerals are arranged according to their peak height from highest to lowest in the spectrum. Abbreviations: Ca, calcite; Qz, quartz; Cl, clay; (b), thermally altered clay

Combustion structure	No. combustion	Sample	FTIR*
Hearth 2	EXP	2	Cl, Ca, Qz
Hearth 2	C2	2	Ca, Cl (b), Qz
Hearth 2	C5	2	Ca, Cl (b), Qz
Hearth 4	EXP	2	Ca, Cl, Qz
Hearth 4	C4	2	Ca, Cl (b), Qz
Hearth 4	C9	2	Ca, Cl (b), Qz

the same area (the surface occupied by the hearth and its surroundings) for different activities (building the hearth, several fuel-burning processes and cooking) can make it difficult to understand the direct relationship between the chemical enrichment of the surface and one of these specific activities. In any case, the analyses make it evident that the hearth building and the use of specific fuels (mainly dung and possibly pine wood) influence the presence of chemical residues on the floor surface, and this has to be taken into account when interpreting archaeological hearths, in particular in contexts in which it is likely that dung was used as a fuel and/or building material. Therefore, it is important to carry out multi-proxy analyses to better understand the origin of

residues. As for the relationship between the presence of fatty acids and pine resin/tar, the analysis of a sample of the burnt traces on the wall on top of hearth 2 (Fig. 8) displayed a value of 1 with spot tests, and the analysis of the total lipid extract carried out by gas chromatography coupled with mass spectrometry following Correa-Ascencio and Evershed (2014) allowed to identify the presence of dehydroabietic acid, considered marker of Pinaceae resin, and retene, considered the marker of the resin heating (Colombini et al. 2005).

Discussion

Various observations of interest could be made during these experimental burnings, with implications for an improved study and understanding of the archaeological record, as well as of past societies and their domestic activities.

Several factors can be considered in the spatial arrangement of combustion structures (Fig. 16): activity distribution, group organisation, space availability, reuse of previous structures, and safety and comfort involving the structures, also taking into account the smoke generated. In this respect, in indoor spaces, openings would have been a key factor to consider to facilitate air currents and smoke evacuation. Combustions in Hearth 2 produced smoke that rose up to the ceiling and either escaped through the window or

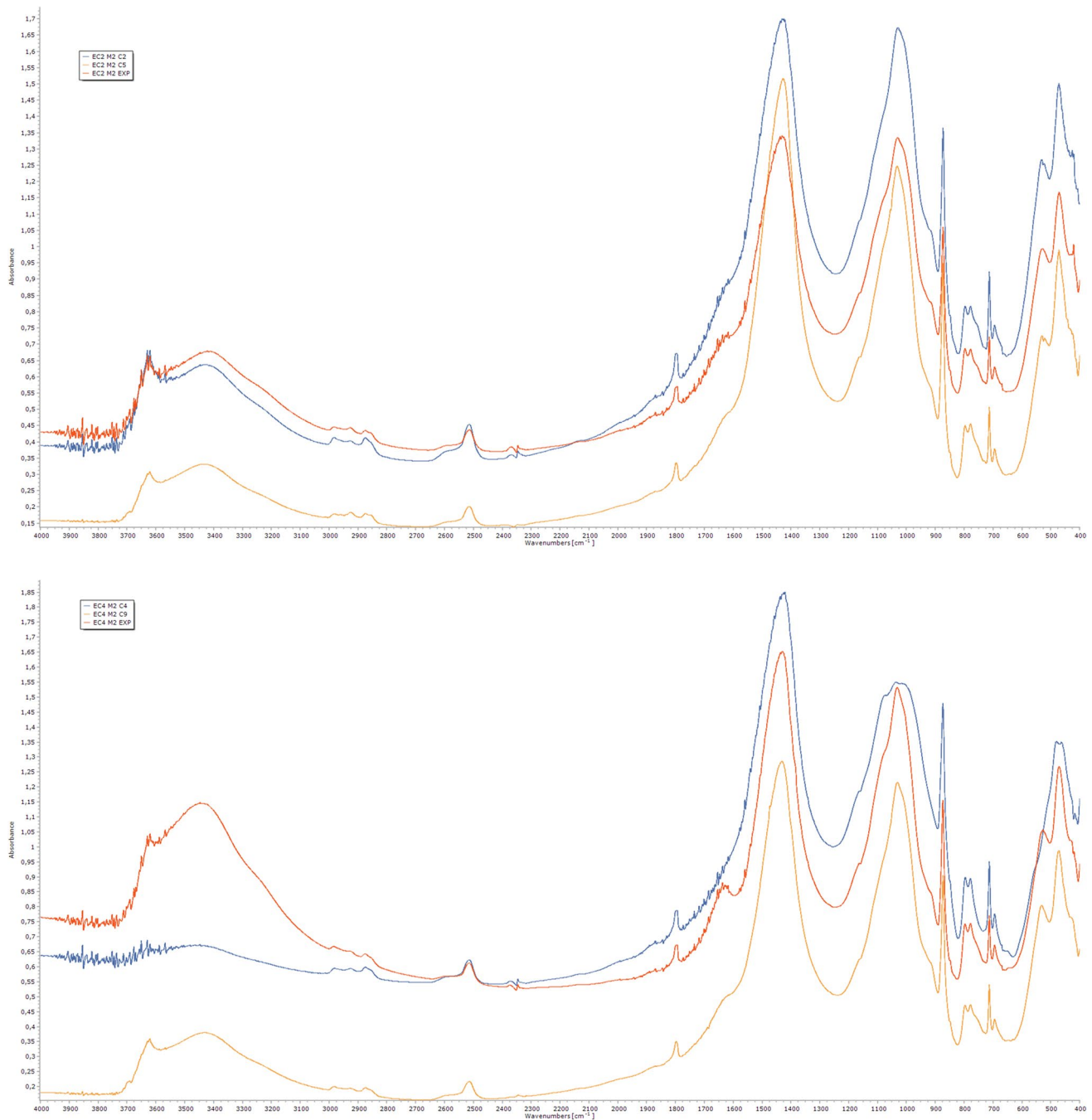


Fig. 14 FTIR results. Above (Hearth 2): red, EXP; blue, C2; yellow, C5. Below (Hearth 4): red, EXP; blue, C4; yellow, C9

accumulated and moved along the interior part of the ceiling up to the door (Fig. 16a). If there was enough air movement (or we fanned the fire), carbonised plant fuel dispersed and even rose with the smoke up to the window. Outdoors, smoke ascended, and its movement depended on the wind direction (in the same way as the flames) and could remain in a kind of column and/or spiral motion that encompassed a wide space around the structure. In the oven used in this study, smoke mainly flowed out through the upper opening

after accumulating in the small vaulted space, although also, to a lesser extent, through the front opening (Fig. 16b). In either case, smoke was more abundant at the beginning of the combustion, when it could have interfered more with the human use of the space where it was lit, but decreased after some minutes and then remained stable.

These observations are of interest for understanding ventilation and smoke evacuation in the past, as windows and openings in roofs are unknown in Iron Age settlements (the

only known openings are doors). In some cases, there may have been windows next to the hearths, as in our Hearth 2. However, smoke was not necessarily perceived as a nuisance, as studied in many social contexts, such as the village of Gourna (Egypt) in the mid-twentieth century described by Hassan Fathy (1970, 162). Openings for smoke evacuation and ventilation could also have drawbacks that were not always socially appreciated; for example, they make it easier for water and animals to enter the home, as shown by ethnoarchaeological research (Beck et al. 2022: 18). Nevertheless, air quality is a factor that should be taken into account, as shown by several previous studies (Shillito 2021).

Combustion processes can be clearly identified macro- and microscopically in the experimental earthen surfaces of the structures, not only in micromorphological observations, but also through FTIR analyses of samples taken through the alterations detected in the clay. This has implications for the archaeological detection of combustion structures.

Preparation layers made of materials such as pebbles or pottery sherds could have helped higher temperatures to be reached and maintained due to increased isolation from the ground or because the heat could be stored in the layer and reflected (Cattani et al. 2015: 38; Fuchs-Khakhhar 2021).

The experimental data record does not allow us to be conclusive in this respect. The micromorphology analyses do not show a significant difference between the microstructure of Hearth 2 (with preparation) and Hearth 4 (without preparation). Neither does the FTIR analysis show a greater alteration of the clays when there is a preparation layer under the combustion surface.

Concerning the use of fuels, the anthracological study detected the species most used in the two hearths, with a marked predominance of oak and pine, which were the wood taxa most used in the experimental fires. The number of undeterminable fragments is representative but not very high in either case (6–7%), and much lower than that we observed in previous analyses of Iron Age combustion structures in the research area, where the indeterminate elements can reach as high as 80% (Belarte et al. 2023). The reason for this difference must reside in the effect of time among other factors in archaeological structures (such as cleaning of the hearths after use, the reuse of fuels or taphonomic processes).

However, interpreting the fuel used on the basis of macrobotanical analysis alone can be misleading and indicates the need for an integrated approach to understanding the use of plant materials. Clearly, phytolith and calcitic microfossil assemblages from experimental hearths allowed us to detect plants and certain “fragile” plant parts that are usually “invisible” in archaeological contexts (e.g. grass leaves/stems, palm leaves) that are not commonly preserved in archaeobotanical records. Moreover, these analyses have been revealed to be especially useful in the accurate interpretation of some of the plant remains identified in the hearths.

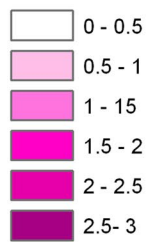
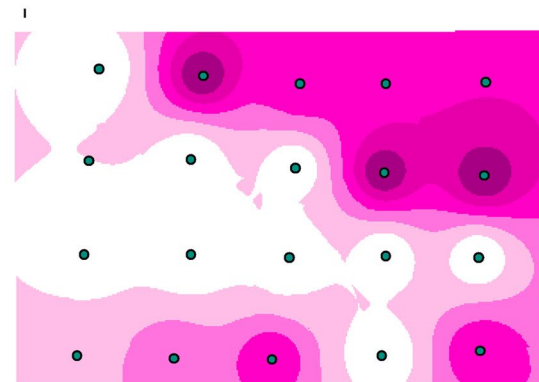
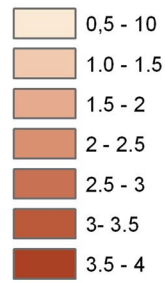
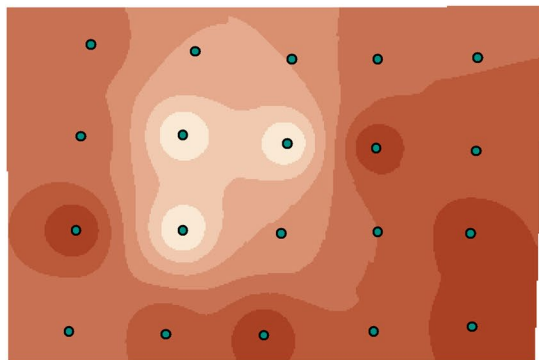
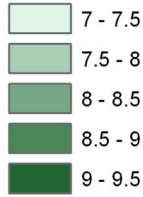
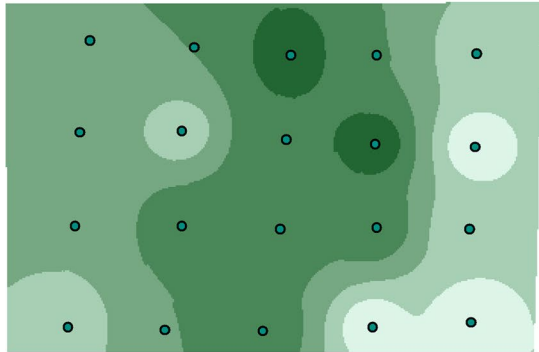
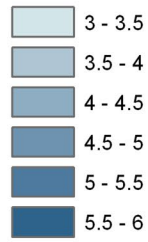
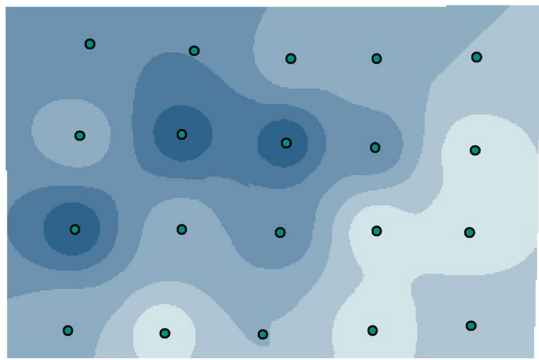
In fact, they are able to detect indicators of faecal matter that are usually difficult to identify on a macroscopic level.

A further challenge to the interpretation of the archaeobotanical record derives from the possible presence of ingested food remains from livestock, when dung has been used as fuel. In our case, the study of the fuel remains after the experimental combustions allowed us to identify olive pits and some shrubs that had not been intentionally used as fuel but had been incorporated into the dung as they were part of the ovicaprine diet.

Differential uses of wood and other plants as fuel, on the one hand, and dung, on the other, are supported by the experiments carried out. Dung, as has also been pointed out in ethnoarchaeological studies, is a fundamental fuel in many societies and allows fires to be stable and last longer, which is especially useful in keeping spaces heated, or for carrying out other activities, such as certain types of food preparation and processing (Kramer 1982: 47; Zapata Peña et al. 2003; Gur-Arieh et al. 2013; Portillo et al. 2017). To be able to be used effectively as fuel, these dung pellets must be completely dried, which reduces their water content and increases their heating potential (Bradbaart et al. 2012: 843). The pellets can be dried by spreading them out in the open air, in the same way as mud bricks (Portillo et al. 2017: 134, Fig. 3a–b). Like mud bricks, dung pellets or “cakes” can also be used for building, as if they were construction blocks (e.g. Anderson and Ertuğ-Yaras 1998; Portillo et al. 2020). Dung is also used as a mortar, mixing it with mud (Steen et al. 2003: 43), acting as temper, or on its own (Shahack-Gross 2011: 212, Fig. 8). As has been already pointed out, during our experimental programme, we experienced the different properties of wood and dung as fuel, reaching lower temperatures with the latter, especially when the weather conditions were cooler and more humid.

A border in the structure, especially when they are indoors, does prevent some of the combusted material used as fuel from moving away from the combustion structure. This dispersion of light-combusted remains of fuel and ash occurs to a much higher degree in outdoor structures and windy conditions and is also influenced by other factors such as human action. In this respect, on a micromorphological level, we observed no ashes and only a few fragments of charcoal on one of the surfaces.

The absence of visible ash and charcoal on the combustion surfaces has often been noted in the archaeological literature and interpreted as an indication that the hearths were cleaned periodically (Belarte 2021). Although rare, some studies refer to the preservation of ash (Karkanis 2021; Braadbaart et al. 2012). In our experimental fires, fuel remains were collected after each combustion, although these were not cleaned thoroughly. This illustrates the difficulty of identifying ashes in archaeological contexts, assuming that the hearths were cleaned periodically. In the case



◀**Fig. 15** Distribution maps (from top to bottom) of phosphates, protein residues, carbohydrates and fatty acids from Hearth 2. The location of samples is visible in the photo and the circles on the distribution maps

of outdoor open fire installations, wind or rain could have completely removed any fuel residue in a short time.

As for chemical residue analysis, the results suggest that chemical residues in the hearth soils may not only depend on the use of hearths for cooking, lighting or heating, but also on the fuel used and the raw materials used to build the hearths. Although the experiment has still limitations and in the future it will be important to carry out a more intensive sampling of the hearths before and after the use of different raw materials and fuel, in order to clarify the relationship between the results of the analyses and the different materials used in these processes, these first results indicate that attention must be paid to the interpretation of the results of hearths found in excavations, and that a multi-proxy approach is needed to better understand the use of these features. This can be linked to the fact that, as previously shown, different activities can leave the same biochemical residues if they are identified with spot tests (Pecci 2003; Pecci et al. 2017). In some cases, the use of analytical techniques that provide a more precise identification of organic residues (i.e. gas chromatography coupled to mass spectrometry, GC–MS) may be needed. These confirm the presence of resin in the burnt traces in the wall next to one of the hearths and more in general could help in better recognising the activities that originated the chemical enrichments. For instance, GC–MS applied to samples rich in fatty acids identified with spot tests could differentiate between pine resin/pitch produced by burning pine wood as a fuel and those from fats of animal origin produced by cooking, therefore differentiating between the two activities (use of fuel vs. cooking). Another example could be if more specific techniques applied to samples rich in protein residues could confirm the presence of dung through the identification of specific biomarkers (e.g. Bull et al. 2002; Vallejo et al. 2022).

In any case, the use of spot tests to analyse hearth surfaces can provide hypotheses (sometimes more than one) on their use that can eventually be better defined by the use of more specific techniques on selected samples or by an approach such as that proposed in this paper, in which fuel specific types could be confirmed by other analyses.

Final remarks

Returning to the questions posed in the introductory section, several concluding remarks can be made.

Firstly, our study does not point to the idea that a preparation layer under the combustion surface, either made of

pebbles or pottery sherds, is clearly related to the attainment of higher temperatures or longer combustions. Many variables were involved in the experimental tasks, including the fuels and weather conditions, in addition to the presence/absence of preparation layers. In further experiments we will reduce the number of variables in order to observe the changes in behaviour of different types of structure. This, together with further analyses of archaeological combustion features, will allow us to come to more solid conclusions in this respect and to better interpret archaeological hearths and their associated spaces.

The experimental studies have shown the potential of combining different techniques applied to the study of combustion structures in order to identify the building materials and processes, the fuels employed and the use of these features.

Micromorphology is especially useful for identifying the building processes, as well as thermoalterations, particularly in combination with FTIR analyses. Integrated macrobotanical and microbotanical evidence from phytoliths and calcitic wood ash pseudomorphs made it possible to identify the fuel remains. Furthermore, calcitic dung spherulites allowed the detection of faecal matter used as a fuel. The current study also points towards the introduction of certain plant remains, such as olive pits, as components of ingested vegetal matter in livestock dung used as fuel, an aspect that should therefore be considered when interpreting fuel use in archaeological contexts.

As for the different uses of fuel, our experiments suggest that dung could have been useful to keep a fire lit for hours (e.g. to keep a space warm), although when higher temperatures were needed (e.g. to boil water), the wood appeared to be more efficient, especially in high humidity conditions. We have observed that when dung was wet, it was not possible to boil water or keep it boiling for a certain number of hours. Thus, it is possible that the use of dung as fuel was related to functions other than cooking, such as heating the spaces, keeping the food warm after cooking or some of the aforementioned uses of smoke that could have been considered by past societies.

The application of spot tests to identify chemical residues suggests that great care has to be taken in the interpretation of hearth chemical enrichment patterns, and that these can depend not only on the use of the hearths themselves (for cooking, heating, etc.), but also on the raw materials used to build them and the burning processes. All this points to the need for an integrated approach to the study of these features.

Finally, we must stress that in this study, we only present the results of the analyses of two experimental combustion structures. Future studies will provide more information and insights to allow improved comparisons with the data recovered from archaeological contexts and to continue towards a better understanding of the past functions and uses of these structures.

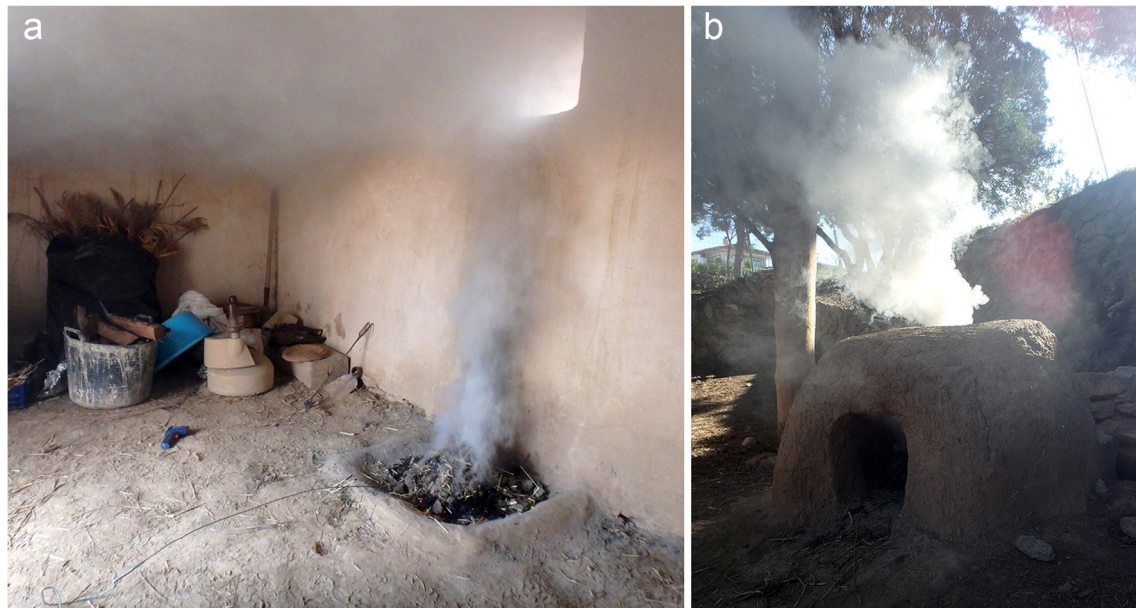


Fig. 16 Smoke behaviour observed during the use of the experimental combustion structures. **a** Indoors in Hearth 2, heading towards the openings and accumulating in the upper part of the room. **b** Outdoors from the oven, dependent on the changing wind

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s12520-023-01772-x>.

Acknowledgements We acknowledge the collaboration of the following institutions and people: Ciutadella Ibèrica de Calafell (Tarragona, Spain) archaeological site, Calafell Town Council, Josep Pou, Georgina Castells, Jordi Morer, Joaquín Fernández, Oriol Cuscó, Silvia Valenzuela, Eduard Solà, Xavi Cid, Lola Ruiz and Laura Giráldez. We are also grateful to Guillermo Marín, who helped in the Phytolith Laboratory at the IMF-CSIC. MPQ holds a Margarita Salas (MARSALAS21-19) postdoctoral contract (Next Generation-EU, Ministerio de Universidades, Universidad de Alicante). MM holds a Beatriu de Pinós (2021BP00103) postdoctoral contract (Universities and Research secretariat of the Ministry of Research and Universities-Generalitat de Catalunya). AG is a JAE Intro fellow at the IMF-CSIC (JAE ICU-21-IMF-02). AP's work is also part of the activities of the ERAAUB (2021 SGR 00696), the IAUB, the INSA-UB (CEX2021-001234-M financed by the Ministerio de Ciencia e Innovación Programme of the Spanish AEI) and the Project PID2020-113409GB-I00.

Author contribution All authors contributed to the study concept and design. The preparation of the first draft of the manuscript was carried out by MCB, MPQ and MM. The paper was written by all the authors. MCB directed the project and obtained the funds; MP and AG conducted the phytolith and calcitic microremains analyses; MM was responsible for the micromorphology of thin sections analyses; CS conducted the FTIR analyses; AP was responsible for the spot tests analyses; SV conducted the anthracological analyses. All the authors participated in the collection of data and the production of text and images; all have read and agreed to the published version of the manuscript.

Funding Open Access funding provided thanks to the CRUE-CSIC agreement with Springer Nature. The TRANSCOMB project (PID2019-104661GB-I00) is funded by the Spanish Ministry of Science and Innovation, and supported by the MIRMED-GIAC (ICAC-URV-UAB) Research Group (2017 SGR 970).

Data availability <https://doi.org/10.34810/data581>.

Code availability Not applicable.

Declarations

Competing interests The authors declare no competing interests.

Ethics approval Not applicable.

Consent to participate Not applicable.

Consent for publication Not applicable.

Conflict of interest The authors declare no competing interests.

Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit <http://creativecommons.org/licenses/by/4.0/>.

References

- Albert RM, Weiner S (2001) Study of phytoliths in prehistoric ash layers using a quantitative approach. In: Meunier JD, Colin F (eds) Phytoliths, applications in earth sciences and human history. A.A. Balkema Publisher, Lisse, pp 251–266

- Albert RM, Shahack-Gross R, Cabanes D, Gilboa A, Lev-Yadun S, Portillo M, Sharon I, Boaretto E, Weiner S (2008) Phytolith-rich layers from the Late Bronze and Iron Ages at Tel Dor (Israel): mode of formation and archaeological significance. *J Archaeol Sci* 35:57–75. <https://doi.org/10.1016/j.jas.2007.02.015>
- Albert RM, Ruiz JA, Sans A (2016) PhytCore ODB: a new tool to improve efficiency in the management and exchange of information on phytoliths. *J Archaeol Sci* 68:98–105. <https://doi.org/10.1016/j.jas.2015.10.014>
- Anderson S, Ertuğ-Yaras F (1998) Fuel fodder and faeces: an ethnographic and botanical study of dung fuel use in Central Anatolia. *Environ Archaeol* 1:99–109. <https://doi.org/10.1179/env.1996.1.1.99>
- Barba L (2007) Chemical residues in lime-plastered archaeological floors. *Geoarchaeology* 22(4):439–452. <https://doi.org/10.1002/gea.20160>
- Barba L, Rodríguez R, Córdoba JL (1991) Manual de técnicas microquímicas de campo para la arqueología. UNAM, Mexico
- Barba L, Ortiz A, Link K, López Luján L, Lazos L (1996) Chemical analysis of residues in floors and the reconstruction of ritual activities at Templo Mayor, Mexico. In: Orna MV (ed) *Archaeological chemistry. Organic, inorganic, and biochemical analysis*. Washington, DC, pp 139–156
- Beck ME, Hill ME, Khandelwal MR (2022) How to keep the home fires burning: a comparative study of cooking hearths for ceramic vessels. *Ethnoarchaeology* 14(1):1–29. <https://doi.org/10.1080/19442890.2022.2084236>
- Belarte MC (2008) Domestic architecture and social differences in north-eastern Iberia during the Iron Age (c. 525–200 BC). *Oxf J Archaeol* 27(2):175–199. <https://doi.org/10.1111/j.1468-0092.2008.00303.x>
- Belarte MC (2021) Forme, fonction et signification des foyers dans la culture ibérique (600-200 av. J.-C.). In: Lamaze J, Bastide M (eds) *Around the Hearth. Ritual and commensal practices in the Mediterranean Iron Age from the Aegean world to the Iberian Peninsula*. De Gruyter, Berlin, pp 255–275. <https://doi.org/10.1515/9783110733662-010>
- Belarte MC, Camañes P, Monrós M, Principal J (2016) Cooking in the Iberian culture (sixth-second century BC): private or public? *J Mediterr Archaeol* 29(2):173–196. <https://doi.org/10.1558/jmea.v29i2.32571>
- Belarte MC, Pastor M, Portillo M, Saorin C, Mateu M, Pecci A, Vila S, Pou J, Castells G, Morer J, Fernández J (2022) Experimental archaeology of Iron Age firing structures from the Western Mediterranean. *EXARC Journal* 2022/2. <https://exarc.net/ark:/88735/10640>
- Belarte MC, Portillo M, Mateu M, Saorin C, Pastor M, Vila S, Pescini V (2023) An interdisciplinary approach to the combustion structures of the Western Mediterranean Iron Age. The first results. Special Issue Firing installations in Mediterranean Late Prehistory: multidisciplinary approaches to their uses and functions. *J Archaeol Sci Rep*. <https://doi.org/10.1016/j.jasrep.2022.103803>
- Berna F, Behar A, Shahack-Gross R, Berg J, Boaretto E, Gilboa A, Sharon I, Shalev S, Shilstein S, Yahalom-Mack N, Zorn JR, Weiner S (2007) Sediments exposed to high temperatures: reconstructing pyrotechnological processes in Late Bronze and Iron Age strata at Tel Dor (Israel). *J Archaeol Sci* 34(3):358–373. <https://doi.org/10.1016/j.jas.2006.05.011>
- Braadbaart F, Poole I, Huisman H, van Os B (2012) Fuel, Fire and Heat: an experimental approach to highlight the potential of studying ash and char remains from archaeological contexts. *J Archaeol Sci* 39:836–847. <https://doi.org/10.1016/j.jas.2011.10.009>
- Brown DA (1984) Prospects and limits of a phytolith key for grasses in the central United States. *J Archaeol Sci* 11:345–368. [https://doi.org/10.1016/0305-4403\(84\)90016-5](https://doi.org/10.1016/0305-4403(84)90016-5)
- Budka J, Geiger C, Heindl P, Hinterhuber V, Reschreiter H (2019) The question of fuel for cooking in Ancient Egypt and Sudan. *EXARC Journal* 2019/1. <https://exarc.net/ark:/88735/10398>
- Bull ID, Lockheart MJ, Elhmmali MM, Roberts DJ, Evershed RP (2002) The origin of faeces by means of biomarker detection. *Environ Int* 27:647–654. [https://doi.org/10.1016/S0160-4120\(01\)00124-6](https://doi.org/10.1016/S0160-4120(01)00124-6)
- Bullock P, Fedoroff N, Jongerius A, Stoops G, Tursina T (1985) *Handbook for soil thin section description*. Waine Research Publications, Wolverhampton
- Cammas C (2018) Micromorphology of earth building materials: toward the reconstruction of former technological processes (Protohistoric and Historic Periods). *Quat Int* 483:160–179. <https://doi.org/10.1016/j.quaint.2018.01.031>
- Canti MG (1999) The production and preservation of faecal spherulites: animals, environment and taphonomy. *J Archaeol Sci* 26:251–258. <https://doi.org/10.1006/jasc.1998.0322>
- Castelleti L, Zimmerman A (1985) Seriation for a spatial factor. An interpretation of the distribution of wood species in a settlement as a hypothesis of the use of the settlements neighbourhood. In: Worrips A, Loving SH (eds) *To pattern the past: Proceedings of the symposium on mathematical methods in archaeology*. Amsterdam, pp 111–118
- Cattani M, Florencia D, Peinetti A (2015) Le strutture di combustione ad uso alimentare nell'età del Bronzo. Dal record archeologico all'archeologia sperimentale. *OCNUS Quaderni della Scuola di Specializzazione in Beni Archeologici* 23:9–43
- Colombini MP, Modugno F, Ribechini E (2005) Direct exposure electron ionization mass spectrometry and gas chromatography/mass spectrometry techniques to study organic coatings on archaeological amphorae. *Mass Spectrom* 40:675–687. <https://doi.org/10.1002/jms.841>
- Correa-Ascencio M, Evershed RP (2014) High throughput screening of organic residues in archaeological potsherds using direct acidified methanol extraction. *Anal Methods* 6:1330. <https://doi.org/10.1039/c3ay41678j>
- Courty MA, Goldberg P, Macphail RI (1989) *Soils and micromorphology in archaeology*. Cambridge University Press, Cambridge
- Courty MA, Allué E, Henry A (2020) Forming mechanisms of vitrified charcoals in archaeological firing assemblages. *J Archaeol Sci Rep* 30:102215. <https://doi.org/10.1016/j.jasrep.2020.102215>
- Fathy H (1970) *Construire avec le peuple*. Sindbad, Arles [Original edition (1969) *Gourna: a Tale of Two Villages*. Ministry of Culture, Arab Republic of Egypt]
- Friesem D, Watzet J, Onfray M (2017) Earth construction materials. In: Nicosia C, Stoops G (eds) *Archaeological soil and sediment micromorphology*. Wiley, Hoboken, pp 99–110
- Fuchs-Khakar C (2021) A focus on the hearth: what a detailed investigation of fireplaces in Boncuklu and Çatalhöyük reveals about Neolithic household practice. *J Archaeol Sci Rep* 39:102900. <https://doi.org/10.1016/j.jasrep.2021.102900>
- Gur-Arieh S, Mintz E, Boaretto E, Shahack-Gross R (2013) An ethnoarchaeological study of cooking installations in rural Uzbekistan: development of a new method for identification of fuel sources. *J Archaeol Sci* 40:4331–4347. <https://doi.org/10.1016/j.jas.2013.06.001>
- Hastorf ChA, Whitehead WT, Johannessen S (2005) Late prehistoric wood use in an Andean Intermontane Valley. *Econ Bot* 59(4):337–355. [https://doi.org/10.1663/0013-0001\(2005\)059\[0337:LPWUIA\]2.0.CO;2](https://doi.org/10.1663/0013-0001(2005)059[0337:LPWUIA]2.0.CO;2)
- Kabukcu C, Chabal L (2021) Sampling and quantitative analysis methods in anthracology from archaeological contexts: achievements and prospects. *Quat Int* 593–594:6–18. <https://doi.org/10.1016/j.quaint.2020.11.004>
- Karkanav P (2021) All about wood ash: Long term fire experiments reveal unknown aspects of the formation and preservation of ash with critical implications on the emergence and use of fire in the past. *J Archaeol Sci* 135:105476. <https://doi.org/10.1016/j.jas.2021.105476>

- Katz O, Cabanes D, Weiner S, Maier AM, Boaretto E, Shahack-Gross R (2010) Rapid phytolith extraction for analysis of phytolith concentrations and assemblages during an excavation: an application at Tell es-Safi/Gath, Israel. *J Archaeol Sci* 37:1557–1563. <https://doi.org/10.1016/j.jas.2010.01.016>
- Kramer C (1982) *Village ethnoarchaeology: rural Iran in archaeological perspective*. Academic, New York
- Loaiza J, Poch R (2015) Muestreo de suelos con énfasis en micromorfología. In: Loaiza J, Stoops G, Poch R, Casamitjana M (eds) *Manual de micromorfología de suelos y técnicas complementarias*. Fondo editorial Pascual Bravo, Medellín, pp 13–27
- McParland LC, Collinson ME, Scott AC, Campbell G, Veal R (2010) Is vitrification in charcoal a result of high temperature burning of wood? *J Archaeol Sci* 37(10):2679–2687. <https://doi.org/10.1016/j.jas.2010.06.006>
- Mateu M, Daneels A (2020) La micromorfología aplicada al estudio del patrimonio construido en tierra. *Gremium. Revista de restauración arquitectónica* 7 (NE2):10–23. <https://doi.org/10.56039/rgne2a03>
- Mateu M, Bergadà MM, Armada XL, Rafel N (2019) Micromorphology of the early Iron Age semi-cemented floors: El Calvari del Molar (Tarragona, NE Spain) as case study. *J Archaeol Sci Rep* 23:746–762. <https://doi.org/10.1016/j.jasrep.2018.11.028>
- Mateu M, Fernández H, Daneels A, Cabadas H, Piña S (2022) Earthen architecture in the Mesoamerican Classic Period: a micromorphological approach to its manufacture process. *J Archaeol Sci* 137:105525. <https://doi.org/10.1016/j.jas.2021.105525>
- Middleton WD, Barba L, Pecci A, Burton JH, Ortiz A, Salvini L, Rodríguez Suárez R (2010) The study of archaeological floors: methodological proposal for the analysis of anthropogenic residues by spot tests, ICP-OES, and GC-MS. *J Archaeol Method Theory* 17:183–208. <https://doi.org/10.1007/s10816-010-9088-6>
- Mulholland SC, Rapp G Jr (1992) A morphological classification of grass silica-bodies. In: Rapp G Jr, Mulholland SC (eds) *Phytolith systematics: emerging issues, advances in archaeological and museum science*. Plenum Press, New York, pp 65–89. https://doi.org/10.1007/978-1-4899-1155-1_4
- Neumann K, Strömberg AEC, Ball TB, Albert RM, Vrydaghs L, Scott-Cummings L (International Committee for Phytolith Taxonomy ICPT) (2019) International Code for Phytolith Nomenclature (ICPN) 2.0. *Ann Bot* 124 (2):189–199. <https://doi.org/10.1093/aob/mcz064>
- Pearsall DM (1988) *La producción de alimentos en Real Alto*. Biblioteca Ecuatoriana de Arqueología, 2. ESPOL-Corporation Editora Nacional, Guayaquil
- Pecci A (2003) Los alimentos y sus residuos químicos. Arqueología experimental para entender actividades prehispánicas. *Quaderni di Thule, III, Atti del XXV Convegno Internazionale di Americanistica*, pp 75–83
- Pecci A (2009) Analisi chimiche delle superfici pavimentali: un contributo all'interpretazione funzionale degli spazi archeologici. In: Volpe G, Favia P (eds) *V Congresso Nazionale di Archeologia Medievale*, Borgo S. Lorenzo, pp 105–110
- Pecci A (2013) Almost ten years of plasters residue analysis in Italy: activity areas and the function of structures. *Period Mineral* 82(3):393–410
- Pecci A (2021) Análisis de residuos químicos en materiales arqueológicos: marcadores de actividades antrópicas en el pasado. *Pyrenae* 52(1):7–54
- Pecci A, D'Andria F (2014) Oil production in Roman times: residue analysis of the floors of an installation in Lecce (southern Italy). *J Archaeol Sci* 46:363–371. <https://doi.org/10.1016/j.jas.2014.03.019>
- Pecci A, Barba L, Ortiz A (2017) Chemical residues as anthropic activity markers Ethnoarchaeology, experimental archaeology and archaeology of food production and consumption. *Environ Archaeol* 22(4):343–353. <https://doi.org/10.1080/14614103.2017.1359354>
- Pecci A, Cau MA, Valdambrini C, Insera F (2013) Understanding residues of oil production: chemical analyses of floors in traditional mills. *J Archaeol Sci* 40:883–893
- Pecci A, Ortiz A, López Varela S (2011) “Tracce” chimiche delle attività umane: distribuzione spaziale dei residui in una abitazione-laboratorio di ceramica a Cuentepec (Messico). In: Lugli F, Stoppiello A, Biagetti S (eds) *Proceedings of the 4th Congress of Ethnoarchaeology* (2006). BAR International Series 2235, Oxford, pp 189–192
- Piperno DR (2006) *Phytoliths: a comprehensive guide for archaeologists and paleoecologists*. AltaMira Press, Lanham
- Piqué R (1996) La gestió dels recursos forestals a la Draga (Banyoles, Pla de l'Estany). In: *I Congrés del Neolític a la Península Ibèrica*. Rubricatum, 1 (vol. 1). Gavà-Bellaterra, pp 57–63
- Piqué R, Vila Moreiras S, Alonso N (2011) Vegetation and firewood uses in the western Catalan plain from Neolithic to the Middle Age. *Saguntum: Papeles del Laboratorio de Arqueología de Valencia N°extra* 13, pp 85–96
- Portillo M, Matthews W (2020) Investigating use of space and human-animal interactions in agricultural built environments: the geo-ethnoarchaeology of livestock dung. In: Otto A, Herles M, Kaniuth K (eds) *Proceedings of the 11th International Congress on the Archaeology of the Ancient Near East*. Harrassowitz Verlag, Wiesbaden, pp 497–508
- Portillo M, García-Suárez A (2021) Disentangling human–plant–animal dynamics at the microscale: geo-ethnoarchaeological case studies from North Africa and the Near East. *Appl Sci* 11:8143. <https://doi.org/10.3390/app11178143>
- Portillo M, Valenzuela S, Albert RM (2012) Domestic patterns in the Numidian site of Althiburos (northern Tunisia): the results from a combined study of animal bones, dung and plant remains. *Quat Int* 275:84–96. <https://doi.org/10.1016/j.quaint.2012.01.024>
- Portillo M, Kadowaki S, Nishiaki Y, Albert RM (2014) Early Neolithic household behavior at Tell Seker al-Aheimar (Upper Khabur, Syria): a comparison to ethnoarchaeological study of phytoliths and dung spherulites. *J Archaeol Sci* 42:107–118. <https://doi.org/10.1016/j.jas.2013.10.038>
- Portillo M, Belarte MC, Ramon J, Kallala N, Sanmartí J, Albert RM (2017) An ethnoarchaeological study of livestock dung fuels from cooking installations in northern Tunisia. *Quat Int* 431 A:131–144. <https://doi.org/10.1016/j.quaint.2015.12.040>
- Portillo M, García-Suárez A, Matthews W (2020) Livestock faecal indicators for animal management, penning, foddering and dung use in early agricultural built environments in the Konya Plain, Central Anatolia. *Archaeol Anthropol Sci* 12:40. <https://doi.org/10.1007/s12520-019-00988-0>
- Portillo M, Dudgeon K, Allistone G, Raeuf Aziz K, Matthews W (2021a) The taphonomy of plant and livestock dung microfossils: an ethnoarchaeological and experimental approach. *Environ Archaeol* 26:439–454. <https://doi.org/10.1080/14614103.2020.1800344>
- Portillo M, Dudgeon K, Anglada M, Ramis D, Llergo Y, Ferrer A (2021b) Phytolith and calcitic spherulite indicators from modern reference animal dung from Mediterranean Island ecosystems: Menorca, Balearic Islands. *Appl Sci* 11(16):7202. <https://doi.org/10.3390/app11167202>
- Portillo M, Morales J, Carrión Marco Y, Aouadi N, Lucarini G, Belhouchet L, Coppa A, Peña-Chocarro L (2021c) Changing plant-based subsistence practices among early and middle Holocene communities in eastern Maghreb. *Environ Archaeol* 26:455–470. <https://doi.org/10.1080/14614103.2020.1829298>
- Pou J, Santacana J, Morer J, Asensio D, Sanmartí J (2001) El projecte d'interpretació arquitectònica de la ciutat ibèrica de Calafell (Baix Penedès). In: Belarte MC, Pou J, Sanmartí J, Santacana J (eds) *Tècniques constructives d'època ibèrica i experimentació arquitectònica a la Mediterrània*. Actes de la I Reunió Internacional

- d'Arqueologia de Calafell (Calafell, 20, 21 i 22 de gener del 2000). *Arqueo Mediterrània*, 6. Universitat de Barcelona, Barcelona, pp 95–115
- Rosen AM (1992) Preliminary identification of silica skeletons from Near Eastern archaeological sites: an anatomical approach. In: Rapp G Jr, Mulholland SC (eds) *Phytolith systematics: emerging issues, advances in archaeological and museum science*. Plenum Press, New York, pp 129–147
- Saorin C (2018) Les estructures de combustió del sector 3 de l'assentament del Tossal de la Vila (la Serra d'en Galceran, la Plana Alta). *Quaderns de Prehistòria i Arqueologia de Castelló* 36:73–81
- Shahack-Gross R (2011) Herbivorous livestock dung: formation, taphonomy, methods for identification and archaeological significance. *J Archaeol Sci* 38:205–218. <https://doi.org/10.1016/j.jas.2010.09.019>
- Shillito LM (2021) Analysis of fine particulates from fuel burning in a reconstructed building at Çatalhöyük World Heritage Site, Turkey: assessing air pollution in prehistoric settled communities. *Environ Geochem Health* 44:1033–1048. <https://doi.org/10.1007/s10653-021-01000-2>
- Steen A, Steen B, Komatsu E (2003) *Built by hand. Vernacular building around the world*. Gibb Smith Publisher, Layton
- Stoops G (2003) *Guidelines for analysis and description of soil and regolith thin sections*. Soil Science Society of America, Madison
- Thompson GB (1984) Wood charcoals from tropical sites: a contribution to methodology and interpretation. In: Hather JG (ed) *Tropical Archaeobotany. Applications and new developments*. Routledge, London, pp 9–34
- Tsartsidou G, Lev-Yadun S, Albert RM, Miller-Rosen A, Efstratiou N, Weiner S (2007) The phytolith archaeological record: strengths and weaknesses based on a quantitative modern reference collection from Greece. *J Archaeol Sci* 34:1262–1275. <https://doi.org/10.1016/j.jas.2006.10.017>
- Tsartsidou G, Lev-Yadun S, Efstratiou N, Weiner S (2008) Ethnoarchaeological study of phytolith assemblages from an agro-pastoral village in Northern Greece (Sarakini): development and application of a Phytolith Difference Index. *J Archaeol Sci* 35(3):600–613. <https://doi.org/10.1016/j.jas.2007.05.008>
- Twiss PC (1992) Predicted world distribution of C₃ and C₄ grass phytoliths. In: Rapp G Jr, Mulholland SC (eds) *Phytolith systematics: emerging issues, advances in archaeological and museum science*. Plenum Press, New York, pp 113–128. https://doi.org/10.1007/978-1-4899-1155-1_6
- Twiss PC, Suess E, Smith RM (1969) Morphological classification of grass phytoliths. *Soil Sci Soc Am Proc* 33:109–115. <https://doi.org/10.2136/sssaj1969.03615995003300010030x>
- Vallejo A, Gea J, Gorostizu-Orkaiztegi A, Vergès JM, Martín P, Sampedro MC, Sánchez-Ortega A, Goicolea MA, Barrio RJ (2022) Hormones and bile acids as biomarkers for the characterization of animal management in prehistoric sheepfold caves: El Mirador case (Sierra de Atapuerca, Burgos, Spain). *J Archaeol Sci* 138:105547. <https://doi.org/10.1016/j.jas.2022.105547>
- Verrecchia EP, Trombino L (2021) *A visual atlas for soil micro-morphologists*. Springer, Cham. <https://doi.org/10.1007/978-3-030-67806-7>
- Vila Moreiras S (2018) *L'explotació dels recursos vegetals a la plana occidental catalana durant la protohistòria a partir de l'anàlisi antracològica (III-I mil·lenni)*. Universitat de Lleida, Lleida. Unpublished PhD
- Weiner S (2010) *Microarchaeology. Beyond the visible archaeological record*. Cambridge University Press, Cambridge
- Zapata Peña L, Peña-Chocarro L, Ibáñez Estévez JJ, González Urquijo JE (2003) Ethnoarchaeology in the Moroccan Jebala (Western Rif): Wood and dung as fuel. In: Neumann K, Butler A, Kahlheber S (eds) *Food, fuel and fields: progress in African archaeobotany*. *Africa praehistorica* 15. Heinrich-Barth-Institut, Köln, pp 163–175

Publisher's note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.