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Phosphatic crusts as macroscopic and microscopic proxies for identifying archaeological animal penning areas

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ABSTRACT

This study introduces new macroscopic and microscopic evidence for identifying archaeological animal penning areas: phosphatic crusts. Despite the importance of herding activities for reconstructing the social, economic, and ecological aspects of ancient communities, evidence for animal penning areas has traditionally relied on faint architectural traces or microscopic indicators that are often challenging to identify in the field. By employing a multidisciplinary approach that combines field observations, geoarchaeology, lipid biomarker, and micro-botanical analyses, this research examines the phosphatic crusts recently identified at the Middle Bronze Age (1650-1300 BCE) site of La Muraiola di Povegliano (Verona, north-eastern Italy).

The analyses uncover the processes behind phosphatic crust formation, highlighting the key role of the concentration of animal ejecta in the cementation of the deposit by nanocrystalline partially carbonated hydroxylapatite. This multi-proxy approach further demonstrates that phosphatic crusts serve as crucial archives for investigating the use of space, livestock management (e.g., free grazing/confinement, livestock species, foddering), and human-animal-environment interactions.

1. Introduction

The identification of livestock penning areas as an indicator of animal management is of paramount importance in the reconstruction of social, economic, and ecological aspects of ancient communities (Fernández-Palacios et al., 2023; Portillo et al., 2020; Kristiansen, 2018, p. 118; Macphail et al., 1997; Portillo et al., 2019). However, so far the identification of penning areas has mostly relied on microscopic indicators that combine geoarchaeological and archaeobotanical proxies, such as soil micromorphology, phytolith, and pollen analysis (Herrera-Herrera et al., 2024; Nicosia et al., 2022; Matthews et al., 2014; Albert et al., 2008; Shahack-Gross et al., 2005; Fernández-Palacios et al., 2023; Alonso-Eguiluz et al., 2023; Polo Díaz et al., 2014; Portillo et al., 2019, 2020). In recent years, these methodologies have been coupled with the analysis of organic compounds, such as faecal biomarkers, to identify traces of faecal pollution derived from domestic animals (Herrera-Herrera et al., 2024; Fernández-Palacios et al., 2024; Pescini et al., 2023; Vallejo et al., 2023; Mackay et al., 2020; Nicosia et al., 2019). The need for microarchaeological and bio/geochemical proxies relies on the faint macroscopic evidence that animal penning activities leave in the archaeological record. In the literature, macroscopic indicators used as evidence for animal gathering enclosures or stables/byre-houses often comprise solely the remnants of postholes demarcating fences or internal partitions (cf. Nicosia et al., 2022, S1 Table; see also Shahack-Gross et al., 2004b; Weiner, 2010, pp. 239–240; Karkanas and Goldberg, 2019, p. 121). This is especially true when burial conditions did not allow for the recovery of macroscopic faecal remains.

This paper sheds light on a macroscopic feature that is directly related to archaeological animal penning areas: phosphatic crusts. A combined approach that involved field data, geoarchaeology, organic compound analysis, and the analysis of botanical microremains (i.e.,

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pollen and phytoliths), was employed to support the hypothesis that the origin of phosphatic crusts was from the gathering of animals. Additionally, this paper aims to elucidate the diagenetic processes that lead to the formation of phosphatic crusts in animal gathering enclosures and to explore their potential in reconstructing ancient livestock management practices and paleoenvironmental aspects. The focus of this paper is the *in situ* phosphatic crusts recently identified and excavated at the Middle Bronze Age (hereafter, MBA; 1650-1350/1300 BCE, see Cardarelli, 2010) Terramara site of La Muraiola di Povegliano (south of Verona, north-eastern Italy). This site provided a unique opportunity to investigate a thick (1.3 m), well-preserved stratigraphic sequence where burial conditions promoted extensive phosphatisation of organic remains.

1.1. Phosphatic crusts in animal penning areas: the state of the art

Geoarchaeological research has demonstrated that phosphate precipitation commonly occurs in oxidised deposits enriched with organic matter and animal droppings, leading to the formation of crusts and other impregnative or intrusive pedofeatures (*sensu* Stoops, 2021; see Karkanas and Goldberg, 2018, p. 334; Shahack-Gross, 2017, p. 270; Shahack-Gross et al., 2004a). Authigenic phosphates has been observed in a variety of archaeological contexts, including cesspits, latrines, caves, and livestock penning areas. In cesspits and latrines, the high phosphate content of human faeces, urine, and food residues is released during decay (Shahack-Gross et al., 2004a, p. 1270), facilitating the formation of authigenic phosphates and the pseudomorphic phosphatisation of botanical remains, such as seeds (Amichay et al., 2019; Carruthers, 2000; Green, 1979; Marshall et al., 2008; McCobb et al., 2001; Murphy, 2014). In cave settings, authigenic phosphates, including calcium (Ca), Ca-aluminium, and Ca-iron phases, are associated with diagenetic processes triggered by the decomposition of bird and bat guano (Cremaschi et al., 2022; Karkanas, 2017; Karkanas et al., 2000; Karkanas and Goldberg, 2010; Shahack-Gross et al., 2004a). In livestock penning areas, phosphatic crusts are likely derived from compacted dung and bedding material enriched with liquid animal waste, which partially transforms carbonates and organic matter into Ca-phosphates (Shahack-Gross et al., 2003, p. 454). The specific phases of authigenic phosphates that form are influenced by several factors, including pH, water availability, phosphorus content, and the abundance of organic matter (Shahack-Gross et al., 2004a; Weiner et al., 2002). While considerable effort has been dedicated to understanding diagenetic processes in caves, the chemical and mineralogical mechanisms underpinning the formation of phosphatic crusts in livestock penning areas remain largely underexplored, with only a few landmark studies suggesting that processes of organic matter decay are likely similar to those observed in caves (e.g., Macphail et al., 2004; Shahack-Gross et al., 2003).

Phosphatic crusts interpreted as indicators of animal penning (in some cases referred to as 'stable' or 'stabling' crusts) have been mainly discussed in the micromorphological literature (Table 1). These features consist of microlaminated plant fragments embedded in a calcium phosphate-rich, autofluorescent (blue light) cement of hydroxylapatite ($Ca_5(PO_4)_3OH$) or carbonated hydroxylapatite ($Ca_5(PO_4,CO_3)_3(OH)$) (see Brönnimann et al., 2017, pp. 62–63; Macphail and Goldberg, 2018).

If phosphatic crusts derived from animal penning are mentioned in micromorphology, to our knowledge no field evidence of them has been published – or recognised as such – so far. This is a crucial aspect: given the scale of micromorphological observation, interpreting localised features as spatially relevant for understanding the ancient use of space becomes rather challenging without a robust connection with field data.

Table 1 summarises the evidence of phosphatic crusts interpreted as evidence of animal gathering enclosures published in the archaeological and ethnoarchaeological literature. The only mention of macroscopic features derives from the experimental site of Butser Farm (UK), in which Iron Age farming techniques are implemented (Reynolds, 1979). At Butser Farm, hard crusts developed on the byre floors after periods of overwintering of cattle and ovicaprids (Cruise and Macphail, 2001, p. 185). Here, "each autumn an animal bedding layer some 40–50 cm thick was prepared from straw and compressed grass pellets; this was renewed halfway through the winter (Reynolds pers. comm.). Loose manure and bedding was cleared out in the spring leaving a crust on the floor" (Macphail and Goldberg, 1995, p. 5). Even though micromorphological and chemical analysis have been conducted on one of these crusts, the available microscopic and chemical data have been questioned (see Canti et al., 2006; and the reply by Macphail et al., 2006). Field images and a detailed description are also missing for the phosphatic crust identified in a modern horse stable at Montainville (FR; Macphail et al., 2004). Moreover, the mineralogical identification of the cement as hydroxylapatite relied solely on its autofluorescence under blue light (Macphail et al., 2004, p. 181).

In archaeological contexts, phosphatic crusts have been noted through micromorphology at the Arene Candide cave (IT), a Neolithic site in which seasonal animal penning activities occurred (Macphail et al., 1997; Wattez et al., 1990). However, the authors just mention "fine yellowish phosphatic layers" in relation to herbivore dung and phytoliths rich deposits, without providing any field view or chemical/mineralogical details (Macphail et al., 1997, p. 82). Cemented Ca-phosphate crusts, rich in phytoliths, have been linked to animal penning areas in micromorphological studies of prehistoric and Medieval sites in Europe and Southwest Asia, though no further information is available (Table 1). A possible phosphatic crust was observed in a Medieval horse stable at Veselí nad Moravou (CZ), but the published photomicrograph shows horizontally-lying vegetal material, with no clear evidence of Ca-phosphate cementation (Dejmal et al., 2014, Fig. 4d). Furthermore, no chemical or mineralogical data support the identification of the cement as hydroxylapatite, aside from its fluorescence under blue light (Dejmal et al., 2014, p. 5).

In other instances, phosphatic aggregates interpreted as reworked crust fragments have been identified in thin section (Table 1), described as 2 mm-long and cemented by "amorphous yellow (likely phosphatic) material" containing "plant fragments and articulated phytoliths, with associated loose phytoliths and amorphous organic matter" (Macphail, 2015). However, in all cases, no field data and mineralogical analysis are provided (Table 1).

In this brief review on phosphatic crusts related to animal penning, it is worth mentioning the Early Iron Age site of KwaGandaganda, a settlement excavated in the 1980s in South Africa. Here, several byres were identified based on the presence of laterally-continuous 30 cm thick dung layers (Whitelaw, 1994, p. 19). Five-cm thick, red and hard crusts were systematically documented beneath the herbivore dung accumulations, interpreted as the byre floors. Whitelaw suggests that "the hard 'floor' […] appeared to be an altered form of the earth underlying the dung, formed through compaction, or more probably, as a result of the heat in the accumulated dung and urine deposits" (1994, p. 20). While no micromorphological analysis was conducted on these 'red crusts', their description clearly parallels the *in situ* phosphatic crusts recently discovered at the Middle Bronze Age site of La Muraiola di Povegliano (§3.1), which is the focus of this paper.

1.2. The Terramara site of La Muraiola di Povegliano

The archaeological site of La Muraiola di Povegliano is located south of Verona (north-eastern Italy), in a portion of the Po Plain characterised by numerous spring-fed rivers. In particular, the site lies within an incised and infilled valley shaped by the Holocene activity of the Tione dei Monti River, which eroded the Late Pleistocene alluvial megafans of the Adige and Mincio Rivers (Fig. 1; Castiglioni, 1997).

In the 1980s, archaeological excavations revealed a large MBA Terramara settlement (Belemmi et al., 1997), a peculiar settlement type that spread in the Po Plain between the MBA and the Late Bronze Age (LBA; Bernabò Brea et al., 1997). Terramara settlements were typically

Table 1

Modern and archaeological sites mentioning the presence of phosphatic crusts or phosphatic crust fragments interpreted as derived from livestock penning activities. Phosphatic crusts formed in cesspits and in cave settings due to the presence of bat guano are not included in the list.

Site	Chronology	Type	Field information (quotation)	Micromophological description (quotation)	Chemical	Mineralogical	References
Butser Farm (UK)	Contemporary	*In situ* phosphatic crust	hard crust on the byre floor	layered plant fragments that are interbedded with silt-size quartz and secondary calcite	Yes	Yes	Macphail et al. (2004); Cruise and Macphail, 2001; Macphail and Goldberg (1995)
Montainville (FR)	Contemporary	*In situ* phosphatic crust	/	very dominantly laminar structured organic deposit (0,9 cm think) that is autofluorescent under blue light […]. It is also dominated by lengthy (5 mm) monocotyledonous plant fragments, and is dark stained in places	/	/	Macphail et al. (2004)
Arene Candide (IT)	Neolithic	*In situ* phosphatic crust	/	fine yellowish phosphatic layers	/	/	Goldberg and Macphail (2006); Macphail et al. (1997)
Los Husos II (ES)	Neolithic	*In situ* phosphatic crust?	/	dung crust from one of the stabling episodes […]. Note rounded yellowish phosphatic aggregates […] embedded in the plant accumulation	/	/	Polo Díaz and Fernández Eraso (2010)
Tel Hazor (IL)	Bronze Age	*In situ* phosphatic crust	/	phosphatic crust that formed following degradation of organic matter. […] intimate association between phosphate and botanic material	/	Partial	Shahack-Gross (2017)
Potterne (UK)	Late Bronze Age- Early Iron Age	*In situ* phosphatic crust	/	calcium phosphate cemented soil containing abundant phytoliths and sporadic diatoms; colourless, non-birefringent, and autofluorescent under UVL cement	Partial	/	Macphail (2000)
Pungrt (SLO)	Early Iron Age	*In situ* phosphatic crust	/	stabling crust with the upper, highly phosphatised, and lower, organic-rich, section	/	/	Prijatelj et al. (2024)
KwaGandaganda (ZA)	Early Iron Age	*In situ* phosphatic crust?	hard 'floor' about 5 cm thick which appeared to be an altered form of the earth underlying the dung, formed through compaction, or more probably, as a result of the heat in the accumulated dung and urine deposits. The red crust occurred beneath all major dung accumulations on the site	/	/	/	Whitelaw (1994)
London Guildhall (UK)	Medieval	*In situ* phosphatic crust	/	intact horizontally layered Poaceae tissues, commonly with abundant phytoliths and long articulated phytoliths intercalated with silt, autofluorescent under blue light	Partial	/	Macphail et al. (2007)
Veselí nad Moravou (CZ)	Medieval	*In situ* phosphatic crust	/	microscopically composed of laminated plant fragments embedded in a calcium phosphate-rich, autofluorescent (blue light) cement of hydroxyapatite	/	/	Dejmal et al. (2014)
London Standset Airport (UK)	Pre-Iron Age	Fragments of phosphatic crust	/	rare to occasional heavily stained yellowish brown sand size fragments of layered plant fragments (up to 0.5 mm)	Partial	/	Macphail and Crowther (2008)
Heybridge (UK)	Late Iron Age-Roman Age	Fragments of phosphatic crust	/	2 mm long fragments of plant fragments and articulated phytoliths, with associated loose phytoliths and amorphous organic matter, that are cemented by amorphous yellow (likely phosphatic) material	/	/	Macphail (2015)

(continued on next page)

Table 1 (*continued*)

Site	Chronology	Type	Field information (quotation)	Micromophological description (quotation)	Chemical	Mineralogical	References
Deansway (UK)	Late Roman Age	Fragments of phosphatic crust	/	phosphatised pieces of soil	/	/	Macphail (1994)

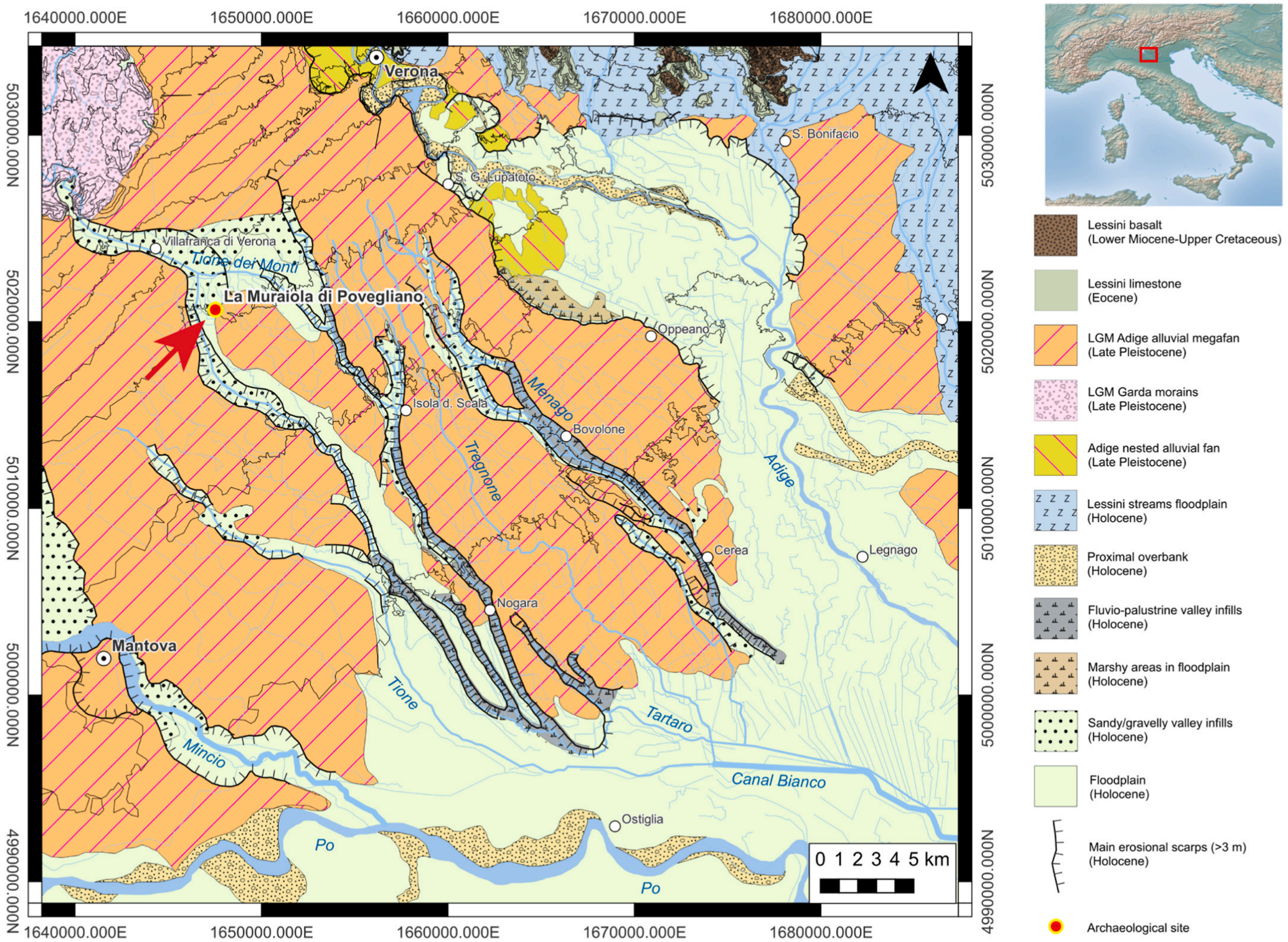


Fig. 1. Geomorphological map showing the floodplain between the Lessini foothill and the present-day Po River. The map is based on Sorbini et al. (1984), Castiglioni (1997), and Carta Geologica d'Italia 1:100.000 sheets n° 48, 49, 62, 63 (modified from Nicosia et al., 2022, Fig. 1). The image of Italy in the upper right corner was obtained from Natural Earth (public domain - naturalearthdata.com).

characterised by a regularly organised living area, surrounded by a ditch and a rampart, with a structured agricultural landscape in their immediate vicinity (Cardarelli, 2010). At La Muraiola, new ongoing excavations started in 2022 (10 × 7 m) near the 1980s excavation sector have uncovered a 1.3 m thick archaeological deposit comprising two main phases (Fig. 2). The oldest phase starts on top of a buried soil and features massive brownish layers, identified through micromorphological analysis as herbivore dung accumulations (Polisca, 2024). The upper phase consists of a sequence of tabular ash-rich layers containing abundant archaeological materials such as pottery fragments, faunal remains, bronze objects, and amber.

Ash-rich layers often contain coprolites and phosphatised vegetal remains, such as wood fragments, seeds and nuts. Within the ash-rich layers, aggregates of hard, phosphatised material are also interspersed (see §3.1). These clumps will be referred to as 'reworked phosphatic fragments' in this paper. Numerous aligned postholes, cooking plates (*sensu* Peinetti et al., 2023), and floor patches are associated with ash-rich layers, suggesting the use of this area for domestic activities.

This article focuses on a structure identified in the field as a possible barn. The perimeter of the structure is defined by aligned postholes, testifying a not-preserved wall that acted as boundary between an internal and an external sedimentation (Fig. 3). Indirect evidence for roofing comes from the absence of micromorphological indicators of poaching (i.e., trampling on wet substrates) in the internal deposits (Rentzel et al., 2017), as well as the recovery of a cluster of thousands of millet grains – likely stored within a structure – found in the conflagration layer that destroyed the barn (SU 185; see Polisca, 2024). Within the structure, two superimposed and laterally continuous phosphatic crusts were identified, namely stratigraphic units (hereafter, SUs) 187 and 410. The crusts are interspersed with layers of trampled herbivore dung mixed with ash (SUs 359, 382, 407; Fig. 3), as revealed by micromorphology (Polisca, 2024). Given their extent, these crusts will

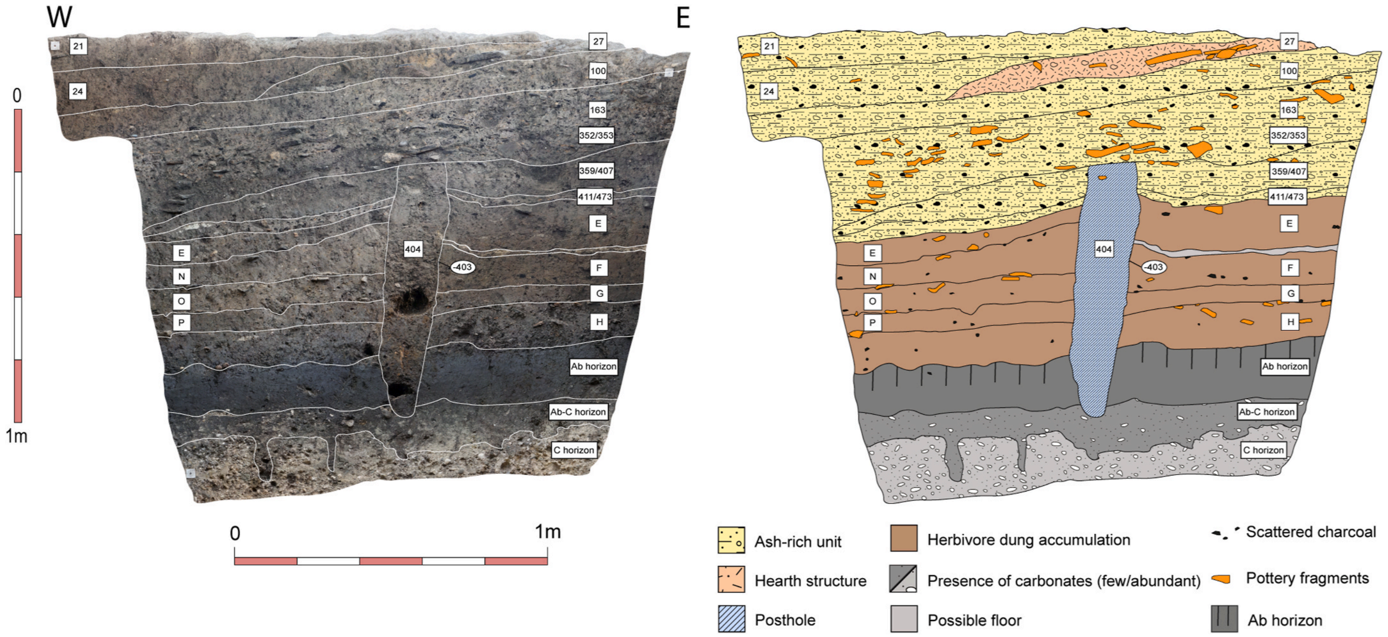


Fig. 2. Stratigraphic profile showing the entire archaeological stratification. On the left, an orthomosaic with the superimposed stratigraphic units distinguished in the field. On the right, an interpretation of the profile based on field data and preliminary micromorphological analysis. Numbers define the SUs already excavated or exposed in planar view, while letters are used for layers observed and described only in profile view (i.e., not yet exposed in plane).

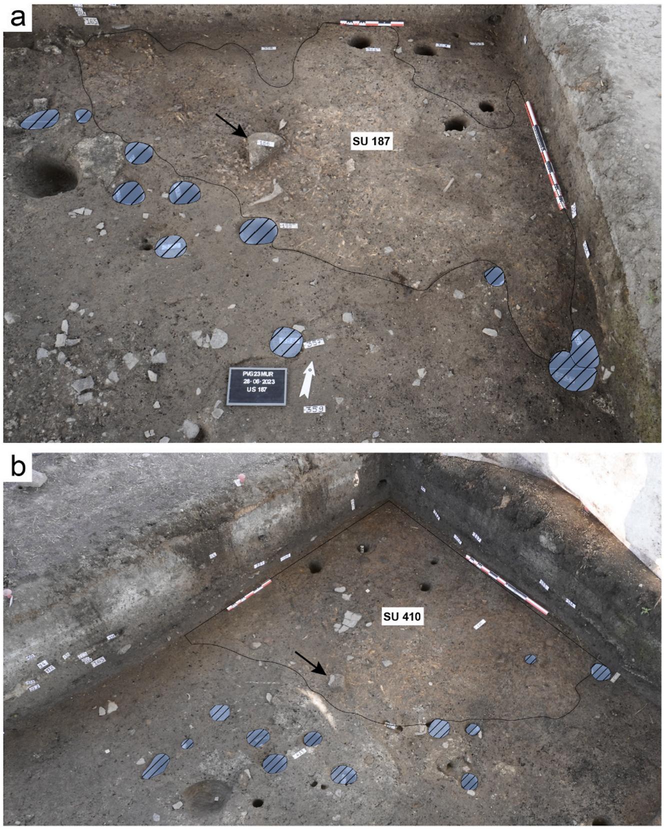


Fig. 3. Field photographs of the *in situ* phosphatic crusts SU 187 ('a') and SU 410 ('b'). The hachured shapes highlight the postholes defining the perimeter of the possible barn. The black lines mark the extent of SUs 187 and 410. In 'b', the arrow points to the sampling location of the micromorphological blocks PVG 80 and PVG 83, containing the *in situ* phosphatic crusts SU 187 and 410, respectively (Fig. 4).

be referred to as '*in situ* phosphatic crusts'. In other areas of the excavation, localised patches of *in situ* phosphatic crusts are also identified, but without a clear relationship with structural elements (i.e., alignments of postholes).

2. Materials and methods

During the 2023 excavation campaign, temporary baulks were intentionally left unexcavated to collect undisturbed and oriented blocks for micromorphological analysis. One of the baulks intersected the internal accretion of the possible barn (Fig. 4a), including the two superimposed *in situ* phosphatic crusts, SUs 187 and 410. Additional micromorphological blocks were collected from localised patches of *in situ* phosphatic crusts, ensuring that both the deposits above and below these crusts were sampled (Fig. 4b). These blocks and thin sections were then chemically mapped using micro-X-ray fluorescence (micro-XRF) spectroscopy. Bulk samples of the phosphatised crusts were also collected from each stratigraphic unit identified in the field for further chemical (micro-Fourier-transform spectroscopy, micro-FTIR; gas chromatography-mass spectrometry, GC-MS), mineralogical (powder-X-ray diffraction, p-XRD), and microbotanical analyses (pollen, phytoliths). Fragments of the *in situ* phosphatic crusts were collected for mesoscopic (3D digital microscopy) and microscopic (Scanning electron microscopy, SEM) observations, as well as chemical analysis through SEM combined with an energy dispersive spectrometer (EDS).

Reworked phosphatic fragments were handpicked during the excavation for the same analyses, to compare their composition with that of the *in situ* phosphatic crusts. An overview of the samples analysed in this study is provided in Table 2.

2.1. Optical microscopy and micro-XRF

Micromorphological samples were air dried (i.e., no acetone replacement) and prepared at the Department of Geosciences of the University of Padova, following the methods of Murphy (1986) and using epoxy resin. The micromorphological description followed the terminology of Stoops (2021). Thin section analysis was done using in plane polarised light (PPL), cross-polarised light (XPL), and observing

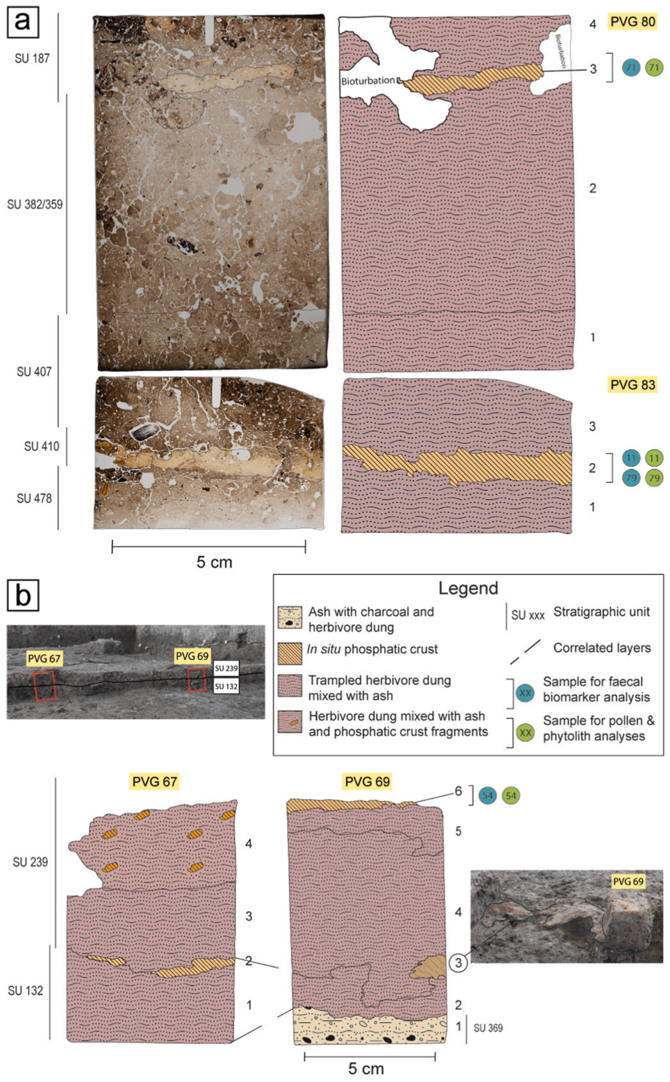


Fig. 4. Thin sections and related interpretation: a) internal accretion of the possible barn. Thin sections PVG 80 and PVG 83 include the *in situ* phosphatic crusts SUs 187 and 410, respectively; b) interpretation of thin sections PVG 67 and PVG 69 that include the patches of *in situ* phosphatic crusts associated with the SUs 239 and 132.

the autofluorescence when excited with blue light (BLF).

Micro-XRF mapping (Bruker M4 Tornado μ-XRF spectrometer, with a rhodium (Rh) X-ray source) was performed on impregnated blocks for thin section manufacturing or directly on uncovered thin sections (Mentzer, 2017). Elemental maps were collected at 50 kV and 200 μA with a vacuum (20 mbar = 2000 Pa). The analyses were conducted with a spot size of 20 μm and a spot distance of 200 μm. Two cycles were done for each map, with a dwell time of 15 mm/s for each measure. For micromorphology block 83, an additional map of a subarea of the sample was collected at 50 kV and 200 μA with a vacuum, a spot distance of 20 μm, and a dwell time of 10 mm/s for two cycles. Data processing was carried out using the Bruker M4 Tornado software.

Intact (i.e., not-powdered, not in chips or in thin sections) fragments of phosphatic crusts and reworked phosphatic fragments were observed without any preliminary treatment using a Keyence VHX7000 3D digital microscope. This microscope enabled three-dimensional imaging of the fragments at various magnifications (from 20× to 6000x), bridging the gap between macroscopic, micromorphological, and SEM observations.

2.2. Powder-XRD

Bulk samples of *in situ* phosphatic crusts and reworked phosphatic fragments were analysed using powder-XRD (p-XRD) for mineralogical identification. To minimise contamination from the greyish rim surrounding the orangish core, which was clearly visible to the naked eye (see below), samples from the reworked fragments were specifically drilled from the orangish portion (i.e., the phosphatic portion, see below) using a Dremel 2050 Stylo. In contrast, samples from *in situ* crusts were handpicked directly in the field and subsequently ground using an agate mortar, as they did not exhibit any macroscopically visible greyish rim above or beneath the crust. The analysis was conducted with a Philips X'Pert Pro diffractometer, equipped with a Co source (40 kV, 40 mA) and Bragg-Brentano parafocusing geometry. The instrument featured a Real-Time Multiple Strip (RTMS) X'Celerator detector. Samples were prepared on a 27 mm diameter circular, flat, zero-background metallic Si sample holder, with sample rotation facilitated by a Bragg-Brentano HD© spinner. Data were qualitatively analysed using High-Score (Plus) software (version 4.9, 2020; Degen et al., 2014) and semi-quantitatively using Rietveld refinement method with Topas v. 7 Bruker. Instrumental broadening profile was refined on Lanthanum boride (LaB_6) standard (National Institute of Standard and Technology). Protocol settings and additional information are provided in the Supplementary Materials (hereafter, 'SM'; SM1).

2.3. Micro-attenuated total reflectance (ATR) FTIR

Given the high spatial resolution of micro-ATR FTIR analysis, point analysis was performed directly on a thin section (PVG 83) to investigate the cementing phase occurring in the *in situ* phosphatic crusts. This allowed characterisation of this phase avoiding the contribution of sediment inclusions (i.e., silicates) and pedofeatures (e.g., secondary carbonates) that were difficult to separate in bulk analysis (i.e., p-XRD). Spectra were collected with a Bruker Hyperion II FTIR microscope equipped with a liquid-nitrogen-cooled LN-MCT detector and a 20x ATR objective with a germanium crystal. For each point analysis 32 scans were acquired, ranging from 4000 to 600 cm^{-1} and with a spectral resolution of 4 cm^{-1}. The analysis spot size is 32 μm. Spectral analysis was performed with the Opus software (Bruker).

2.4. SEM-EDS

To examine the sample texture and characterise the elemental composition of *in situ* phosphatic crusts and reworked phosphatic fragments at higher magnifications than those achievable with micro-XRF, intact fragments were analysed by SEM, using a FEI-Inspect F coupled with an energy dispersive spectrometer (EDS) INCA X-Act. The samples were coated with 5 nm of gold, and images were produced using a back-scattered electron (BSE) detector. EDS data were collected at 25 keV and quantified using Oxford Instruments INCA software.

2.5. Faecal biomarker analysis (GC-MS)

Six samples of phosphatic crusts were analysed using GC-MS to detect traces of faecal steroids from the lipid fraction. The yellow/orange material of interest was separated from the cemented greyish area of the dry samples (see §3.1) using a Dremel 2050 Stylo, yielding approximately 1–1.5 g of fine powders. The analytical procedure for faecal biomarkers analysis followed the methodology proposed by Battistel et al. (2015), with modifications outlined in Bortolini et al. (2024). The concentrations of all steroids were reported in ng g^{-1} or as a percentage of the total amount of steroids analysed ($\sum_{tot}$ steroid), in order to standardise the data. The comparison of the relative abundances of steroids was performed using a Student's t-test, after verifying normality and variance homogeneity with the Shapiro–Wilk and Fisher tests, respectively. The chosen significance level was $\alpha = 0.05$.

Table 2
Comprehensive list of samples analysed in this article. For each sample, the type (i.e., *in situ* phosphatic crust/reworked phosphatic fragment), the stratigraphic unit (SU), and the analyses conducted are specified.

General information				Analysis							
Type	Thin section	SU	Bulk	Micromorphology	3D digital microscopy	Micro-XRF	P-XRD	Micro-ATR FTIR	SEM	Microbotany	Faecal biomarker analysis
***In situ* phosphatic crust**	**PVG 69**	239	Bulk 54	●	●	●	●			●	●
	PVG 69	132	Bulk 55	●	●	●					
	PVG 80	187	Bulk 71	●	●	●	●			●	●
	PVG 83	410	Bulk 78, Bulk 79	●	●	●	●	●	●	●	●
	PVG 84	410	Bulk 11	●	●	●				●	●
	PVG 85	410	/	●	●	●					
Reworked phosphatic fragment	**PVG 64**	245	Bulk 84	●	●	●			●		
	PVG 65_1	132	Bulk 85	●	●	●	●			●	●
	PVG 65_2	132	Bulk 86	●	●	●	●		●		
	PVG 66	132	/	●		●					
	PVG 73	184	/	●		●					
	PVG 74	184	Bulk 61	●	●	●	●			●	●
Control samples	/	187	Bulk 70							●	
	/	359	Bulk 72							●	
	/	185	MF248							●	

2.6. Palynological analysis

For palynology, six subsamples of *in situ* phosphatic crusts (SUs 410, 187, 239) and two reworked phosphatic fragments (from SUs 132, 184) were processed (Table 2). A few grams (2.6–0.6 g) of material were powdered and then processed following Dal Corso (2018), modified without HF treatment and with addition of SPT-heavy liquid separation (density 2.35 g/cm^3). Microcharcoals between 10 and 250 μm were counted on a sum of 100 *Lycopodium* spores. Counting was carried out with a Zeiss Axiolab microscope at 400x and at 630x magnifications. Counts reached 300 pollen grains from terrestrial plants; when preservation was poor, counts reached the correspondence in pollen of 100 *Lycopodium* spores. Pollen percentages were calculated on total terrestrial pollen, excluding wetland plant pollen, pollen massulae, unidentifiable corroded grains, and fern spores, for which relative values were obtained on total terrestrial pollen plus the excluded *taxon* itself. Pollen massulae of multiple grains of the same pollen type were counted as 'one' since often the exact number of grains was not discernible. Standard pollen atlases (Reille, 1992, 1995, 1998) and pollen keys (Beug, 2004; Moore et al., 1991; for Chicorieae, see Florenzano et al., 2015) were used for identification.

2.7. Phytolith analysis

Nine samples (Table 2) were processed for siliceous phytolith analysis, including the same six samples prepared for palynology and GC-MS. Additionally, three sediment samples from the same stratigraphic sequence were studied for comparison: bulk 72/SU 359 (i.e., the deposit below the *in situ* phosphatic crust SU 187), bulk 70/SU 187 (i.e., the non-mineralised sediments above the crusts), and MF248/SU 185 (i.e., a unit representing a conflagration event that burnt a concentration of broomcorn millet grains within the barn and destroyed the structure). Sample processing followed Madella et al. (1998), with adaptations for calculating the acid-insoluble fraction (AIF) as in Lancelotti (2018). The AIF is a parameter used for phytolith concentration after Albert et al. (1999). Counts of morphotypes were performed at 400x magnifications in temporary slides, a minimum of 350 identifiable phytolith cells per sample were counted; multicellular aggregates (i.e., silica skeletons) were counted separately. Phytolith nomenclature, taxonomic and anatomical attributions follow ICPN 2.0 (International Committee for Phytolith Taxonomy, 2019) and references in SM8.

3. Results

3.1. Field evidence

In the field, the *in situ*, laterally continuous, phosphatic crusts SUs 187 and 410 appeared as orangish (10 YR 6/8, brownish yellow) hard concretions, delimited by an alignment of postholes. These crusts extended beyond the excavation limits, covering an area of approximately 6.50 m^2. Their thickness ranged from 2 to 4–5 cm, with a sturdy, compact texture (SM2). The surface of the crusts consists of irregular, pluricentimetric polygons separated by 1–2 mm-wide fissures (Fig. 5a). Phosphatised wood fragments and animal bones can be cemented within the crusts (Fig. 5b). Elsewhere in the excavation, other smaller patches of *in situ* phosphatic crusts were identified (i.e., SUs 132, 239). These features shared similar thickness, colour, and hardness with the laterally continuous crusts, though they typically measured no more than 30 cm in size and had irregular shapes in planar view (Fig. 4b).

The reworked phosphatic fragments, instead, are platy aggregates (Fig. 5c–e), likely resulting from compaction or trampling. These fragments are found within ash-rich layers and are associated with pottery shards, animal bones, coprolites, and phosphatised wood fragments. They are hard and compact, making them easy to handpick during excavation. Macroscopically, the fragments have an orangish core (10 YR 6/8, brownish yellow), similar to that of the *in situ* phosphate crusts, and a whitish/greyish coating (10 YR 6/2, light brownish grey) that cements ash, charcoal fragments, and mm-sized phosphatised wood fragments (Fig. 5c–e). This distinct coating helps differentiate them from coprolites, which typically also have a different morphology (cf. Shillito et al., 2020).

3.2. Geoarchaeological analyses (p-XRD, micro-ATR FTIR, micromorphology, micro-XRF, SEM-EDS)

The p-XRD spectra from *in situ* phosphatic crusts exhibit sharp Bragg peaks related to crystalline materials and broad diffraction features, indicative of nanocrystalline or amorphous phases. Specifically, the broad peaks were associated with the presence of nanocrystalline and possibly non-stoichiometric hydroxylapatite, which constitutes the primary cementing phase of the crusts (Fig. 6; Table 3, and SM3 report the results of the Rietveld refinement and a semi-quantitative estimation of mineralogical phases). The background modulation displays large humps, suggesting the presence of an amorphous phase, likely

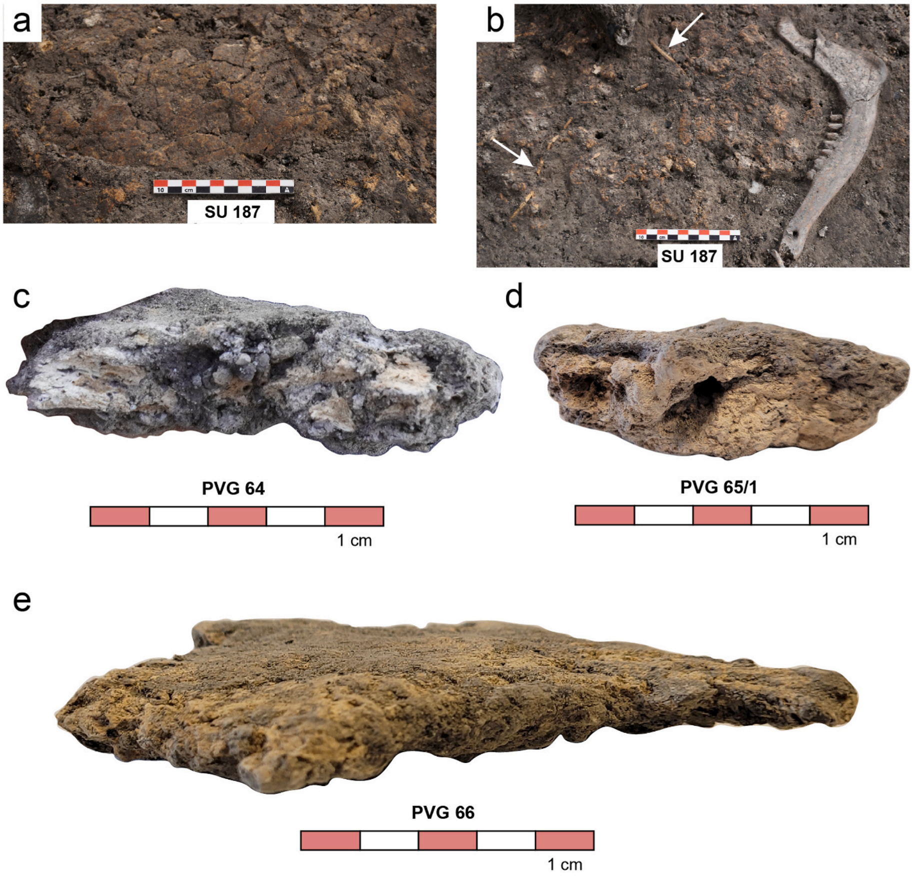


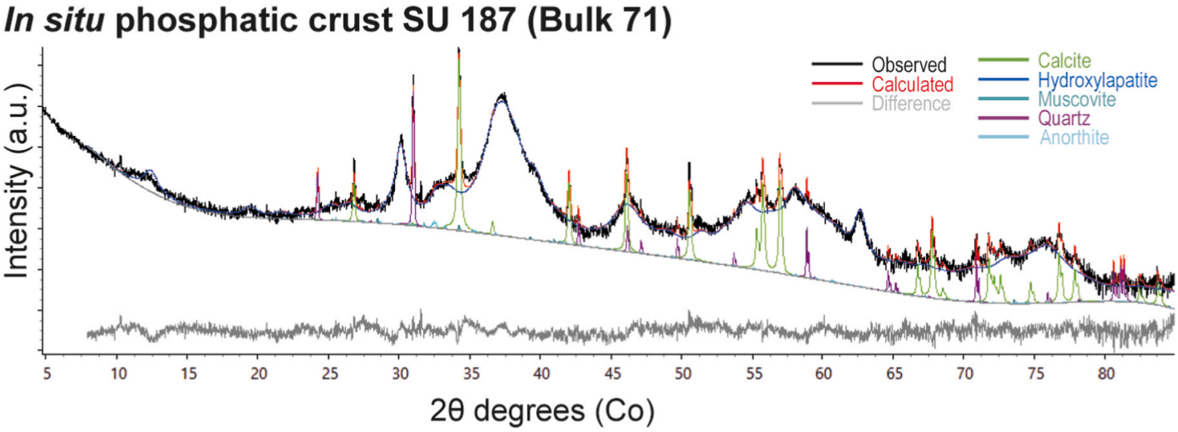
Fig. 5. Details of *in situ* phosphatic crusts and photographs of reworked phosphatic fragments: a-b) field photograph of the *in situ* phosphatic crust SU 187. In ‘a’, notice the polygonal structure characterising *in situ* phosphatic crusts, while in ‘b’ the presence of mineralised wood twigs (arrows) and of a bovid mandible associated with the crust; c-e) field photographs of reworked phosphatic fragments. Notice the presence of a whitish cemented material (calcite and phytoliths) coating the yellowish (hydroxylapatite) portion area.

associated with opal phytoliths, which are found in high concentrations within the crusts (see §3.5). In addition to hydroxylapatite, other mineralogical phases are present: mainly calcite, but also quartz, feldspar, phyllosilicates (e.g., mica, biotite, clinochlore), dolomite, and amphibole. While calcite derives from faecal spherulites, limestone fragments, and post-depositional processes (see below), the other mineral phases are related to geogenic sediments cemented within the crust and/or contamination from the surrounding deposits (see below).

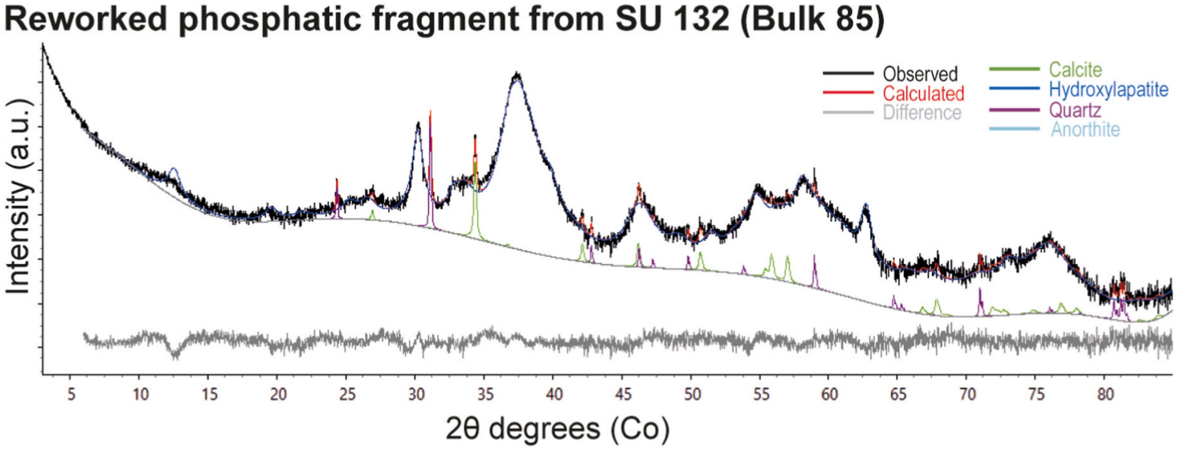
Micro-ATR FTIR analysis allowed for a better characterisation of the hydroxylapatite cement composing the *in situ* phosphatic crusts. The analysis was performed on different points on the thin section (Fig. 7a–c): i) A reference spectrum was obtained from a void filled with epoxy resin that embeds the entire thin section (point 1) to account for its signal, which partially overlaps with other phases in subsequent analyses; ii) A reference spectrum from a micritic grain (point 2) to record the signal of calcite; iii) A point analysis from an area of the phosphatic crust enriched in secondary calcite (point 3), as revealed by micromorphology; iv) Seven point analysis on the phosphatic crust (points 4 to 10).

The spectrum from the epoxy resin (point 1) revealed characteristic vibrational bands associated with hydroxyl groups, amines, and aromatic rings (Fig. 7d; Pannico et al., 2024). The spectra from points 2 and 3 exhibited the distinctive absorption peaks of calcite, with major bands at approximately 1400, 870 and 712 cm^{-1} (Fig. 7e; Dal Sasso et al., 2016) and broad absorption bands in the regions 1100-1000 and 790-770 cm^{-1} that can be reasonably associated to Si-O vibrational modes in clay minerals and/or phytoliths (Madejová and Komadel, 2001). Spectra from the phosphatic crust (points 4 to 10) exhibited absorption bands attributable to the epoxy resin, superimposed with additional bands. To separate the contribution of the resin from that of the sample, spectra were baseline-corrected and normalized (see SM4 for further details). Prominent absorption bands detected between 900 and 1150 cm^{-1} can be mostly attributed to the vibrational modes of phosphate groups in an apatitic environment (Fig. 7d), although possible overlapping with Si-O vibrational modes may occur (see SM4). Absorption at 870 cm^{-1} and in the range 1400–1490 cm^{-1}, which exceeds that of the epoxy resin, can be assigned to carbonate groups (Fig. 7d). The second derivative analysis of the band at 870 cm^{-1} for these spectra reveals its splitting and shift towards higher wavenumbers with respect to the signal of calcite, peaking at 872 and 880 cm^{-1}. These frequencies are characteristic of the vibrational modes of carbonate ions substituting in the hydroxylapatite structure, indicating a partial carbonation of the hydroxylapatite phase, as observed for biogenic apatite and synthetic analogues (Carmona et al., 2020; Scaggion et al., 2024).

In thin section, *in situ* phosphatic crusts (SUs 132, 187, 239, 410) consist of a yellowish cement that is optically isotropic (XPL) and autofluorescent (BLF; Fig. 8a–d). These characteristics align with the identification of hydroxylapatite. The hydroxylapatite cement consists of subrounded aggregates of nanocrystals, as observed by SEM (BSE) and with the 3D digital microscope (Fig. 9 and SM5) and confirmed by p-XRD analysis. It contains numerous phytoliths, with a strongly expressed horizontal orientation, and pseudomorphic articulated botanical remains composed of hydroxylapatite (Fig. 8a, c, and Fig. 9). Faecal spherulites are typically very rare and distributed randomly. Mineral inclusions such as quartz and mica are sporadic. In the crust,



In situ phosphatic crust SU 187 (Bulk 71)



Reworked phosphatic fragment from SU 132 (Bulk 85)

Fig. 6. XRD spectra. Above, XRD spectrum of the *in situ* phosphatic crust SU 187 (bulk 71) and below the spectrum of a reworked fragment from SU 132 (bulk 85).

Table 3

Relative abundance (wt%) of the minerals identified via p-XRD in all the samples presented in this study. The XRD spectra not shown in Fig. 6 are available in SM3.

Type	Stratigraphic unit	Sample	Hydroxylapatite [$Ca_5(PO_4)_3OH$]	Calcite [$CaCO_3$]	Quartz [SiO_2]	Feldspar [(K,Na,Ca)(Al, Si)$_4$O$_8$]	Phyllosilicates [$XY_{2\text{-}3}Z_4O_{10}(OH, F)_2$]	Dolomite [CaMg (CO$_3$)2]	Amphibole [$A_{0\text{-}1}B_2C_5T_8O_{22}(OH, F)_2$]	Diopside $MgCaSi_2O_6$
***In situ* phosphatic crust**	239	Bulk 54	50.3	19.4	1.8	5.0	1.7	4.6	1.0	–
	187	Bulk 71	85.9	10.6	2.9	0.4	0.2	–	–	–
	410	Bulk 78	54.1	31.5	10.6	1.9	1	0.5	–	0.4
Reworked phosphatic fragments	184	Bulk 61	90.1	4.9	3.4	0.8	0.5	0.3	–	–
	132	Bulk 85	95.8	2.2	1.9	0.1	–	–	–	–
	132	Bulk 86	88.7	8.5	1.9	0.9	–	–	–	–

pseudomorphic voids after vegetal remains and vertical planar voids are present, sometimes with sparite coatings (Fig. 8c). Both the upper and the lower boundaries of the *in situ* phosphatic crusts are abrupt, marked by a lack of phosphatic cementation (Figs. 8 and 10).

Above and beneath the crusts, articulated phytoliths are frequent. They lie horizontally in superimposed rows, and show evidence of snapping (Fig. 8e and f). Phytoliths are associated with dispersed faecal spherulites and microsparite. In general, above and beneath the crusts, faecal spherulites are more frequent than within the crusts, as well as wood ash crystals. Wood ash is always present in the deposits in which phosphatisation processes are documented at La Muraiola di Povegliano. Localised areas are cemented by calcite in the form of hypocoatings and various generations of micrite and sparite coatings and infillings (Fig. 8g and h). This cementation tends to be more frequent or intense beneath the crusts.

Micro-XRF mapping confirms the micromorphological data (Fig. 10 and SM3). Micro-XRF maps do not show a clear gradient in the concentration of phosphorus (P) within the crusts. However, in the case of SU 410, the concentration is higher at the base of the crust and slightly decreases towards the top (Fig. 10g–i). Interestingly, the silicon (Si) signal within the crusts is extremely low compared to the surrounding groundmass, which contains frequent articulated phytoliths and common silicate minerals (see below). In contrast, the signal for sulphur (S), likely derived from the vegetal material that originally composed the

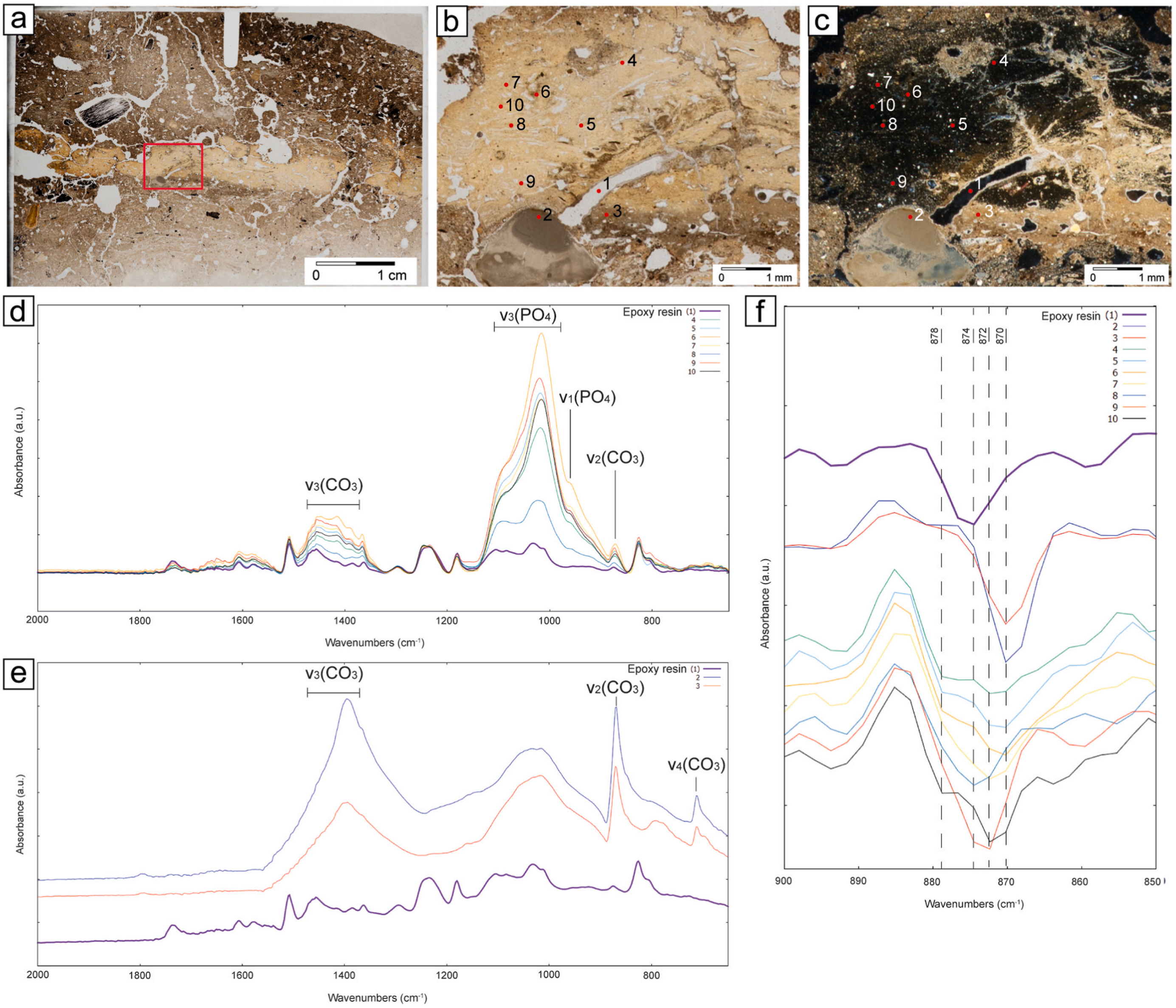


Fig. 7. Micro-ATR FTIR spectra: a) scan of thin section PVG 83, showing the *in situ* phosphatic crust and the locations of 'b' and 'c' (rectangle); b-c) Detailed views of (a) in PPL and XPL, indicating the sampling points and their corresponding numbers; d) baseline-corrected ATR-FTIR spectra, normalized against the epoxy resin spectrum (1), of point analysis in the phosphatic crust (4–10); major vibrational modes of phosphate and carbonate groups are highlighted; e) raw ATR-FTIR spectra of the epoxy resin (1), of a micritic grain (2) and of a portion of phosphatic crust enriched in calcite (3); vibrational modes of carbonate ions within the calcite structure are highlighted; f) second derivative of the ATR-FTIR spectra in the region 900-850 cm -1, showing the position of maximum absorbance (minimum of the second derivative) of vibrational bands related to the ν_2 (CO_3) mode for the point analysis in calcite-enriched areas (2–3) and in the phosphatic crust (4–10). For comparison, the second derivative of the epoxy resin spectrum (1) is also reported.

crusts (cf. Nicosia et al., 2022), shows the opposite trend.

Above and beneath the crusts, the prevailing chemical signal is Si together with the typical elements in silicates (e.g., Al, Fe, K, and Ca). This confirms that the quartz, feldspar, and phyllosilicates identified through p-XRD mostly originate from contamination from the surrounding sediments. The signal of P, instead, is nearly absent or very low in the silicates-rich areas compared to the crusts. Localised increase in the signal of Ca is documented in close association with the secondary carbonates mentioned above.

Reworked phosphatic fragments feature an orangish core exhibiting characteristics similar to those of the *in situ* phosphatic crusts (Figs. 6 and 8i–l; see also SM3 and SM5). Chemical and mineralogical analyses confirm the presence of hydroxylapatite in the orangish core of the fragments. Conversely, XRF maps clearly show the dominance of Ca and the absence of P in the whitish coating/greyish coating around the core (SM3). Micromorphology reveals that this Ca signal derives from the cementation by calcium carbonate of the surrounding groundmass, which often contains horizontally aligned phytoliths arranged in multiple superimposed rows (Fig. 8k and l). These features closely resemble the deposits associated with the *in situ* phosphatic crusts (Fig. 8g and h). Considering p-XRD data, reworked phosphatic fragments exhibit a considerably lower presence of calcite and silicates compared to the *in situ* crusts (Table 3).

3.3. Faecal biomarker analysis

In all the samples analysed via GC-MS, the most abundant compounds are 5β-stigmastanol (25% ± 10) and epi-5β-stigmastanol (63% ± 15), while the concentrations of the other investigated steroids, in particular phytosterols (i.e., campesterol, stigmastanol and β-sitosterol),

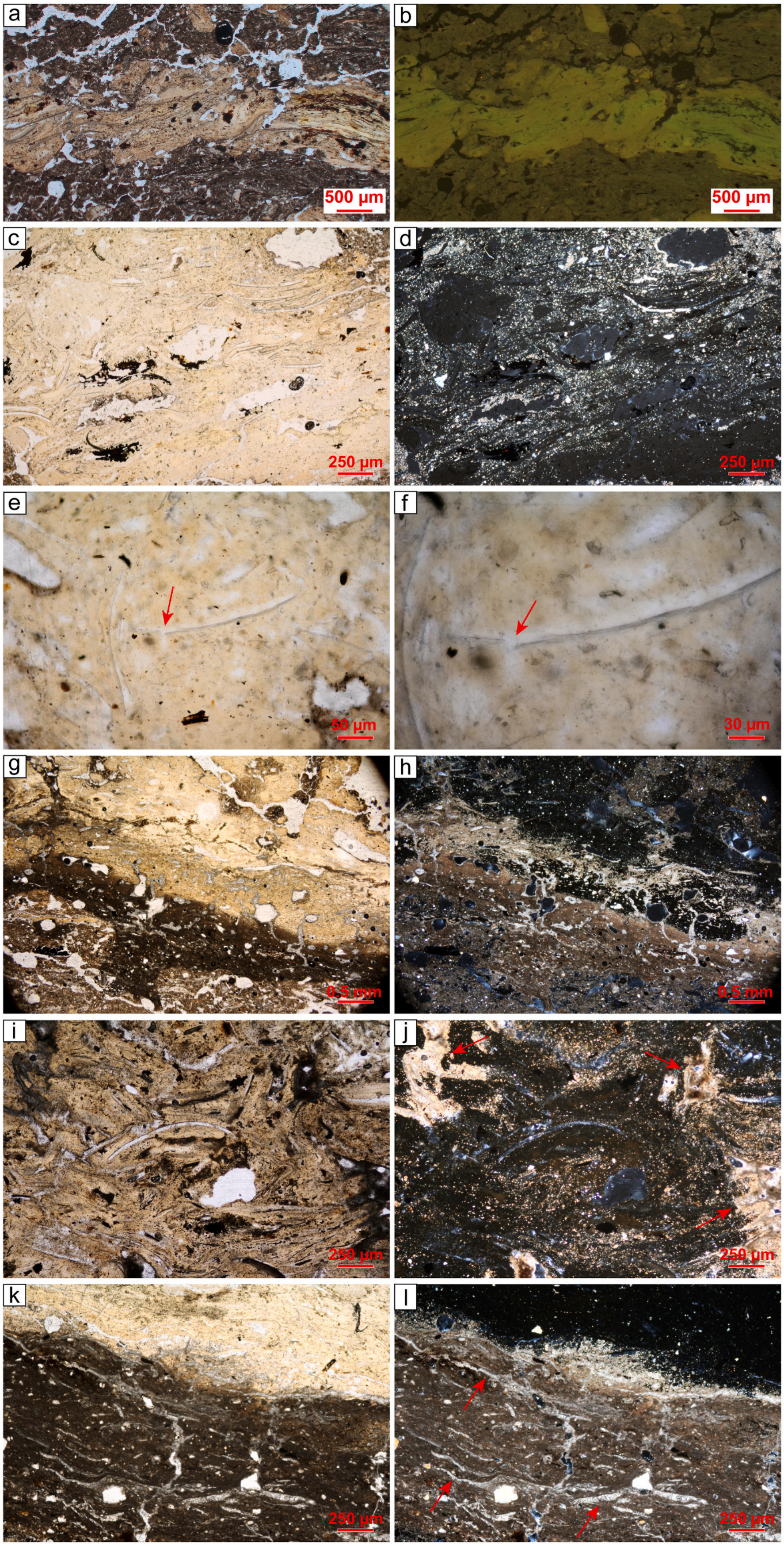


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Fig. 8. Photomicrographs of *in situ* phosphatic crusts and reworked phosphatic fragments: a) sample 67, SU 132: *in situ* phosphatic crust showing numerous horizontally-lying articulated phytoliths, PPL; b) same as 'a', but BLF. Notice the strong autofluorescence of the phosphatic crust when excited with blue light; c) sample 80, SU 187: detail of an *in situ* phosphatic crust. Notice the yellowish phosphatic cement (hydroxylapatite) as well as the abundance of phytoliths and pseudomorphic voids oriented parallel to each other, PPL; d) same as 'c', but XPL. Notice that the hydroxylapatite cement is optically isotropic; e) sample 85, SU 410: snapped articulated phytoliths embedded in a phosphatic cement; f) detail of 'e', PPL; g) sample 83, SU 410: cementation of calcite beneath an *in situ* phosphatic crust, PPL; h) same as 'g', but XPL. Notice the presence of coatings and infillings of calcite within planar voids and vughs; i) reworked phosphatic fragment PVG 65-2, SU 132: hydroxylapatite micromass containing numerous horizontally-lying articulated phytoliths and pseudomorphic voids (cf. 'a'), PPL; j) same as 'i', but XPL. Notice the presence of calcite hypocoatings and infillings (arrows); k) reworked phosphatic fragment PVG 66, SU 132: detail of the deposit cemented by calcium carbonate that is attached to the reworked phosphatic fragment (cf. 'e'), PPL; l) same as 'k', but XPL. Notice the presence of infillings of calcite within planar voids (for example, see arrows; cf. 'h'). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

when higher than the method detection limit (MDL), are not remarkable (see SM6). The low concentration of phytosterols is partially due to the higher methodological limit of quantification encountered for Δ^5-sterols in these analyses (as reported in SM6). Nevertheless, when quantifiable (e.g., in sample bulk 79/SU 410), the concentration of β-sitosterol was about 6 and 17 times lower than those of 5β-stigmastanol and epi-5β-stigmastanol, respectively. Considering the large interspecies and individual variability in modern mammal faeces, these findings are consistent with the literature (Bortolini et al., 2024; Prost et al., 2017). Furthermore, the low amount of β-sitosterol may also result from post-depositional microbial degradation to 5α-stigmastanol (Bull et al., 2002), which can reduce β-sitosterol levels in the samples. Among 5β-zoostanols, no statistically significant differences are detected between the samples related to *in situ* phosphatic crusts and reworked phosphatic fragments for 5β-stigmastanol, epi-5β-stigmastanol, and coprostanol, as demonstrated by the Student's t-test which provided p-values of 0.61, 0.75, and 0.27, respectively. Only epi-coprostanol values are slightly lower in *in situ* phosphatic crusts compared to reworked phosphatic fragments ($p\text{-value} = 0.021$). It has been demonstrated that 5β-stigmastanol and epi-5β-stigmastanol in faeces are indicative of a plant-based diet typical of herbivores (Herrera-Herrera et al., 2024; Lerch et al., 2022; Vázquez et al., 2021) as well as a vegan or vegetarian human diet (Aries et al., 1971; Reddy et al., 1998). In general, 5β-stigmastanol tends to be more abundant than epi-5β-stigmastanol (Bortolini et al., 2024; Fernández-Palacios et al., 2024). The ratio R:

$$R = \frac{coprostanol + epi - coprostanol}{coprostanol + epi - coprostanol + 5\beta - stigmastanol + epi - 5\beta - stigmastanol}$$

has been proposed to distinguish a plant-based diet from an omnivore/carnivore diet (Bortolini et al., 2024), where $R < 0.29$ is typically associated with herbivores, whereas $R > 0.65$ characterises humans with a mixed-diet. In our study, R ranges between 0.02 and 0.14 (Fig. 11c), which is consistent with a plant-based diet typical of herbivores (such as ovicaprids, cattle or equines). Although a vegan or vegetarian human diet cannot be fully excluded based on these results, contextual data and available literature on human diet in the northern Italian Bronze Age (e.g., Varalli et al., 2022, 2016) suggest that this hypothesis is unlikely.

In addition, the ratio epi-5β-stigmastanol/5β-stigmastanol determined in the phosphatic crusts analysed in this work (2.9 ± 1.3) is significantly higher ($p\text{-value} < 0.05$) than those obtained in modern herbivore dung (0.6 ± 0.4; see Bortolini et al., 2024) (Fig. 11). The only exception is the *in situ* phosphatic crust SU 239 (bulk 54/SU 239), that shows an epi-5β-stigmastanol/5β-stigmastanol ratio of 0.8 (Fig. 11). The fact that in most of the samples the epi-5β-stigmastanol/5β-stigmastanol ratio is higher than in modern herbivore dung may be explained considering an *in situ* epimerisation of 5β-stigmastanol under anaerobic environmental conditions (Lerch et al., 2022, p. 1142; von der Lühe et al., 2018, p. 233), whereas the lower value in the sample bulk 54/SU 239 (i.e., 0.8) may reflects a slower rate of epimerisation.

Since 5β-stigmastanol and epi-5β-stigmastanol are mainly produced in the gut of mammals and, in larger amounts, in herbivores, faecal biomarker analysis clearly suggests that the source of steroids in the phosphatic crusts is attributable to herbivores. However, the unambiguous identification of the particular species or families is not possible through faecal steroids.

3.4. *Palynological analysis*

Pollen preservation varied significantly, with concentrations ranging from a maximum of 37,711 pollen grains/g in bulk 11/SU 410 (*in situ* crust) to a minimum of 3582 pollen grains/g in bulk 61/SU 184 (reworked phosphatic fragment; Fig. 12a). Pollen counts were particularly low in samples from reworked phosphatic fragments (31 pollen grains in bulk 61/SU 184 and 65 in bulk 85/SU 132) and in one sample from an *in situ* crust (27 pollen grains in bulk 71/SU 187). The pollen diagram shows percentage values (Fig. 12c), while raw counts are provided in SM7.

Arboreal pollen is scarce (average 4.8%) in the record. Tree species typical of the hygrophilous riparian woodland, such as alder (*Alnus*) and willow (*Salix*), and to the mesophile floodplain woods, like deciduous oak (deciduous *Quercus* gr.) and hazel (*Corylus*), are represented. Another shrub, cornelian cherry (*Cornus mas*), is also attested in the *in situ* phosphatic crust SU 410 (bulk 11). Pine (*Pinus*) pollen, wind-pollinated and prone to long-distance transport, likely reflects a regional signal from the Alpine forelands. Cerealia-type pollen is ubiquitous, reaching values around 6.8% in the *in situ* phosphatic crusts and up to 16% in the reworked fragments. Synanthropic forbs, such as mugwort (Artemisia), plants of the goosefoot family (Amaranthaceae), and thistles (*Cirsium/Carduus*), have been found, indicating arable fields, fallowland, and disturbed ruderal areas. Notably, thistle pollen, with large grains and a thick and recognisable exine (Beug, 2004), occurs in all samples except bulk 61/SU 184. The most abundant pollen is that of Cichorieae (chicory tribe of the Asteraceae family; Fig. 12b), accounting for 38.7%–66% of the record, with the highest value in bulk 11/SU 410. Cichorieae produce easily recognisable pollen with echinate, fenestrate exine that in these samples falls in the size range of medium pollen grains (26–44 μm), attesting plants such as *Cichorium*, *Taraxacum*, *Sonchus* among others (Florenzano et al., 2015, Table 1). The good preservation of Cichorieae pollen is evidenced by intact grains (Fig. 12c, top). In bulk 11/SU 410 and bulk 54/SU 239, massulae of multiple Cichorieae grains derived from anthers were found. In the Mediterranean, Cichorieae pollen is often indicative of pastures and grazing pressure, particularly when associated with wild grass (Poaceae) pollen, which in this record ranges between 8% and 29% (Florenzano et al., 2015; Mercuri et al., 2013). In this study, additional pastoral indicators include pollen

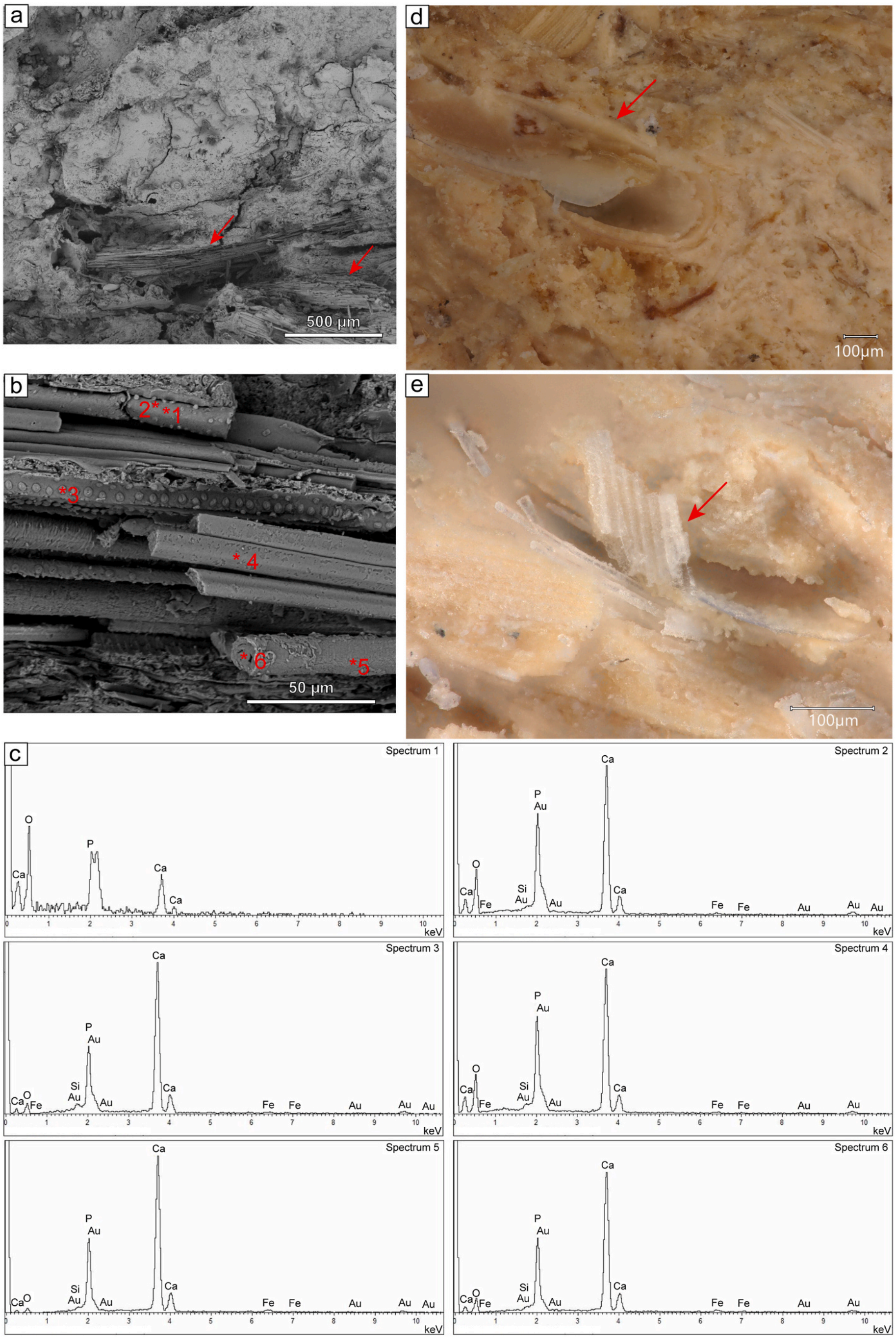

Fig. 9. SEM (BSE) images and 3D digital microscope photographs: a) SEM image showing an overview of a fragment of an *in situ* crust (bulk 79/SU 410). Notice the presence of articulated botanical remains (arrows) embedded in a dense groundmass composed of hydroxylapatite; b) SEM image showing a detail of the articulated botanical remains from the same fragment of *in situ* phosphatic crust shown in ‘a’. The spots indicated with an asterisk were investigated by SEM-EDS (see ‘c’); c) EDS spectra of the spots indicated with an asterisk in ‘b’. The composition of all the spectra confirms the pseudomorphic substitution of hydroxylapatite in vegetal tissues; d) optical image of hydroxylapatite groundmass embedding aggregates of phytoliths (arrow) from an *in situ* phosphatic crust (bulk 55/SU 132); e) optical image taken from an *in situ* phosphatic crust fragment (bulk 71/SU 187), where hydroxylapatite groundmass associated with articulated phytoliths and pseudomorphic substitution of hydroxylapatite after vegetal tissues and organs (arrow) can be observed.

from common knapweed (*Centaurea nigra* type, average 5.3%), plantain (*Plantago lanceolata* type) and meadow buttercup (*Ranunculus acris* type), attested in the *in situ* phosphatic crusts, and alfalfa (*Medicago* type). Plants typical of calcareous dry grasslands, such as white laceflower (*Orlaya grandiflora*; Fig. 12b), small scabious (*Scabiosa columbaria*), and rockrose (*Helianthemum*), are present in the *in situ* phosphatic crusts and reworked phosphatic fragment bulk 61/SU 184, further reflecting grazing pressure in open habitats (Perego et al., 2011). The pollen of other plants of the Apiaceae (carrot family), Fabaceae (pulses family), and Caryophyllaceae (pink family) that occur in different kinds of open environments is also present. Within *in situ* phosphatic crusts, massulae with several grains of Cichorieae, Apiaceae, and Scrophulariaceae (figwort family) have been found. Wetland plants are sparsely represented, with few pollen grains of sedges (Cyperaceae) in bulk 71/SU 187 and bulk 61/184, and waterwort (*Elatine*) in sample bulk 61/SU 184. The pollen of Cyperaceae is quite large and thin, and as a

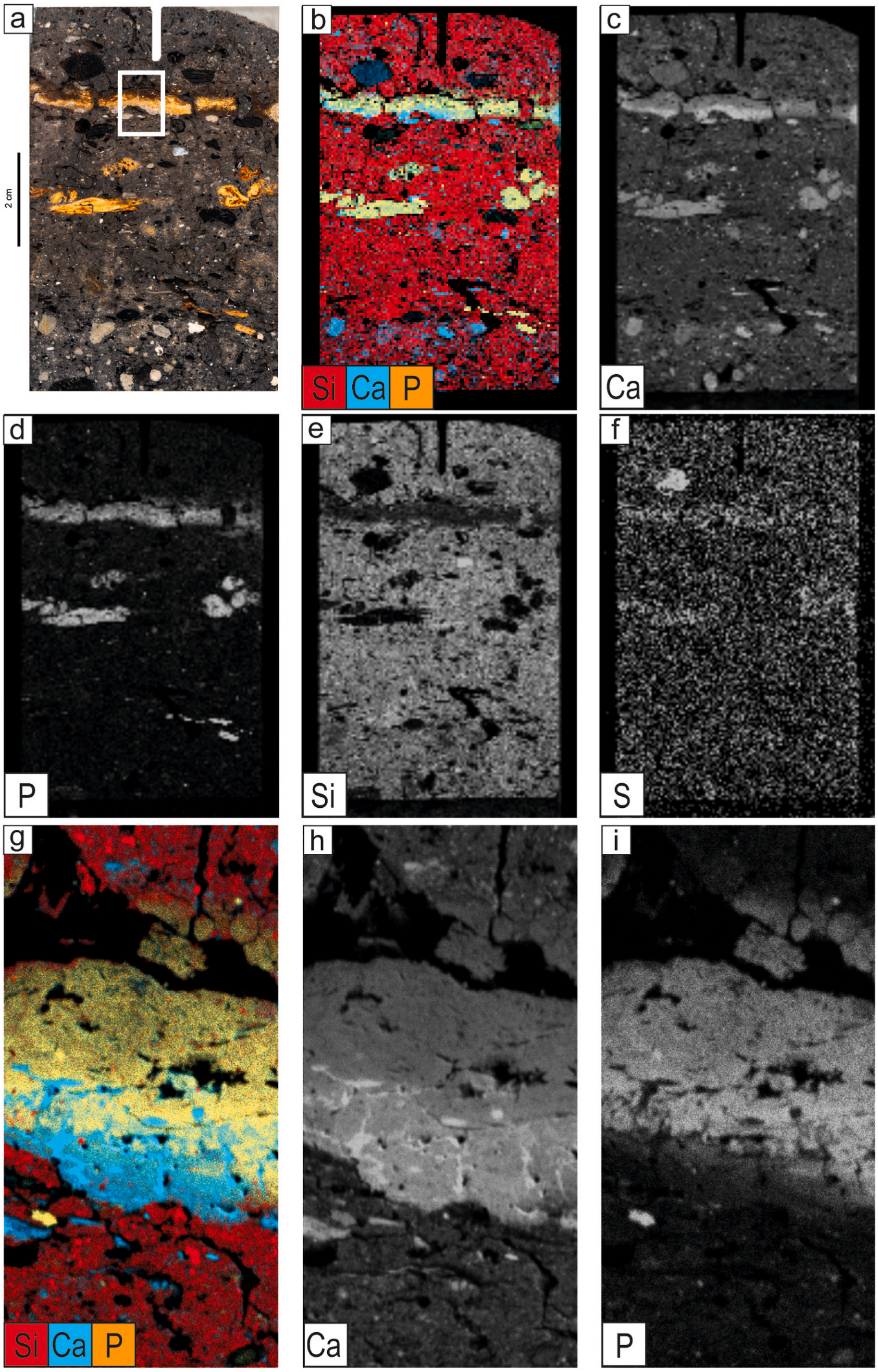

Fig. 10. Micro-XRF maps of the micromorphological block PVG 83, containing the *in situ* phosphatic crust SU 410: a) photograph of the block. The white rectangle indicates the position of the detailed micro-XRF maps of the *in situ* phosphatic crust SU 410 reported in (‘g’-‘i’); b) coloured micro-XRF maps showing the abundance of P, Si, and Ca; c-f) micro-XRF maps of specific elements. Notice the close relationship between the concentration of Ca and P in the crust and in the crust fragments located below and the slight enrichment in S within the crust with respect to the surrounding matrix; g-i) detailed coloured micro-XRF maps of the phosphatic crust SU 410, showing the abundance of P, Si, and Ca (‘g’) and of specific elements (‘h’-‘i’). Notice the significant presence of Ca below the phosphatic crust related to the calcite cementation observed in thin section.

result, it may not preserve well in dry contexts. Few monolete fern spores were found within the *in situ* phosphatic crusts.

3.5. Phytolith analysis

The analysed samples are extremely rich in phytoliths (Fig. 13a and b and SM8), with concentrations exceeding 5 million per g of AIF in all but the control sample bulk 71/SU 359. The highest concentrations, above 20 million per g of AIF, are recorded in bulk 11/SU 410 and bulk 71/SU187 from *in situ* phosphatic crusts.

The assemblage comprises 25 morphotypes of single-celled phytoliths, six morphotypes of multicellular phytolith aggregates, rare sponge spicules (in control sample bulk 72/SU 359) and diatoms (Fig. 13c; SM8). Phytoliths predominantly indicate grass (Poaceae) origins. The

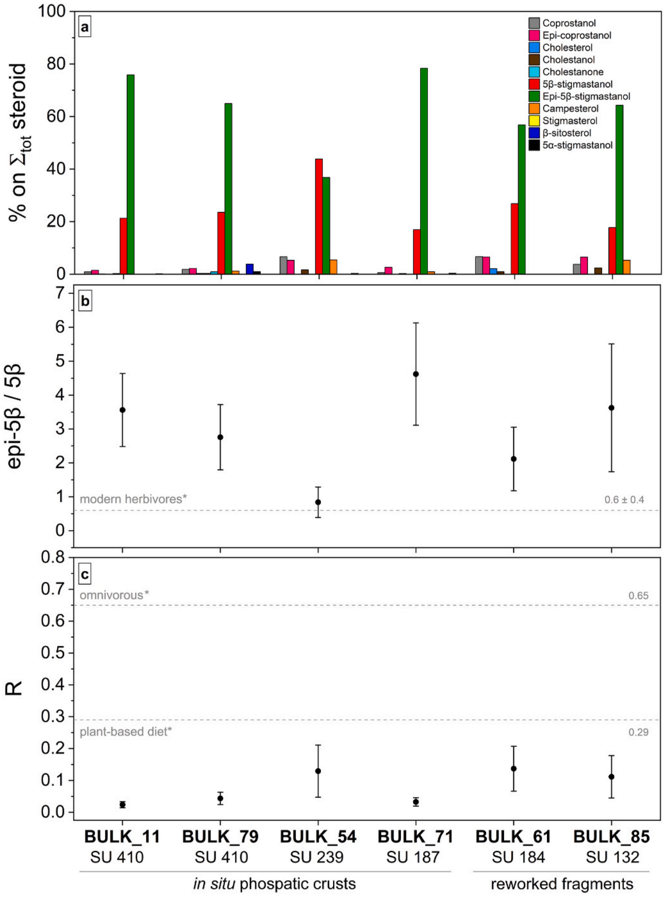

Fig. 11. Faecal biomarker analysis of *in situ* phosphatic crusts and reworked phosphatic fragments via GC-MS: a) percentage of the total amount of steroids analysed (S_{tot} steroid) in the six samples of phosphatic crusts; b) ratio between epi-5β-stigmastanol (epi-5β) and 5β-stigmastanol (5β) that is higher than in modern herbivore dung (0.6 ± 0.4; see Bortolini et al., 2024); c) ratio R that assumes values < 0.29 in all the six samples, showing a plant-based diet typical of herbivores.

group of GSSCP (Grass Silica Short Cell Phytoliths) consisting of rondels, trapezoid and crenate is ubiquitous in the record and is commonly found in grasses of the Pooideae subfamily, which includes C3 cereals such as barley and wheat (International Committee for Phytolith Taxonomy, 2019). Similarly, the GSSCP cross, bilobates, polylobate, usually linked to the Panicoideae sub family that includes C4 grasses and cereals such as millets, are also well-represented (International Committee for Phytolith Taxonomy, 2019). Another common GSSCP is saddle, typical of wild C4 grass such as *Cyanodon dactylion* of the subfamily of Chloridoideae. This subdivision is not strict and some of these types (e.g., saddle, bilobates, polylobates) can be found also in other Poaceae, such as common reeds (*Phragmites australis*; Guo et al., 2012; Ollendorf et al., 1988). Multicellular stacked bulliforms, indicative of leaves of common reed (see Ramsey et al., 2018, 2016), are observed in the *in situ* phosphatic crusts. Single bulliform flabellate and blocky types, characteristic of monocotyledon leaves (i.e., grasses and sedges in temperate environments), are frequent throughout the record. Frequent morphotypes derived from grass inflorescences, such as elongate dendritic, elongate dentate, papillate, andellipsoidal echinate type referring to hair base (Ball et al., 2016; Dal Corso et al., 2018), likely indicate pooid cereal chaff. This interpretation is supported by the presence of *Cerealia*-type pollen (see above) and charred macrobotanical remains (D'Aquino, 2024).

Interdigitating multicellular aggregates of millets (Laugier et al., 2022; Lu et al., 2009; Madella et al., 2016; Weisskopf and Lee, 2016) are also common. The latter has been found in control samples (well-represented in SU 185), in the adjacent SU 187, and in SU 132. elongate entire phytoliths, also as multicellular aggregates, are abundant and suggest grass culms/leaves, though they are not exclusive to grasses (Lancelotti and Madella, 2012). Few morphotypes represent dicotyledonous arboreal plants and forbs, such as multicell isodiametric cells (also known as honeycombed and jigsaw types) from dicot leaves (An and Xie, 2022; Ramsey et al., 2016), which occur in the *in situ* phosphatic crusts and reworked phosphatic fragments. Sedge-specific morphotypes (e.g., sedge-type cone) are very rare, although some elongate dentate and bulliform types can also derive from sedges (International Committee for Phytolith Taxonomy, 2019; Novello et al., 2012). Overall, the assemblage is dominated by grasses and is quite homogenous, even though multicellular aggregates prevail in the *in situ* phosphatic crusts and reworked phosphatic fragments relative to control samples. GSSCPs from C3 and C4 grasses, both wild and domesticated, are very frequent. However, saddle, particularly abundant in SU 410, indicates wild grasses and is associated with a reduced presence of cereal chaff within the crusts (Fig. 13c).

To further investigate the record and identify potential associations among samples, a correspondence analysis was performed (Fig. 14). The results show that control samples from SUs 185 and 187, along with the reworked phosphatic fragments, cluster with morphotypes form pooid and panicoid inflorescences (i.e., cereal chaff of pooid and panicoid cereals, and monocot leaves). This kind of material was likely stored, processed, and discarded during daily food preparation activities. The sample from the *in situ* phosphatic crust SU 187 is also in this cluster but closer to other millet/panicoid GSCCPs. In contrast, samples from crusts SUs 410 and 239, as well as the control sample from SU 359, scatter in another area of the plot. This area is associated with morphotypes from leaves of wild/domesticated monocots and wild dicots, suggesting a greater contribution of wild plant resources to the assemblage's formation. Morphotypes from sedges and common reeds are rather sparse.

4. Discussion

Micromorphological, mineralogical, chemical, and microbotanical data confirm that the hard, orangish crusts identified in the field resulted from *in situ* animal penning activities. This interpretation is supported by several lines of evidence.

1. The crusts contain faecal spherulites and a notable quantity of articulated phytoliths, which exhibit a strongly expressed horizontal distribution and parallel orientation. These features are consistent with the trampling of herbivore dung, possibly mixed with litter and/or forage material (Nicosia et al., 2022; Shahack-Gross, 2017);
2. Biomarker analysis reveals a significant faecal signal associated with mammals on a plant-based diet (i.e., herbivores), as evidenced by the presence of 5β-phytostanols. The signal of undigested vegetal remains (i.e., campesterol, stigmasterol, β-sitosterol), possibly linked to accumulations of fodder or bedding material, is extremely low, thereby refining the interpretation of the deposits as composed almost exclusively of trampled herbivore dung;
3. Botanical microfossils further support this interpretation. The extremely high concentration of phytoliths, both as single cells and in multicellular aggregates, is a typical signature of herbivore dung and dung-rich deposits (e.g., Dunseth et al., 2018; Égüez et al., 2020; Portillo et al., 2020, 2014), particularly when grasses are the primary component of the animals' diet. Moreover, palynological analysis indicates a dominance of pastoral indicators from open and disturbed habitats, including secondary dry grasslands, alongside pollen massulae derived from flower anthers. This evidence is in line with the interpretation of the material as herbivore dung (e.g., Dunseth et al., 2018; Perego, 2015).

The compositional similarities between *in situ* phosphatic crusts and reworked phosphatic fragments reveal that the latter are in fact

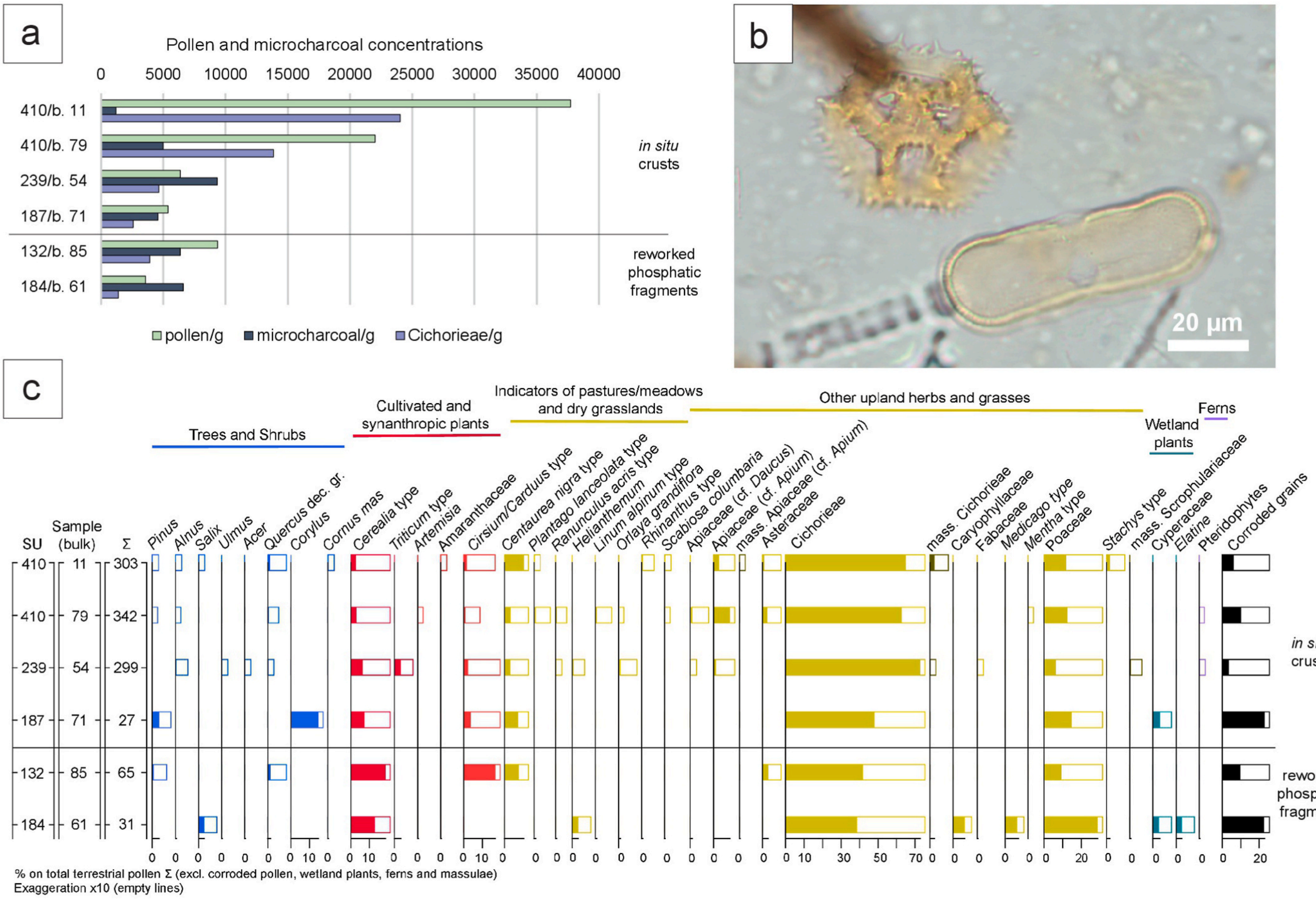

Fig. 12. The palynological record of the phosphatic crusts and reworked phosphatic fragments from La Muraiola di Povegliano: a) total pollen, Cichorieae pollen and microcharcoal concentrations; b) microphotographs of pollen grains of Cichorieae (top) and *Olarya grandiflora* (bottom); c) diagram of relative pollen percentage values.

reworked fragments of phosphatic crusts, likely resulting from the periodic cleaning of animal penning areas. The considerably lower presence of calcite and silicates in reworked fragments compared to the *in situ* crusts can be attributed to variations in sampling protocols: p-XRD samples from the fragments were drilled specifically from the orangish portion (i.e., the hydroxylapatite core), whereas *in situ* crust samples were handpicked in the field. As a result, the latter likely include contamination from overlying and underlying sediments, as well as from the secondary carbonates observed at the crust interfaces (see above).

Below, we summarise and discuss our main interpretations regarding: (1) the diagenesis of herbivore dung deposits leading to the formation of phosphatic crusts, and (2) the archaeological implications, with a focus on reconstructing past herding practices and providing insights for contemporary excavation practice.

4.1. Formation processes of phosphatic crusts and taphonomic considerations

Our evidence indicates that the phosphatic crusts identified at La Muraiola di Povegliano derived from compacted herbivore dung enriched in liquid animal waste (Brönnimann et al., 2017, pp. 62–63). This process is in line with previous knowledge on degradation of excrements that was expected to be valid also in animal penning deposits (Shahack-Gross, 2017, pp. 268–270; Shahack-Gross et al., 2004a). Herbivore excreta contains significant amounts of P and Ca that have not been absorbed in the digestive tracts of the animals. Phosphorus is present mainly in solid faeces, while urine contains significantly lower values of P (Christensen and Sommer, 2013). At La Muraiola, dissolved wood ash can be considered an additional source of P and Ca, as it is common in the stratigraphic units associated with phosphatic crusts and, more broadly, with phosphatised organic remains (Karkanas, 2021, pp. 25–26). Similarly, dissolved faecal spherulites can have provided an additional source of Ca (Canti and Brochier, 2017). Solid herbivore faeces also include large quantity of lignin, derived from the animals' diet (Foth and Turk, 1972, p. 330) and therefore contain significant amounts of S (cf. Nicosia et al., 2022).

In modern stable/byre deposits, the reaction between phosphate ions (PO_4^{3-}) in aqueous solution with Ca^{2+} has been documented, forming Ca-phosphates, such as apatite ($Ca_5(PO_4)_3$), hydroxylapatite ($Ca_{10}(PO_4)_6(OH)_2$), monetite ($CaHPO_4$), and brushite ($CaHPO_4 \cdot 2H_2O$) (Shashvatt et al., 2017, pp. 483–484; Christensen and Sommer, 2013, pp. 59–60). When animals are stalled in enclosed spaces, trampling and consequent compaction of the deposit accumulated on the floor can create anaerobic conditions (Foth, 1990, p. 243). These conditions are the ideal environment for microbial degradation of organic matter mediated in particular by sulphate-reducing bacteria (SRBs), several *taxa* of which have been identified in modern stable/byre deposits (Karnachuk et al., 2021; St-Pierre and Wright, 2017). Considering that the role of bacteria and, in particular, of SRBs in the precipitation of Ca-phosphates has been assessed in ancient and modern phosphorites deposits (Bailey et al., 2013; Crosby and Bailey, 2012) and in laboratory experiments (Hirschler et al., 1990a, 1990b; Lucas and Prévôt, 1985), it is likely that the process of Ca-phosphate precipitation can be also mediated by microbial activity (cf. Krajewski et al., 1994). A relevant factor affecting phosphate precipitation and transformation is pH (Secco et al., 2015, p. 59; Lindsay, 1979). In general, in recently deposited

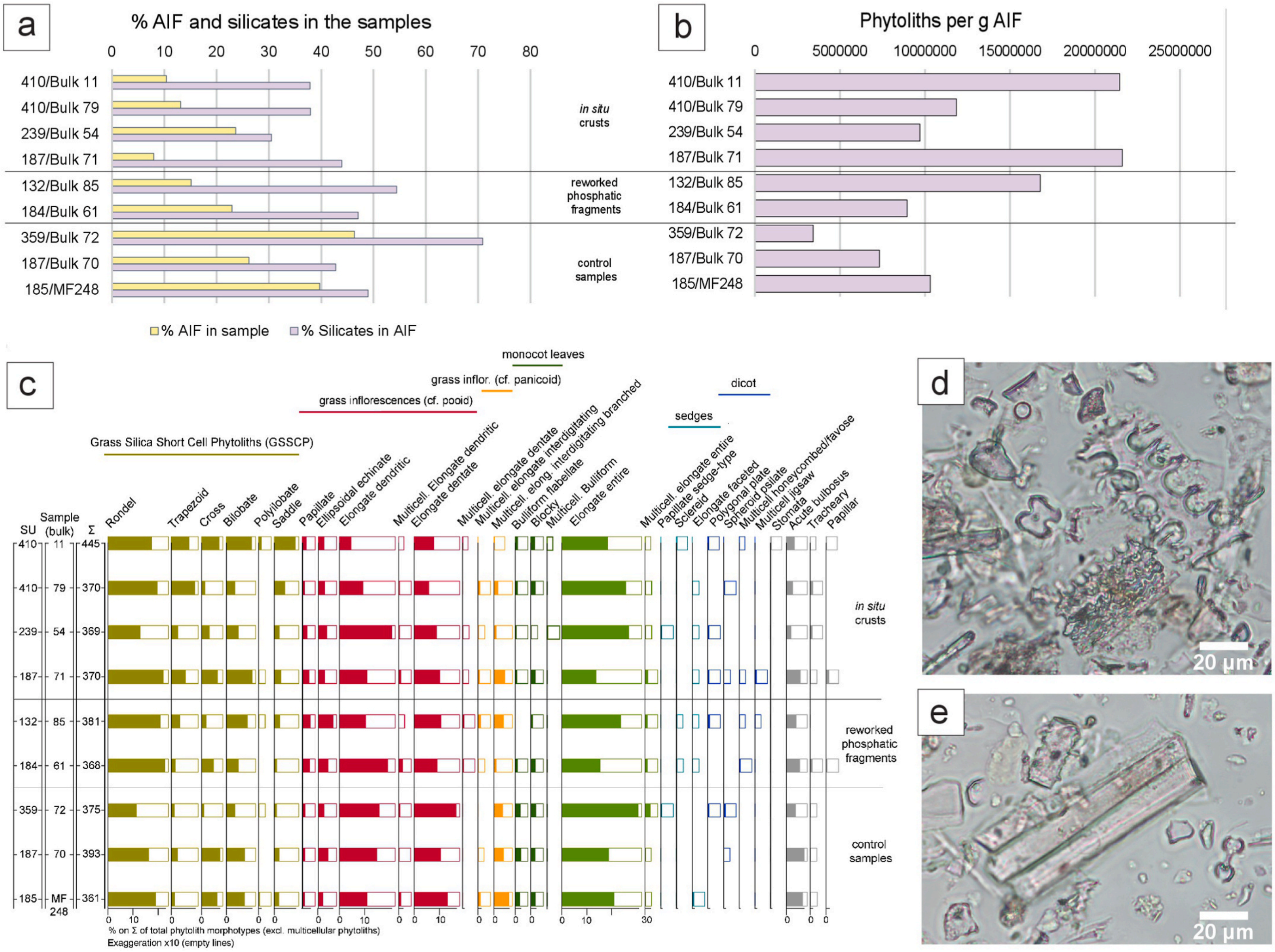

Fig. 13. The record of siliceous microfossils from the *in situ* phosphatic crusts, reworked phosphatic fragments, and contiguous control samples of La Muraiola di Povegliano: a) percentage of AIF in the sample and of silicates in the AIF; b) phytolith concentration per g of AIF; c) diagram with relative values of the morphotypes comprising the phytolith record; d) common morphotypes include BILOBATES, RONDELS and ELONGATE DENDRITIC; e) another common morphotype is ELONGATE ENTIRE (here as multicellular aggregate).

mixtures of animal faeces and urine pH is around 7.5, even though anaerobic conditions can contribute to the formation of organic acids, further lowering the pH. On the other hand, with time, the oxidation of the deposits leads to the degradation of organic matter and the decrease of the acids in solution, thereby increasing the pH (Christensen and Sommer, 2013, pp. 51–53). Above pH of 7.2, phosphates ions tend to precipitate as insoluble Ca-phosphate compounds that are slowly converted to apatite/hydroxylapatite through dissolution and reprecipitation processes (Schoumans, 2014; Foth, 1990, p. 199). Previous research demonstrated that brushite formation is favoured at pH lower than 6, while above 6 the formation of minerals of the apatite group is also favoured (Secco et al., 2015). Similarly, at pH above 7, monetite converts into hydroxylapatite, which is the most stable Ca-phosphate in neutral to slightly alkaline conditions (Prado Da Silva et al., 2001).

Similar processes to those documented in modern stables and byres likely took place at La Muraiola di Povegliano. The temporary occurrence of neutral to slightly acidic conditions during phosphatic crust formation is suggested by the dissolution of faecal spherulites, as evidenced by their significantly lower abundance within the crusts compared to the surrounding herbivore dung deposits (cf. Macphail et al., 2004). Rapid cementation by Ca-phosphates is indicated by the presence of phosphatised organic remains within the crusts. This aligns with observations from the experimental site of Butser Farm, where hard crusts formed annually on a byre floor after overwintering periods of cattle and ovicaprids (see Macphail and Goldberg, 1995, p. 5). A rapid precipitation process is also consistent with studies on modern phosphorites, where microbial activity has been demonstrated to play a key role (Bailey et al., 2013).

A subsequent local rise in pH is evidenced by the precipitation of secondary carbonates as micrite, hypocoatings, coatings, and infillings. This process, likely driven by the onset of oxidising conditions and the abundance of dissolved Ca^{2+} (i.e., from dissolved ash and faecal spherulites and circulating water), resulted in pedofeatures forming above and below the crusts. The presence of coatings and infillings within the phosphatic crusts indicates that hydroxylapatite precipitation preceded that of secondary carbonates. Further evidence of a pH rise derives from the identification of partially carbonated hydroxylapatite as the only Ca-phosphate (cf. Shahack-Gross et al., 2004a, p. 1266).

The rise in pH during the formation of phosphatic crusts is also likely responsible for the oxidation of pollen grains and spores, many of which are unidentifiable due to thinning of the exine and loss of diagnostic features (see ‘corroded grains’ in Fig. 12c). Consequently, the very high percentage values of pollen types with thick exine and easily recognisable grains, such as Cichorieae and *Cirsium/Carduus* type, could lead to their overrepresentation in the relative abundance record. This taphonomic bias, however, does not deny the relevance of the

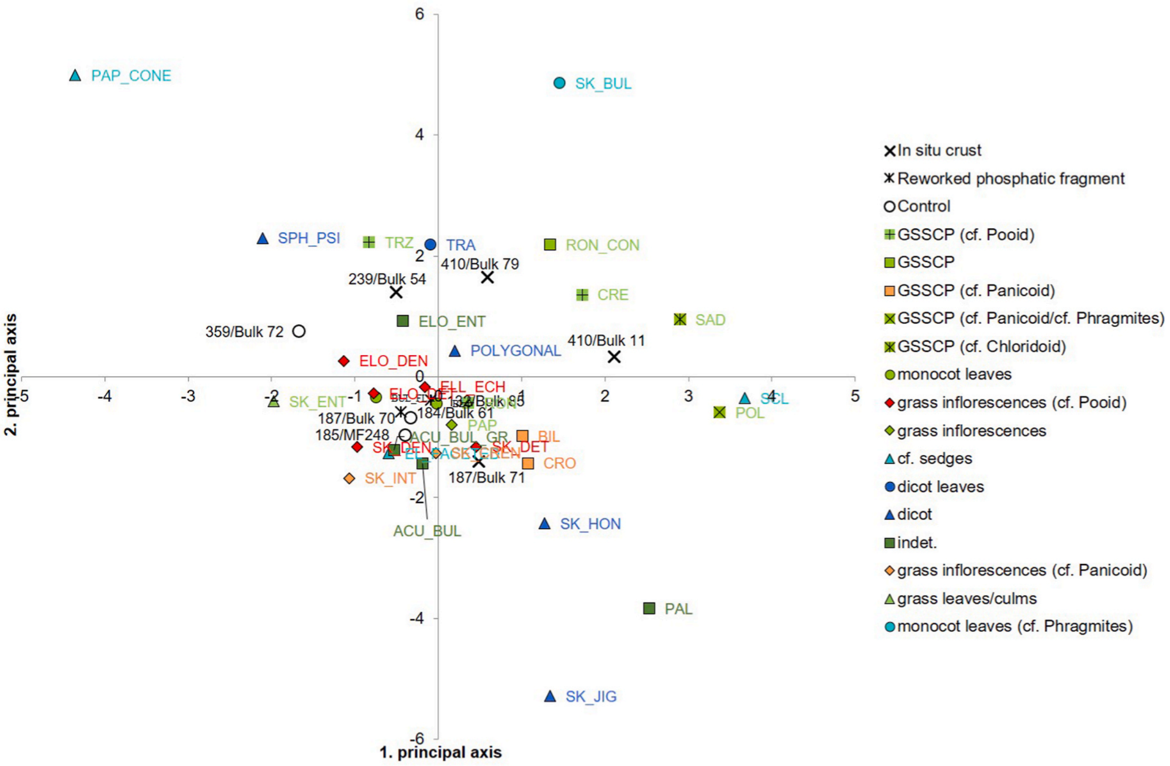

Fig. 14. Correspondence analysis of the samples from La Muraiola di Povegliano based on their phytolith assemblage of single cells and multicellular aggregates. Axis 1 and 2 give respectively 30% and 23% of the explanation.

abovementioned plants, further attested by the presence of massulae, as indicative of grazing pressure (Florenzano et al., 2015). Nevertheless, phosphatic crusts represent a promising archive for pollen in well-drained (i.e., non-waterlogged) alkaline deposits. This is due to their rapid formation in a temporary acidic microenvironment, and most of all, to the concentration of plant material within herbivore dung (for good pollen preservation in dung and cesspits see also Langgut et al., 2016; Langgut and Finkelstein, 2023).

The presence of reworked aggregates of phosphatic crusts within ash-rich deposits provides insight into the practice of cleaning of animal penning areas, presumably after periods of overwintering (i.e., at the onset of the warm season; cf. Butser Farm). Cleaning activities disrupted the phosphatic crusts and, as a consequence, their fragments were discarded outside the structure, inadvertently becoming incorporated into domestic waste (cf. Macphail, 2015). The two superimposed *in situ* phosphatic crusts (i.e., SUs 187, 410) likely escaped this practice.

4.2. *Archaeological implications*

4.2.1. *An overlooked archive: the uniqueness of phosphatic crusts from La Muraiola in the Italian Bronze Age*

Considering the Italian Bronze Age (2200-950 BCE), despite the economic importance of livestock, evidence of animal penning areas or structures is extremely sporadic (cf. Debandi, 2021, p. 145). In southern Italy, a series of pens have been investigated at the Early Bronze Age village of Nola – Croce del Papa (Naples province), a site sealed by the Avellino eruption. These enclosures hosted sheep, cattle, pigs, and several pregnant goats, which were crammed in a wooden cage (Albore Livadie et al., 2020; Pizzano and Genovese, 2020). In northern Italy, byre-houses have been identified at the MBA 1–2 settlement of Oppeano (Verona province), thanks to dung accumulations discovered within domestic structures (Nicosia et al., 2022). In the Terramara culture, LBA structures possibly used as byres and animal pens were found in external or marginal portions of the sites of Beneceto (Parma province; Bernabò Brea et al., 2022), Pragatto (Modena province; Miari et al., 2019), and Santa Rosa di Poviglio (Reggio Emilia province; Maini, 2021; Mariani and Cremaschi, 2021, p. 101). At Santa Rosa di Poviglio, micromorphological analysis identified an animal penning area thanks to trampled herbivore dung accumulations on the living surface (Cremaschi et al., 2004, p. 123). However, these accumulations were not distinctly related to any clear structure. Similar micromorphological evidence also characterised the Terramara site of Castellaro del Vhò (Cremona province; Ottomano, 2001) and the coeval settlement of Monte Castellaccio d'Imola (Bologna province; Cremaschi and Ottomano, 1996). Finally, the existence of pens was supposed based on remote sensing in other Terramara sites, such as Cavo Molinella/Fenilone and Maccaccari, in unstructured areas within the settlement (Tirabassi, 2003, p. 69).

This brief overview emphasises the remarkable significance within the context of Italian protohistory of the identification of phosphatic crusts as the macroscopic evidence of animal penning areas. When properly identified, phosphatic crusts serve as an invaluable archive for the investigation of animal management practices, also encompassing aspects such as living conditions, economic choices, and human-environment interactions.

4.2.2. *Implications about herding practices through fodder remains*

Formed by the concentration of plant material ingested by domestic animals, phosphatic crusts provide rich microfossil records that offer insights on foddering strategies and, indirectly, the environmental setting available for herding. The palynological and phytolith assemblages highlight the selection of vegetal resources from three main environments. These suggest a predominantly anthropogenic open landscape with patchy woodlands surrounding the site, reflecting the impact of MBA agropastoral activities (see e.g., Cremaschi et al., 2016; Dal Corso, 2018; Dalla Longa et al., 2019; Mercuri et al., 2006b, 2006a; Perego, 2015; Perego et al., 2011; Ravazzi and Valsecchi, 2001; Rottoli, 2001). The majority of pollen and phytoliths derive from grasses and forbs that grow in dry open habitats (e.g., *Orlaya grandiflora*), which, in the context of an alluvial plain, correspond to drained areas where mesophile woodlands could grow, had the land not been used for agropastoral activities. The dominance of Cichorieae and *Cirsium/Carduus* pollen types suggests abundance of these plants, possibly testifying overgrazing of pastureland (Florenzano et al., 2015; Mercuri et al., 2013). Many plants from cultivated land, such as arable weeds, and species from disturbed anthropogenic environments, could also serve as food/fodder and be enhanced by trampling and nutrient-rich soil resulting from animal passage. A third environment represented in the palynological record, though less so in the phytoliths, is that of floodplain mesophile and hygrophilous woodlands, which could provide leafy hay in winter and woodland pastures (see Jakobitsch et al., 2023; Karg, 1998; Mercuri et al., 2006b; Perego, 2015). Despite the site's proximity to springs, wetland vegetation is not well represented in the crust records: while selective corrosion of Cyperaceae pollen is a possibility, in the well-preserved phytolith assemblage, sedge phytoliths are relatively rare. Fodder provided by sedges and other wetland plants is known to be less nutritious than other resources, though it is available year-round (Sucholas et al., 2022). Thus, we can hypothesise that other types of resources were preferred for feeding the animals at La Muraiola.

Defining the seasonality of herding practices is challenging. Unlike individual coprolites and pellets (Fuks and Dunseth, 2021), phosphatic crusts reflect a broader timeframe of plant material ingestion, which could also include stored fodder, possibly including some pollen from pollen rain. Seasonal gathering of plants that bloom in early spring is represented by hazel catkins, which provide nutrient-rich plant material at the end of winter. However, most of the pollen from wild plants found in the crusts corresponds to species that flower in spring and summer, a time when animals could have been taken to pastures, possibly involving daily movements of livestock. The possibility that summer herbs were harvested and stored for winter cannot be ruled out, as suggested for the Early/Middle and Final Bronze Age in northern Italy (Karg, 1998, p. 93; Pini et al., 2021).

Pollen and phytoliths also indicate cereal processing at the site, a daily activity that generated substantial discarded material. Cereal pollen may have been dispersed locally during the final stage of crop processing (Bower, 1992), such as the dehusking of emmer and einkorn which are common at the site (D'Aquino, 2024). Phytoliths from cereal inflorescences, instead, attest the presence of the plant material itself. "Chaff phytoliths are abundant in all samples, suggesting that cereal by-products were likely used as fodder component. They are slightly more frequent in reworked fragments and in the control samples identified through micromorphology as herbivore dung deposits mixed with hearth rake-out (SUs 359 and 187; see Fig. 4a). This can indicate that domestic waste disposal may have contributed to the presence of chaff in these deposits.

While millet phytoliths are present in the crusts, it remains unclear whether millet was used as fodder. The charring of clusters of broomcorn millet (*Panicum miliaceum*) caryopses during the fire that destroyed the barn (SU 185) likely indicates the storage of these grains for human consumption. C/N stable isotope analysis has already demonstrated the role of millet in complementing the diet of contemporary MBA communities in the region (Varalli et al., 2022). In this context, additional C/N stable isotope analysis of faunal remains is required to further investigate the role of C4 plants in the animals' diet.

4.2.3. Implications for archaeological excavation practices

In situ phosphatic crusts appear in the field as orangish, cemented deposits. Their colour and compaction could easily be misinterpreted as evidence of exposure to heat, as noted for the Early Iron Age settlement of KwaGandaganda (Whitelaw, 1994). However, these crusts actually result from the cementation of herbivore dung by hydroxylapatite.

The fact that, to our knowledge, no field evidence of phosphatic crusts has been published so far can be explained by different factors: (1) the concept of 'phosphatic crust' has predominantly been discussed in micromorphological literature, potentially leading non-specialists to overlook this feature and struggle with accurate interpretations in the field; (2) *in situ* phosphatic crusts were likely often removed during the periodical cleaning of animal penning areas, leaving behind only reworked fragments; (3) these reworked fragments may be mistaken for coprolite fragments, particularly when their morphology has been modified by trampling and no further analysis, such as GC-MS, has been conducted; (4) *in situ* phosphatic crusts may be misinterpreted as heated surfaces; (5) their limited thickness (a few cm) and possibly patchy distribution may go unnoticed during fieldwork. Additionally, we must consider the potential oversight of unclear aspects when publishing excavation data, particularly given the lack of available comparisons.

When properly identified, phosphatic crusts can serve as a key feature for identifying structures built directly on the ground, particularly in those contexts in which stilted structures were commonly used (i.e., Terramare culture; see Bernabò Brea and Cremaschi, 1997), or when evidence of ground-level structures is not clearly discernible.

In parallel, reworked fragments of phosphatic crust highlight the role of removal, backfilling, and dumping activities in the formation processes of domestic archaeological deposits. This finding also underscores that the archaeological record should not be interpreted as a continuous sequence of deposition. Moreover, while negative features such as pits are often visible in the field, they may not account for all 'removal' processes. Reworked phosphatic crust fragments illustrate that such processes are not always directly observable during excavation and may sometimes only be inferred indirectly.

The interdisciplinary nature of this study highlights the importance of a multi-proxy approach, starting in the field, for unlocking the insights phosphatic crusts can provide. When identifying a hard, orangish crust, we recommend taking detailed photographs as reference material for scholars and field archaeologists. It is also crucial to record the crust's thickness, dimensions, and spatial relationships with structural elements (e.g., walls, postholes) to determine whether the space was roofed (e.g., a byre, barn, or stable) or enclosed by lightweight, unroofed structures (e.g., a corral or fence). The presence of mineralised vegetal matter, such as wood fragments or seeds, within the crust can help distinguish phosphatisation from rubefaction caused by heating.

For post-excavation analysis, we suggest sampling for micromorphology. Uncovered thin sections and chips from these samples can also be valuable for complementary chemical (micro-XRF, micro-Raman, micro-FTIR) and mineralogical (micro-XRD) analyses. Additionally, we recommend collecting fragments from the crust near the micromorphology sample, as well as from other areas, for extracting biomarkers and botanical microfossils. Collecting samples from multiple areas can help assess lateral variability. To avoid contamination, sampling for biomarkers and botanical microfossils must be performed using sterilised metal tools and clean nitrile gloves. In the laboratory, we suggest using a precision tool to drill the orangish core of the fragments collected in the field, aiming to exclude potential secondary carbonates such as those observed above and below *in situ* crusts (see above).

In summary, this study has demonstrated that.

1. Micromorphological and mineralogical techniques are fundamental for understanding the formation processes of phosphatic crusts, suggesting the role of herbivore excrement;
2. Faecal biomarkers are essential for validating this hypothesis. They also enable to evaluate potential contributions from human, porcine, or canine excrement, which would suggest a different use of the space, particularly in the case of human inputs. Humans and pigs produce significant amounts of coprostanol, whereas dogs produce cholesterol. These can be easily distinguished from herbivore inputs through the analysis of sterols and stanols (Baeten et al., 2012; Bull et al., 2002);
3. Botanical microfossils offer key insights into herding practices and human-animal-environment interactions, especially in well-drained sites with difficult preservation of organic materials.

Depending on specific research interests, one may explore one or more of these aspects in greater detail.

5. Conclusion

The identification of archaeological features associated with animal management has long been challenging, particularly without a robust microarchaeological approach (Weiner, 2010, pp. 239–240). Therefore, the outcomes of this study expand beyond the site-specific results, offering new methodological tools for identifying ancient animal penning areas. In particular, the macroscopic 'visibility' of phosphatic crusts can help bridge the gap between the importance of herding in ancient economies and the scarce archaeological evidence of structures associated with this activity, as seen in the case of the Italian Bronze Age (see §4.2.1; see also De Grossi Mazzorin, 2013a; 2013b; Debandi, 2021).

Exhibiting an orangish colour and hard texture, phosphatic crusts should not be mistaken for rubified surfaces caused by heating. Their colour and hardness result from hydroxylapatite cementation of trampled herbivore dung and, possibly, bedding and fodder material. Hydroxylapatite likely forms through the transformation of less-stable Ca-P minerals, a process also observed in modern stable/byre deposits. The diagenetic processes involved in the formation of phosphatic crusts make them valuable archives for studying livestock management. They preserve faecal biomarkers, phytoliths, and pollen, the latter of which is often poorly preserved in well-drained prehistoric contexts due to detrimental burial conditions (Campbell et al., 2011, p. 6). The periodic cleaning of animal penning areas can lead to the recovery of only reworked fragments of phosphatic crusts during excavations. However, this study has shown that these fragments can provide results comparable to those from *in situ* phosphatic crusts. Consequently, they can be considered the same as a valuable 'artefact' (*sensu* Miller, 2011) for reconstructing past human activities and human-animal-environment interactions.

In a future perspective, it remains to be explored whether phosphatic crusts primarily form in enclosed roofed spaces (i.e., barns, byres, or stables) or can also form in open areas where livestock is concentrated (i.e., animal pens or corrals). Identifying additional *in situ* crusts will be crucial for answering this question, providing new field and laboratory data. Further analysis of samples from other phosphatic crusts from La Muraiola di Povegliano will deepen our understanding of the natural resources exploited during herding activities. From a diachronic perspective, this will allow for an evaluation of changes in landscape exploitation over the course of the settlement's lifespan, as well as shifts in the use of space at the site.

CRediT authorship contribution statement

Federico Polisca: Writing – review & editing, Writing – original draft, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Marta Dal Corso:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. **Maela Baldan:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. **Mara Bortolini:** Writing – review & editing, Writing – original draft, Investigation, Formal analysis, Data curation. **Dario Battistel:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Investigation, Formal analysis, Data curation. **Gregorio Dal Sasso:** Writing – review & editing, Formal analysis, Data curation. **Francesca Gherardi:** Writing – review & editing, Writing – original draft, Investigation, Formal analysis, Data curation, Conceptualization. **Matthew Canti:** Writing – review & editing, Writing – original draft, Formal analysis. **Giorgio Piazzalunga:** Investigation, Formal analysis. **Cristiano Nicosia:** Writing – review & editing, Writing – original draft, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Data availability statement

The authors confirm that the data supporting the findings of this study are available within the article and its supplementary materials.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jas.2025.106207.

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