

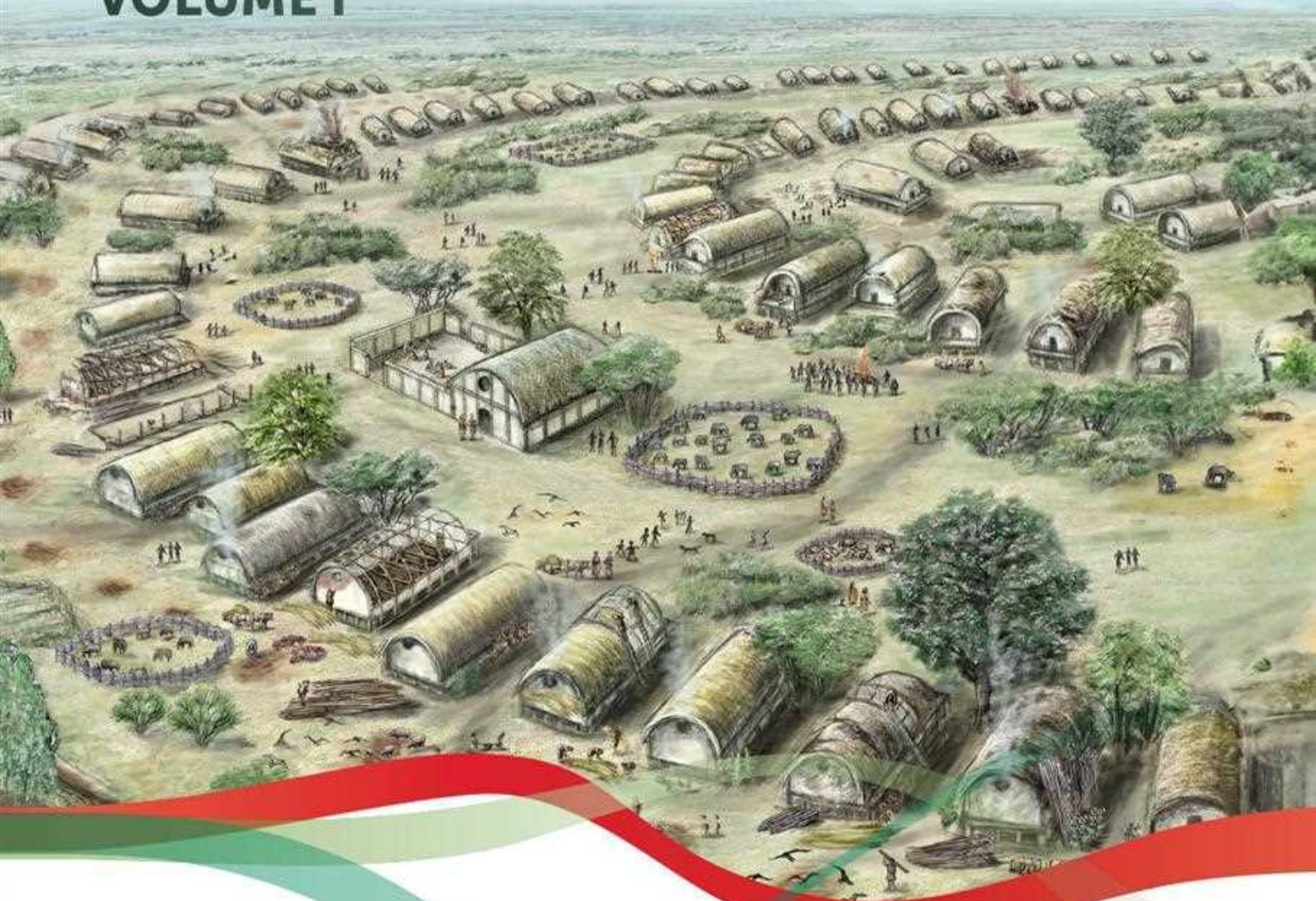
Edited by:

**ROBERT HOFMANN, WIEBKE KIRLEIS, JOHANNES MÜLLER, VITALII RUD,
STANISLAV ŤERNA†, MYKHAILO VIDEIKO**

From Ros to Prut:

TRANSFORMATIONS OF TRYPILLIA SETTLEMENTS

VOLUME I



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Foreword of the series editors

With this book series, the Collaborative Research Centre Scales of Transformation: Human-Environmental Interaction in Prehistoric and Archaic Societies (CRC 1266) at Kiel University enables the bundled presentation of current research outcomes of the multiple aspects of socio-environmental transformations in ancient societies. As editors of this publication platform, we are pleased to be able to publish monographs with detailed basic data and comprehensive interpretations from different case studies and landscapes as well as the extensive output from numerous scientific meetings and international workshops.

The book series is dedicated to the fundamental research questions of CRC 1266, dealing with transformations on different temporal, spatial and social scales, here defined as processes leading to a substantial and enduring reorganization of socio-environmental interaction patterns. What are the substantial transformations that describe human development from 15,000 years ago to the beginning of the Common Era? How did interactions between the natural environment and human populations change over time? What role did humans play as cognitive actors trying to deal with changing social and environmental conditions? Which factors triggered the transformations that led to substantial societal and economic inequality?

The understanding of human practices within often intertwined social and environmental contexts is one of the most fundamental aspects of archaeological research. Moreover, in current debates, the dynamics and feedback involved in human-environmental relationships have become a major issue, particularly when looking at the detectable and sometimes devastating consequences of human interference with nature. Archaeology, with its long-term perspective on human societies and landscapes, is in the unique position to trace and link comparable phenomena in the past, to study human involvement with the natural environment, to investigate the impact of humans on nature, and to outline the consequences of environmental change on human societies. Modern interdisciplinary research enables us to reach beyond simplistic monocausal lines of explanation and overcome evolutionary perspectives. Looking at the period from 15,000 to 1 BCE, CRC 1266 takes a diachronic view in order to investigate transformations involved in the development of Late Pleistocene hunter-gatherers, horticulturalists, early agriculturalists, early metallurgists as well as early state societies, thus covering a wide array of societal formations and environmental conditions.

In the last decade, archaeologists from Kiel University carried out intensive fieldwork and collaborative efforts with colleagues from Ukraine and Moldova to analyse population agglomeration in Trypillia mega-sites some 6000 years ago. Multiple monographs and papers have been published on this exciting phenomenon. Still, new aspects of Trypillia societies are to be discovered. The two volumes of STPAS 19 compile – under the title “From Ros to Prut, Transformations of Trypillia settlements” – archaeological, geophysical, archaeobotanical, archaeozoological and geoarchaeological contributions on the economy, settlement patterns, material

culture and dating of Chalcolithic societies in the territory of present-day Ukraine and Moldova. Organised across three test regions spanning an East-West transect, the two volumes bring together in-depth investigations of selected key sites and regional reconstructions (Volumes 1 and 2), which are used as the basis for a cross-regional comparison (Volume 2). This comparison, based on the many pieces of the puzzle compiled here for the different test regions, enable a deeper understanding of the particularities, but also the overarching commonalities, in the process of socio-ecological transformations of the Trypillia societies and their environment in diachronic perspective, as well as on spatial scale.

We are thankful to the multiple authors and the graphic designers Esther Thelen, Ralf Opitz and Carsten Reckweg for their deep engagement with the volumes and Sara Krubeck for harmonising the texts and reference lists. We also want to thank Karsten Wentink and Eric van den Bandt from Sidestone Press for their responsive support in realising these volumes and Nicole Taylor for organising the entire publication process in her experienced and high-quality manner.

Wiebke Kirleis and Johannes Müller

*To the memory of Stanislav Ľerna,
a cherished colleague and inspiring friend.*



Contents

Foreword of the series editors	5
Foreword of the editors	9
TRYPILLIA RESEARCH DECOLONISATION	13
1. Modern Trypillia Transformation research: decolonising through new research concepts, methods and results	15
<i>Johannes Müller</i>	
MAIDANESTSKE AND SINYUKHA REGION (REGION A)	21
2. Report on the fieldwork of 2016 in the Trypillia mega-site Maidanetske: Investigations on the development and internal structuring	23
<i>Robert Hofmann, Johannes Müller, Wiebke Kirleis, Mykhailo Videiko, Hans-Rudolf Bork, René Ohlrau, Natalia Burdo, Liudmyla Shatilo, Vitalii Rud, Stefan Dreibrodt, Knut Rassmann, Mariia Videiko</i>	
3. Geophysical Investigations at Maidanetske	107
<i>Natalie Pickartz, Tina Wunderlich, Erica Corradini, Knut Rassmann, Dennis Wilken, Wolfgang Rabbel</i>	
4. Geoarchaeological analyses on daub pieces from Maidanetske – A treatise on reconstructing burning temperatures of houses and daub processing	115
<i>Stefan Dreibrodt, Sarah Martini, Robert Hofmann, Marta Dal Corso, Wiebke Kirleis, Johannes Müller</i>	
5. The geoarchaeological record of the Chalcolithic Trypillian mega-site Maidanetske, central Ukraine	137
<i>Stefan Dreibrodt, Sarah Martini, Robert Hofmann, Marta Dal Corso, Wiebke Kirleis, Johannes Müller</i>	
6. Worked osseous materials from the site of Maidanetske: is that all there is?	157
<i>Andreea Țerna</i>	
7. Plant economy and local environment at the Trypillia mega-site Maidanetske from botanical macro-remains	167
<i>Marta Dal Corso, Wiebke Kirleis</i>	

8. X-Ray computer microtomography (XRay-μCT) as a fast non-invasive technique for compositional studies of burnt prehistorical materials: results and implications from burnt daub at Maidanetske, central Ukraine	191
<i>Stefan Dreibrodt, Christopher Heilmann, Marta Dal Corso, Pia Bodden, Robert Hofmann, Mykhailo Videiko, Johannes Müller, Wiebke Kirleis, Astrid Holzheid</i>	
9. Archaeozoological and taphonomic examinations carried out on faunal remains from Ukrainian-German excavations at Maidanetske from 2013 to 2016	217
<i>Norbert Benecke, Robert Hofmann, Mykhailo Videiko, Johannes Müller</i>	
10. The contribution of Chalcolithic terrestrial snail shells from Maidanetske to environmental reconstruction	251
<i>Frank Schlütz, Marta Dal Corso, Wiebke Kirleis</i>	
11. Before Talianki: large Trypillian sites built before the largest mega-sites in the Sinyukha basin	257
<i>Robert Hofmann, Liudmyla Shatilo</i>	
12. What was the significance of small Trypillia settlements? Report on a test excavation in Moshuriv 1, Sinyukha river basin, Ukraine	309
<i>Robert Hofmann, Liudmyla Shatilo, Mykhailo Videiko</i>	
13. Trypillia mega-sites vicinity: the chronology of several small sites of the Southern Buh left bank region	323
<i>Mykhailo Videiko, Vitalii Rud, Robert Hofmann, Vladyslav Chabaniuk</i>	

VOLUME II contains a further 8 chapters, organised into the following sections:

BUG-DNIESTER INTERFLUVE (REGION B)
PRUT/DNIESTER INTERFLUVE (REGION C)
TRYPILLIA MACROSCALE
CONCLUSION

8. X-Ray computer microtomography (XRay- μ CT) as a fast non-invasive technique for compositional studies of burnt prehistorical materials: results and implications from burnt daub at Maidanetske, central Ukraine

Stefan Dreibrodt, Christopher Heilmann, Marta Dal Corso, Pia Bodden, Robert Hofmann, Mykhailo Videiko, Johannes Müller, Wiebke Kirleis, Astrid Holzheid

Abstract

In this paper we present results from an XRay- μ CT study on burnt daub samples unearthed at the Trypillian mega settlement site of Maidanetske, central Ukraine. The volume and characteristics of pores of eight pieces of burnt daub were measured via XRay- μ CT using the SkyScan1172 program and the raw data were evaluated using the NRecon program. The analysed daub pieces display porosities between $7.5 \pm 1.9\%$ and $22.3 \pm 4.3\%$. Thorough visual inspection of the pictures resulted in estimations of organic remains imprints and non-organic cavities. The former consist of cereal grains, glumes, culm fragments and spikelet forks/rachis segments; the latter consist of voids, cracks, and inhomogeneities. The content of the organic remains was calculated by subtracting the cracks from the total porosity. The resulting contents of organic remains in the daub pieces totals between $0.6 \pm 0.1\%$ and $15.1 \pm 6.0\%$. Archaeological daub classification, phytolith analysis and geochemical composition validated the XRay μ CT results. Given the non-invasive character and the manageable effort of using this method, XRay μ CT might be considered as a promising supplemental technique for the internal study of burnt artefacts.

Additional outcomes of this multidisciplinary daub study include information on daub processing technology and resource use as well as giving some ideas about social interaction within Trypillian communities. Chaff was preferentially used for outer walls of houses. Urine was probably added to the daub mass to mix it and make it malleable. This indicates a certain standardisation of daub production, which is

to be tested by further analysis. Since the harvest remains used as organic temper were present in large amounts annually, and storage pits filled with harvest remains are lacking so far in the extensive Trypillian archaeological record, this suggests that Trypillian house building was probably a well-planned communal activity which followed the annual cereal harvest.

Introduction

Burnt daub has been a research object of prehistorical archaeology for a long time. Research questions have concerned, for example, aspects of architecture, the provenance of building materials, the amount of working effort, the burning environment or geophysical mapping of single houses and complete settlements (*e.g.* Shaffer 1993; Stevanović 1997; Chapman 1999; Tringham 2005; Fries-Knoblach 2009; Lichter 2016; Pickartz *et al.* 2019; Chapter 4, this work, Vol. I).

Burnt daub is easily accessible for investigations as it is often present at prehistoric archaeological excavations in large amounts. Analysis of burnt daub pieces has so far been focussed on macroscopical classification, since compositional analysis after crushing the daub pieces or studying daub in thin sections is very time-consuming. In this paper we present a new approach which allows rapid insight into burnt daub internal composition using X-ray μ CT. This pilot study was supplemented by traditional methods such as macroscopical daub classification, phytolith analysis and geochemical analysis (ped-xrf) to validate the results and to check the potential of the X-ray μ CT method to deliver additional archaeobotanical data and ideas about plant resource management at a Chalcolithic Trypillian mega site in central Ukraine (Maidanetske).

Materials and methods

Study site

The burnt daub pieces analysed in this study were unearthed during archaeological excavations at Maidanetske, a Chalcolithic mega-site in central Ukraine. These early agglomerations of Chalcolithic people resulted in an incomparable amount of archaeological remains of buildings in earthen architecture, *i.e.* daub (Kruts 1989; Shmaglij and Videiko 2005; Menotti and Korvin-Piotrovskiy 2012; Chapman *et al.* 2014; Müller 2016; Müller *et al.* 2016b; Pickartz *et al.* 2019).

Large Trypillian settlements (so-called mega-sites) have been found especially in the Bug-Dnieper interfluve, *i.e.* the north Pontic steppe regions of central Ukraine (*e.g.* Kruts 2012; Wengrow 2015; Müller *et al.* 2016b; Gaydarska *et al.* 2019). Maidanetske (Cherkasy region, Ukraine), with an area of 200 ha, is one of the largest Trypillian mega-sites, having had up to 15,000 inhabitants and being occupied for ca. 200–350 years, with a peak in house occupation during the century between ca. 3800 and 3700 BCE (Müller *et al.* 2016a; Ohlrau 2020; Shatilo 2021). It is evident that reliable food supply is a relevant precondition for the existence of such mega-sites that are considered proto-urban, building upon an agrarian subsistence economy (*e.g.* Pashkevich and Videiko 2006; Harper 2011; Shukurov *et al.* 2015; Ohlrau *et al.* 2016).

The use of systematic extensive flotation programmes to acquire charred archaeobotanical material from archaeological stratigraphy containing cereal grains and chaff parts is a recent development and can help improve our knowledge of plant use in Trypillia (Kirleis and Dal Corso 2016; Dal Corso *et al.* 2019; Terna *et al.* 2019). In particular, excavations of deep pits next to house floors are highly

beneficial and bring numerous finds to light. This suggests that pedogenetic processes produce a taphonomic bias, which may affect the preservation of charred organic remains in most of the settlement/house stratigraphy that lies less than a metre below the modern soil surface (Dreibrodt *et al.* 2022). Recent archaeobotanical and zooarchaeological studies highlight the relevance of cereal crops, as indicated by, *e.g.*, intensive use of cereal by-products and the herding of domestic animals (Dal Corso *et al.* 2018; Dal Corso *et al.* 2019; Kirleis and Dal Corso 2016).

Archaeobotanical research history on the Trypillia culture has studied plant imprints of cereal by-products and sometimes imprints of individual grains in ceramics and daub as a main source for reconstruction of the plant economy (Yanushevich 1975; Yanushevich 1978; Pashkevich and Videiko 2006 and references therein). Plant imprints in daub from Neolithic and Chalcolithic sites have been recently re-visited (*e.g.* An *et al.* 2018). The aim of this paper is to develop a rapid non-invasive procedure to quantify the amount and characterise the types of cereal by-products in the daub used for the construction of Trypillian houses.

Methods

Daub sampling and classification

At Maidanetske, similarly to other Trypillian settlements, daub from walls and other architectural parts has been preserved until present, since most of the houses were burnt and buried by the subsequent Chernozem formation (Dreibrodt *et al.* 2022). During the excavation of House 44 at Maidanetske, the locations, masses and types of daub were systematically documented with point coordinates and recorded in a grid of $1 \times 1 \text{ m}^2$ cell sizes (Müller *et al.* 2017). Details of the advanced archaeological classification of the daub are given as supplementary information (Suppl. 1).

Eight daub samples from House 44 (Trench 51) were chosen that represent three material types (Fig. 1). The sample and unit number, location in the trench, macroscopic properties and sample code of the μ CT laboratory are listed in Table 1.

The floor plan of the house follows a common template of houses of the Tomashovka local group: on top of a massive clay platform, the main floor of the house is located, with an anteroom in the northeast, a main room in the southwest, a podium on the southeastern longitudinal wall of the main room, a furnace in the northern corner of the main room and a central clay installation.

X-ray microtomography

Seven of the eight samples investigated were chaff-tempered daub fragments of Material Types 2 and 3 and were selected to acquire detailed information about the organic temper components, the daub porosity, and the spatial distribution of the components in the daub samples, using up to three individual ROI (region of interest) sections. As a control, one sample was chosen representing non-chaff-tempered daub (Material Type 1).

The selected daub samples are of various sizes and irregular in shape. Since the sample chamber of the X-ray microtomograph allows the processing of samples with a maximum diameter of 34 mm and a maximum height of 55 mm, rectangular bars with dimensions of (x, y) $\sim 20 \times 20 \text{ mm}$ were cut out of the daub pieces. The top and bottom surfaces were left untreated in order to preserve unaffected the internal structure of the daub; this resulted in various heights (z) of the bars, ranging from $\sim 25 \text{ mm}$ to $\sim 55 \text{ mm}$ (see Fig. 2).

X-ray microtomography characterises internal structures two- and three-dimensionally. Because X-ray microtomography is a non-destructive technique,

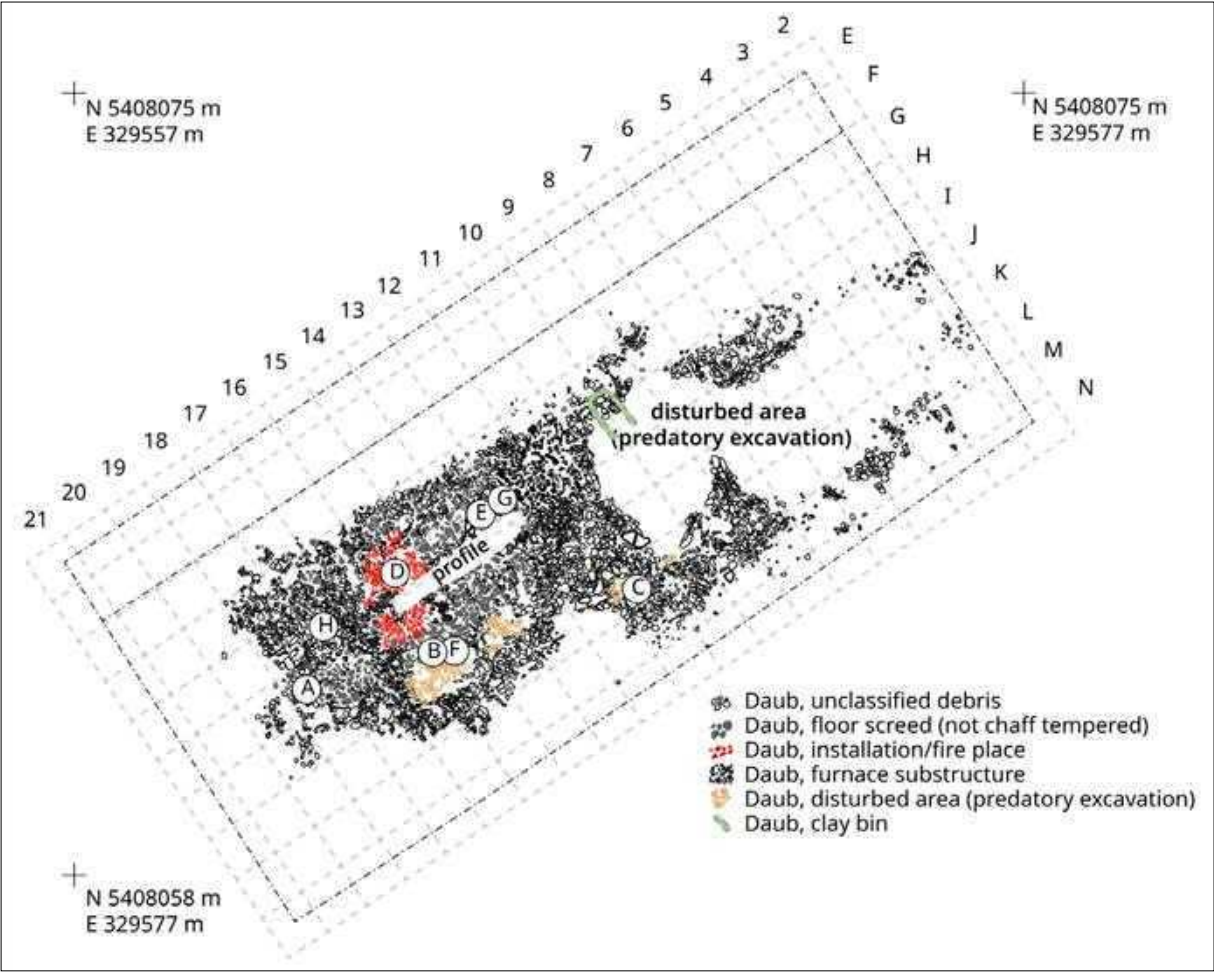


Figure 1. Excavation plan of House 44 in Maidanetske with location of the eight investigated samples. Coordinates refer to the UTM coordinate system (zone 36N) and WGS 84 ellipsoid.

Find-ID	Feature-ID	Location	Daub category ¹	Material type ²	μCT # ³	Architectural context	Organic temper
51394	51003	I 13	1	3	E	Floor sub-layer	Plant-based temper
51366	51003	J 18	1	2	A	Exterior wall?	Plant-based temper
51416	51009	I 15	1	2	D	Clay installation	Plant-based temper
51413	51009	I 12	2	1	G	Floor screed	Non-plant-based temper
51392	51003	I 17	4	3	H	Clay installation	Plant-based temper
51390	51003	K 15	4	3	F	Podium	Plant-based temper
51402	51003	K 15	5	2	B	Podium	Plant-based temper
51379	51003	L 11	5	2	C	Exterior wall?	Plant-based temper

Table 1. Selected daub samples and related classifications.
¹ classification after Müller et al. (2017); ² see text for more details; ³ internal labelling by μCT laboratory.



Figure 2 (above). Daub samples machined to $\sim 20 \times 20$ mm rectangular bars.

it has a decisive advantage over wavelength dispersive X-ray fluorescence spectrometry and thin section petrography when investigating archaeological finds. Volume fractions of phases (inclusions) in the temper can be characterised and in addition 3D visible orientation of pores can be detected and classified. This allows, *e.g.*, conclusions to be made about the manufacturing procedure of the pottery (for more details see Menne *et al.* 2020). X-ray microtomographic devices consist of an X-ray source, a rotating sample holder and a detector. The X-rays penetrating through the sample are absorbed differently by the various phases present in the sample and are displayed space-resolved at the detector. The differences in X-ray absorption correspond to the densities of the present phases. When reconstructing the 2D and 3D images, grey values based on the degree of X-ray absorption are assigned to each pixel (2D bitmap) and voxel (grid in 3D space), respectively. Pores with the density of air are displayed in black or dark grey, while phases with high densities (*e.g.* clay, mineral and/or rock fragments used as matrix and temper, respectively, in ancient pottery) appear in light grey or even white (for more details regarding methodology, see Kahl *et al.* 2013 and references therein).

Sample-specific measurement routine and data processing

The X-ray microtomography scans in this study were carried out using the SkyScan 1172 system (SkyScan, Belgium) at the Institute for Geosciences, Kiel University, Germany. The following parameters were used: beam energy of 100 kV, beam current of 100 μ A with an aluminium-copper filter, sample rotation (360°) with a step width of 0.5° , and medium resolution (detector resolution: 17.3 μ m per pixel). Given the fragility of the daub samples, these were placed inside a frame of modelling clay and then mounted on the sample holder. The vertical maximum extension of a single scan corresponds to a height of 20 mm. The majority of the analysed daub samples are taller (Fig. 2). The sample holder was moved in a vertical direction and multiple scans were conducted (oversize scans) to scan the tall samples. After the data acquisition, the 2D and 3D images were reconstructed using the program NRecon (SkyScan). For each sample ~ 1000 stacked layered images were generated and correspondingly more for oversize scans (up to 3000 stacked layered images). Each layered image consists of isometric voxels (volumetric pixels) whose sizes depend on the detector resolution. Therefore, the stacked images give volumetric information. The SkyScan software tools CTAn and CTVol were used to decode the data into black and white images as well as to visualise and calculate the individual volumes of the various phases present, extracted from the binarised images. Hence, continuous image volume inspections were undertaken using computer-aided morphometry and quantitative image analysis. The reconstructed scans were apportioned to individual ROI sections, not only to keep the computing time manageable but also to allow direct comparison of the various daub samples.

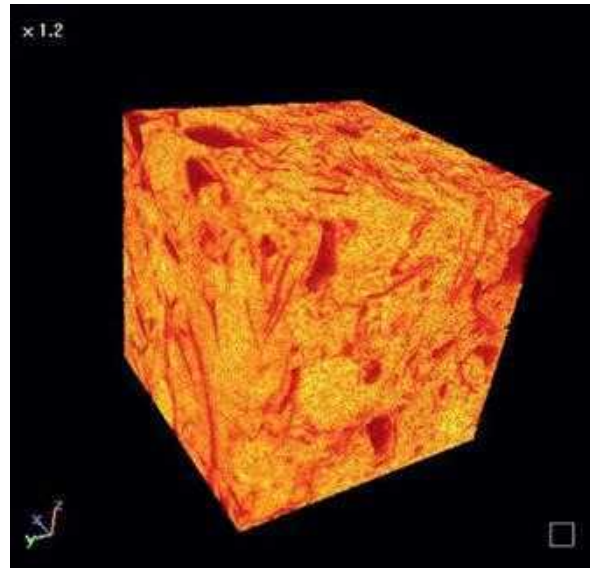


Figure 3. Representative virtually cut cube (μ CT#: A). Please note: (i) $x, y, z = 1$ cm; (ii) the grey tone image is coloured for better visibility.

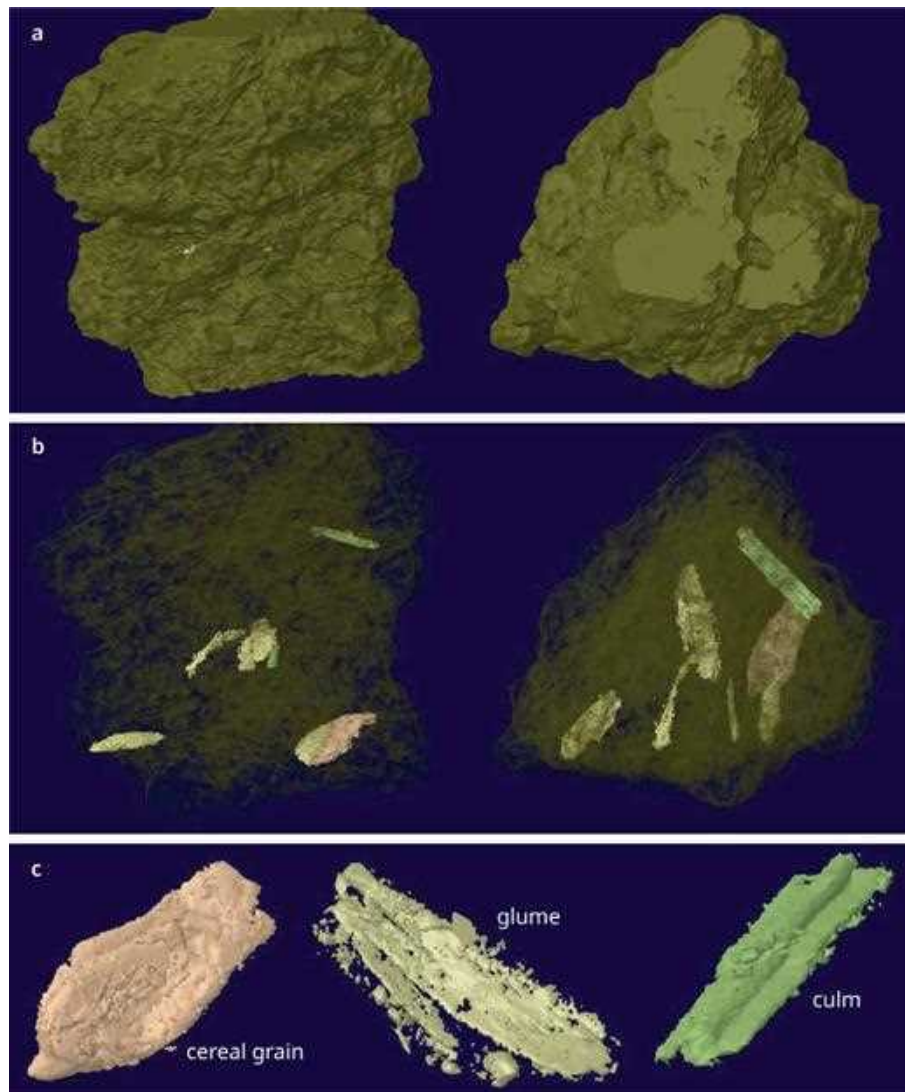


Figure 4. Correlation of pores of supposed organic origin in daub to mm-sized organic components: (a) 3D reconstruction of the outside surfaces of the daub as side view (left) and top view (right); (b) Unmasked pores using translucent matrix (left: side view; right: top view); (c) Distinct shapes of pores assigned to various organic components (mm-sized).

This was done using the CTVol software 're-slice' tool. Depending on the length of the daub samples, up to three cube-shaped areas ($1 \times 1 \times 1$ cm) were virtually extracted from the reconstructed 3D images. Figure 3 is a representative example of one of these cubes. As the cubes were cut virtually, there was no mechanical damage to the surfaces and edges.

The virtually cut cubes are mainly used to derive the portion of the used organic temper from the total fraction of porosity, based on a standardised volume ($1 \times 1 \times 1$ cm) to better compare the various daub samples. The method developed to determine organic temper proportions is described in detail below.

Assignment of pore shapes to specific organic components and determination of total volume of organic fraction

Organic components were used as temper in the daub, as attested by macroscopic observation of plant imprints and by phytolith analysis (Dal Corso *et al.* 2019). The organic components either decomposed over the years or were burnt to ash during the final burning of the houses, leaving behind impressions that are now seen as specifically shaped pores in the ancient daub samples. Figure 4 illustrates the various steps required to correlate pores of organic origin with the organic components after X-ray microtomography scans. First, a 3D image of the daub must be reconstructed (Fig. 4a). To unmask the pores, the matrix has to be displayed translucently (Fig. 4b). For better visualisation, some of the pores in Figure 4b are coloured. Finally, optical enlargement allows the pores to be assigned to various anatomical plant parts such as cereal grains, glumes, and culms (Fig. 4c).

The total pore volume consists not only of organic temper but also of cracks, vesicles and micropores formed during the manufacturing process of the daub and its use as building material. As a result, almost all objects (pores of varying origin) are interconnected and it is not possible to distinguish them automatically, other than by the volume of the micropores. In this study, micropores are defined as pores with volumes of less than 0.05 mm^3 , as the majority of all assigned fragments of organic parts are larger than 0.05 mm^3 . Fragments of organic parts are those organic components that are only preserved as several pieces and are no longer preserved as one piece in their original shape, as exemplified in Figure 4c. The SkyScan software CTAn allows automatic subtraction of pores with pre-defined volumes. All pores with volumes of less than 0.05 mm^3 , *i.e.* all micropores, were subtracted from the total pore volume. To be able to subtract either the organic components or the cracks and vesicles from the total pore volume, it has proven useful to manually encircle cracks and vesicles rather than organic components, due to their complexities of shape. Thus, the proportions of organic temper in the studied daub samples (*i.e.* all pores of organic origin) were derived by subtracting the volumes of all cracks, vesicles and micropores from the total pore volume.

To verify not only the volume fractions calculated by the Skyscan software but also to ensure that the total pore volume is not significantly influenced by the manufacturing procedure of the daub, a quantitative test to verify the calculated volume fractions was performed. Menne *et al.* (2020) validated the calculated volume fractions. They conducted a quantitative test by placing ink filler balls with standardised diameters into a plastic container of known volume. This allows not only a precise calculation of the pore volumes or cavities as artificial porosities but also of the total ink filler ball volume. Figure 5 illustrates (a) the ink filler balls inside the plastic container and (b) the 3D view of the absorbed X-rays. All ink filler balls are made of glass, except one ball that is made of plastic and is easily recognisable in Figure 5b as a darker sphere, due to the lower density of plastic compared to glass. Menne *et al.* (2020) confirmed the software-based calculated pore volume by manually calculating the volumes.

Figure 5. Validation of software-based volume fractions: (a) 97 ink filler balls (diameter: 2.5 mm) in the plastic container; (b) 3D view of the X-ray microtomography scan; Figure is modified after Menne et al. (2020).

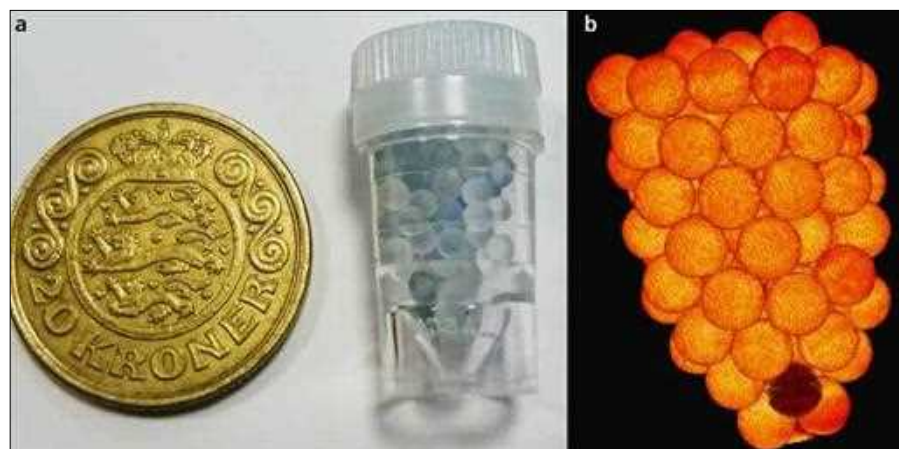
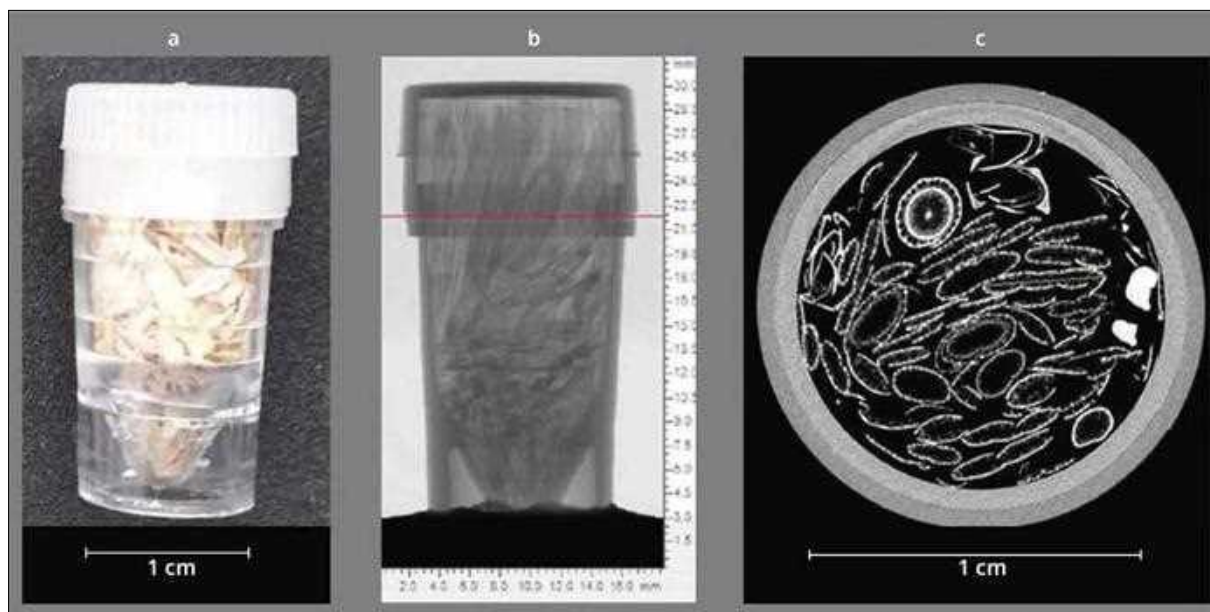


Figure 6. Daub bricks after air drying (left) and firing at 550°C (right).



To test if manufacturing procedures influence the total pore volume, experimental daub bricklets were produced by mixing loess, culms and glumes of einkorn (*Triticum monococcum*, a hulled wheat seen in the Trypillia period; Kirleis and Dal Corso 2016) with water, in proportions similar to those of the daub samples from Maidanetske. The volume ratios were 1400 ml (loess) : 250 ml (culm fragments) : 250 ml (glumes), corresponding to 20 g (loess) : 0.31 g (culm fragments) : 0.47 g (glumes). The solid mixture was worked with the aid of water to ensure a certain stiffness that would allow further handling. Finally, rectangular bricks with dimensions of $\sim 1 \times 1 \times 4$ cm were formed (Fig. 6). X-ray microtomography scans were carried out after air-drying the daub bricklets for 2 weeks at room temperature. The bricklets were then placed in a preheated (50°C) muffle furnace, fired under oxidised conditions (air) for 2 hours at 550°C and 800°C respectively, after which the X-ray microtomography scans were repeated.

To ensure proper recognition and classification of the organic components, culms, glumes and spikelets of einkorn were placed in a plastic container and X-ray microtomography scans were performed (Fig. 7). The culms to glumes ratio was identical to the final brick mixtures. The filled and more rounded objects in Figure 7c are whole cereal grains, the elongated objects are culm fragments and the more flat and linear appearing objects are glumes.



The known contours of the raw materials (Fig. 7c), allowed the recognition of components in the fired experimental daub bricklets as not yet carbonised organic matter. Figure 8 displays 2D layers of the reconstructed 3D images of an unfired daub bricklet (Fig. 8a, c) and the same daub bricklet fired at 550°C (Fig. 8b, d). Care was taken in displaying transverse sections at very similar vertical positions, for better comparison of the unfired and fired textures, *i.e.* Figures 8a and 8b as well as Figures 8c and 8d are 2D layers at similar vertical positions, respectively. However, Figures 8c and 8d are intentionally vertically off-set from Figures 8a and 8b. Two important observations can be made based on the optical appearance and morphometric analyses: (1) about 50% of the organic components remained unchanged, *i.e.* those components did not burn, instead the grains survived the firing of the bricklets at either 550°C or 800°C; (2) neither further cavities nor additional cracks formed from the firing of the daub bricklets in the experiment. This implies that the increase in total pore volume is closely correlated to the burning of the organic components.

Figure 7. Analysis of raw material used in experimental daub bricks: (a) Culm-glume-spikelet mixture of einkorn in plastic container; (b) Microtomography scan; (c) 2D layer of the reconstructed 3D image. Whole cereal grains and fragments of culms and glumes are visible (see text for more details).

Portable energy dispersive X-ray fluorescence (ped-xrf) measurements

Total elemental contents were measured on daub samples from Maidanetske by a ped-xrf analyser (NITON XL3t 900-series device, Thermo Scientific Niton Analyzers; see also Chapter 4, this work, Vol. I). 77 sub-samples from 32 daub pieces of two unearched buildings were analysed (19 from House 44, including the samples analysed by X-Ray tomography; 13 from House 111). In addition, 62 soil samples from Maidanetske and the surrounding landscape were analysed.

Sample preparation followed Lubos *et al.* (2016) and Dreibrodt *et al.* (2017). After drying the samples at 35°C for 2 weeks, they were gently disintegrated with mortar and pestle. The finer than 2 mm fractions were homogenised in an agate mill (<60 μ m). Samples were transferred into plastic tubes which were then covered with a 4 μ m film. Measurements were performed in a lead-mantled measurement chamber with He-flotation in the detector unit of the device. The measurement mode ‘mining Cu/Zn’ was applied. The following filter modes of the device were used: main filter 40 kV, 50 mA; high filter 50 kV, 40 mA; low filter 20 kV, 100 mA; light filter 6 kV, 100 mA. The total measurement time was 300 s (main filter, high filter, low filter: 60 s each, light filter: 120 s). If present in measurable amounts, the device measures the content of 35 main and trace elements and in addition calculates the amount of unmeasured residues (‘bal’). Based on calibration with

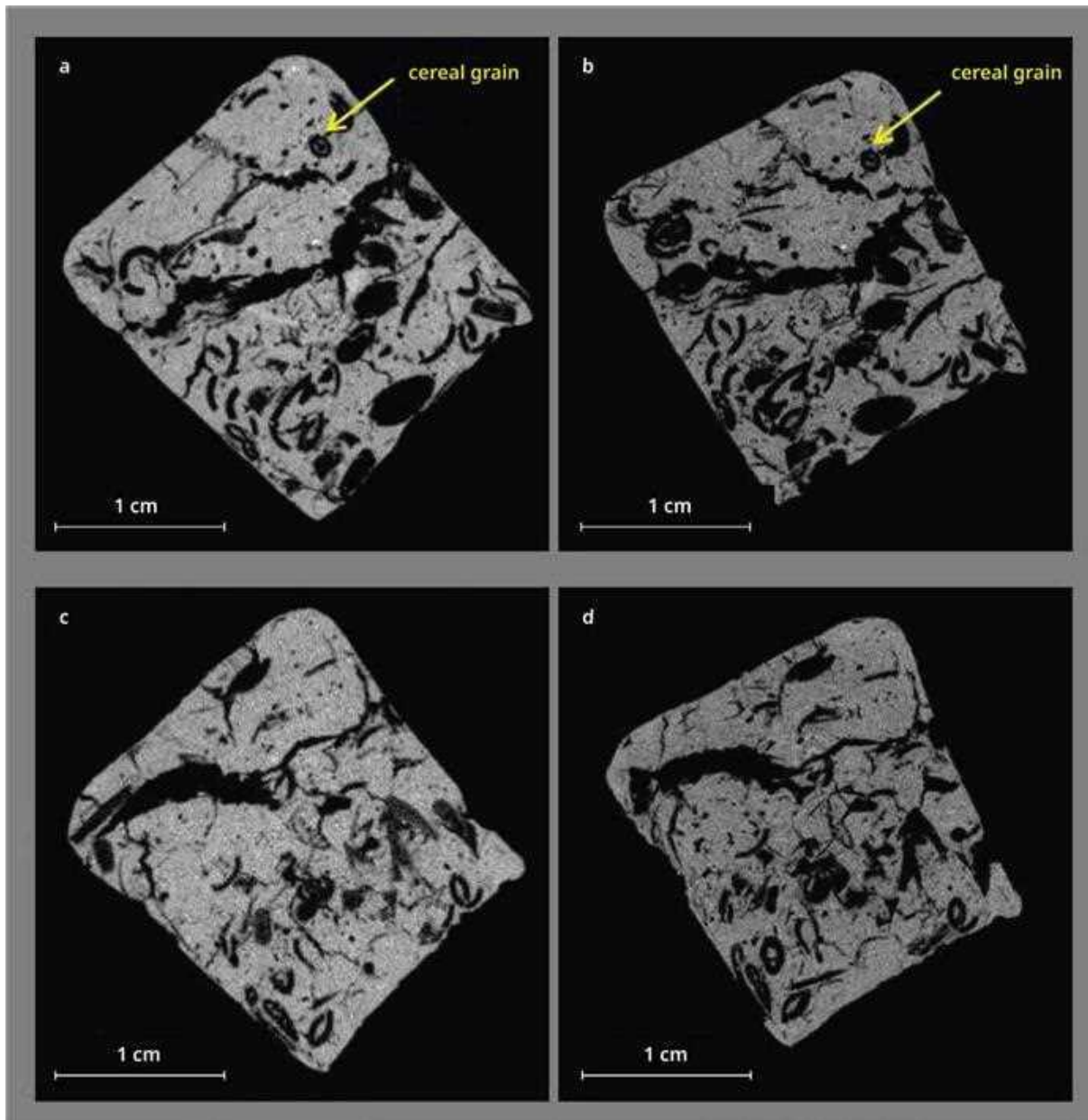


Figure 8: 2D layers of the reconstructed 3D images before and after firing at 550°C: (a) and (c) unfired; (b) and (d) fired; transverse sections are at similar vertical positions: (a & b) and (c & d). Please note: some organic components (e.g. cereal grains) do not show a change in their microtomography signal after firing.

internal standards, the NITON XL3t 900-series device also gives an estimation of measurement errors for each element. The only elements considered here were those with measured values with relative standard errors of ~15% or less. A certified standard sample (GBW07411) was measured regularly to check the reproducibility of the measurements (e.g. ageing of the X-ray source).

Phytolith analysis

From the eight daub pieces analysed by X-Ray tomography, 13 phytolith samples were taken (see Suppl. 2 for more details). Results from samples 51394 and 51402 were published in Dal Corso *et al.* (2018). In all cases, a portion of daub was subsampled from the middle of the piece, after being freshly opened and then disintegrated using mortar and pestle. 2.5–4 g of material was processed after Madella *et al.* (1998) using heavy liquid (sodium polytungstate/SPT) separation. Where possible, a minimum number of ca. 200 phytolith single cells (excluding unidentifiable

phytoliths and multicellular aggregates) were counted at 400x magnifications under a light microscope and used as the base for percentage values. Samples poor in phytolith content resulted in lower counts; the counting was limited to ca. 100 oculars. For the identification and attribution to plant groups and/or parts see Dal Corso *et al.* (2018) and references therein. Phytolith morphotypes are named according to the International Code for Phytolith Nomenclature 2.0 (ICPT 2019). In the present study, only single phytolith cells are considered, although multicellular aggregates of phytoliths in anatomical positions (also called silica skeletons) have also been found (Dal Corso *et al.* 2018). The single cells record has been chosen because of easier comparability of relative proportions of morphotypes in the assemblage of each sample.

Results

X-ray microtomography of daub samples of House 44

Table 2 provides an overview of the qualitative amounts of the organic components (cereal grains, glumes, culm fragments, and spikelet fork/rachis segments), voids, cracks and inhomogeneities. Neither cereal grains nor significant amounts of glumes, culm fragments and spikelet fork/rachis segments were found in Sample 51413 (Material Type 1). This is in accordance with its classification as a non-chaff-tempered daub sample. Cereal grains were detected in half of the samples of Material Type 2 (51366, 51416, 51402, 51379) and in all studied samples of Material Type 3 (51394, 51392, 51390). Glumes are the dominant organic component in all samples of Types 2 and 3. No obvious sequence exists regarding the relative amounts of the other organic components, although most of the samples of both types contain slightly more spikelet fork/rachis segments compared to culm fragments. The same lack of grouping exists regarding the presence of voids, cracks and inhomogeneities. Table 3 summarises the quantitative morphometric information.

The distribution of organic components in all samples of Types 2 and 3 is heterogeneous, resulting in large variations of both porosities and organic components. The averages of most porosities and of absolute and relative organic contents, respectively, are identical within 1σ standard deviation in all the classes studied. The daub sample of Type 1 (non-chaff-tempered daub) has a very low porosity ($7.5 \pm 1.9\%$) and, thus, marginal organic components, which can be attributed to very few culm fragments (see Tab. 2). The total volume of organic components, now present as distinct shapes of pores and easily distinguishable from voids and cracks, is significant.

Total elemental contents of the studied daub pieces

The results of the ped-xrf measurements are shown in Supplement 3 and in Figure 9. The evaluation of the data shows that 15 elements (soil) and 13 elements (daub: Zr, Y, Sr, Zn, Fe, Mn, Ti, Ca, K, Al, P, Si, Mg), respectively, are measured with a relative error of $<15\%$ and could be considered appropriate for a statistical analysis.

A comparison of the mean values (box-plots) of indicative elemental ratios is given in Figure 9a. Considering the context of the study, a focus here is on biogenic silica. Geochemical analysis can provide data about biogenic silica in sediments (*e.g.* Kylander *et al.* 2011; Dreibrod and Wiethold 2015) and within archaeological deposits (*e.g.* Lubos *et al.* 2013). While biogenic silica concentration can be measured in the eluate of alkaline solvents directly (*e.g.* Engström and Wright 1984; Lubos *et al.* 2013) the application of elemental ratios based on xrf-measurements has been

Sample #	μ CT # ¹	Total volume ²	Cereal grains ³	Glumes ⁴	Culm fragments ⁴	Spikelet fork/rachis segments ⁴	Voids ⁴	Cracks ⁴	Inhomogeneities ⁴
51394	E	9849	yes	+++	++	○	+	○	+
51366	A	8449	no	++	+	+	++	+	+
51416	D	10556	no	+++	++	○	○	++	-
51413	G	13683	no	-	+	○	○	○	++
51392	H	7569	yes	++++	○	++	+	+	○
51390	F	5588	yes	++++	○	+	++++	○	+
51402	B	12069	yes	++++	○	+	+	○	+
51379	C	6078	yes	+++	+	++	+	○	-

Table 2. Organic components in studied daub samples.

¹ internal labelling by μ CT laboratory; ² total volume of scanned sample in mm³; ³ presence or absence of cereal grains in studied samples; ⁴ optical description of scanned sample (- : absent; ○ : barely recognisable and classification questionable; +/++/+++ /++++ : organic components clearly identified with increasing relative amounts of different organic components in individual samples).

Sample #	μ CT # ¹	Total porosity ²	Total porosity [mm ³] ³	Porosity of ROI sections ⁴	Organic content of ROI sections ⁵	Relative organic content ⁶	Avg. porosity ⁷	Avg. organic content ⁸	Avg. rel. org. content ⁹	Avg. total organic volume [mm ³] ¹⁰
51394	E	15.8	1556	20.4 / 19.8 / 12.8	18.5 / 18.7 / 8.2	90.9 / 94.3 / 64.2	17.7 ±4.2	15.1 ±6.0	83.1 ±16.5	1293 ±257
51366	A	11.0	929	14.4 / 11.9 / 10.1	13.3 / 10.1 / 4.7	92.1 / 83.9 / 46.5	12.1 ±2.2	9.4 ±4.3	74.2 ±24.3	689 ±226
51416	D	17.5	1847	14.5 / 17.1 / 21.3	7.4 / 13.5 / 14.8	51.0 / 78.8 / 69.5	17.6 ±3.4	11.9 ±4.0	66.4 ±14.2	1226 ±262
51413	G	7.7	1054	8.8 / 8.3 / 5.3	0.6 / 0.5 / 0.7	6.4 / 5.7 / 13.0	7.5 ±1.9	0.6 ±0.1	8.4 ±4.0	88.5 ±42.2
51392	H	13.8	1045	13.8 / 18.6	12.8 / 17.2	92.4 / 92.5	16.2 ±3.4	15.0 ±3.1	92.5 ±0.1	967 ±1.05
51390	F	21.3	1190	17.3 / 25.3 / 24.2	9.0 / 15.3 / 10.8	52.0 / 60.4 / 44.8	22.3 ±4.3	11.7 ±3.2	52.4 ±7.8	624 ±92.8
51402	B	14.7	1774	17.1 / 15.9 / 12.5	13.3 / 14.5 / 9.1	77.6 / 91.3 / 73.4	15.2 ±2.4	12.3 ±2.8	80.8 ±9.4	1433 ±167
51379	C	15.0	912	15.7 / 15.9	14.2 / 13.9	90.1 / 87.3	15.8 ±0.1	14.1 ±0.2	88.7 ±2.0	809 ±18.2

Table 3. Quantitative morphometric information of studied daub samples.

¹ internal labelling by μ CT laboratory; ² porosity [%] of entire scanned sample; ³ porosity [mm³] of entire scanned sample; ⁴ porosity [%] in up to 3 individual sections (ROI = region of interest); volume of ROI was standardised (1000 mm³); ⁵ percentage of organic components related to porosity in individual ROI sections; ⁶ relative amount of organic components [%]; ⁷ average porosity of combined individual ROI sections with 1 σ standard deviation [%]; ⁸ average amount of organic components of combined individual ROI sections with 1 σ standard deviation [%]; ⁹ average relative amount of organic components with 1 σ standard deviation [%]; ¹⁰ average total organic volume with 1 σ standard deviation [mm³].

established as a quick alternative, delivering additional multi-elemental datasets (Lubos *et al.* 2016; Dreibrodt *et al.* 2017). Silica (Si) is present in an amorphous form in plants (phytoliths) but also in minerals of the soil at the site. Thus, ratios of Si with other elements, limited to the minerals of a sample, could be considered to reflect the amount of excess (= biogenic) silica. Figure 9a gives a comparison of the mean values of Si:Ti, Si:Al, Si:Zr, and Si:K ratios in the soils of the site (considered to reflect the resource for daub production) and the daub pieces. T-tests of the mean values of the

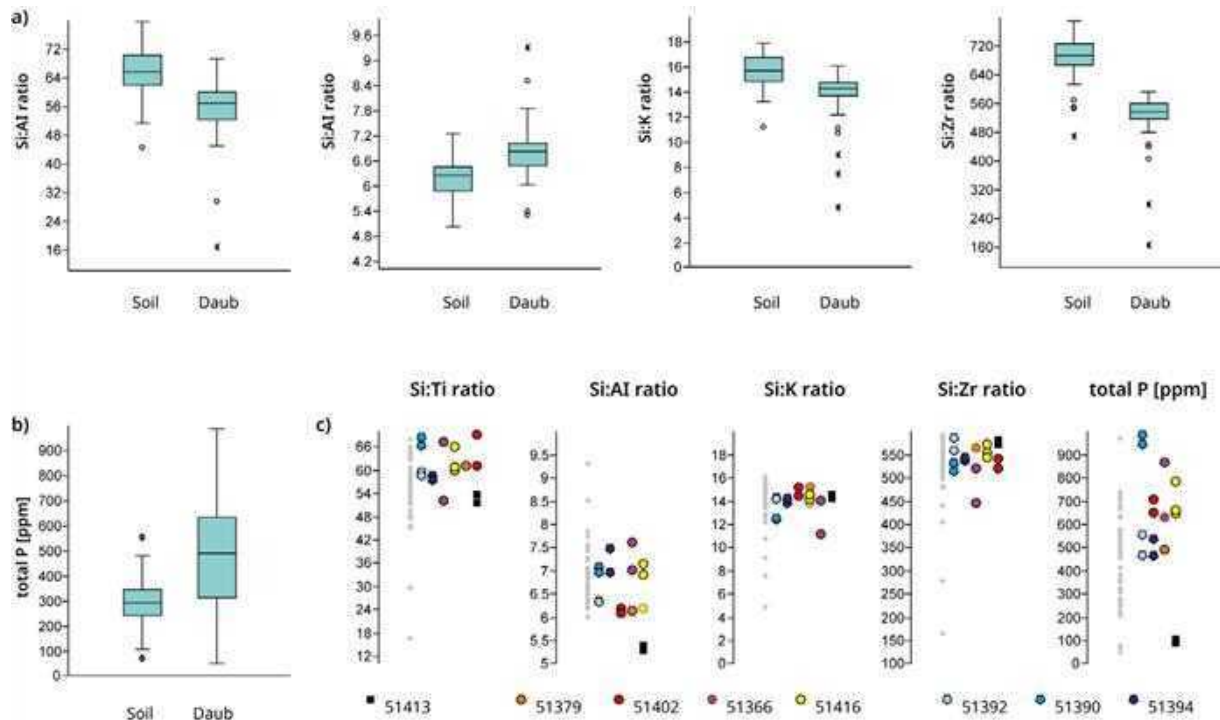


Figure 9. Geochemical properties of the daub from Maidanetske ($n=77$) compared to the local soils ($n=62$): (a) boxplots of elemental ratios considered to reflect the content of excess silica (biogenic silica); the differences in mean values are significant according to the t -tests; (b) total phosphorus content of daub and soil; (c) geochemical composition of the daub samples of House 44 and the building in Trench 111. Coloured symbols highlight the daub samples studied with μ CT and phytolith techniques. Note that the symbols indicate the material types of the analysed daub: square = Type 1, circle = Type 2, pentagon = Type 3.

studied daub pieces and soils from the site show that the displayed differences are significant. Whereas there are enrichments of Si in the soils compared to the daub visible in Si:Ti, Si:Zr and Si:K ratios, the opposite is the case for Si:Al mean values.

Another difference is shown in the values of the total phosphorus contents (Fig. 9b). The mean values of the samples display a significantly higher P content in the daub samples compared to the soils at the site (significance approved by t -test).

Figure 9c shows all daub values for the aforementioned elemental values and the total phosphorus content for the studied daub pieces. The samples analysed by μ CT and used in the phytolith analysis are marked with coloured symbols. It is obvious that the daub pieces selected for the study reflect a wide spectrum of daub present at the site, given their elemental composition.

Results of phytolith analyses

The phytolith record is rich and is composed of 15 different morphotypes (Fig. 10; Suppl. 2).

Single cell phytolith counts range from 205 and 420 per sample, with the exception of two samples with poor phytolith content (51413: 7 phytoliths; 51390b: 42 phytoliths). Those two samples are excluded from the following description of results. As expected from a previous study (Dal Corso *et al.* 2018), the phytolith record mostly refers to Poaceae. Grass short cell phytoliths produced in Poaceae are mostly represented by ubiquitous RONDELS (average 21.2%; maximum value 38.8% in Sample 51390a; minimum 5.4% in Sample 51402) and frequent TRAPEZOID/CRENATE phytoliths (average 2.6%; max. 11.2% in 51394; absent in 51416). These

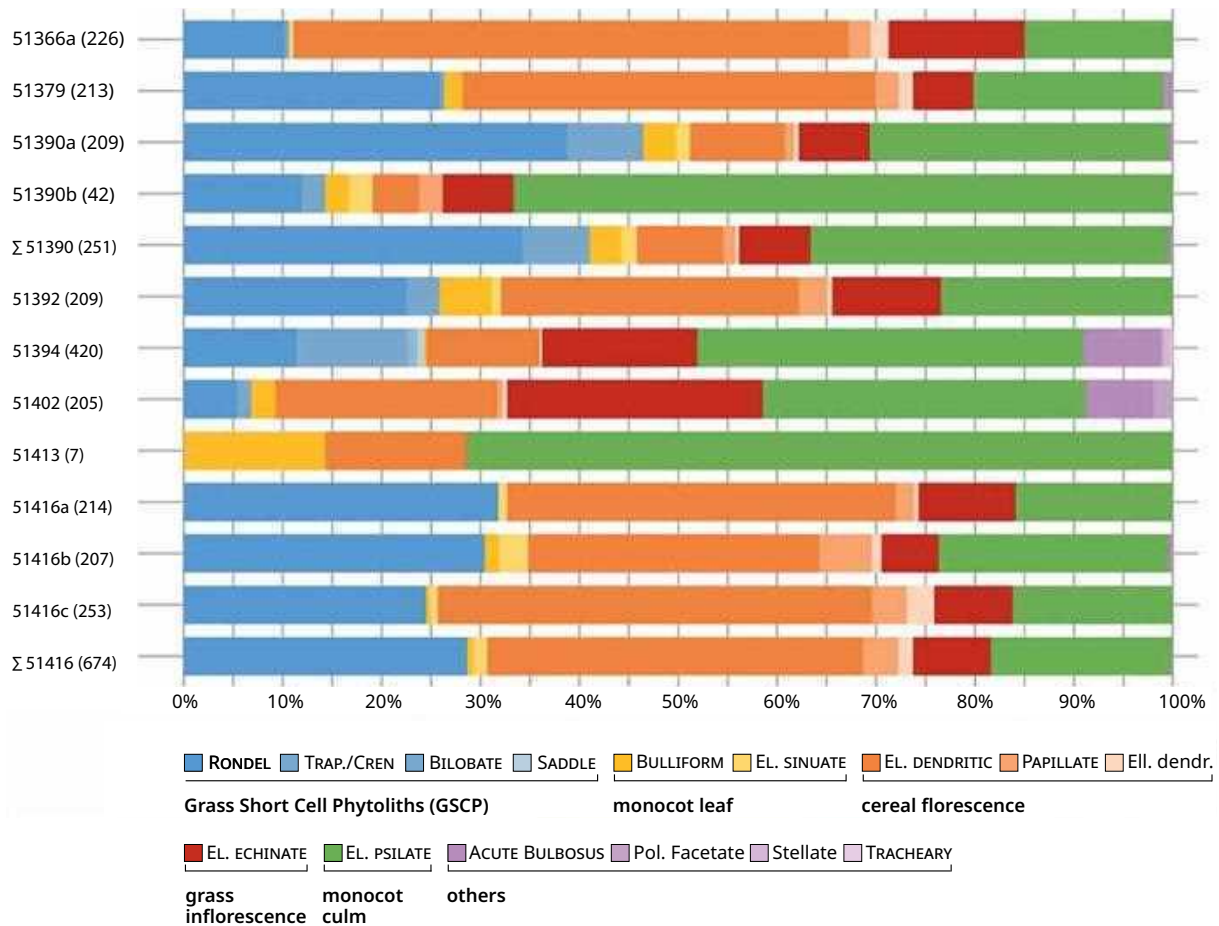


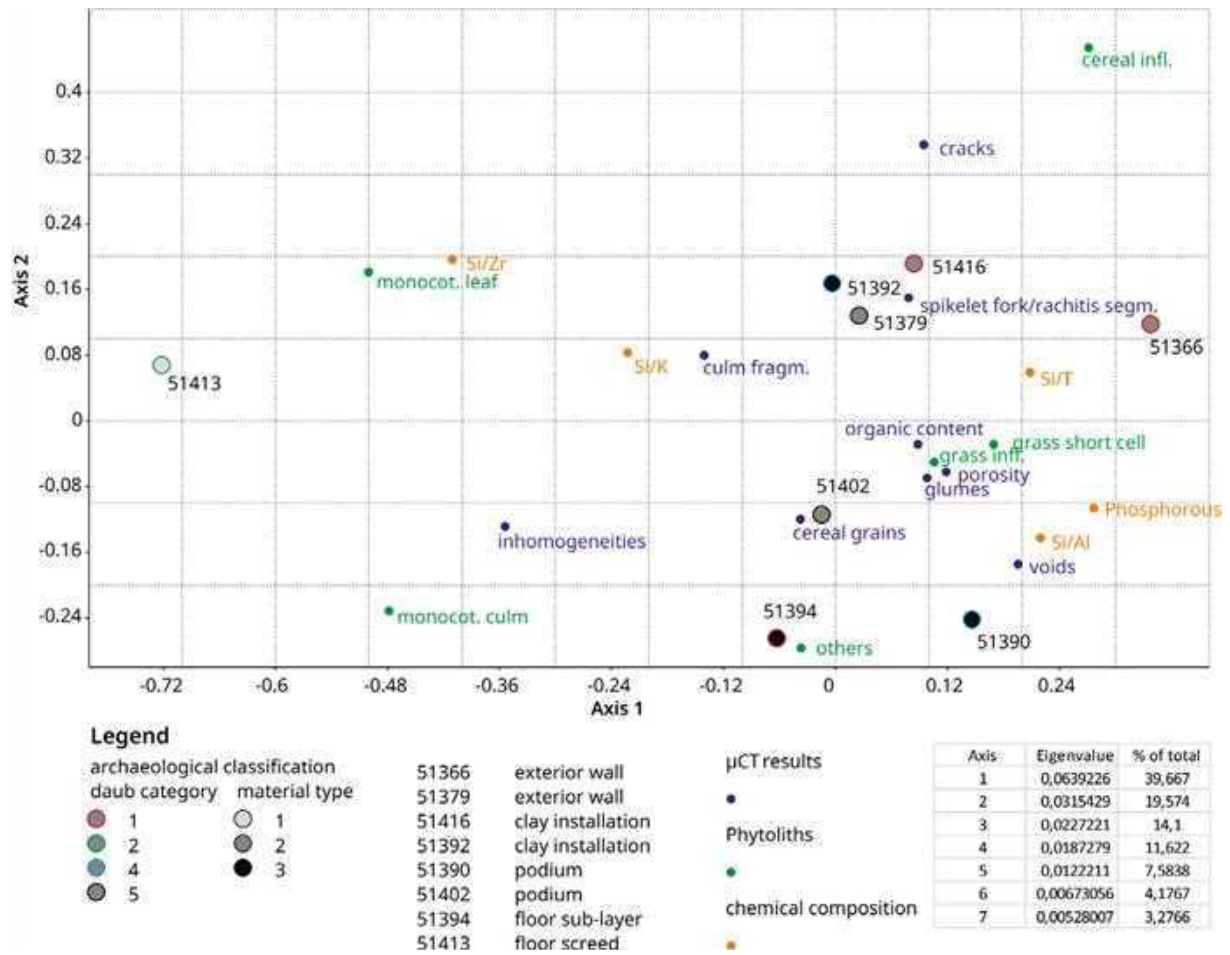
Figure 10. Summarised assemblages of phytolith single cells.

morphotypes are typical of pooid grasses and cereals (Ball *et al.* 1999). BULLIFORMS FLABELLATE from monocot leaves are rare (average 2.7%; max. 5.3% in 51392; absent in 51366a and 51416a). ELONGATE SINUATE from monocot leaves are also rare (average 1%; max 2.9% in 51416b). ELONGATE ENTIRE are ubiquitous and very frequent (average 31.3%; max. 39% in 51393; min. 15% in 51366a). They can be found in different plant groups and in different grass parts; they are especially abundantly produced in grass culms (Lancelotti and Madella 2012), *i.e.* they can originate from straw (Pető and Vrydaghs 2016). ELONGATE DENDRITIC are also ubiquitous and very abundant (average 26.9%; max. 56.2% in 51366a; min. 9.6% in 51390a). These phytoliths derive from light chaff parts (glumes, lemma, palea and awns), obtained from cereal ears (Ball *et al.* 2016; Pető and Vrydaghs 2016). Similarly, ELONGATE DENTATE phytoliths are commonly attributed to inflorescences and in minor part leaves of Poaceae too, although not only of domesticated cereals. They are common in this record (average 9.6%; max. 25.9% in 51402; min. 5.8% in 51416b).

Discussion

In this section, the μ CT results of the daub pieces are compared with daub (phytoliths, chemical composition) and possible implementations of ancient human behaviour and socio-technological aspects are discussed.

The correspondence analysis of the whole dataset (Fig. 11) shows that the main results of XRay- μ CT measurements (porosity, voids, organic content, cereal grains, glumes) plots in one cluster, with main compositional proxies for organic temper contents (grass phytoliths, phosphorus content, excess silica (Si:Al, Si:Ti)). On the



other hand, other features such as cracks or inhomogeneities considered to result partly from the burning process plot outside this cluster. Thus, porosity and the deduced organic content measured by Xray- μ CT correlate with the amount of organic temper added during the daub production, which backs up the validity of the XRay- μ CT results. Some differences in the datasets might result from sampling volumes (e.g. cereal grains measured in XRay- μ CT and cereal inflorescence in phytolith assemblages). Whereas the μ CT and ped-xrf composition rely on a large part of the analysed daub samples, phytolith sampling was restricted to small amounts of the daub pieces. Some variability is visible within different phytolith sub-samples of the daub pieces (Suppl. 2). A comparison with the macroscopic archaeological daub classification, also integrating functional aspects of the daub, indicates that main categories (daub category 2, Material Type 1) are related to daub composition. The remainder of macroscopically differentiated daub types does not reflect well the daub's physical, microfossil and geochemical properties. The daub category classes (1, 4, 5) and material type classes (2, 3) show a large overlap in Figure 11 instead of forming distinct clusters. Looking at different architectural components, exterior walls (51366, 51379), together with daub from interior components of clay installation (51416, 51392), plot in one group. Other house interior daub components such as podium (51390, 51402) and floor layers (51394) plot in a different group. Whether this indicates a kind of standardisation of source materials in daub production (as mainly indicated by phytolith analysis) or not is discussed in the following sections.

Figure 11. Correspondence analysis of the daub samples. Numerical data on μ CT data (porosity, organic content), phytolith assemblages and geochemical composition have been transformed to ordinally scaled data (classes).

Socio-economic and technological insights from construction materials based on the phytoliths record

Phytoliths allow a quantifiable comparison of different plant parts that are otherwise not quantifiable (van der Veen 1999; Pető and Vrydaghs 2016). This brings the study of daub a step closer to an understanding of the use and management of by-products in prehistoric architecture. According to the phytolith record, three groups of samples can be distinguished that show good correspondence with the material type classification assigned by archaeologists. In the first category, Sample 51413, characterised by absence of plant temper visible macroscopically (Material Type 1), contains almost no phytoliths. The second group of phytolith samples includes samples 51366a, 51379, 51402, 51416a–c that are characterised by abundant indicators of chaff (ELONGATE DENDRITIC average 38.8%, ELONGATE DENTATE 11.5%) and that originate from daub pieces of Material Type 2, described as solid and with evident voids from organic plant temper. The third group of phytolith samples concerns 51390a–b, 51392, 51394, and presents evidence of an admixture of both chaff (ELONGATE DENDRITIC average 12.9%, ELONGATE DENTATE 9.6%) and straw (from culms, ELONGATE ENTIRE 39.1% vs. 20.4% of the average value in the previous group; from leaves, BULLIFORM FLABELLATE AND BLOCKY 2.8% vs. 1% and TRAPEZOID 6.2% vs. 0.4% in the previous group). This third group of samples corresponds to daub of Material Type 3, crumbly with visible porosity due to plant temper.

When considering the phytolith content in respect to architectural contexts, the two samples from external walls (51366a, 51379) show mostly chaff used as temper. In contrast, from the samples originating from podium and clay installation inside the house, in some cases the daub has mostly chaff (51402, 51416a–c) and in other cases an admixture of chaff and straw (51390a–b, 51392, 51394). According to this preliminary picture, it seems that chaff was preferred for building the most critical parts of the house architecture, such as external walls that are exposed to different weather conditions and that have to provide stability to the whole house construction. Interior clay installations that are less important for the house architecture as a whole could have been built at the time of house construction or at a later stage, depending on availability and function, either by using pure chaff as plant temper or an admixture of chaff and straw.

From a socio-economic perspective, emmer and einkorn, proven to be the main cereals in the macro-botanical record of Maidanetske (Dal Corso *et al.* 2019), are two species usually stored as spikelets and dehusked prior to cooking (Hillman 1984). If the chaff of such hulled wheat was such valuable construction material, it seems reasonable that it had to be collected and stored in a systematic way. Two options seem to be reasonable, either as piecemeal dehusking as part of routine household activities, or as a bulk, large-scale, collective scheduled action. Alternatively, house constructions could have been conducted as well-planned societal action immediately after the annual cereal harvest. The lack of pit records filled with harvest residues could be read as an indicator for the latter scenario; this would support the scenario of a longer duration of inhabitation of Trypillian mega-sites (Ohlrau *et al.* 2016; Müller *et al.* 2017; Hofmann *et al.* 2019).

Daub material and processing: insights from geochemical composition and porosity of the daub

Besides the indication that our selected daub pieces reflect the variety of a larger number of daub pieces at Maidanetske, additional clues might be offered by the geochemical composition of the analysed daub pieces and soil samples.

Assuming the use of the soils and loess deposits locally available for daub manufacturing, significant differences in the elemental ratios (Si:Ti, Si:Al, Si:Zr, Si:K; Fig. 9a) could be explained by an alteration of the daub samples during the burning process, a selective extraction of the building material or an alteration of the material during the processing of daub production. None of the compared elements is known to become lost in larger amounts during combustion processes. A selective extraction of material enriched in Ti, Zr or K also seems unlikely since the whole site is underlain by loess with a thickness of >2m. Processing that includes water saturation of the daub material renders the most probable process that could have resulted in the observed results. By watering and intensive mixing of the daub material before its application as plaster on the wooden walls, a separation of the mineral assemblage could have happened. During this process a part of the clay minerals may have been lost from the daub mixture by flotation. Clay might easily go into suspension when water is added to the mineral organic temper mixture. Aluminium, in comparison, is to a large extent bound to clay minerals and probably becomes lost through supernatant water suspension. Zirconium and titanium are mostly bound in heavy minerals with a comparable high density ($>4 \text{ g}\cdot\text{cm}^{-3}$) and may be assumed to be able to become enriched by flotation. Potassium could be considered to behave in a way that falls in between the above two cases, since considerable amounts could be present in clay minerals (mica/illite) but additional K might occur in feldspars. The order of enrichment observed in the daub compared with the soil samples (Zr⁺⁺, Ti⁺⁺, K⁺, Al⁻) thus probably reflects the loss of clay minerals due to the flotation process of the daub preparation.

The significantly higher phosphorous content of the analysed daub pieces compared to the soil samples probably reflects the addition of dung or urine during the processing of the daub (Fig. 9b). Since no sperulites have been detected in the phytolith assemblages, urine is the more probable source of phosphorus in this present case.

Comparing the xrf elemental composition of the daub with the porosity results, additional evidence emerges (Fig. 11). Although the number of measurements is small, the observed linear dependence of the Si:Ti ratio, the Si:Al ratio and the total phosphorus content to the porosity of the daub samples (as derived from μ CT measurements) implies a close relationship between the porosity of the samples and organic admixtures included during the daub processing. This makes the μ CT method that was applied a promising approach to quantify the amount of organic temper in the daub samples. Furthermore, of the three groups shown by phytoliths and material types, only the first group shows distinctly lower porosity in μ CT scans compared to the other two groups, whose porosity results are quite homogeneous and similar (see Tab. 3).

The results from the material analysis of the daub pieces from Maidanetske suggest a standardised technology: during daub processing, similar amounts of plant material, sediment and a liquid (probably urine) were added. Plant temper has structural functions of providing more elasticity, thermic isolation and increased stability to wall structures, with even drying helping to prevent cracks from forming (Newton 2004; Bonnaire 2014). It is possible that different requirements account for the floor screed, which in this case did not require organic tempering in order to achieve smooth and even floor surfaces. This is also known for other samples of floor screeds in Maidanetske (see Müller *et al.* 2017 for more details). The use of urine as a liquid has been seen from ethno-archaeological work elsewhere. To test the extent of technological standardisation, a larger number of daub pieces needs to be analysed in future studies.

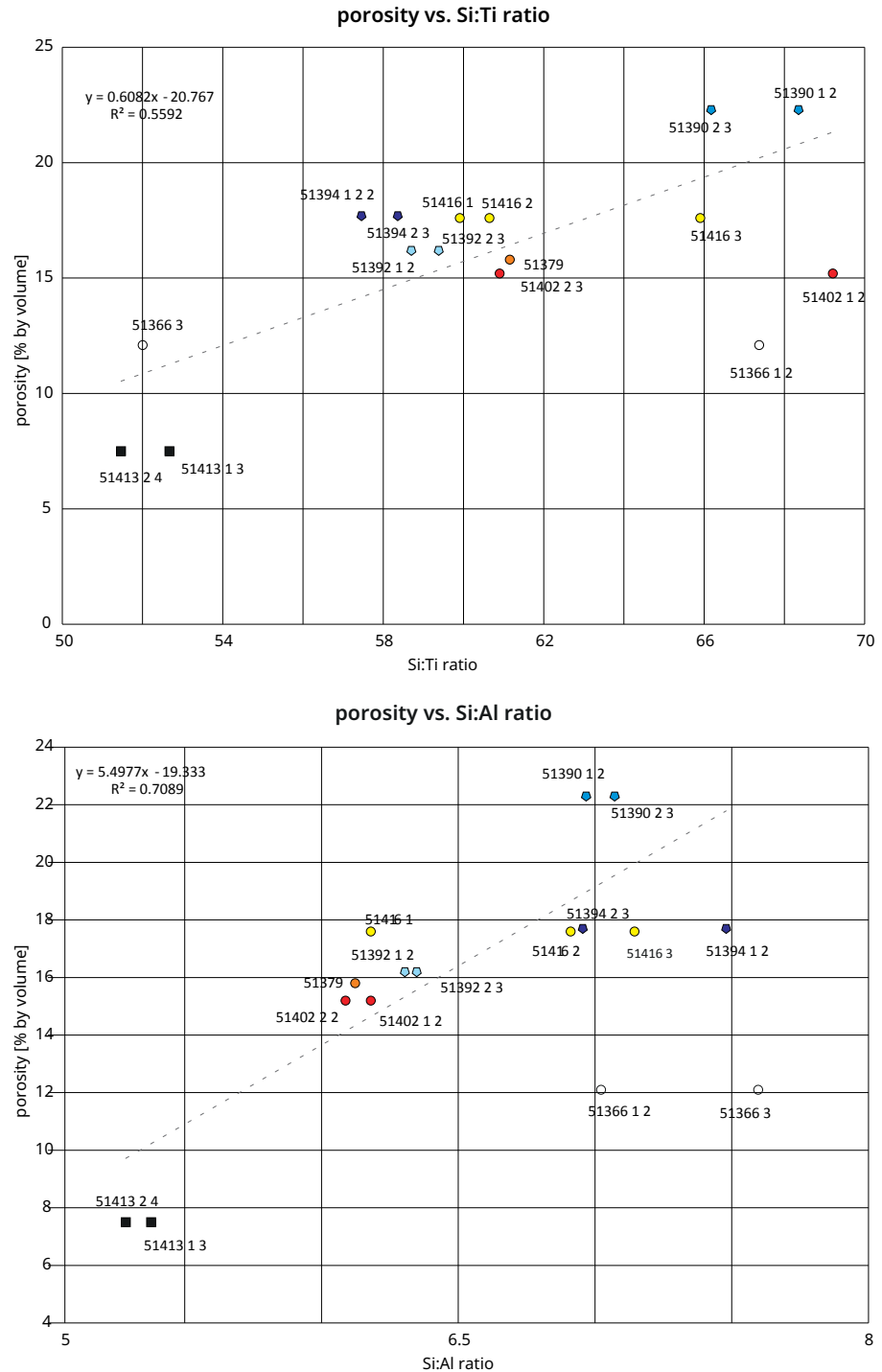


Figure 12. Linear regressions between Si:Ti and Si:Al ratios and total phosphorus content with the porosity values of the analysed daub samples. The symbol coding is identical to Fig. 9.

Possible connections between agricultural field size and use of harvest residue amounts in daub

Different models for the reconstruction of palaeo-economy, land-use and the carrying capacity for Trypillian sites have been developed (Bibikov 1965; Kruts 1989; Gaydarska 2003; Nikolova and Pashkevich 2003; Pashkevich and Videiko 2006; Harper 2012; Ohlrau *et al.* 2016; Dal Corso *et al.* 2019). All agree that resource availability was a given and that carrying capacity was never reached. As regards

the soil resource, data from the investigated site do not indicate excessive depletion by soil erosion during Chalcolithic times (Dreibrodt *et al.* 2020).

In this section we relate the amount of harvest and the associated field sizes necessary to explain the amount of organic temper in the daub (Tab. 3) to calculations of field sizes necessary to supply the estimated population of the Chalcolithic mega-site with nutrition.

The average cereal remains content of the daub was first related to the daub mass included in a normal size one- and two-storey house (Pickartz *et al.* 2019) that is assumed to have been inhabited by five people (Ohlrau *et al.* 2016). By relating this amount of cereal remains to an assumed field size, 0.03 to 0.3 ha of agricultural fields would have been needed to account for the amount of organic temper of one house (Suppl. 4). This equals 0.0006 to 0.06 ha/capita. The construction of two-storied houses (Korvin-Piotroskiy and Menotti 2008; Johnston *et al.* 2018; Ohlrau 2020) would demand 0.36 to 0.6 ha/house, equal to 0.07 to 0.12 ha/capita.

In a second step, the calculation was related to the number of contemporaneous houses. The following calculations refer to the maximum phase of ca. 1,550 contemporaneous houses (time period 3800–3700 BCE) estimated by Ohlrau (2020) and Dal Corso *et al.* (2019) based on a detailed analysis of the available chronological data. A total of 3,100 houses were ascribed to the settlement period in question. A lifespan of ca. 50 years per house was assumed. Thus, an annual average of 62 houses must have been built to maintain the total number of 1,550 houses (total number of houses (3,100) divided by lifespan of houses (50 years)). Accordingly, a minimum size of 0.03 to 0.3 ha/house $\times$ 62 (if two-storied houses are considered: $0.06\text{--}0.6 \times 62$) of arable land area is required annually to obtain sufficient by-products for clay tempering needed for house construction. This is equivalent to 1.9 to 18.6 ha per year (one-storey house) or 3.7 to 37.2 ha per year (two-storey house).

Ohlrau *et al.* (2016, Tab. 3) consider a requirement of 0.3 ha/capita/year to achieve food security. Converted into estimates for the arable land necessary to feed the inhabitants of 1,550 houses with 5 inhabitants, this amounts to 2,325 ha of field size per year considering the whole period in consideration.

The size of agricultural fields to account for the amount of cereal remains used in house construction (1.9–37.2 ha per year) represents 0.1 to 1.6% of the area of agricultural fields necessary to meet the nutritional requirements of the contemporaneous mega-site population.

The major archaeological result of this pilot study relates to the resource management of a mega-site. In order to obtain the resources for the tempering of clay for house construction, no special cultivation processes were necessary. The chaff was available to the inhabitants of the large settlement as a by-product of cereal cultivation.

In addition to the evidence for prolonged occupation that can be inferred from radiocarbon dates (Müller *et al.* 2016a), the inventory of houses (Ohlrau 2020), and the absence of smaller settlements in the vicinity of Maidanetske, the results presented of the daub analysis provide additional arguments against the interpretation of large Trypillian settlements as temporary seasonal collection camps. Approximately 1.4 m³ (one-storey houses) and 4.6 m³ (two-storey houses) of crop residues would have had to be carried to the site to construct the houses at Maidanetske. This poses a certain logistical problem for the settlers, who are said to have gathered together from a surrounding region of unknown size.

Conclusions

A main aim of our study was to test the feasibility using XRay- μ CT analysis to contribute to compositional analyses of burnt daub from the Chalcolithic mega-site settlement of Maidanetske, central Ukraine. Based on morphometric studies of the pores, not only can the amount of organic temper be quantified and distinguished from other kinds of porosity, but in addition, various components of the organic temper were classified successfully. The XRay- μ CT results have been supported by comparison with results of archaeological classification, phytolith analysis and geochemical analysis. The development of standardised quantification and classification protocols for the XRay- μ CT measurement in order to limit the amount of data processing and time and thus enable the analysis of larger numbers of burnt artefacts was a rewarding undertaking.

Detailed multidisciplinary analysis of burnt daub pieces from Maidanetske revealed in addition information on harvest residue use, daub processing and gave further hints to social interaction of Trypillian communities as regards plant use and construction of houses. The preference for using different plant compounds in different parts of the architecture (*e.g.* cereal inflorescence in house outer walls) and results from processing of the daub (*e.g.* geochemically indicated addition of urine to the daub mass) indicates a certain level of standardisation for house construction. The extent of this standardisation of house construction should be verified by further investigations. The selection of specific harvest residues (chaff) for the organic tempering of houses by Trypillian people indicates a well organised social interaction between the ancient farmers which probably took place after the annual cereal harvest was finished. A calculation of the total amount of harvest residues needed to construct the recorded number of houses in the mega-site of Maidanetske implies that this need was met by the yearly harvest of the fields supporting the inhabitants with nutrition.

The XRay- μ CT method is a promising tool to make use of the high potential of the largely available daub archives of archaeological sites to reconstruct different aspects of crop use, archaeo-technology and ancient social interaction.

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Supplementary materials

The supplementary materials are freely available online under a CC BY 4.0 licence, in the Kiel University research data repository opendata@uni-kiel.de: <https://doi.org/10.57892/100-55>

Supplement 1: (text, tables, figure)

Classification scheme after Müller *et al.* (2017) and advanced classification with subdivisions in material characteristics and architectural features.

Supplement 2: (figure and table)

Sampling for phytoliths analyses and phytolith record from daub pieces.

Supplement 3: (table)

Geochemical composition (ped-xrf) of analysed daub and soil samples from Maidanetske.

Supplement 4: (table and text)

Background data for calculation of arable land size solely used to produce chaff for daub manufacturing.

References

- An, T., Pashkevich, G. and Jones, M., 2019. Re-examining millet impressions in Usatovo clay materials from NW Black Sea region, Ukraine. *Archaeological and Anthropological Sciences*, 11, 3201–3211. Available from: <https://doi.org/10.1007/s12520-018-0718-3>
- Ball, T.B., Gardner, J.S. and Anderson, N., 1999. Identifying inflorescence phytoliths from selected species of wheat (*Triticum monococcum*, *T. dicoccon*, *T. dicoccoides*, and *T. aestivum*) and barley (*Hordeum vulgare* and *H. spontaneum*) (Gramineae). *American Journal of Botany*, 86 (11), 1615–1623. Available from: <https://doi.org/10.2307/2656798>
- Ball, T., Chandler-Ezell, K., Dickau, R., Duncan, N., Hart, T.C., Iriarte, J., Lentfer, C., Logan, A., Lu, H., Madella, M., Pearsall, D.M., Piperno, D.R., Rosen, A.M., Vrydaghs, L., Weisskopf, A. and Zhang, J., 2016. Phytoliths as a tool for investigations of agricultural origins and dispersals around the world. *Journal of Archaeological Science*, 68, 32–45. Available from: <https://doi.org/10.1016/j.jas.2015.08.010>
- Bibikov 1965: Биби́ков, С.Н., 1965. Хозяйственно-экономической комплекс развитого Триполья. *Советская археология*, 1965 (1), 48–62.
- Bonnaire, E., 2014. Use of Crop-Processing By-Products for Tempering in Earthen Construction Techniques. In: A. Chevalier, E. Marinova and L. Peña-Chocarro, eds. *Plants and People. Choices and Diversity through Time*. EARTH 1. Oxford, Philadelphia: Oxbow Books, 282–286.
- Chapmann, J., 1999. Deliberate house-burning in the prehistory of Central and Eastern Europe. In: A. Gustafsson and H. Karlsson, eds. *Glyfer och arkeologisk rum – en vänbok till Jarl Nordbladh. Gotarc Series A 3*. Göteborg: Göteborg University, Department of Archaeology, 113–126.
- Chapman, J., Videiko, M.Y., Hale, D., Gaydarska, B., Burdo, N., Rassmann, K., Mischka, C., Müller, J., Korvin-Piotrovskiy, A. and Kruts, V., 2014. The Second Phase of the Trypillia Mega-Site Methodological Revolution: A New Research Agenda. *European Journal of Archaeology*, 17 (3), 369–406. Available from: <https://doi.org/10.1179/1461957114Y.0000000062>
- Dal Corso, M., Out, W.A., Ohlrau, R., Hofmann, R., Dreibrodt, S., Videiko, M., Müller, J. and Kirleis, W., 2018. Where are the cereals? Contribution of phytolith analysis to the study of subsistence economy at the Trypillia site Maidanetske (ca. 3900-3650

- BCE), central Ukraine. *Journal of Arid Environments*, 157, 137–148. Available from: <https://doi.org/10.1016/j.jaridenv.2018.06.009>
- Dal Corso, M., Hamer, W., Hofmann, R., Ohlrau, R., Shatilo, L., Knitter, D., Dreibrodt, S., Saggau, P., Duttmann, R., Feeser, I., Knapp, H., Benecke, N., Videiko, M., Müller, J. and Kirleis, W., 2019. Modelling landscape transformation at the Chalcolithic Tripolye mega-site of Maidanetske (Ukraine): Wood demand and availability. *The Holocene*, 29 (10), 1622–1636. Available from: <https://doi.org/10.1177/0959683619857229>
- Dreibrodt, S. and Wiethold, J., 2015. Lake Belau and its catchment (northern Germany): A key archive of environmental history in northern central Europe since the onset of agriculture. *The Holocene*, 25 (2), 296–322. Available from: <https://doi.org/10.1177/0959683614558648>
- Dreibrodt, S., Furrholt, M., Hofmann, R., Hinz, M. and Cheben, I., 2017. P-ed-XRF-geochemical signatures of a 7300 year old Linear Band Pottery house ditch fill at Vrable-Ve'lké Lehemby, Slovakia – House inhabitation and post-depositional processes. *Quaternary International*, 438, 131–143. Available from: <https://doi.org/10.1016/j.quaint.2017.03.054>
- Dreibrodt, S., Hofmann, R., Sipos, G., Schwark, L., Videiko, M., Shatilo, L., Martini, S., Saggau, P., Bork, H.-R., Kirleis, W., Duttmann, R. and Müller, J., 2020. Holocene soil erosion in Eastern Europe-land use and/or climate controlled? The example of a catchment at the Giant Chalcolithic settlement at Maidanetske, central Ukraine. *Geomorphology*, 367, 107302. Available from: <https://doi.org/10.1016/j.geomorph.2020.107302>
- Dreibrodt, S., Hofmann, R., Dal Corso, M., Bork, H.-R., Duttmann, R., Martini, S., Saggau, P., Schwark, L., Shatilo, L., Videiko, M., Nadeau, M.-J., Grootes, P.M., Kirleis, W. and Müller, J., 2022. Earthworms, Darwin and prehistoric agriculture-Chernozem genesis reconsidered. *Geoderma*, 409, 115607. Available from: <https://doi.org/10.1016/j.geoderma.2021.115607>
- Engström, D.R. and Wright, H.E., 1984. Chemical stratigraphy of sediments as a record of environmental change. In: E.Y. Haworth and J.W.G. Lund, eds. *Lake Sediments and Environmental History. Studies in Palaeolimnology and Palaeoecology in Honour of Winifred Tutin*. Leicester: Leicester University Press, 11–67.
- Fries-Knoblach, J., 2009. Hüttenlehm als Quelle zu Bauweise und Gestaltung eisenzeitlicher Gebäude. In: P. Trebsche, C. Eggl and I. Balzer, eds. *Architektur. Interpretation und Rekonstruktion. Beiträge zur Sitzung der AG Eisenzeit während des 6. Deutschen Archäologie-Kongresses in Mannheim 2008*. Langenweißbach: Beier & Beran, 31–53.
- Gaydarska, B., 2003. Application of GIS in settlement archaeology: an integrated approach to prehistoric subsistence strategies. In: О.Г. Корвін-Піотровський, В.О. Круц and С.М. Рижов, eds. *Трипільські поселення-гіганти. Матеріали міжнародної конференції / Tripolian settlements-giants. The international symposium materials*. Київ: Корвін Пресс, 212–216.
- Gaydarska, B., Nebbia, M. and Chapman, J., 2019. Trypillia Megasites in Context: Independent Urban Development in Chalcolithic Eastern Europe. *Cambridge Archaeological Journal*, 30 (1), 97–121. Available from: <https://doi.org/10.1017/S0959774319000301>
- Harper, T.K., 2011. Visualizing Agricultural Production during the Eneolithic: A Case Study from the Tripol'ye Giant-settlement of Tal'yanki. *Chronika*, 1, 27–32 [and 62–63 for Fig. and Tabs.]. Available from: <https://www.chronikajournal.com/resources/ChronikaOne%28online%29.pdf>
- Harper, T.K., 2012. *Sustaining Talianki: a Model of Eneolithic subsistence economics at a giant-settlement of the Western Tripolye Culture, Ukraine*. [Unpublished M.A.

- thesis. Department of Anthropology, State University of New York at Buffalo]. Available from: <https://doi.org/10.13140/RG.2.2.10409.44644>
- Hillman, G.C., 1984. Interpretation from archaeological plant remains: the application of ethnographic models from Turkey. In: W. van Zeist and W.A. Casparie, eds. *Plants and Ancient Man. Studies in palaeoethnobotany. Proceedings of the sixth symposium of the International Work Group for Palaeoethnobotany, Groningen 30 May-3 June 1983*. Symposium of the International Work-Group for Palaeoethnobotany 6. Rotterdam, Boston: A.A. Balkema, 1–42.
- Hofmann, R., Müller, J., Shatilo, L., Videiko, M., Ohlrau, R., Rud, V., Burdo, N., Dal Corso, M., Dreibrodt, S. and Kirleis, W., 2019. Governing Tripolye: Integrative architecture in Tripolye settlements. *PLoS ONE*, 14 (9), e0222243. Available from: <https://doi.org/10.1371/journal.pone.0222243>
- ICPT 2019: International Committee for Phytolith Taxonomy (Neumann, K., Strömberg, C.A.E., Ball, T., Albert, R.M., Vrydaghs, L. and Cummings, L.S.), 2019. International code for phytolith nomenclature (ICPN) 2.0. *Annals of Botany*, 124 (2), 189–199. Available from: <https://doi.org/10.1093/aob/mcz064>
- Johnston, S., Diachenko, A., Gaydarska, B., Nebbia, M., Voke, P., Bondar, K., Litkevych, V. and Chapman, J., 2018. The experimental building, burning and excavation of a two-storey Trypillia house. *Past. The Newsletter of the Prehistoric Society*, 89, 13–15. Available from: <https://www.prehistoricsociety.org/publications/past/89>
- Kahl, W.-A., Hinkes, R., Feeser, V. and Holzheid, A., 2013. Microfabric and anisotropy of elastic waves in sandstone – An observation using high-resolution X-ray microtomography. *Journal of Structural Geology*, 49, 35–49. Available from: <https://doi.org/10.1016/j.jsg.2013.01.006>
- Kirleis, W. and Dal Corso, M., 2016. Trypillian Subsistence Economy: Animal and Plant Exploitation. In: J. Müller, K. Rassmann and M. Videiko, eds. *Trypillia Mega-Sites and European Prehistory 4100-3400 BCE*. Themes in Contemporary Archaeology 2. London, New York: Routledge, 195–205. Available from: <https://doi.org/10.4324/9781315630731>
- Korvin-Piotroskiy and Menotti 2008: Корвин-Пиотровский, А. and Менотти, Ф., eds., 2008. *Трипольская культура в Украине. Поселение-гигант Тальянки*. Киев: Институт археологии НАН Украины.
- Kruts 1989: Круц, В.А., 1989. К истории населения трипольской культуры в междуречье Южного Буга и Днестра. In: С.С. Березанская, ed. *Первобытная археология. Материалы и исследования*. Київ: Издательство “Наукова думка”, 117–132.
- Kruts, V., 2012. Giant-settlements of Tripolye culture. In: F. Menotti and A.G. Korvin-Piotrovskiy, eds. *The Tripolye Culture Giant-Settlements in Ukraine. Formation, Development and Decline*. Oxford: Oxbow Books, 70–78. Available from: <https://doi.org/10.2307/j.ctvh1dvmn.8>
- Kylander, M.E., Ampel, L., Wohlfarth, B. and Veres, D., 2011. High-resolution X-ray fluorescence core scanning analysis of Les Echets (France) sedimentary sequence: new insights from chemical proxies. *Journal of Quaternary Science*, 26 (1), 109–117. Available from: <https://doi.org/10.1002/jqs.1438>
- Lancelotti, C. and Madella, M., 2012. The ‘invisible’ product: developing markers for identifying dung in archaeological contexts. *Journal of Archaeological Science*, 39 (4), 953–963. Available from: <https://doi.org/10.1016/j.jas.2011.11.007>
- Lichter, C., 2016. Burning Down the House – Fakt oder Fiktion? In: K. Bacvarov and R. Gleser, eds. *Southeast Europe and Anatolia in prehistory: Essays in Honor of Vassil Nikolov on his 65th anniversary*. Universitätsforschungen zur prähistorischen Archäologie 293. Bonn: Dr. Rudolf Habelt, 305–317.
- Lubos, C., Dreibrodt, S. and Bahr, A., 2016. Analysing spatio-temporal patterns of archaeological soils and sediments by comparing pXRF and different ICP-OES ex-