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Revealing the invisible dead: integrated bio-geoarchaeological profiling exposes human and animal remains in a seemingly 'empty' Viking-Age burial

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A R T I C L E I N F O

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A B S T R A C T

Recent investigations of an apparently 'empty,' partly disturbed Viking chamber grave in Denmark (Fregerslev II, dated around the mid-10th century CE) provided an opportunity to develop a novel multi-scale and multi-method analysis of burial and post-burial processes. To overcome the limitations of poor preservation of artefacts and bones, and the lack of a clear macrostratigraphic sequence, we integrated multi-proxy analyses of organic and inorganic materials to study the spatial architecture, burial, and post-depositional processes, including soil chemistry (inductively coupled plasma mass spectrometry - ICPMS, portable X-ray fluorescence spectrometer - pXRF), soil micromorphology, archaeobotany (wood, seeds, fruits, phytoliths), palynology (pollen, non-pollen palynomorphs), and faecal lipid biomarkers. The results enabled the detailed characterisation, spatial analysis, and sequencing of burial deposits, and the identification of post-depositional factors responsible for the poor preservation of the burial. Soil, phytolith and pollen data indicated that the base of the grave was covered with a matting of plant material, and there was no wooden floor. Faecal biomarkers detected substantial amounts of faecal matter, most probably originating from horse faeces, suggesting that a horse died *in situ*, and trace amounts of pig faeces, which are more likely to have been trampled into the grave. Enriched phosphorus concentrations could be linked to the bodies in the northern and southern sector of the grave. Furthermore, enrichment in lead was found where metal objects were recovered. The findings from Fregerslev II show that integrating high-resolution approaches to the analysis of poorly preserved burial contexts can fundamentally transform archaeological interpretations.

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1. Introduction

In temperate regions, fluctuating moisture, anaerobic and acidic (pH < 7) conditions can compromise the preservation of burials, leading to the destruction of bone remains, and the dissolution of bone minerals in extreme cases (e.g., Dent et al., 2004; Janaway, 2008; High et al., 2016). Understanding the micro-context is critical as complex soil processes and properties influence the deposition, movement and transformation of organic matter, solutes, water, and air on and through the soil. The burial of organic and inorganic materials affects soil processes and might shape the way key soil properties change. Oxygen, temperature, moisture, pH, and organic matter content influence the colonisation of fungal decomposers (Haslam and Tibbett, 2009), insect larvae (e.g., Anderson and Cervenka, 2002) and the nature and amount of organic matter from body decomposition (Stolt and Lindbo, 2010; Rousk et al., 2009). Enhanced biological activity can be expected in and around inhumations and grave goods made of organic materials (Turner-Walker, 2007). Studies of burial decay products in northern Europe show increased phosphorus (P) and microscopic organic matter (including excremental) in the sedimentary matrix, deriving from the decomposition of the body and grave goods. Iron (Fe) nodules are also common, and the formation of redoximorphic features might be enhanced by the microbial activity in grave fills (Lang, 2014: 205–207). The degradation of skeletal remains can leave 'soil silhouettes' (or colour stains; Biek, 1963) such as, for example, those recorded in Viking graves at Hesby, Norway (Macphail and Goldberg, 2017: 482–484), and the early medieval graves at Sutton Hoo, England (Bethell & Carver, 1987; High et al., 2016), where soil acidity prevented bone preservation.

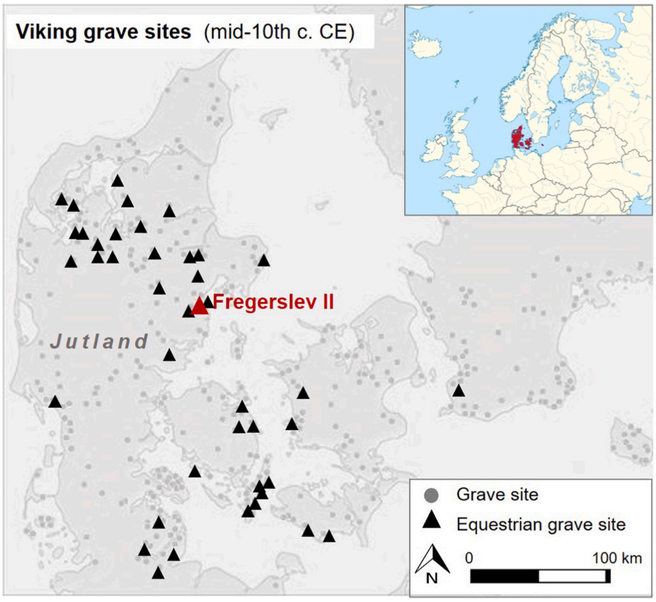

Fig. 1. Viking grave sites contemporary to Fregerslev II. Base map adapted from denstoredanske. dk; distribution of equestrian graves from Pedersen (2014).

The Jutland region, Denmark, is well known for Bronze Age burial mounds, well-preserved oak-coffin burials, and Iron Age bog bodies (Broholm and Hald, 1939; Breuning-Madsen et al., 2003; Asingh and Lynnerup, 2007; Holst and Rasmussen, 2013; Frei et al., 2015). However, the regional sandy and acidic soils result in poor preservations of burials under aerobic conditions (Breuning-Madsen et al., 2003). Recent excavations of a Viking-Age chamber grave at Fregerslev (Fig. 1)

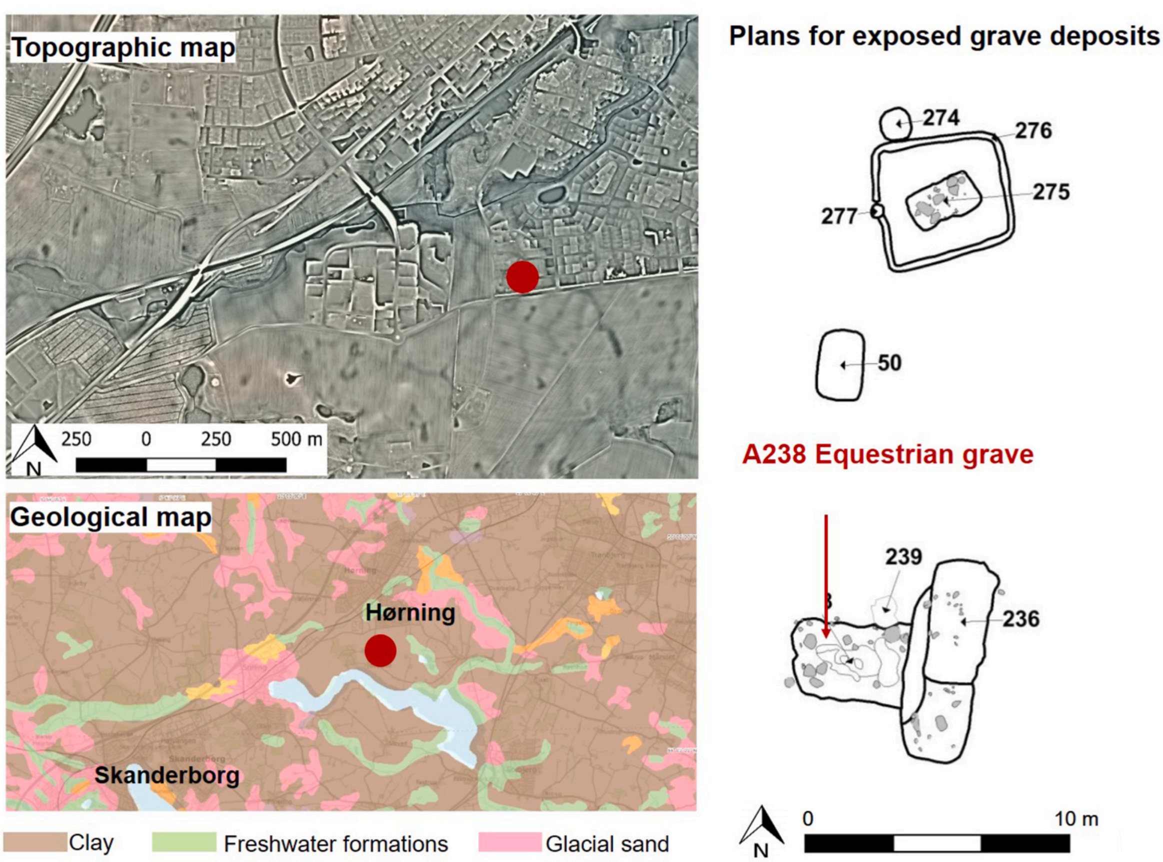

Fig. 2. Location of Fregerslev II; digital elevation model with a residual relief model (Stott et al., 2019); geological soil type map (after GEUS); the three graves recorded at Fregerslev II (adapted from Bagge, 2016).

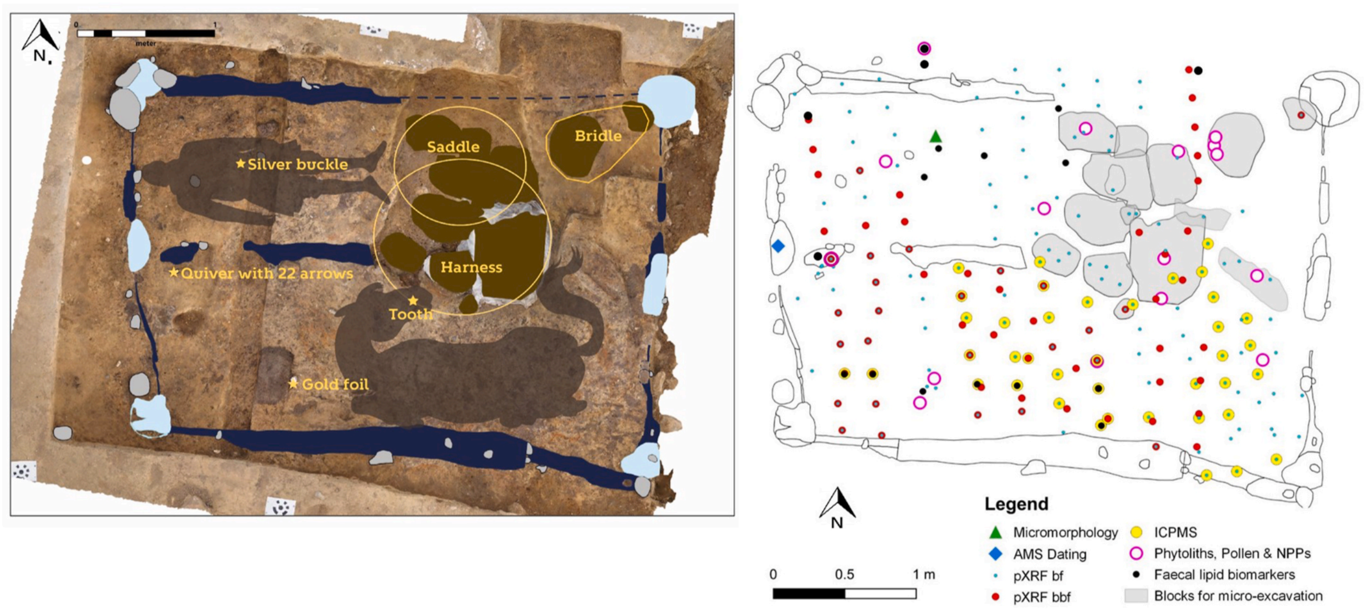

Fig. 3. Left: Aerial photograph of the 2017 trench showing the location of main finds and interpretative model of the grave; right: plan of the trench showing the sample locations.

uncovered a seemingly elite burial with an exquisite horse harness with gilded and silver-laid fittings, a quiver with 22 arrows, and a silver brooch, but no skeletal remains (Bagge and Hertz, 2021; Bagge, 2019, 2020). Artefact distribution suggested the presence of a human and a horse, but it was impossible to determine the spatial organisation of the interior of the grave, or whether other animals and organic objects were present, as is common in other high-status, Viking-Age chamber graves (Iversen, 1991; Price, 2008; Price et al., 2019). A multi-scale sampling and analytical programme was then developed to detect vital information about the burial that could not be retrieved using standard archaeological methods. Here, we present the results of integrated, multi-element geochemistry (portable X-ray fluorescence spectrometry: pXRF, ultra-trace inductively coupled plasma mass spectrometry: ICPMS), soil micromorphology, archaeobotany (wood, seeds, fruits and phytoliths), palynology (pollen and non-pollen palynomorphs), faecal lipid biomarkers (5β-stanols), and our evaluation of the potential of this approach to elucidate other poorly preserved burials worldwide.

2. Research context

2.1. Environmental context

Denmark has a temperate, humid Atlantic climate with an average temperature of 8.3 °C (DMI, 2019). The regional landscape settings and paleoecology of Jutland are well described (Holst et al., 2018; Søe et al., 2017a). Fregerslev is situated on an elevated plateau of Late Glacial loamy tills and relatively flat relief with several small lakes and fenlands, and intersecting E-W glacial tunnel valleys. In the 10th century CE, the region was covered by forests with primarily beech, oak, and hazel (Søe et al., 2017b). 19th-century cadastral maps (e.g., 1816, Fregerslev By, Hørning 1,040,553, 1:4000, Geodatastyrelsen-Danish Geodata Agency, https://hkpn.gst.dk/) show the Fregerslev site on farmland, but since it has been gradually engulfed by development around the modern town of Hørning (Fig. 2). This area is covered by a well-drained loamy parent material, consistent with slightly acidic Luvisols (Breuning-Madsen and Jensen, 1992). The site was later truncated by modern tillage, acidified by organic and chemical fertilisers, and compacted by farm machinery, with associated changes in soil moisture content, including pseudogley features from temporary stagnating water. In 2015, a new road and a deep pipeline were placed about 15 m to the north of the grave. If there was originally a burial mound on top of the chamber grave (e.g., as at Stengade II, Gødsvang; see Pedersen, 2006), there is no record of it, or when it was removed.

2.2. Equestrian burials in Viking-Age Denmark

In Denmark, most of the eighty recorded equestrian burials of the Viking Period were found in the 19th and early 20th centuries, and few were professionally excavated later (Pedersen, 1997, 2014). This burial practice is distinctive of the mid-10th century CE (925–975), when the basic pattern of equestrian burials becomes rather standardised: the individual was commonly placed with personal items and weapons in the northern or central part of the burial, and a gaming board and table setting placed to the right side. Sacrificed animals such as horses and/or dogs were laid in the foot end of the grave, on top of the grave, or in a pit next to the grave (Pedersen, 2021). Horse remains were found in forty-eight equestrian graves and dogs in nine graves (Pedersen, 2014, Find list 10). Horse is by far the most commonly observed sacrificed animal in burials across Viking Scandinavia, Britain, Ireland, and Iceland (Sikora, 2004). Recent DNA analysis of horse remains in Viking graves showed that they were predominantly male (Nistelberger et al., 2019).

2.3. The archaeological record from Fregerslev

The region around Fregerslev has known sites dating from the late Neolithic, late Bronze Age and late Iron Age periods. The largest valley, Illerup Ådal, was an important Iron-Age sacrificial landscape since the 1st century CE (Holst et al., 2018). Lake sediments reflect extensive land-use by humans and animals from the 1st to 11th century CE, related to a religious control of this valley (Søe et al., 2017b). The Viking-Age records include a burial ground with seven, simply furnished graves less than 1 km west of Fregerslev (Bagge, 2020; Bagge and Hertz, 2021), and a late Viking-Age equestrian grave at Ravnholt, c. 3.5 km east (Brøndsted, 1936: no. 49). Medieval field systems are present around Fregerslev II and several pits (some reaching up to 7 m in diameter) with no known purpose were dug here in the 18th and 19th centuries.

Prompted by land development, several seasons of field excavations at Fregerslev from 2012 onwards uncovered a richly furnished chamber grave (Fregerslev II) and two other unfurnished graves. In 2017, *The*

Fig. 4. Fregerslev II: top left, the burial deposit exposed during the last excavation season (2017), after clearing the topsoil; top right, measurement of pXRF *in situ* alongside Profile T79; bottom: sketch of Profile T79 showing the main stratigraphic units.

Viking from Fregerslev project implemented a novel, multidisciplinary analytical program in the last phase of excavation. The chamber grave (9.4 m^2) contained an impressive range, quality, and quantity of finds, including a wooden quiver with 22 arrows, a silver brooch, and remains of a wooden horse saddle, bridle, harness, and leather fragments possibly from a leather pouch or nosebag (Bagge, 2019, 2020; Bagge and Hertz, 2021). Over 700 small silvered and gilded metal fittings, primarily from the bridle, were found in the northeast part of the chamber, suggesting that the burial was that of a high-status individual dating to around the mid-10th century CE. The equestrian fittings do not appear to have been placed on the horse, a layout also observed in other equestrian graves. Traces of wooden planks and postholes, one containing a plank fragment, and nails, indicated the presence of a chamber grave with a lengthwise partition wall, which probably separated the deceased from the grave goods. No physical remains of the individual(s) were recovered and only one fragment of a tooth of a larger herbivore (probably horse) was found on the grave floor, just south of the harness and not in contact with it. If the tooth is indeed from a horse, its position suggests that the horse was unharnessed before its deposition in the grave (Fig. 3). Informed by the distribution of finds, a tentative reconstruction proposed the placement of the individual in the northwest part of the grave, and, next to the harness fittings, a horse or horse skull in the southern part of the grave (Fig. 3). Few features or finds were recorded in the seemingly "empty" space in between.

3. Materials and methods

3.1. Field excavation methods and sampling approaches

In 2012, mechanical excavating in advance of development removed approximately 15–20 cm of topsoil and exposed remains of three burials (Fig. 2), including the chamber grave (A238). Subsequently, an area of c. 17,000 m^2 was excavated to expose the chamber grave and its surroundings. During the final field season (2017), an area of c. 13 × 14 m was re-exposed and excavated by trowel (Figs. 3 and 4). The burial pit was only 15–28 cm deep, and clearly truncated by ploughing and mechanical digging. The fill of the grave was excavated and recorded in 3–7 cm spits, with additional 3D documentation when features were encountered. In the northeast corner, concentrations of metal objects were lifted in 13 large blocks covered with plaster for micro-excavation under controlled laboratory conditions. The grave fills were wet-sieved using a 3 mm mesh, and multi-scale sampling was applied to materials and features found in the basal fill of the grave (Fig. 3; Table 1). Undisturbed block samples for soil micromorphology and loose bulk soil samples were subsampled in the laboratory for geochemistry, macrobotanical remains (wood, seeds and fruits), phytoliths, pollen and non-pollen palynomorphs (NPPs), and faecal lipid biomarkers (Sulas et al., 2021; Orfanou et al., 2021; Out et al., 2021; Out et al., forthcoming).

In the field, 131 *in situ* geochemical measurements were performed using a portable Bruker S1 Titan 800 tube-based X-ray fluorescence spectrometer (pXRF; Orfanou et al., 2021) over a 25 cm^2-grid covering the basal fill (the burial 'floor'), additional features of interest, and the soil below and outside the grave cut for reference (control) purposes. The results of *in situ* pXRF measurements guided sub-sampling in the laboratory for ultra-trace inductively coupled plasma mass spectrometry (ICPMS) (Becker, 2005; Neff, 2017) to characterise the upper grave fill, basal fill/floor, and sediments associated with the main finds (quiver, saddle, bridle, and concentrations of metal objects). Combined, these methods provide a rich range of quantitative and qualitative data (Entwistle et al., 1998; Radu and Diamond, 2009; Horta et al., 2015; Sulas et al., 2019; Trant et al., 2021).

Table 1
List of archaeological layers and samples associated with them.

Context	Location	Description	Artefacts	Sampling	n. of samples per method							
					pXRF	ICPMS	Thin section	Wood	Phytoliths	Pollen	NPPs	Lipid
Local reference soil	Just north of the excavation trench	Mixed, organic-rich soil over glacial till consistent with regional Luvisols	None	Contextual	4	2	1		1			3
			None	Contextual			1					
Excavation Trench												
Grave fill				20 × 30 cm grid	64		1					
Mixed grave basal fill (floor)	Across the trench (Layer 12)	Layer 12	None	20 × 30 cm grid	64	37	1		2			14
Feature blocks												
Artefact concentrations and features in the basal fill (floor) were lifted as blocks for subsampling and micro-excavation under controlled laboratory conditions.			Silver brooch; gold foil; remains of saddle, harness, bridle, metal fittings	Contextual	39			105	11	10	2	

3.2. Laboratory analyses

3.2.1. Multi-element geochemistry and soil micromorphology

Additional pXRF readings (n = 39) were taken from the soil blocks under controlled laboratory conditions. All pXRF measurements were then calibrated by correlation with the ICPMS measurements (SM A1.1, A2.1). Three undisturbed block samples were thin-sectioned for micromorphological analysis (SM A1.2, A2.3): one fom just outside the trench as a control to characterise local soils; 2) from the grave fill; and 3) from a soil sequence, comprising a B horizon underlying the grave cut, the basal fill/burial floor and the upper grave fill. The thin sections were analysed using a polarizing microscope and described following international standards (Bullock et al., 1985; Stoops, 2003). The identification and interpretation of features in thin section followed guidelines from reference literature (Macphail and Goldberg, 2017; Nicosia and Stoops, 2017; Stoops et al., 2010) and relevant, comparative case studies.

3.2.2. Archaeobotanical and palynological analyses

Wood, seeds, fruits, phytoliths, pollen, and non-pollen palynomorphs (NPPs) were analysed from the grave fills (SM A1.3, A2.4, A2.5).

A total of 105 wood remains, collected opportunistically by hand during the excavation of the block samples, were identified. Since wood finds were classified as artefacts and wood identification is usually destructive, only small fragments were available for identification. All wood fragments were studied using a stereomicroscope and a reflected light microscope with a magnification up to 500x. Identification was based on Schweingruber (1990).

The grave yielded charred grains of wheat and barley, including hulled barley (Out et al., 2021), but due to their preservation condition and the presence of similar finds outside the grave, these grains were considered to be potentially intrusive and are excluded from the results discussed below. Analysis of phytoliths, pollen and NPPs was conducted to establish the presence and nature of plant and biogenic materials in the grave (SM A1.3, A2.4, A2.5). The sampling strategy was informed by the distribution of archaeological finds and resource considerations.

A total of fourteen soil samples were collected for phytolith analysis: one control from the soil/sediment just outside the grave approximately at the level of the grave bottom, eight from the grave fill (*in situ* and from the soil blocks aiming to cover the entire grave), and five from two soil blocks (X140 and X974) that contained remains of the bridle and leather fragments (interpreted as a possible a nosebag). Phytoliths from the control and grave fill samples were chemically extracted, while the material from the soil blocks, consisting primarily of phytoliths, was mounted directly onto slides. Phytoliths were classified according to the International Code for Phytolith Nomenclature (ICPN 2.0; Neumann et al., 2019).

The same soil samples used for phytolith extraction were subsampled for pollen analysis. The ten pollen (sub)samples selected were evenly distributed across the bottom of the basal fill and concentrated near the leather remains possibly from a nosebag (X140, X974). Pollen extraction was performed according to Fægri et al. (1989), and identification followed Beug (2004) and reference collection at Moesgaard Museum. In addition, two (sub)samples from the block preserving the remains of a quiver were subjected to combined analysis of pollen and NPPs: one from the inner edge of the quiver and one from between the arrow shafts. These were prepared following a modified protocol for maximum diversity of palynomorphs (Enevold, 2018; Enevold et al., 2019), and NPP identification followed published literature (van Geel, 2001 and references provided in SM A1.3).

3.2.3. Faecal lipid biomarker analysis

Fourteen soil samples from the grave floor were analysed for faecal lipid biomarkers (5β-stanols) to identify the presence of faecal material and to determine its main species of origin (SM A1.4). Three control samples were taken from just outside the grave cut to determine the

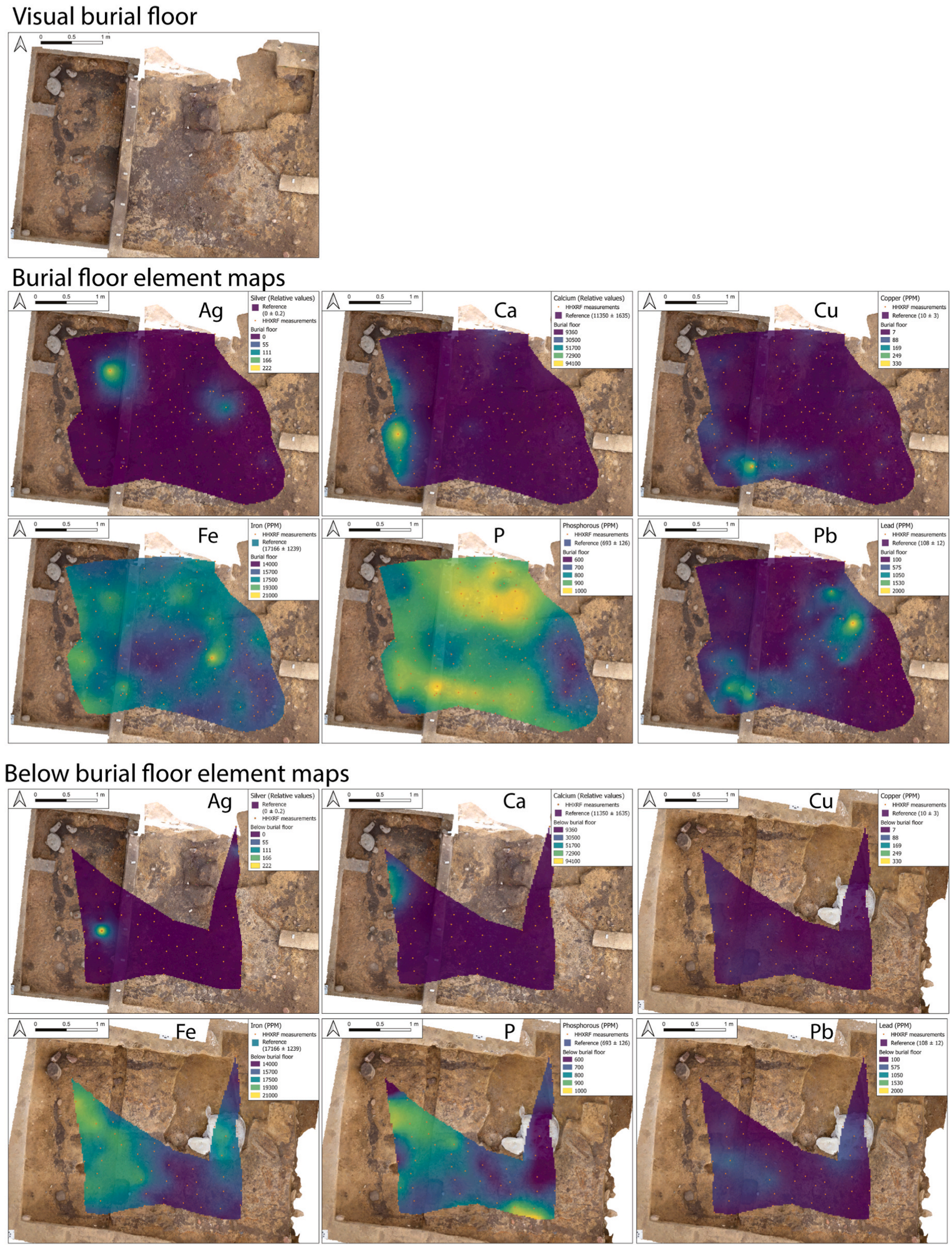

Fig. 5. Elemental concentrations of Ag, Cu, Fe, P, and Pb in the burial floor layer and the soil below it.

concentration of 5β-stanols in the 'natural' soils adjacent to the grave and to set thresholds above which samples should be considered to be impacted by the addition of animal faeces (Prost et al., 2017; Harrault et al., 2019). Sampling within the grave fill targeted locations provisionally ascribed to the inhumation burial in the northern part of the chamber, and those showing P enrichment based on the calibrated pXRF analysis. In summary, eleven 5β-stanol compounds (coprostanol, epicoprostanol, 5β-brassicastanol, 5β-lichestanol, 5β-epibrassicastanol, 5β-campestanol, 5β-epicampestanol, Δ^{22}-stigmastanol, Δ^{22}-epistigmastanol, 24-ethylcoprostanol and 24-ethylepicoprostanol) were extracted with organic solvents, isolated by column chromatography on silica, and analysed by gas chromatography coupled with a mass spectrometer (SM A1.4, Fig. 4; cf. Harrault et al., 2019). Faecal fingerprints were then determined based on the 5β-stanol distributions and

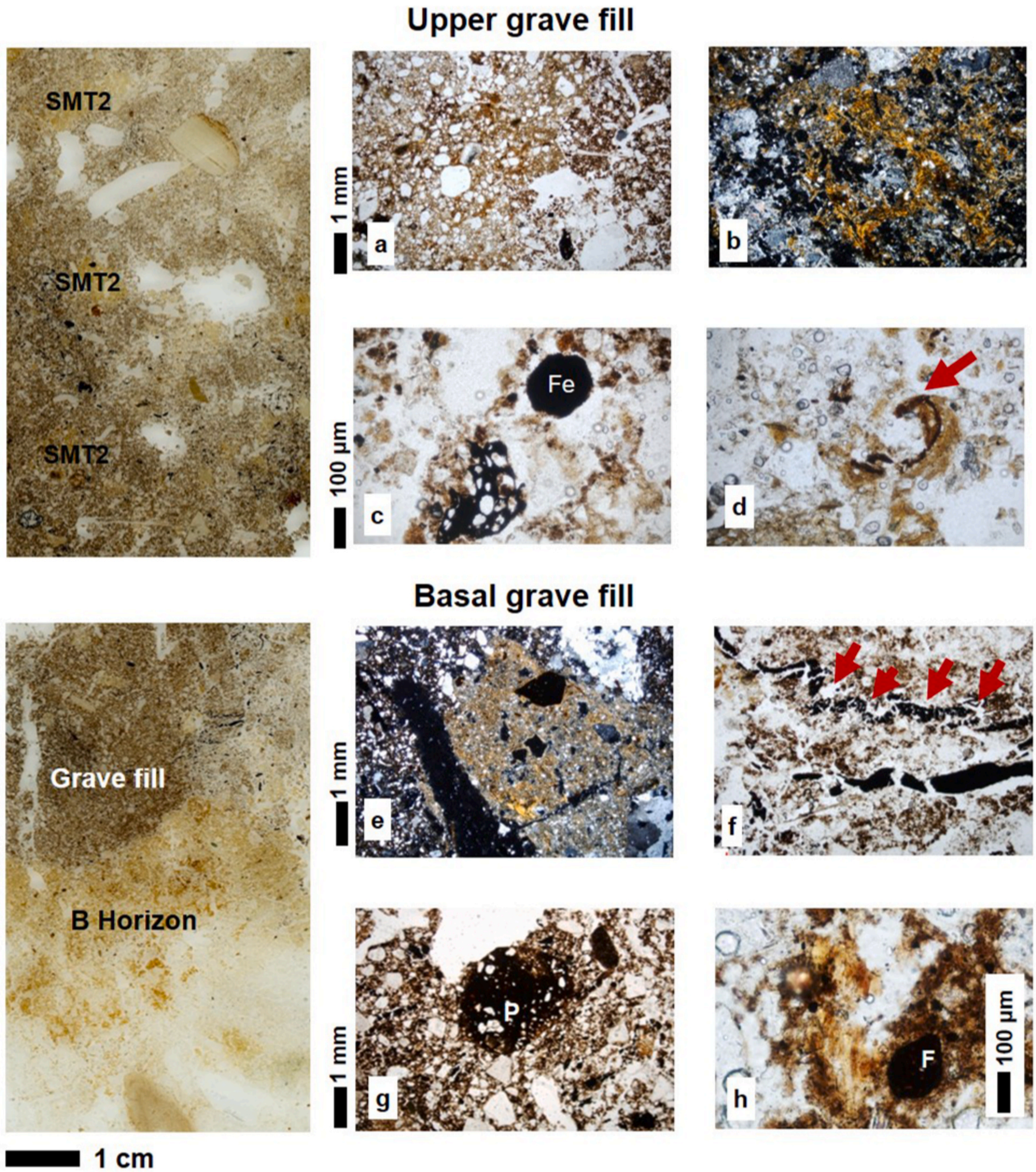

Fig. 6. Microstructure and key pedofeatures of the grave fills. Left, scans of the thin sections from the grave fill and B horizon; right micrographs: a. structure and porosity exhibiting different degree of iron impregnation, PPL (plane polarized light); b. mix of different soil microfabric types, XPL (crossed-polarized light); c. iron typic nodule (Fe), PPL; d. iron-manganese hypocoating (red arrow), PPL; e. mix of different soil microfabric types, XPL; f. horizonal distribution of plant remains with red arrows indicating potential direction of compression, PPL; g. potsherd (P), PPL; h. amorphous organic matter and fungal spore (F), PPL. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

statistically compared with a database of species-specific faecal fingerprints derived from reference samples of humans and the most relevant domestic animals in a southern Scandinavian context: dog, cow, goat, sheep, and horse. As the 5β-stanol-based toolbox developed by Harrault et al. (2019) provides reliable and cost-effective (not requiring an additional saponification step, LL extraction and GC-MS analysis) identifications of past sources of faecal inputs, other faecal steroid biomarkers, such as bile acids, were not used in this study (Bull et al., 2002; Prost et al., 2018). A direct comparison of 'traditional' stanol ratio proxies with the fingerprint toolbox used in this study can be found in Fig. 8b and A2.6.

4. Results

Full results are presented in Supplementary Material A2.

4.1. *Establishing a baseline for local conditions, anthropogenic markers and taphonomic indicators*

Reference soil samples consisted of a loamy till soil with evidence of clay illuviation and pseudogley features in the B horizon. Concentrations of phosphorus (P) at 671 ± 31 mg/kg and copper (Cu) at 12 ± 5 mg/kg are consistent with Danish soil properties influenced by recent agricultural liming and fertilization of the glacial parent material (e.g.,

Table 2
Archaeobotanical and palynological results by context.

Method	Sampling strategy	Sample location	Preservation	Organism group	Organism part	Taxa	Function and/or interpretation
Wood	Few finds from excavation + finds from block samples	Bottom of the grave, various locations	Uncarbonized, partly mineralized	Woody taxa	Trunk/branches	Oak (*Quercus* sp.) Maple (*Acer* sp.) Coniferous wood (Gymnospermae) Pine (*Pinus* sp.) Unidentified deciduous wood, not oak or maple	Burial chamber Saddle Quiver Quiver + Arrow Quiver
Macroremains	Finds from block samples	Dispersed through grave	Carbonized	Grasses, cultivated	Fruits	Wheat (*Triticum* sp.) and barley (*Hordeum vulgare*), including hulled barley (*H. vulgare* var. *vulgare*)	Association with grave unclear
			Carbonized	Potential arable weeds	Fruit	Black bindweed (*Fallopia convulvulus*)	Association with grave unclear
			Carbonized	Indet.	Tuber	Indet.	Association with grave unclear
Phytoliths	Systematic spot samples from excavation and block samples; sediment samples	Bottom of grave	Silicified	Predominanlty grasses	Culms and leaves	Poaceae	Bedding
		Block samples with leather remains (poss. nosebag)	Silicified, articulated (multi-cellular)	Grasses, presumably cultivated	Chaff	Predominantly oat (*Avena* sp.)	Fodder for horse
Pollen	Systematic spot samples from excavation and block samples; sediment samples	Bottom of grave	Uncarbonized	Grasses, wild (predominant) and cultivated	Pollen	Poaceae, barley/rye type (see SM A2.5.2)	Bedding
		Bottom of grave	ditto	Herbs, including potential arable weeds and potential food plants	Pollen	E.g. cabbage family, mugwort, yarrow, chicory, ribwort plantain, spearwort and thistle (see SM A2.5.2)	Bedding
		Bottom of grave	ditto	Woody taxa	Pollen	Maple, alder, birch, hazel, beech, pine, oak, willow, lime and elm (see SM A2.5.2)	Presumably deposited by wind onto the plants used for bedding; not representing grave goods present in the grave.
		Block samples with leather remains (poss. nosebag)	ditto	Grasses, cultivated	Pollen	Predominantly oat/wheat type (*Avena/Triticum* type)	Fodder for horse
NPPs	Two sediment samples from block samples	Quiver	ditto	Fungi	Fungal fruit bodies (sporocarps) and spores	Various types (see SM A2.5.2)	Decaying nutrient-rich material (e.g. wood and/or leather)

Ingerslev, 1997). Micromorphological analysis of the reference soil profile next to the northeast end of the excavation trench showed an AB horizon of a mixed, organic-rich soil developing over a glacial till substratum. Mixing of different material was recorded in thin section where three main soil microfabric types (SMT) were identified, ranging from an organic-rich and highly bioturbated fine sandy, clayey silt (SMT1 and SMT2, 70%, Ah horizon) to a clayey very fine sand with common clay coatings (30%, Bt horizon). The overall moderate to strong iron-impregnation and the amorphous conditions of the organic fraction suggest relatively strong but contingent and varying oxidizing conditions. The AB horizon yielded much lower concentrations and different types of phytoliths than those recorded in the grave fill, as discussed below.

4.2. Stratigraphic sequence and spatial organization of finds and features

Excavations exposed a stratigraphic sequence of three main deposits (Fig. 4) at c. 20 cm below the topsoil. The grave fill varied in thickness from c. 15–28 cm and was exposed in its entirety, revealing a number of features, including possible cuts from post-depositional disturbance. However, the spatial organisation and concentrations of artefacts suggest that this disturbance was relatively localised, and likely to be associated with animal burrowing and the collapse of the grave.

4.3. Archaeological sediments

4.3.1. Soil chemistry

Correlations of the pXRF and the ICPMS results showed patterns of elevated copper (Cu), iron (Fe), phosphorus (P), and lead (Pb) in both the basal fill and the soil beneath it (Fig. 5). Two areas had very enriched levels of P compared to natural background levels (Reimann et al., 2014) but did not correlate with any of the other mapped elements. In contrast, calcium (Ca) had a very uniform distribution, suggesting little influence by artefacts or human/animal remains in the grave. Silver (Ag) was enriched where a small silver buckle was found, while Pb showed a peak where the bridle fittings were found in the northeast part of the grave. A strong enrichment of P, Cu and Pb was detected in the southwest sector, where very few artefacts were found, possibly indicating the former location of decomposed organic materials and very thin, fully degraded objects. However, humic staining (often associated with iron concretions), typically found in inhumations (e.g., Bethel and Carver, 1987; Aspöck and Banerjea, 2016) and present elsewhere in this grave, was not observed in the southwest sector, possibly because of the aerobic conditions within the burial since construction, as seen in other prehistoric burials (Thomsen et al., 2008).

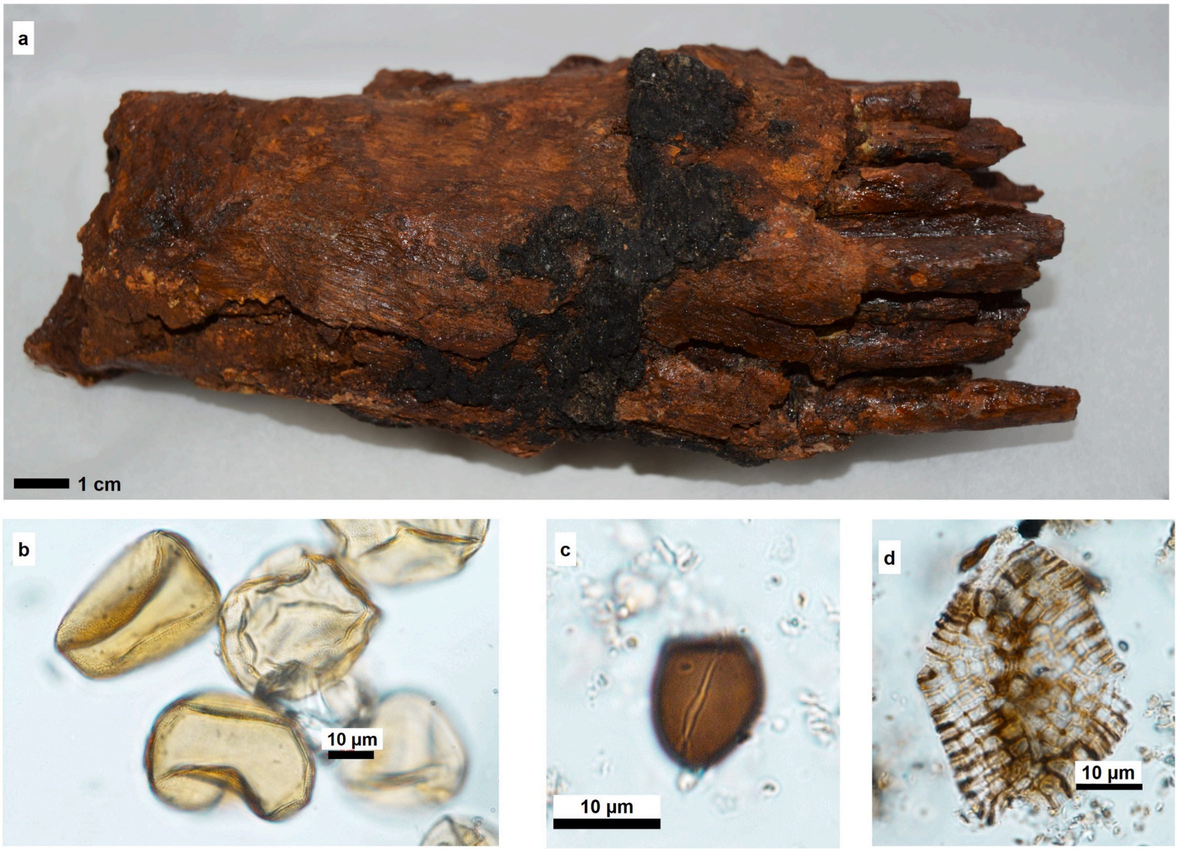

Fig. 7. Wood, pollen and NPP finds: a. remains of the quiver (Moesgaard Museum, C. Odderskov); b. cereal pollen (*Triticum-Avena* type) from chaff; c. ascospore from coprophilous fungus (*Sporormiella* sp.); d. fungal fruit body.

4.3.2. Soil micromorphology

Analysis of thin sections identified four main categories of materials and features: general anthropic debris, burning, compression and redox (Fig. 6). The redox features observed point to different rates of soil water and air changes, with typic, sharp-edged iron nodules indicating they were associated with relatively fast redox processes, and orthic iron-manganese nodules forming *in situ* as a result of slower bio-chemical processes (Lindbo et al., 2010). In thin section, the basal fill contained two distinctive deposits: 1) the top part featured a medium to fine sandy clay matrix, rich in organic matter, similar to the SMT 1 observed in the reference soil sample (Fig. 6a), covering 2) a mixed medium to fine sandy clay with a moderately developed subangular microstructure and low organic content (Fig. 6b). The latter is characterised by a relatively high porosity (15%, mainly planar and vughy), common illuvial clay (coatings and infillings), rare iron (typic) nodules and iron-depleted domains (Fig. 6a,c). This material is likely to derive from a Bt horizon, similar to the one observed in the reference sample. However, the distinctive vughy porosity, the iron-depleted domains, and the presence of typic iron nodules strongly point to changing soil water and air conditions due to redox processes (Wang et al., 2015). No archaeological materials were detected in this thin section, and charcoal was only present in negligible amounts.

In thin section, the grave fill contained mixed fabrics and anthropogenic inclusions. Most of the slide (c. 70%) was dominated by very fine sandy silt, similar to the SMT 1 observed in the reference sample, mixed with discrete, clay-rich soil aggregates (c. 30%, Fig. 6e). In the dominant fabric, the weakly developed, complex (granular to crumb) microstructure was associated with packing voids and predominant medium to fine grains (quartz, feldspars), but gravel and rock fragments (limestone and biotite) were also present, unlike in the other samples. Common inclusions consisted of charcoal (50–500 μm) and micro-charcoal, ferrous plant pseudomorphs of parenchyma tissue, phytoliths and fungal sclerotia (common in A horizons); fragments of a stone flake (flint), potsherds and burnt clay (Fig. 6g and h). In some instances, plant residues appear compacted into horizontal bands (Fig. 6f). The fine groundmass was characterised by abundant amorphous organic matter and iron impregnation. Textural pedofeatures were dominated by redoximorphic nodules, and a few calcite nodules were also present. Discrete fragments of clay-rich fabric probably derive from the same source as the SMT 2 observed in the reference sample. In sum, the grave fill was composed of the local soil, with soil fabrics originating from different horizons mixed by digging and back-filling, and incorporating organic matter from the original topsoil and litter layer, and residual anthropogenic material that had been present in the surface soils at the time of the burial.

4.3.3. Macrobotanical remains

The basal grave fill yielded uncarbonized wood. Most wood was preserved in partially mineralized state due to the presence of metal grave goods and nails in the chamber construction, except for two elements found during the trench excavation (Table 2; SM A2.4). The assemblage consisted of wood and possible wood (n = 69 identifications). Deciduous wood was predominant (37 of 69 certain wood identifications) including oak (*Quercus* sp., n = 18), maple (*Acer* sp.), and at least one more taxon indicated by the presence of scalariform perforation plates. Wood associated with both the quiver and one arrow contained coniferous wood, including pine (probably *Pinus sylvestris*). The quiver was also partly made of deciduous wood other than oak (with scalariform perforation plates; not hazel). The wood from the saddle was identified as maple (*Acer* sp.).

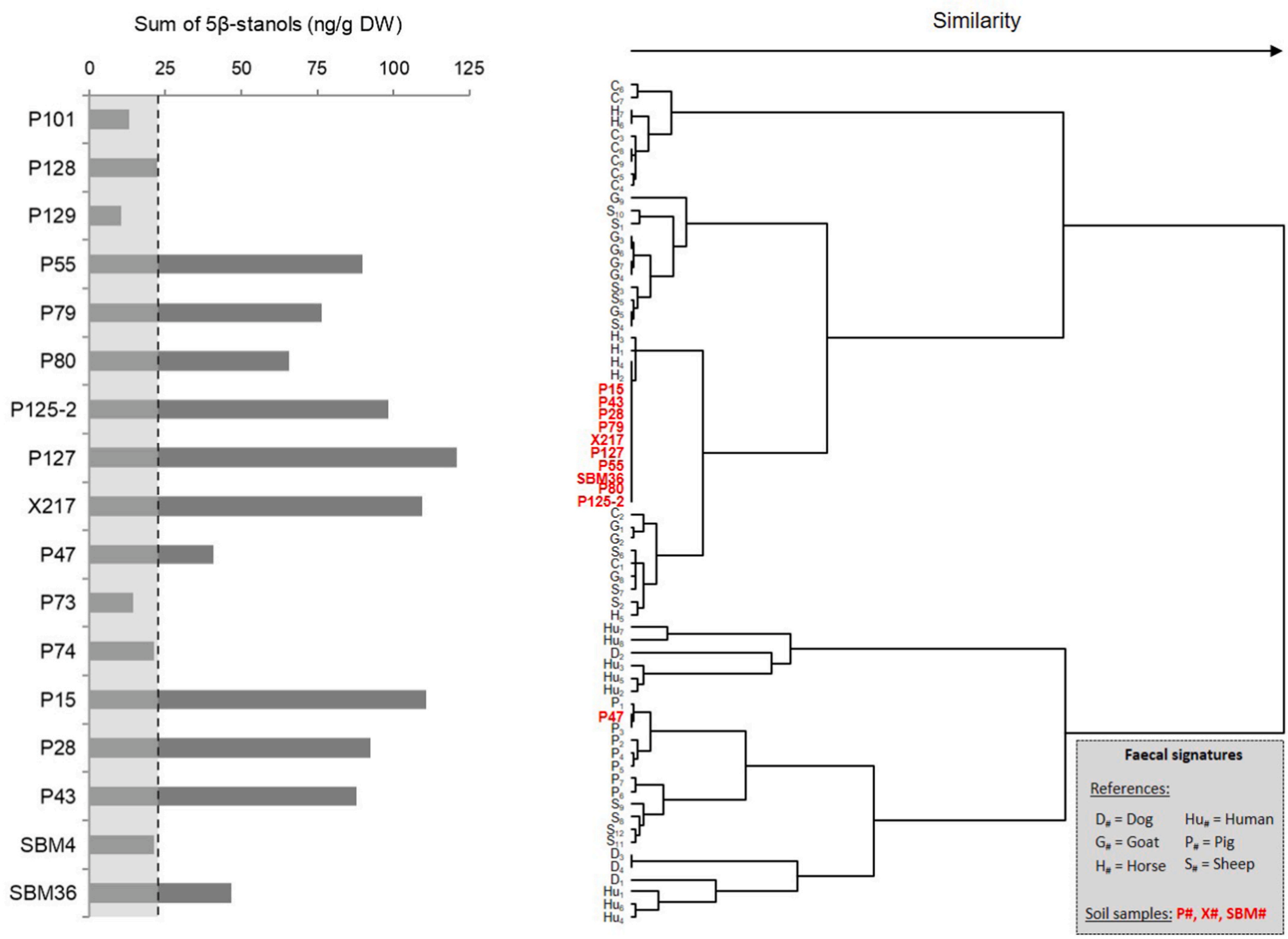

Fig. 8. Faecal lipid biomarkers: a. left, sum of 5β-stanols; right, hierarchical cluster; b. left, fragmentograms from two different samples (P80 with a suggested horse signature and p47 with a suggested pig signature); right, partial chromatograms of a steroid standard mixture (top) and a reindeer faeces lipid extract used as internal reference (second from the top). Fragmentograms of the lipid extract from sample P15. TIC = partial total ion current analysed in SIM mode. IS 2 = injection standard (5α-Cholestane). IS 1 = recovery standard (5β-Cholan-24-ol). Greyed compound names display unstudied steroids. Fragmentogram colours are purely graphical. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

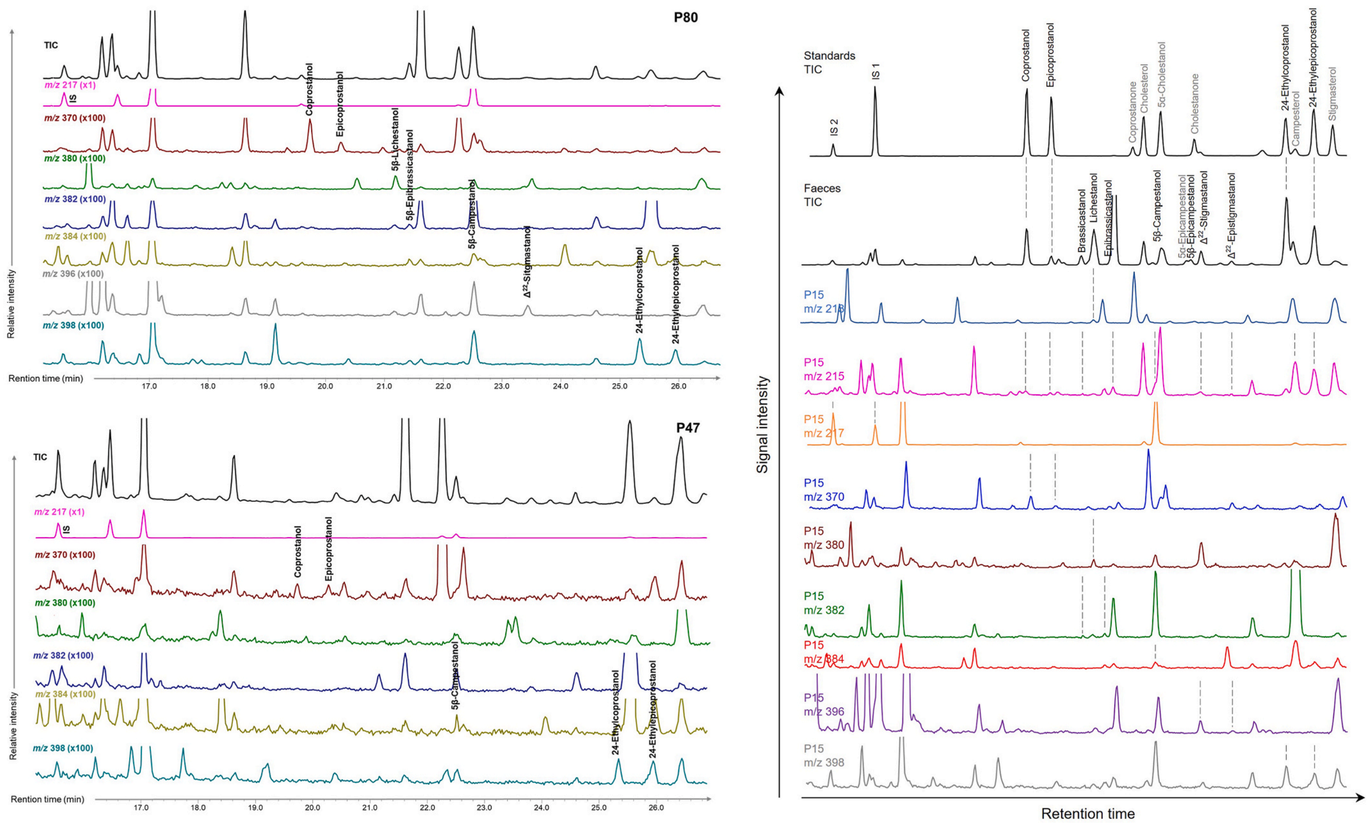

Fig. 8. (*continued*).

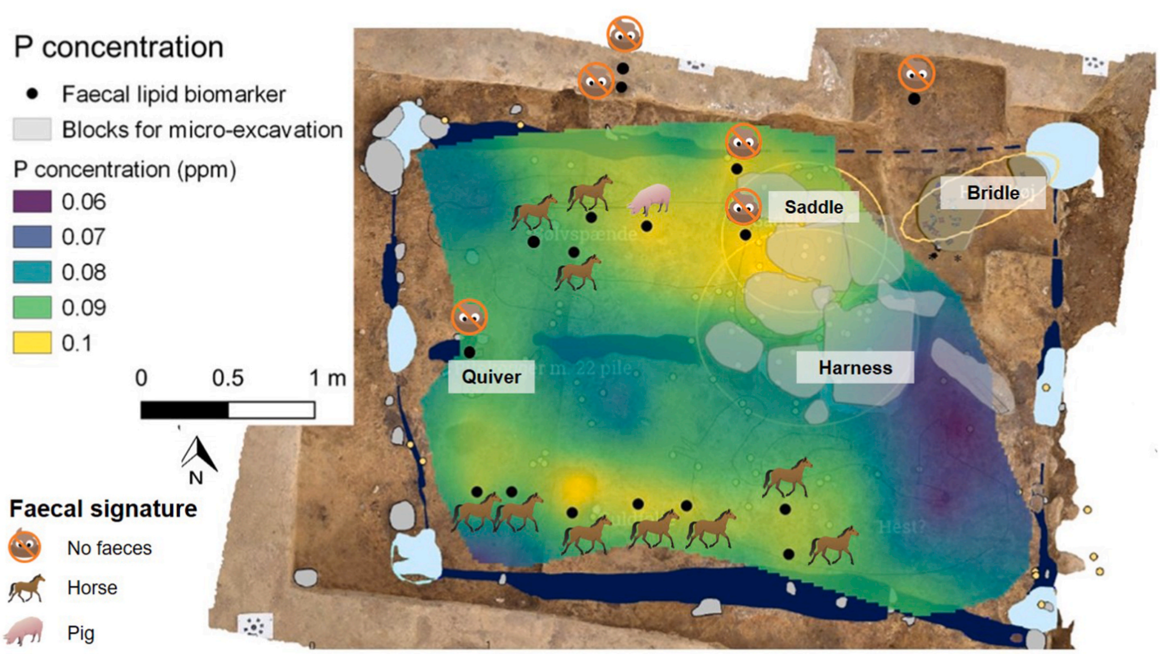

Fig. 9. Distribution of faecal lipid biomarker and P concentration.

4.3.4. *Microbotanical remains (phytoliths, pollen, and NPPs)*

The microbotanical signature from the basal fill was dominated by grasses and dryland herbs, while tree/shrub species and wetland herbs were present in small quantities. The phytolith samples yielded primarily (disarticulated) grass short cells (c. 70–80% per sample; mainly rondels, crenates) and long cells (elongate psilates and few elongate dentates) that point to the presence of culms and leaves of Pooid grasses (Table 2; full results presented in Out et al., forthcoming). Comparison with the control sample indicates that this concerns deposited material, presumably reflecting use of the nearby vegetation. The pollen assemblages varied highly within the grave, yet no more than it might be expected as a natural variation in a bedding of hay (Behre, 1981). The

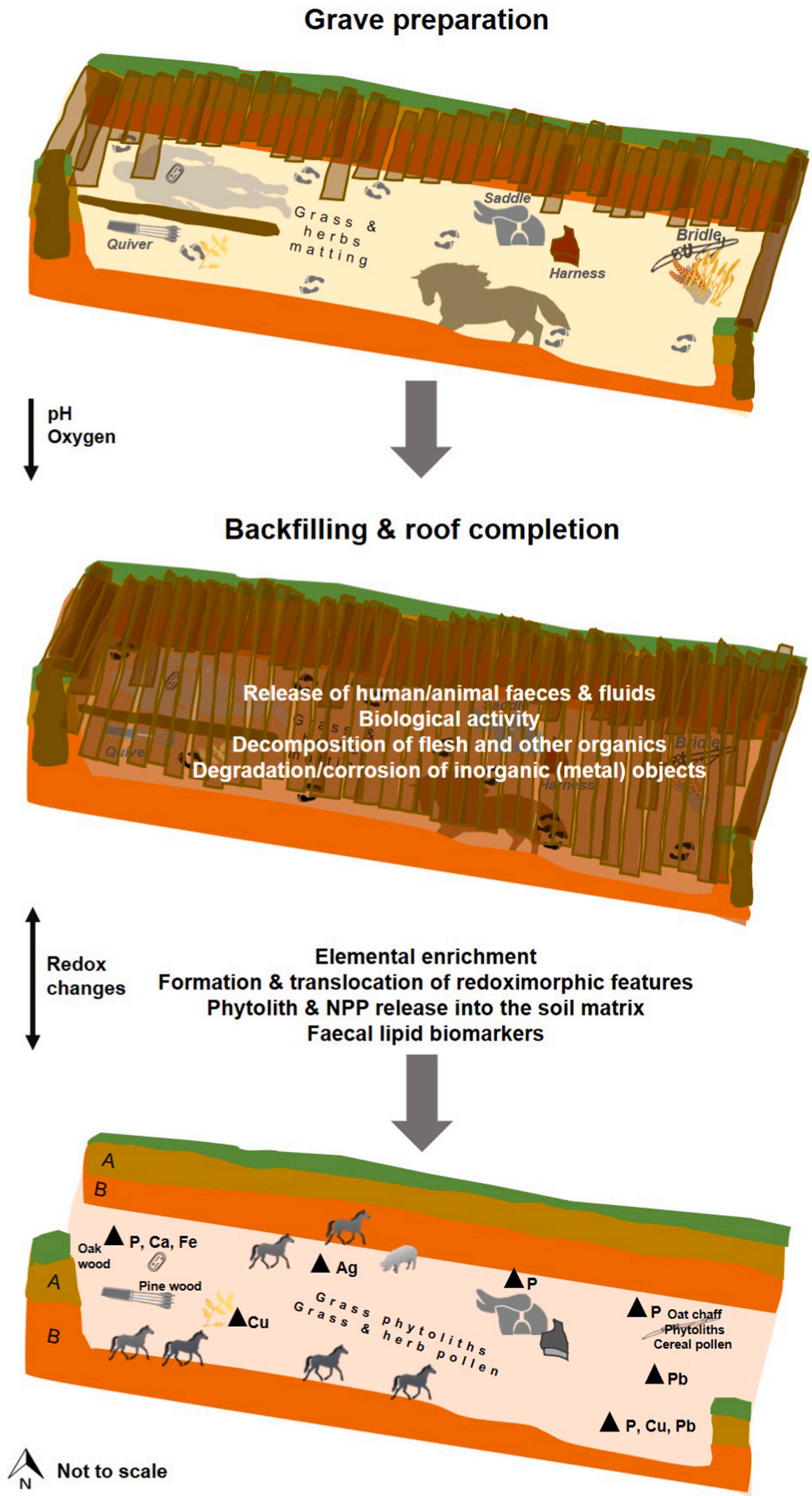

Fig. 10. Simplified model of grave establishment and post-depositional processes.

pollen assemblage was dominated by dryland herbs and grasses, with variable amounts of pollen from trees/shrubs, dwarf shrubs and wetland herbs (SM A2.5.2) (Out et al., 2021).

The area around the bridle and leather fragments (possible nosebag) was characterised by multiple, fine layers of silicified chaff, exceptionally well preserved, and yielded very high frequencies of oat/wheat pollen grains (up to 97%). Phytoliths were present in this chaff as (articulated, multi-cellular) silica skeletons of elongate dendritics from Pooid grasses, predominantly oat (94%). The soil sample from the edge of the quiver yielded a relatively high frequency of pollen as well as NPPs. The pollen assemblage had an overall combination that indicated a lining of hay/straw. Fungal spores, fungal fruit bodies and charcoal fragments were also found with relatively high frequencies in this sample (Fig. 7; SM A2.5.2).

4.3.5. Faecal lipid biomarkers

5β-Stanols were found in all seventeen soil samples analysed, but concentrations were very low (11–22 ng/g of soil) in the three control samples taken outside of the grave cut (Fig. 8). With 22 ng/g used as a threshold below which the grave fill samples were considered not to have received faecal inputs, samples P73, P74, P101, P129 and SBM4 were eliminated from the dataset, and are not discussed below. Concentrations of 5β-stanols in the remaining samples ranged from 41 to 228 ng/g, and there was a weak linear positive trend between P values and compound diversity (number of 5β-stanol compounds), indicating that the P values were partially derived from the faeces in the burial deposit, but were probably also derived from other decomposing organic remains (Fig. 8).

Up to ten different 5β-stanols were found in each of the grave fill samples, and samples showed similar relative distributions of compounds (ranges from 10 to 16% for coprostanol, epicoprostanol, lichestanol, epibrassicastanol, 5β-campestanol, Δ^{22}-stigmastanol, 24-ethylcoprostanol and 24-ethylepicoprostanol; see Fig. 8b and SM A1.4, Fig. 2). Hierarchical cluster analysis of the faecal lipid fingerprints from the grave fill and reference human and animal faecal samples revealed two main clusters: 1) an omnivore cluster, in which sample P47, in the northern part of the chamber, clustered with reference pig faecal fingerprints; and 2) a herbivore cluster, in which all of the remaining grave samples clustered with reference horse faecal fingerprints (Figs. 8 and 9). It is notable that both the compound diversity (5) and the concentration of 5β-stanols (41 ng/g) in the soil sample identified with pig faeces, were relatively low, even though pigs readily produce 5β-stanols from the sterols in their diet (Leeming et al., 1996). In contrast, horses are much less effective at producing 5β-stanols—yet the concentrations of faecal lipids in the soil samples identified with horse faeces was very high. It is therefore likely that only the horse faecal lipids represent an animal sacrificed during the burial ceremony, while the pig faecal lipids represent a trace amount of faecal material, derived from the nearby ground surface, which was trampled into the lower surface of the grave cut when the burial was being prepared and the bodies were being laid out.

5. Discussion

5.1. Burial environment and post-depositional processes

Micromorphological, phytolith and palynological results indicate that the burial was placed within a prepared surface dug into the B horizon of a fine sandy clay soil that originally might have been covered with grass and other plant material. The grave fill yielded patterned enrichment of selected elements that distinguished it from the underlying soil and the reference samples outside of the grave cut. In thin section, the presence of different soil microfabric types and the degree of mixing observed suggest that the soil material dug out to create the grave chamber was also used to backfill it. Phytoliths of grass culms and leaves, and herbal pollen from across the bottom of the grave, together with compressed plant residues seen in thin section, might be indicative of a grass and herb bedding laid on the floor of the chamber. Furthermore, the thin section of the grave fill included abundant charcoal, microcharcoal and rare artefacts (lithics, potsherds, etc.), consistent with residual domestic material commonly found in topsoils of Viking-Age sites.

Oak wood remains, including a roof-bearing plank and fragments around nail remains, match the common use of oak for different purposes, including as a construction material, in other Viking-Age burials (Grieg, 1928; Malmros, 2009; Müller-Wille, 1976; Roesdahl, 1977: 130–132). The chamber would have provided a closed space, sheltering the grave contents from light, rainfall, and, for a while, pedogenic processes and other agents of post-depositional change. A change from exposed (during pre-burial preparation and the burial ceremony itself) to protected conditions would influence the contact of the grave contents with soil water and air (Fig. 10). The combination of acidic pH (from local Luvisols) and organic matter from the grave goods and the inhumations might have enhanced microbial activity (Turner-Walker, 2007), influencing redox processes (Lang, 2014: 205–207) and speeding up the decomposition, dissolution and leaching of bone and other organic materials (Rousk et al., 2009; Tjelldén et al., 2018; Thomsen et al., 2008). This is supported by the features seen in the thin section from the grave fill, which include common amorphous organic matter and fungal sclerotia, and distinctive redoximorphic features probably linked to the decomposition and dissolution of the flesh and bone. Redoximorphic features also originate from decay processes, and they might be linked to enhanced biological activity (Lang, 2014: 205–207), but might also result from temporary flooding of the burial chamber, following rainstorms for example (see e.g., Puy et al., 2016). The presence of fungal sclerotia in thin sections from the grave fill and fungal fruiting bodies in the palynological assemblage from the quiver is likely to be associated with decomposing organic matter (Hawksworth and Wiltshire, 2011).

5.2. Burial practices

The multi-scale results revealed important details about the type, placement and preservation of grave goods and inhumations at Fregerslev II. Macrobotanical remains of oak, pine, maple and other deciduous wood, include local and non-local resources. In this grave, pine is only attested in the quiver and one arrow shaft. Indeed, pine is one of the suitable taxa for making arrows because of the characteristics of the wood (Beckhoff, 1965). Also, at the nearby late Iron Age site of Illerup, half of the 250 arrows identified were made of pine, probably imported (Jensen and Nørbach, 2009). While finds of saddles in other Viking-Age graves show that no specific wood was selected for this purpose (see e.g., Grieg, 1928; Szabó et al., 1985; Müller-Wille, 1987; Sørensen, 2001), the use of maple (*Acer* sp.) at Fregerslev II finds an important parallel in the saddle from a grave at Grimstrup, which was also made of maple (*Acer* cf. *platanoides*; Malmros, 2009; Stounmann, 2009).

In terms of spatial organisation, the area next to the saddle and bridle in the northern sector of the grave is characterised by high P enrichment that, together with microbotanical indicators of a grass and herb bedding, might be reflecting the presence of a human body, matching the orientation suggested so far (head to the west), with horse fittings laid at the foot. To the east of the saddle, remains of a bridle and leather (possibly from a nosebag) yielded a concentration of oat chaff phytoliths and high amounts of cereal pollen. P enrichment and faecal lipids associated with a horse signature in the southwest and south sectors of the chamber are located slightly west of the presumed position of a horse (based on the finding of remnants of a possible horse tooth). High concentrations of horse-associated faecal lipids suggest that a horse was sacrificed and buried as part of the funerary rite. As soon as a human or animal dies, muscles relax, and bowels and bladders empty; these processes can be accompanied by the release of fluids and waste from the bodies. Where humans or other animals are killed, such as in the case of ritual sacrifices, their faeces might be released and faecal residues might be preserved *in situ*. Viking-Age graves could take hours, days, or even weeks to prepare, and the body of the dead was often treated before burial (Price, 2010). It is therefore unlikely that faeces of the person (or people) honoured in the burial would have a significant impact on the faecal material found in the grave fill. The lack of faecal biomarkers associated with humans in the Fregerslev II grave therefore suggests that (a) the person (or people) interred in the grave died elsewhere, and (b) no humans were sacrificed at the grave site during the burial ceremony itself.

Enrichment hotspots of Ag, Cu and Pb are likely associated with the presence of specific items. Ag enrichment, for example, matches the location of a silver brooch, while Cu concentrates where the gold foil was found. Containers or tableware in the grave might have made from organic materials, with only minor functional and decorative metal mounts.

6. Conclusions

The integration of multiple methods at Fregerslev II provided unique records for understanding and reconstructing the character and processes of a burial hampered by poor preservation conditions. Geochemical mapping captured ephemeral signatures of depositional and taphonomic processes that were instrumental to the construction, degradation and preservation of the burial and its contents. Soil records reflected environmental conditions that prevented the preservation of bone from human and animal bodies. In Viking-Age burial practices, the body was not treated in view of long-term preservation. At Fregerslev II, enhanced P concentrations and fungal remains reflected the decomposition of a human body and, likely, a horse. Faecal lipid analysis established the presence of pig faeces (in trace amounts) and horse faeces, the latter in high enough quantities to suggest that a horse might have been sacrificed at the grave site.

Soil, archaeobotanical, and palynological records further showed that the grave fill consisted of local sediments, and the inhumation and grave goods were placed over a matting of hay/stray grass stalks and leaves, and herbs. Movement of people and goods during the deposition would explain the ubiquitous mixing observed at the bottom of the grave, and the trace amounts of faecal lipids associated with pigs. A concentration of phytoliths and pollen from oat might be associated with food for the horse. Wood identification confirmed the presence of local taxa commonly used in contemporary Viking-Age graves (e.g., oak), but also non-local taxa such as pine, which must have been imported or traded for, and might therefore be an indicator of the wealth or prestige reflected in or communicated by the burial. Thus combined, the records returned a portrait of a high-status grave, comparable to contemporary equestrian burials. The remains had decomposed to the point, however, where artefacts and biological remains alike were rendered all but untraceable by traditional excavation methods. The integrated multi-proxy analysis of organic and inorganic materials enabled a detailed characterisation of the burial, demonstrating how a high-resolution approach can transform archaeological interpretation.

Our sampling and analytical program employed methods that remain largely peripheral in commercial and professional archaeology. At Fregerslev II, continuous dialogue and interactions between the museum and the field excavation team, conservators, and researchers enabled developing, refining, and adjusting analytical and sampling strategies, while negotiating the pace and resources of museum-led excavations with the time and investment requirements of archaeological science methods. We hope this study demonstrates the feasibility and value of integrating multiple archaeological science methods, and the mutual benefits of collaboration between museums and university-based researchers to maximise the recovery and interpretation of archaeological remains.

Declaration of competing interest

None.

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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.2022.105589.

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