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Making the Most of Soils in Archaeology. A Review

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Abstract
Sediments serve as an archive of human and animal activity and environmental conditions through their physical and chemical properties as well as captured biological traces. Archaeologists have been extracting information from archaeological soils and sediments for decades, but recent technological developments, such as the analysis of lipid biomarkers, proteins, and ancient DNA from soil and the diversification of approaches necessitate a re-examination of standard field practice and a renewed emphasis on soil and sediments as archaeological materials. This review paper brings together a range of specialists to introduce cutting-edge approaches to analysing soils and sediments. From the large to the small scale, pioneering methods can complement established soil analytical methods to address issues of soil formation and erosion processes, heritage preservation, mobility, domestication, land use, human-environmental interactions, cultural and biological complexity, and ecosystem legacies. Soil analyses are poised to enable archaeologists to ask new questions and generate innovative hypotheses in an interdisciplinary research framework.

Keywords
Sediments, micromorphology, lipid biomarkers, palaeoproteomics, sedimentary aDNA, soilscapes

Zusammenfassung – *Die optimale Nutzung des Erkenntnispotentials von Böden in der Archäologie: Ein Überblick*

Sedimente dienen aufgrund ihrer physikalischen und chemischen Eigenschaften sowie der in ihnen enthaltenen biologischen Spuren als Archiv menschlicher Aktivitäten und Umweltbedingungen. Seit Jahrzehnten extrahieren Archäolog*innen Informationen aus Böden und Sedimenten, aber die jüngsten technologischen Entwicklungen wie die Analyse von Lipid-Biomarkern, Proteinen und alter DNA im Boden, sowie die Diversifizierung dieser Ansätze, erfordern eine Überprüfung der Standardpraxis im Feld und eine erneute Hervorhebung der Bedeutung von Böden und Sedimenten als archäologische Materialien. Dieser Überblick stellt ausgehend von der Expertise unterschiedlicher Fachleute die modernsten Ansätze der Boden- und Sedimentanalyse vor. Vom großen bis zum kleinen Maßstab können bahnbrechende Methoden etablierte Bodenanalyseverfahren ergänzen, um Fragen der Bodenbildung und Erosionsprozesse, der Bewahrung des kulturellen Erbes, der Mobilität, der Domestizierung, der Landnutzung, der Wechselwirkungen zwischen Mensch und Umwelt, der kulturellen und biologischen Komplexität und der Hinterlassenschaften von Ökosystemen zu behandeln. Aufgrund von Bodenanalysen können neue Fragen gestellt und innovative Hypothesen in einem interdisziplinären Forschungsrahmen entwickelt werden.

Schlüsselbegriffe
Sedimente, Mikromorphologie, Boden-Biomarker, Paläoproteomik, sedimentäre aDNA, Bodenlandschaften

1. Introduction

Soils and sediments are inconstant, changing components of the Earth's surface that play a fundamental role in reconstructing palaeoenvironments and archaeological landscapes.[1] Soils and sediments are also an archive of past cultural and non-cultural events, from volcanic eruptions to domestication and early agriculture, storing the microremains of

1 Bede et al. 2015. – Crombé, Verhegge 2015. – Kluiving et al. 2016.

settlement and production activities,[2] traces of pollution,[3] evidence of ecological changes, and the environmental impact of cultural behaviour.[4]

Despite the relevance of these traces, systematic applications of geochemistry and geo-biochemistry of soils and sediments are still underused in archaeology. This is all the more regrettable in light of the rapidly expanding toolbox of conjoined methods from geo, bio, and eco sciences, which allow us to target new high-potential proxies such as lipid biomarkers,[5] ancient human and environmental DNA (aDNA),[6] and ancient proteins.[7] Full exploitation of the soil archive remains sporadic. Keith W. Kintigh and colleagues[8] set out 25 grand challenges for archaeology to address the fundamental nature of human societies, of which the emergence of complexity, resilience, mobility, and human-environment interactions are just a few examples. Organic and inorganic microtraces in sediments, and microstructure of deposits, can contribute data to each of these challenges.

Soils and sediments are steadily evolving materials with very heterogeneous ages, origins, and formations. Soils form in place on some parent material, which can include sediments, and are spatially immobile. Pedogenesis (soil formation) occurs through the combined influences of climate, topography, and biological and geochemical processes on the parent material over time. A well-developed soil profile will have a distinct sequence of soil horizons. In contrast, layering in sediments is related to depositional events. Sediments are mineral particles formed by weathering of rocks and then transported by water, wind, ice, gravitation, people, or animals. Some sediment clasts can travel thousands of kilometres by air, while others experience shorter transport by river, lake, or seawater; some originate from local sources, transported by a variety of processes including local surface runoff or human activity, and some components may have formed autochthonously.[9] Accordingly, different soils and sediments can have very different properties, inherited from the different source regions. Some sediment grains could be many millions of years old, possibly (re-)eroded and (re-) deposited in several cycles, while others, formed during soil formation or by calcium precipitation or other geochemical processes, are very young.

This variability becomes even more relevant when considering soil/sediment mixing by bioturbation (e.g. rodents, earthworms, roots), cryoturbation, and along desiccation cracks. Earthworms and the roots of many plants can reach several metres in depth, disturbing deeper levels of soils and palaeosols and moving particles up or down. In addition to 'natural' processes, the anthropogenic effects on erosion, sedimentation, and pedogenesis, such as forest clearance, animal husbandry, water management, and agricultural practices, often act much faster and with more energy than non-cultural processes.[10] The geogenic, biogenic, and anthropogenic processes that act on sediments can be synchronous with deposition (syn-depositional) or post-depositional. Soils and sediments can be influenced by various amounts of syn- to post-depositional erosion, generating sediment deposition elsewhere as colluvium or increased sediment load in waterways. During depositional and post-depositional phases, variations in many large-scale and small-scale factors, including relief, climate, hydrology, mineral inputs, vegetation, and fauna can influence soils and sediments. In addition, soil formation (pedogenesis) frequently occurs as a post-depositional process on cultural deposits. Due to the nature of evolution and alteration, sediments/soils form discontinuous, patchy layers, highly variable in horizontal and vertical extent.[11]

To get the most out of soils and sediments, therefore, requires a solid understanding of formation processes and depositional environments, as well as the analytical techniques available in the second decade of the 21st century. In this article, we have three objectives: i) to advocate a conceptual reframing of soils and sediments as archaeological materials, ii) to review cutting-edge approaches to soil analysis, including recommendations for sampling and storage (see Tab. 1), and iii) to make suggestions for a re-examination of standard field practices and integration with established soil analytical methods to move towards a fully integrated bio-geoarchaeology framework, thus making the most out of archaeological soils and sediments.

2. Sedimentology

Sediments serve as archives from which to extract pollen, phytoliths, diatoms, black carbon, datable organics,

2 Migliavacca et al. 2013. – Pecci, Barba, Ortiz 2017. – Salisbury 2017.
3 Martínez Cortizas et al. 2016. – Lentz et al. 2020.
4 Karkanas et al. 2011. – Schumacher, Schier, Schütt 2016.
5 Bull, Betancourt, Evershed 2001. – Kovaleva, Kovalev 2015. – Zocatelli et al. 2017.
6 Slon et al. 2017. – Crump et al. 2021.
7 Oonk, Cappellini, Collins 2012.
8 Kintigh et al. 2014.
9 Pope 2013.
10 Zalasiewicz et al. 2019.
11 Pope 2013.

Method	Research possibilities	Sample type / quantity	Limitations	References
Sedimentology	Stratigraphy, depositional environments, human/animal activities, formation processes. Particle size, mineralogy, magnetic susceptibility, soil organic carbon, soil nitrogen, pH, carbonates. Extraction of pollen, phytoliths, diatoms, black carbon, datable organics, biomolecules, and other proxies	Core Bulk (loose); quantity depends on desired analyses; for multiple analyses, up to 1000 g may be needed Block samples; size depends on research questions and sediment types (larger blocks in looser sediments)	Most analytical methods require the destruction of a sample	Leopold, Völkel 2007; Gerlach, Eckmeier 2012; Nicoll, Murphy 2014; Vrydaghs, Ball, Devos 2016; Dreslerová et al. 2019; Jansen et al. 2019; Rick et al. 2022
Micromorphology	Micro-stratigraphy, micro-structure, mineralogy, walking surfaces, formation processes. Extraction of DNA	Block samples; plaster preferred to Kubiena tins	Coarse-grained sediments can lose structural integrity	Courty, Goldberg, Macphail 1989; Stoops 2014; Macphail, Goldberg 2017; Karkanas et al. 2019
Trace-element chemistry	site boundaries, activity zones, task areas, areas, spatial organization, land use, manuring, 'empty' burials	Bulk (loose); c. 3–5 g per analysis; in-situ pXRF	Contamination from leaching; ambiguity from overlapping activities; best as complementary data	Holliday, Gartner 2007; Wilson, Davidson, Cresser 2008; Lubos, Dreibrodt, Bahr 2016; Salisbury 2016; Salisbury 2017; Šmejda et al. 2018
Total C / N	depletion or enrichment due to agricultural and pastoral activities, ancient manuring, nutrition/diet	Bulk (loose); c. 5 g per analysis	C3 and C4 plant proportions influence $\delta^{13}C$ values; climate during decomposition influences $\delta^{15}N$ values; acid treatment may fractionate nitrogen	Beach et al. 2011; Terwilliger et al. 2011; Lauer et al. 2014; Sandor et al. 2022
Fatty acids (lipids) Faecal and plant leaf waxes	animal husbandry, land use, manuring, species change, vegetation change, environmental monitoring	Bulk (loose); c. 5 g per analysis	contamination from plasticizers; pedological conditions influence lipid preservation	Bull et al. 2000; Killops, Killops 2005; Shillito et al. 2011; Gerlach et al. 2012; Prost et al. 2017; Schirrmacher et al. 2019; Patalano, Zech, Roberts 2020
aDNA	evolutionary history (human, animal, plant), ecosystem monitoring and reconstruction, biodiversity	Cores or bulk samples from terrestrial, marine and lake sediments	contamination, degradation	Taberlet et al. 2012; Slon et al. 2017; Giguet-Covex et al. 2019; Edwards 2020; Kanbar et al. 2020; Vernot et al. 2021; Zavala et al. 2021
Amino acids (proteins)	human occupation, activities, animal husbandry, land use	Bulk (loose)	contamination, degradation	Oonk, Cappellini, Collins 2012; Cappellini et al. 2018; Hendy et al. 2018

Tab. 1. Summary of methods discussed in the text, with research questions and limitations.

biomolecules, and other environmental proxies.[12] Therefore, a thorough understanding of landscape formation processes, providing context for these archives, should be an integral part of any bio-geoarchaeological approach, in addition to being fundamental to reconstructions of land use and palaeoenvironments. Classical sedimentological analysis, based on coring campaigns and examination of exposed profiles,[13] represents one of the most powerful approaches to understanding formation processes that play out on the landscape level as well as mapping the complex entanglement of cultural and non-cultural drivers of these processes.

This analysis provides information about geomorphology, erosion/accumulation history, hydrological activity, and topographic changes.[14] Data is generally collected from exposed profiles, cores or drillings, including vibracores, hydraulic percussion drills, and Russian corers, among others. When using plastic or polycarbonate tubes within the corer, the resultant cores can be stored and subsampled for many of the other methods presented in this paper. Analysing the sediment grains themselves aids in determining the differences between sedimentation and erosion, depositional alterations such as (de)calcification or bioturbation and argillipedoturbation, and the identification of palaeosurfaces and phases of landscape stability or activity in the form of buried topsoils or redeposited soil sediments. Analyses include one or more of sediment colour, grain size, mineralogical composition, magnetic susceptibility, soil organic carbon, soil nitrogen, pH, and carbonates, as well as chemical enrichment.[15] Sediment particle size is typically measured using a laser granulometer or nested sieves. Soil organic matter and carbonate content are quantified through total carbon analyses.[16]

The application of these methods to reconstructing palaeolandscapes and human settlements, as well as the effects of both cultural and non-cultural processes on the archaeological record, is exemplified in the following studies. Studies of coastal plains in southern and central Italy, for example, demonstrated that sedimentation in these plains biases our understanding of archaeological settlement patterns; at the same time, sedimentation serves as a proxy for determining the relative sustainability of ancient agriculture on coastal plains.[17] In the coastal karst setting of the ancient Roman city of Apsorus (modern Osor) in the Adriatic Sea, airborne laser scanning (ALS) and airborne laser bathymetric (ALB) data combined with lithostratigraphic documentation, radiocarbon dating, and X-ray diffraction (XRD) of karst sinkhole sediments from vibracores allowed for the reconstruction of the palaeocoastline and the early onset of erosion.[18] Investigations of the fluvial hinterland of the ancient salt mine at Chehrābād, located at an altitude of c. 1450 m in the northwestern Iranian Plateau, used high-resolution digital elevation models, geotechnical rotary drilling, sedimentological analyses, and radiocarbon dating to document the long-term effect of centuries of irrigation on the fluvial landscape.[19] In another example, from the Ecse-halom burial mound in eastern Hungary, particle size, magnetic susceptibility, soil organic and carbonate content, and thin-section microscopy indicated that layers originated from the immediate vicinity of the mound, but have different characteristics than present-day soils. Results also suggest continuous salinization of the Hortobágy marshlands throughout the Holocene.[20] In the Seille Valley in eastern France, an integrated approach relying largely on borehole surveys, sedimentological analysis, geochronology, and palynology revealed the fundamental impact of prehistoric salt production on the hydrological regime and subsequently on landscape formation.[21] The scale of landscape alteration was such that these early industrial activities still influence land use and hydrology today.

3. Sediment Thin-section Microscopy

Micromorphology is the study of undisturbed, oriented, and resin-impregnated soil/sediment samples, ground to a thickness of 30 μm and observed under a petrographic microscope, which is a transmitted light microscope with a polarizing filter.[22] The identification and description of the soil/sediment components – their nature, geometry, and spatial arrangement – facilitates the recognition of distinct depositional environments and agents, thus providing a detailed understanding of the genesis of investigated contexts.

Since the 1970s and 1980s, micromorphological studies have been growing within archaeology.[23] Investigated topics include the reconstruction of past environments and land management strategies, the integrity and preservation of archaeological sequences, spatial differentiations at the

12 E.g. Gerlach, Eckmeir 2012. – Nicoll, Murphy 2014. – Vrydaghs, Ball, Devos 2016. – Jansen et al. 2019.
13 Rick et al. 2022.
14 Leopold, Völkel 2007. – Dreslerová et al. 2019.
15 Bede et al. 2015. – Draganits et al. 2019.
16 E.g. Nejman et al. 2018.
17 Attema 2017.
18 Draganits et al. 2019.
19 Draganits 2020.
20 Bede et al. 2015.
21 Riddiford et al. 2016.
22 Courty, Goldberg, Macphail 1989. – Macphail, Goldberg 2017.
23 Stoops 2014.

intra-settlement and intra-building scales, experimental and ethno-geoarchaeological studies aimed at defining pathways of deposition and degradation of specific materials as well as their distinctive features in thin section (e.g. mudbricks, dung, combustion features), the use of resources, and the study of ancient technologies.[24]

Other research focuses on the presence/absence of material residues from daily-life activities and how people dealt with these materials (e.g. by discarding, recycling, or incorporating them into the built environment). Examples of such investigations include middens, pits, ditches, constructed and informal floors, streets, open spaces, and penning areas.[25]

The main contribution of these studies lies in the reconstruction of the complex relationship between people, animals, and their physical surroundings, in turn offering insights into past living conditions, health as well as socially and culturally-driven perceptions of wellbeing and propriety. Also, in combination with other lines of evidence (e.g. organic chemistry, parasitology, mycology), the microstratigraphic study of waste, rubbish, and cleaning practices has the potential of addressing past health challenges, thus stimulating new interdisciplinary avenues for archaeological research.

By providing crucial microcontextual, microstratigraphical information, micromorphology is particularly well suited for multi-proxy research. For this reason, sampling strategies must preserve the structural integrity and orientation of the sediment blocks. Once in the laboratory, samples can be temporarily stored either at room temperature – in dry and ventilated places – or cold conditions (i.e. refrigerated), depending on the moisture and organic content. However, since weathering and mechanical disturbances eventually affect unconsolidated samples, at present the best way to ensure their long-term preservation is by consolidating them with resin.

The process of impregnating a block with resin is irreversible, and microsampling of loose sediment for further tests (e.g. gas chromatography-mass spectrometry (GC-MS), x-ray diffraction (XRD), ^{14}C) should be carried out before the impregnation. Other measurements (e.g. micro-computed tomography (µCT), micro-x-ray fluorescence spectroscopy (µXRF), micro-Fourier transform infrared spectroscopy (µFTIR), µRaman spectroscopy, etc.) can be carried out directly on the thin sections (with no coverslip) or by drilling the blocks.[26] However, one recent study suggests that GC-MS and GC-isotope ratio mass spectrometry (GC-IRMS) can be performed on dust drilled from impregnated slabs.[27] Another recent paper suggests that aDNA can be successfully extracted from impregnated blocks, directly linking genetic information with archaeological and ecological records on a microstratigraphic scale.[28]

4. Multi-element Chemistry

Anthropogenic processes, combined with the chemical and physical properties of soils, allow for the accumulation of chemical residues as indicators of past human activities. Many different soil characteristics and chemical analyses can be used to examine anthropogenic markers in the sediment archive, including phosphates, trace elements, plant nutrients, soil organic carbon, and biomarkers. Established methods of inorganic chemistry, including soil phosphate analyses and multi-element inductively coupled plasma mass spectroscopy or optical emission spectroscopy (ICP-MS or OES), along with recent developments in portable, handheld x-ray fluorescence (pXRF), provide information on site boundaries, activity areas, spatial organization, and land use.[29] Colorimetry tests are useful for soil phosphates, albeit providing qualitative results.[30]

The established process for inorganic multi-element soil chemistry involves the chemical digestion of a soil sample using acid, followed by measurement of the elements using ICP-MS or ICP-OES. Which acids are best suited for this has been subject to extensive debate,[31] with quasi-total extraction and sequential extractions emerging as the most reliable and replicable methods.[32] Handheld pXRF is now being widely used for soil analyses, returning total elemental composition comparable to total and quasi-total chemical extractions.[33]

Soil chemistry has contributed to understanding the uses and organization of space. Plazas at Mesoamerican settlements most likely served as multi-purpose areas, hosting

24 See Macphail, Goldberg 2017, and references therein.
25 See, recently, Macphail et al. 2017. – Koromila et al. 2018. – Furlan, Bonetto, Nicosia 2019. – Borderie et al. 2020. – Brönnimann et al. 2020. – Lisá et al. 2020. – Portillo, García-Suárez, Matthews 2020. – Shillito, Mackay 2020.

26 Nicosia, Stoops 2017. – Karkanas et al. 2019.
27 Rodríguez de Vera et al. 2020.
28 Massilani et al. 2022.
29 Holliday, Gartner 2007. – Wilson, Davidson, Cresser 2008. – Salisbury 2016. – Šmejda et al. 2018.
30 Holliday, Gartner 2007.
31 Middleton, Price 1996. – Wells 2004. – Vyncke et al. 2011.
32 Wilson, Cresser, Davidson 2006. – Wilson, Davidson, Cresser 2008.
33 Lubos, Dreibrodt, Bahr 2016. – Horák et al. 2018. – Šmejda et al. 2018. – Dreslerová et al. 2020.

markets, rituals, and feasts, based on the results of phosphate colorimetry, ICP-OES, and pXRF.[34] Soil chemistry conducted using pXRF at Tel Burna in the southern Levant identified several activity areas, including copper working.[35] Soil phosphates have been included in several attempts to locate prehistoric garden plots and agricultural fields.[36]

Future research combining established multi-element methods with soil biomarkers and micromorphology will strengthen the interpretational power of soil chemistry. Lipids and multi-element chemistry have been used together, for example, to identify activity areas in a Danish Iron Age longhouse.[37] Subsamples for trace element and lipid analysis can be taken from block samples prior to resin impregnation. The continued development of these methods, and potentially lower costs, should contribute to increased visibility of geochemical applications.

5. C/N Isotopes

Carbon (C) and nitrogen (N) are important elements, especially in terms of depletion or enrichment due to agricultural and pastoral activities and the ability to link these to isotope values in human and animal bone and cereal grains.[38] Traditional methods for soil carbon have employed wet or dry combustion methods to determine total carbon by measuring captured CO_2,[39] or to determine organic carbon through titration or loss on ignition using a muffle furnace and digital scales;[40] thermogravimetric analysers automated the process with computerized analysis and outputs. Current analytical techniques for percent total organic C and percent total N use combustion and elemental analysers, often linked to isotope-ratio mass spectrometers for $\delta^{13}C$ and $\delta^{15}N$.[41]

Results of studies on specifically archaeological soils indicate that anthropogenic activities result in increased C and N values.[42] For example, indications of C and $\delta^{15}N$ enrichment were recovered in relict topsoils found in excavated pit infillings.[43] In East Africa, before c. 1200 BP, changes in $\delta^{15}N$ most likely due to decreased precipitation were found in conjunction with changes in $\delta^{13}C$ associated with changes in the quantity of C_4 relative to C_3 plants.[44] Analysis of C and N isotopes from buried relict topsoil layers would significantly improve our interpretations of ancient ecosystems, manured agricultural plots, and human-environmental interactions. Soil and sediment archives provide archaeological material for analysis in the absence of preserved seeds and grains. Moreover, ^{14}C dating of the soil organic carbon, in conjunction with $\delta^{13}C$ analysis, increases the confidence that chronologies and interpretations are complementary.[45]

6. Soil Biomarkers

Approaches using lipid biomarkers to derive information about the history of a soil are particularly useful since information may be obtained in the absence of any morphological evidence. The specificity of particular biomarkers for different faecal sources makes them a valuable resource for environmental monitoring as well as archaeology.[46] A defining characteristic of biomarkers is the retention and stability of structural traits indicating the biogenic source, despite diagenesis,[47] although pedological conditions influence biomarker preservation.[48]

Methods for lipid analyses vary depending on the type of compound(s) being targeted. Pre-analysis preparation of samples typically involves several extraction and clean-up steps. In general, the process involves extraction into a solvent system of medium polarity such as dichloromethane (DCM)/methanol (2:1 *v/v*) to acquire a total lipid extract (TLE). Subsequently, the TLE is chromatographically separated into polarity-based fractions containing the target compounds, which are then derivatized with additional reagents (to ensure gas chromatographic amenability), and analysed by some form of chromatographic instrumentation, most commonly GC-MS.[49]

Faecal biomarkers (5β stanols and, to a lesser extent, bile acids), have proved to be reliable and environmentally recalcitrant indicators of cultural activity and therefore have been widely deployed in the archaeological sciences.[50] In addition to manuring studies,[51] geochemical biomarkers have aided in reconstructing palaeoenvironmental conditions

34 Coronel et al. 2015.
35 Šmejda et al. 2018.
36 Roos, Nolan 2012.
37 Hjulström, Isaksson 2009.
38 E.g. Kanstrup et al. 2014. – Dreslerová et al. 2021.
39 Nelson, Sommers 1982.
40 Dean 1974.
41 Beach et al. 2011. – Lauer et al. 2014. – Dreslerová et al. 2021.
42 Beach et al. 2011. – Lauer et al. 2014. – Sandor et al. 2022.
43 Lauer et al. 2014.
44 Terwilliger et al. 2011.
45 van der Plicht, Streurman, van Mourik 2019.
46 Walker et al. 1982. – Dinel, Schnitzer, Mehuys 1990. – Bethell, Goad, Evershed 1994.
47 Peters, Moldowan 1993.
48 Bull et al. 2000. – Killops, Killops 2005.
49 Elhmmali, Roberts, Evershed 1997. – Bull et al. 1999. – Gerlach et al. 2012.
50 Evershed et al. 1997. – D'Anjou et al. 2012. – White et al. 2018.
51 Bull et al. 1999. – Simpson et al. 1999.

and other human activities.[52] Faecal biomarkers provide data on pastoral practices and land use in France,[53] animal husbandry and uses of dung in Anatolia,[54] and plants as a significant component of Neanderthal diet in Spain.[55] They have long been used to make basic distinctions between different animal groups and/or species such as humans, ruminants, and pigs; this approach has recently been extended and refined to increase the range of animal species (e.g. reindeer, lemming, goat, sheep, horse, moose, dog, pig, goose, donkey), significantly increasing its usefulness for research on early domestication and animal husbandry.[56]

Other lipids are also being used as proxies for a range of activities in various archaeological contexts at multiple analytical scales. For example, combined archaeological and experimental data revealed that fatty acids recovered from ancient hearths most likely derive from the burning of large animal bones.[57] Similar to faecal biomarkers, *n*-alkanes and plant sterols from leaf waxes can exhibit chemical signatures specific to different plant types and can survive in sediments for thousands of years or more.[58] They can be used to reconstruct plant communities and species changes, such as from lacustrine to terrestrial or from forest to grassland.[59] Carbon and hydrogen stable isotopes comprising these compounds can be used to infer palaeoclimate variability[60] and resultant expansion and contraction of forests.[61] In another application, *n*-alkanes indicated significant Neolithic biomass burning.[62]

7. Ancient DNA from Sediments

The analysis of ancient sediment DNA (*seda*DNA) from terrestrial, marine, and lake sediments has become an increasingly powerful tool for understanding past ecosystems, biodiversity, and evolutionary history as it enables the examination of DNA from many different taxa (flora, fauna, and microorganisms) from each sample and can be applied across large temporal ranges.[63] This has been demonstrated through various studies, including, for example, the dating of the appearance of a viable ice-free corridor between Beringia and North America[64] and tracking changes in the arctic ecosystem during the last interglacial.[65]

The basic processing of *seda*DNA involves three steps: (1) DNA extraction; (2) data generation through (i) metabarcoding (sequencing of amplicons targeted for taxa of interest), (ii) shotgun sequencing (direct sequencing of DNA libraries), or (iii) enrichment of DNA libraries for specific genomic targets by hybridization capture; and (3) data analysis (data authentication and taxa identification).[66] Methodological studies in *seda*DNA have increased our understanding of how DNA is bound to various sediment components,[67] but there are still open questions surrounding *seda*DNA taphonomy. Current known temporal limits are similar to skeletal remains at over 300,000 years in cool environments.[68] Studies have also found minimal evidence of DNA leaching, but large impacts of bioturbation.[69] It is therefore critical to work closely with micromorphologists, chronologists, and geologists to evaluate stratigraphic integrity. Detailed studies on the impacts of sampling locations and flow rates on lake *seda*DNA emphasize that large sample sets are needed to accurately understand past environments.[70]

Recently, *seda*DNA has been integrated with archaeological data to study patterns of human occupation.[71] This was taken a step further when it was demonstrated that hominin DNA could not only be recovered from Pleistocene sediments,[72] but also be used to reconstruct population histories and directly compare them to changes in climate and faunal diversity.[73] Moreover, aDNA has been successfully extracted from impregnated block samples and uncovered thin section slides, expanding the range of potential sampling strategies.[74] *Seda*DNA studies will continue to provide new insights on past eco-diversity and how it was shaped by changes in climate.

8. Palaeoproteomics from Sediments

Much like lipids and DNA, proteins are important biomarkers that should become fundamental to archaeological research. An increasing body of evidence and models

52 Hjulström, Isaksson 2009. – Shillito et al. 2011. – Prost et al. 2017.
53 Zocatelli et al. 2017.
54 Portillo, García-Suárez, Matthews 2020.
55 Sistiaga et al. 2014.
56 Prost et al. 2017. – Harrault et al. 2019.
57 Kedrowski et al. 2009.
58 Patalano, Zech, Roberts 2020.
59 Schwark, Zink, Lechterbeck 2002. – Zech et al. 2010. – Schatz et al. 2011.
60 Schirrmacher et al. 2019. – Patalano, Zech, Roberts 2020.
61 Wurster et al. 2010.
62 Eckmeier, Wiesenberg 2009.
63 Giguet-Covex et al. 2014. – Parducci et al. 2017.
64 Pedersen et al. 2016.
65 Crump et al. 2021.
66 Edwards 2020.
67 Giguet-Covex et al. 2019. – Kanbar et al. 2020.
68 Rawlence et al. 2014. – Slon et al. 2017.
69 Edwards 2020.
70 Edwards 2020. – Vernot et al. 2021.
71 Bálint et al. 2018.
72 Slon et al. 2017.
73 Vernot et al. 2021. – Zavala et al. 2021.
74 Massilani et al. 2022.

demonstrates that proteins can be recovered from ancient contexts and geographic regions with generally poor preservation of ancient biomolecules.[75] Soils are an abundant archaeological artefact and may function as a sink for molecules such as proteins, which hold specific information about their origin, enabling the detection of human occupation and activities.[76] This information yield is often limited by contamination and degradation. Proteins have been presumed to be especially prone to microbial degradation, as they have a high nutrition value for soil organisms.[77] However, just as DNA molecules can successfully survive degradation by absorption onto mineral matrices and adsorption on clays and to humic substances in the soils,[78] so can proteins.[79] Moreover, studies have shown that amino acids are more stable than nucleic acids in many environments.[80] Nevertheless, although the potential of proteins as archaeological biomarkers is widely appreciated and already used for a variety of different archaeological materials,[81] the applicability of soil proteomics to archaeological soil material is still in its infancy and has a great need for testing and development. A first exploratory study done in 2012 investigated the effects of different soil components on the fraction of proteins in soils, the isolation efficiency of different reagents, and how the detection and identification of proteins in soils are affected by protein retention, isolation reagents, and co-isolated soil particles.[82] Since then (as far as the authors are aware), no substantial progress has been made in the utilization of ancient proteins from archaeological soils. Recently, soil proteomics analysis has been applied to investigate the soil textile imprints of a tomb at the Dahekou Cemetery site in China.[83] More work is necessary to fully develop the methodology and exploit the great potential of this biomarker in archaeological soils and sediments.

9. Sampling and Storage Recommendations

Collecting and storing sediments for multi-element chemistry, phosphates, magnetic susceptibility, and total carbon are relatively straightforward and easily done by archaeologists in most field settings. Samples should be air-dried or freeze-dried as soon as possible to limit organic activity, such as the continued action of tiny insects and microorganisms or germination of seeds. These samples can be stored indefinitely at cool temperatures. The greatest concern is rapid or extreme changes in temperature or humidity.

These older methods of sampling for inorganic soil chemistry are inadequate for current capabilities involving biomolecules. One immediate methodological aim in archaeological soil chemistry, and geoarchaeology more broadly, should be to establish new and standardized sampling and storage methods focused on the preservation of biomolecules, in particular those collected directly from archaeological contexts. For sediment cores collected in sealed tubes, this is not as problematic. In most other cases, geoarchaeologists should be able to take *in situ* measurements before sampling, for example using pXRF or magnetic susceptibility.[84]

Sampling schemes should be developed with archaeologists and geochronologists to discuss research questions and ensure as far as possible the stratigraphic integrity of the material. With the right sampling approach, soil samples can be taken for both aDNA and protein analysis together. Samples for inorganic chemistry can later be subsampled and freeze-dried to expedite analysis, or parallel sampling should be undertaken. In caves and other terrestrial archaeological sites, samples should be taken from exposed archaeological profiles in a grid-like pattern (approximately every 10 cm or adapted to the specific situations and research questions) if possible, or in multiple columns of block samples, to facilitate microstratigraphic analysis of sample locations. Including samples from above and below each layer of interest is critical for understanding the context of the results. To limit the number of samples tested, a preliminary screening may be completed of 1–2 samples per layer of interest to determine the success of DNA and/or protein preservation at the site. Sampling is also possible from drill cores, following similar considerations.

While sampling, minimization of modern contamination is essential. We encourage the use of plastic (non-latex) gloves, facemasks, and hair coverings when sampling, with frequent glove changes (ideally between samples, at minimum when they are visibly dirty). Furthermore, no wool, silk, rubber, or leather should be worn, and skin and hair should be covered at all times when sampling and handling samples, as protein and DNA from these sources could potentially contaminate the samples. If lipid analyses are to be conducted, then the

75 Cappellini et al. 2018.
76 Oonk, Cappellini, Collins 2012.
77 Cappellini et al. 2018.
78 Nielsen, Calamai, Pietramellara 2006. – Levy-Booth et al. 2007.
79 Quiquampoix et al. 1993. – Zang et al. 2000. – Nielsen, Calamai, Pietramellara 2006.
80 Buckley 2019.
81 Hendy et al. 2018.
82 Oonk, Cappellini, Collins 2012.
83 Li, Zhu, Xie 2021.

84 Kainz 2016. – Šmejda et al. 2018.

introduction of any extraneous chemical compounds, e.g., plasticizers and other additives from plastic implements or containers, should be avoided. Storage in furnaced glassware or, as a minimum, uncontaminated aluminium foil is recommended. Clean surfaces and equipment are essential. Metal or plastic tools are best washed with bleach solution or 70 % ethanol, and baked glassware should be used. Generally, all measures taken to reduce contamination from sampling/excavation, through storage and laboratory analysis should always be reported and described in publications. A thorough guide to handling palaeoproteomic samples in the lab, including a detailed explanation of why the use of non-latex gloves is necessary, is given in Jessica Hendy and colleagues.[85] When sampling vertical profiles, start with the bottommost sample in a column and remove the surface of the material with a sterile scalpel or spatula. Then sample about 0.5–5 grams of sediment into a sterile plastic tube or bag. Fresh tubes and scalpels should be used for each sample collected.

Vacuum freeze-drying should be considered the standard for the storage of small sediment samples because it has the advantage of preserving the sample's chemical and biological structures. The method was used for soil biomarker analyses in the Faroe Islands.[86] Another study revealed that vacuum freeze-drying minimized errors in mercury (Hg) fraction analysis, yielding Hg values close to those from fresh samples, as compared to air-drying and oven-drying.[87] Currently, vacuum freeze-drying is being investigated as a possible alternative for long-term storage of sediment cores, although the project is in a preliminary stage and the feasibility of this preservation strategy still needs to be fully evaluated.[88] Research is needed to assess the effect of the vacuum on stratigraphic integrity in cores. The impacts of different storage conditions on the survival of aDNA and proteins are not fully studied, but we recommend storing sediments in cool environments, ideally a refrigerator, or frozen if they were collected in permafrost. Lake sediment samples and any samples with highly organic layers should be stored at 4°C, ideally in a cold room, as soon as possible after sampling on site. Soil samples for aDNA or protein analysis should ideally be stored in a freezer at -80°C to avoid bacterial growth until processing. Freeze-thaw cycles should be avoided. The potential of impregnated micromorphological blocks as repositories of trace element and biomolecular data[89] will be resolved through additional testing but looks promising.

10. Summary: Analysis for the Future

Landscapes, soils, and sediments have for too long been solely treated as the backdrop of human existence, rather than as elements that can capture a wide range of traces of behaviours and cultural practices. Soil deposits store incredible amounts of information generated by both cultural and non-cultural processes. Information stored in the sediment archive includes the macro- and microstratigraphy, chemical signatures, isotopes and biomarkers, aDNA, and proteins. Bio-geoarchaeological work of the last decades has shown that new methods acquire data that was previously unavailable, and address new questions that were previously barely imaginable.

To get the most out of soil means incorporating micro-scale methods into a discipline for which the destruction of soil archives – excavation – is the primary approach. In practice, it is difficult to store vast quantities of soil from excavations without a clear goal and strategy, and it is also not necessary. Here, we present an outline of current methods and research questions they can address, with the goal of inspiring archaeologists to integrate smart soil sampling and storage in their research design. An interdisciplinary, integrative approach with the joint elaboration of questions and selection of the best methods is essential for making real progress. Methods are constantly being developed and in flux, and it remains a challenge to integrate the widest possible range of approaches and methods from increasingly smaller samples, by finding ways to produce several strands of evidence from single samples by applying different methods in optimal sequence and curating samples appropriately. Advances in extracting trace element and biomolecular data from thin-section blocks offer hope for consolidated sampling and storage in the future. However, we must emphasize that sedimentary palaeoproteomics, particularly extraction from terrestrial archaeological contexts, is underdeveloped in comparison with other methods.

Soils and sediments are the records that hold information about the lives of humans, animals, plants, and microbes, as well as their dynamic relationships with each other and the geosphere. It is time to make the most out of soils for archaeology, and the most of the archaeological record by flipping our methodological paradigm. Instead of discarding soils and sediments to get to objects of interest, sediments must become the objects of interest, analysed through the plethora of new technologies.

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85 Hendy et al. 2018.
86 Curtin et al. 2021.
87 Liu et al. 2019.
88 Enevold et al. 2019.
89 Rodriguez de Vera et al. 2020. – Massilani et al. 2022.

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Data Availability Statement

Data sharing does not apply to this article as no new data were created or analysed in this study.

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