



Thematic review

Advances in Archaeological Soil Chemistry in Central Europe

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ABSTRACT

Analytical technologies for the evaluation of archaeological soils have developed rapidly in recent decades, and now support a range of innovative research and interpretations of archaeological sites and landscapes. Established methods, including phosphates and multi-element ICP-MS/OES, have provided interpretations of the use of space within settlements and houses, and the function of specific archaeological features. Recently, portable X-Ray Fluorescence has been introduced to archaeological soil science, but published results have generated knowledge gaps. The correspondence between archaeological geochemical anomalies and specific human activities is partly dependent on geology (including sediment type and relative acidity and permeability of the soil), topography, and formation processes, as well as influence of human activities. At the same time, which elements, and fractions of elements, are measured is largely dependent on instrument parameters and extraction methods. This paper provides an overview of archaeological soil chemistry in Central Europe, and the current state-of-the-art, followed by an assessment of future developments in archaeological soil chemistry, molecular biogeochemistry, and the significance of geoarchaeology in multi-disciplinary research.

1. Introduction

Soils and sediments are archaeological materials, archaeological resources that contain an archive of past events and cultural behaviours. For nearly one hundred years, archaeologists and soil scientists have accessed parts of this archive through applications in archaeological soil chemistry. Central Europe has been home to several key developments in archaeological soil chemistry, from the earliest applications of soil phosphate analysis to recent practical application of portable X-ray Fluorescence spectroscopy. Established methods, including phosphate analyses and multi-element ICP-MS/OES, have provided interpretations of the use of space within settlements and houses, and the function of specific archaeological features. As archaeology continues to evolve as a discipline, micro-remains are becoming increasingly important sources of data about past human mobility, diversity, production, consumption, and human ecodynamics. These data come from a range of sources, including both human and animal

remains, primarily bones and teeth (proteomics, Charlton *et al.*, 2019; strontium isotopes, Giblin *et al.*, 2013), and portable artefacts (*e.g.* starch grains on grinding stones and ceramics, Duke *et al.*, 2018; ceramic residue analysis Dunne, *et al.*, 2019; residues on lithics, Rots *et al.*, 2015).

Chemistry of soils and sediments have been recognized, and utilized, for a longer time. In addition to soil phosphate analysis, soil chemical methods for archaeology traditionally include multi-element chemistry, soil pH, magnetic susceptibility, and soil organic carbon and nitrogen. Recently, research in the Central European Palaeolithic have combined these, for example, assessing carbon, nitrogen, and magnetic susceptibility at the Pod Hradem cave to aid in interpreting soil formation and potential climatic changes (Nejman *et al.* 2018; 2020). Nevertheless, full exploitation of the soil archive remains sporadic.

Cultural soilscape (Salisbury, 2016; Wells, 2006) hold ancient human and environmental DNA (Slon *et al.*, 2017; Willerslev *et al.*, 2003), biomarkers (Kovaleva and Kovalev, 2015; Zocatelli *et al.*, 2017), traces of pollution (Martínez Cortizas *et al.*, 2016; Veron *et al.*, 2014), evidence of ecological changes and the environmental impact of

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cultural behaviour (Schumacher *et al.*, 2016; Sprafke, 2016), archaeological site formation processes (Nicoll and Murphy, 2014), raw material provenance for components of ceramic matrices (Riebe and Niziolek, 2015), and the contamination of cultural heritage and artefacts by modern pollution (Nord *et al.*, 2005). Continued development of archaeological biogeochemistry is beginning to recognize the archaeological potential of proteomics, genomics, and other biomarkers in the soil archive.

Several reviews of archaeological soil chemistry have been published (Oonk *et al.*, 2009a; Wilson *et al.*, 2008); of especial import was the detailed summary of soil phosphate methods in archaeology by Holliday and Gartner (2007). A vast body of literature exists for archaeological soil chemistry (Table 1). This review of the history and development of archaeological soil chemistry focuses on examples from Central Europe, and the current state-of-the-art, followed by an assessment of future developments in molecular biogeochemistry and the significance of geoarchaeology in multi-disciplinary research. Currently, trends are changing from the traditional roles of archaeological soil chemistry – site prospection and delimiting habitation areas – into a complex, multi-disciplinary endeavour integrating various strands of

geoarchaeology, bioarchaeology, and environmental studies. This trajectory needs geoarchaeologists to contribute to diachronic and synchronic examinations of archaeological landscapes and human-ecodynamics.

2. Archaeological soil phosphates in Central Europe

The history of archaeological soil chemistry extends over a century (Arrhenius, 1931) and across the globe, from Alaska (Knudson *et al.*, 2004) to the Levant (Šmejda *et al.*, 2018) and to Australia (Fanning *et al.*, 2018). The link between ancient human occupation and increased soil phosphate content were first noted in late 19th or early 20th century agricultural soil surveys (Russell, 1915). Publication of methods and results began when Swedish agronomist Olaf Arrhenius recognized and recorded the relationship between surface finds, human occupation, and soil phosphate levels while conducting agricultural soil testing, and posited a causal correlation between enhanced soil phosphates and human settlements (Arrhenius, 1929; 1931). The method was applied to Swedish habitation sites by Schnell (1932), who sampled along transects radiating outward from an assumed

Table 1. Selected references for archaeological soil chemistry, with focus on Central Europe (arranged chronologically).

Type of study	References
Soil chemistry & soil science	Feigl, 1960; Bowen, 1979; Füleký, 1983; Kabata-Pendias and Pendias, 1984; Sposito, 1998; Sparks 1996; 2003; Holliday 2004.
Early archaeological investigations	Arrhenius, 1929; 1931; Lorch, 1930; 1940; 1941; 1951; Schnell, 1932; Christensen, 1935; Bandi, 1945; Stoye, 1950; Lutz, 1951; Dauncy, 1952; Pelikán, 1954; Dietz, 1957.
Methodological reviews and histories	Sjöberg, 1976; Eidt, 1977; Keeley, 1981; Bethell and Máté, 1989; Zöllitz and Heinrich, 1990; Walker, 1992; Kondratiuk and Banaszuk, 1993; Bjelajac <i>et al.</i> , 1996; Aston <i>et al.</i> , 1998; Klamm <i>et al.</i> , 1998; Leonardi <i>et al.</i> , 1999; Haslam and Tibbett, 2004; Wells 2004a; Holliday and Gartner, 2007; Wilson <i>et al.</i> , 2008; Oonk <i>et al.</i> , 2009a; Pastor <i>et al.</i> , 2016.
Applications in Central Europe	
Phosphate	Gundlach, 1961; Schwarz, 1967; Grimm, 1971; Sjöberg, 1976; Kieffmann, 1979; Zöllitz, 1980; 1982; 1983; 1986; Gebhardt, 1982; Majer, 1984; Zimmermann, 1995; 2001; 2008; Stäuble and Lünig, 1999; Majer, 2004; Sarris <i>et al.</i> , 2004; Ernée, 2005; Ernée and Majer, 2009; Hlavica <i>et al.</i> , 2011; Schreg and Behrendt, 2011; Petřík <i>et al.</i> , 2012; Salisbury, 2012; Lauer <i>et al.</i> , 2013; Salisbury <i>et al.</i> , 2013; Weihrauch <i>et al.</i> , 2017; Weihrauch and Söder, 2020; Weihrauch <i>et al.</i> , 2020.
Multi-element	Hejzman <i>et al.</i> , 2011; 2013a; 2013b; Gauss <i>et al.</i> , 2013; Salisbury, 2013; Pető <i>et al.</i> , 2015; Lubos <i>et al.</i> , 2016; Salisbury, 2016; Dreibrodt <i>et al.</i> , 2017; Šmejda <i>et al.</i> , 2017; 2018; Horák <i>et al.</i> , 2018; Janovský and Horák, 2018; Pankovská <i>et al.</i> , 2018; Pető <i>et al.</i> , 2019; Dreslerová <i>et al.</i> , 2020.
Future directions	
Lipid biomarkers	Bull <i>et al.</i> , 2000; Bull <i>et al.</i> , 2002; Schwark <i>et al.</i> , 2002; Killops and Killops, 2005; Zech <i>et al.</i> , 2010; Schatz <i>et al.</i> , 2011; Sistiaga <i>et al.</i> , 2014; Prost <i>et al.</i> , 2017; Zocatelli <i>et al.</i> , 2017; Harrault <i>et al.</i> , 2019; Schirrmacher <i>et al.</i> , 2019; Patalano <i>et al.</i> , 2020; Portillo <i>et al.</i> , 2020.
Isotopes	Bogaard <i>et al.</i> , 2007; D'Anjou <i>et al.</i> , 2012; Abell <i>et al.</i> , 2019; Bataille <i>et al.</i> , 2020; Snoeck <i>et al.</i> , 2020.
Sediment aDNA	Hebsgaard <i>et al.</i> , 2009; Giguët-Covex <i>et al.</i> , 2014; Madeja, 2015; Thomsen and Willerslev, 2015; Parducci <i>et al.</i> , 2017; Slon <i>et al.</i> , 2017; Brunson and Reich, 2019; Epp <i>et al.</i> , 2019; Nejman <i>et al.</i> , 2020.
Proteomics	Oonk <i>et al.</i> , 2012.

central point in a given site and constructed isopleth maps of the phosphate values to delimit site boundaries. Following this, archaeologists in Central Europe and elsewhere have conducted soil phosphate analyses for archaeological survey and site interpretations for nearly a century, with continuing methodological developments.

Early archaeological soil chemistry focused primarily on phosphates for four reasons: First, it was the first element explicitly connected to prehistoric human habitation, and subsequent studies exploited this discovery. Second, it is essential to life and therefore can be found in and deposited by anything organic, as primary, secondary, or *in situ* refuse. Inputs include food preparation wastes such as bone, meat, fish, and plants, wood ash, human or animal burials, and urine and faeces of humans and animals. Therefore, human habitation areas will have higher levels of soil P than surrounding areas with no habitation. Conway (1983) demonstrated that human occupation could result in annual increases in concentrations of P from 1–10%. Third, phosphates tend to accumulate quickly, have low solubility and a strong ability to fix within the soil profile. In favourable soil conditions, P remains stable and is likely to be retained, even in disturbed soils, for millennia without appreciable leaching. Fourth, in its most basic form, phosphate testing is fast and inexpensive compared with almost every other analytical technique, and can be conducted in the field during survey or excavation.

2.1 The first 60 years of archaeological soil phosphate analysis

Arrhenius's methods were quickly applied to prehistoric and medieval sites in Central Europe by German geographer Walter Lorch (1940; 1941; 1951). Lorch sampled along regular transects across and around ancient settlements, used a laboratory colorimetric method to measure phosphate content, and graphed the results. By comparing graphs of density and distribution, and interpreting the different profiles, Lorch persuasively argued that variability in phosphate was due to different subsistence economies in the Palaeolithic, Neolithic, and metal ages. Other research projects soon followed. Bandi (1945) used Lorch's method to locate a medieval site in Switzerland. Dietz (1957) used a method of sulphuric acid in test tubes visually compared to standards to examine a small plot of land with surface material indicating prehistoric activity, looking for evidence of organic waste deposits.

The next major advance in archaeological soil phosphate studies came with the development of a spot-test, or ring-chromatography test, by Friedrich "Fritz" Feigl in Vienna. In his comprehensive two-volume compendium on chemical spot-tests, Feigl (1960) recommended the highly toxic and corrosive nitric acid (HNO_3) to prepare an ammonium molybdate solution, and the toxic and flammable benzidine as a reducing agent to measure inorganic phosphate from geological samples. Gundlach (1961) modified Feigl's method to increase the speed and safety when testing soil from boreholes at prehistoric sites. Gundlach retained the nitric

acid for digestion, but switched to the organic and relatively harmless ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) for reduction, making it more stable and eliminating the need for flame and ammonia in field conditions. In any acid-molybdate method, PO_4 reacts with molybdate to form phosphomolybdic acid (yellowish in colour); in Gundlach's method, phosphomolybdic acid is reduced by ascorbic acid to form a blue complex. Gundlach conducted his tests in the field, with the filter paper and drop bottles attached to a pole stuck in the ground next to his borehole (depicted in Gundlach, 1961 p.736, Figure 1), and noted that the entire process takes approximately 90 seconds. Gundlach ranked the results on a scale of 1–5, from none to very high levels of P.

Schwarz (1967) used the Gundlach method and established a field methodology for sample collection, collecting samples in plastic bags, collecting enough soil to run multiple tests and to determine colour and grain size, and collecting samples by layer. Schwarz conducted large-scale surveys near the San Bernardino Pass in southern Switzerland, taking samples at 30–50 m intervals. Two suggestions regarding research methodology are of note from the Schwarz's paper. First, he observed that some information about land-use, previous archaeological investigations, and geomorphology are essential to planning a chemical survey campaign. Second, he suggested that phosphate surveys would be more useful when complemented by geophysical surveys or test trenching, and warned that carrion pits, where local villagers dispose of diseased animals, should not be mistaken for prehistoric habitations (Schwarz, 1967, pp.58–61). Schwarz's sampling approach has been criticized (Sjöberg, 1976, p.449) because of his suggestion that samples be taken along natural lines, such as field boundaries, rather than on a regular grid or transect system.

Kiefmann (1979) conducted large-scale phosphate mapping in East Holstein and arrived at two significant conclusions. First, soil phosphate content is influenced by both changing land-use and pedogenesis. That is, archaeological soil chemistry requires some understanding of both cultural and non-cultural formation processes, including soil formation. Second, Kiefmann found that different extraction methods yielded different P concentrations and distributions.

Zölitz (1980) pioneered the use of variogram modelling as a statistical method for analysing soil phosphate results. Majer (1984) developed a 3-point relative scale for measuring results, and noted that archaeologists were most interested in the anthropogenic enhancement of soil phosphate, rather than the total quantity of P. Phosphate surveys successfully identified residential areas in both prehistoric and medieval sites (Grimm, 1971; Zölitz, 1982; 1983; 1986).

Eidt refined Gundlach's method further, first by replacing nitric acid with hydrochloric acid (HCl) (Eidt, 1973) and then by developing a bath to stop the chemical reaction, so that results could be archived (Eidt, 1977). Eidt found that HCl is superior to both nitric acid and sulphuric acid when extracting P in laboratory tests, and observed that the use of HCl for both field and lab analyses would improve comparability of results. He also noted that the question of

which acid was superior in most cases had been raised by both Feigl (1960) and Murphy and Riley (1962), and the answer was probably that both nitric and sulphuric acids are oxidizers, whilst producing a blue colour from phosphate and molybdenum requires a reducing process (Eidt, 1973, p.207).

By the 1990s, spot-test methods were standardized, a few specific extraction formulas for colorimetry had been widely adopted, and several critiques and reviews were being published (Bethell and Máté, 1989; Bjelajac *et al.*, 1996; Klamm *et al.*, 1998; Kondratiuk and Banaszuk, 1993; Zólitz and Heinrich, 1990). In Germany and in the Czech Republic, soil phosphate analyses were frequently used (Majer, 2004; Stäuble and Lünig, 1999; Zimmermann, 1995), but were rarely used elsewhere in Central Europe.

2.2 Recent developments of soil phosphate analysis in Central Europe

Despite acknowledgement of the contribution of soil phosphate mapping for settlement archaeology (Zimmermann, 2001), the increasing sophistication of aerial remote sensing and geophysical prospection led to wide-scale adoption of alternative survey methods at both the site and regional scale, and a concomitant decrease in soil chemistry survey. In part, this can be explained by recognizing that phosphate spot testing, the method fastest

and requiring the least training and financing, is also the least informative method; Zimmerman (2001) called it inappropriate. As a survey method, inappropriate is perhaps unfair for a tool that identifies areas of possible human settlement activity while enabling students to participate in sample collection and analysis during field projects. Archaeological soil phosphate has been the largest and most common application of geochemistry in archaeology, with examples at the regional and micro-regional scales (Nuñez and Vinberg, 1990; Salisbury, 2012; Thurston, 2001; Zólitz, 1982). Challenges remain, however, including fertilization (Weihrauch *et al.*, 2017) and heterogeneous environments (Weihrauch and Söder, 2020).

Although an essential component of site prospection and settlement archaeology, soil phosphate analysis is largely restricted to identifying activity or habitation areas where large quantities of organic matter were deposited, such as detecting boundaries of settlements, household clusters, and activity zones (Salisbury, 2016; Sarris *et al.*, 2004; Zimmermann, 2008). Pav (available P) was used to delineate site boundaries at a series of Late Neolithic and Early Copper Age settlements in the Körös Region of eastern Hungary (Figure 2; Salisbury, 2012; 2016).

P analysis was applied at the excavation of an early medieval house at Schalkstetten in South Germany. Samples from a 1 m interval grid revealed three areas of elevated P. One

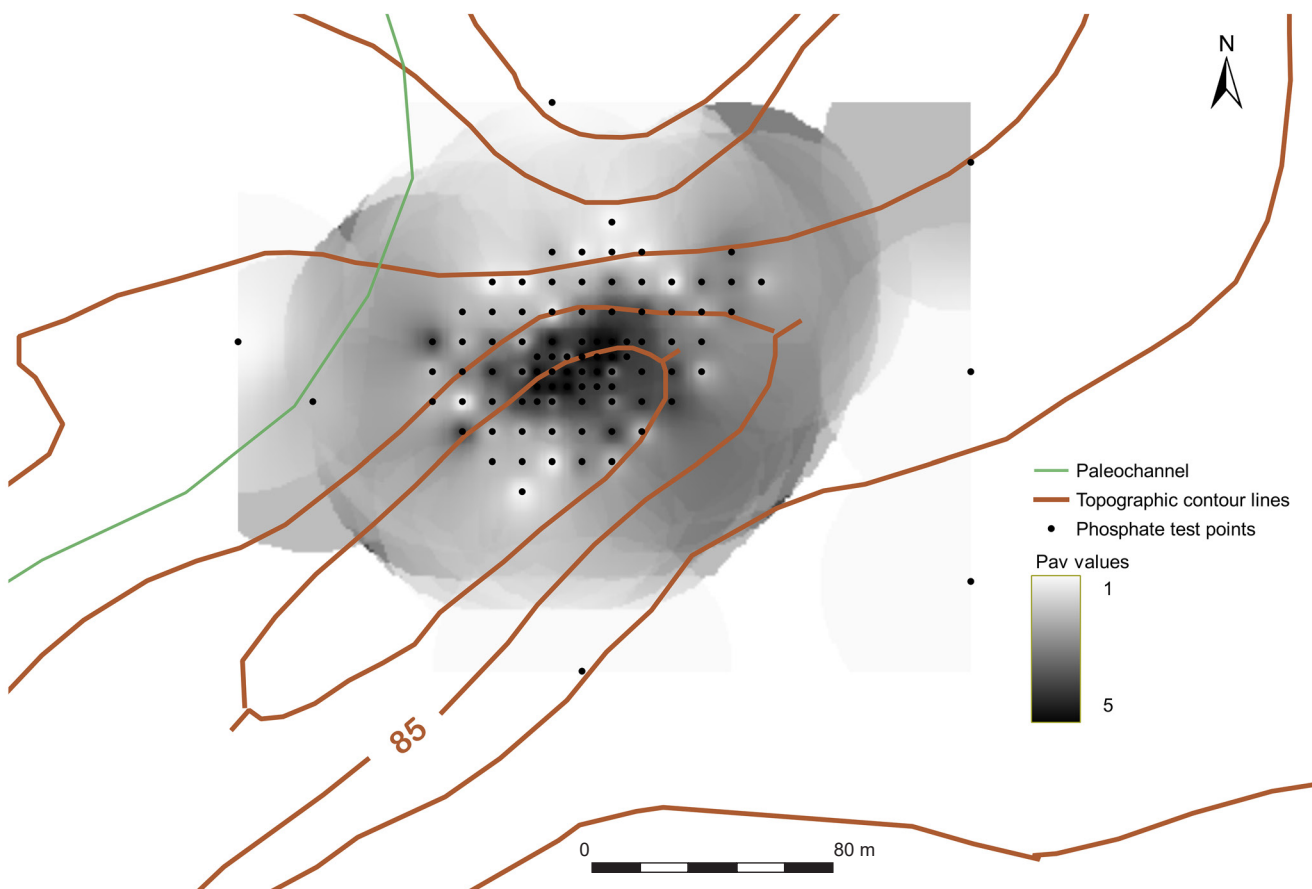


Figure 1. Distribution of Pav (available P) at the Early Copper Age Tiszapolgár settlement of Mezőberény-68 on an elevated loess ridge on the south side of a palaeochannel in the Körös Region of eastern Hungary.

of these areas was associated with a hearth and interpreted as indicating food preparation. The other two areas could not be interpreted with certainty, although knowledge of typical organization of medieval houses into domestic areas and stables led to a plausible conclusion that a large area of P enrichment on one end of the structure marked the stable (Schreg and Behrendt, 2011). In another example, soil P was successful in delimiting vertical stratigraphy and layers with anthropogenic inputs, even when those layers could not be distinguished visually (Ernée, 2005). Ongoing developments in soil P analysis in Central Europe indicate that the method not only remains useful, but has the potential to overcome some restrictions – for example, through the use of oxalate-extractable P (P-ox) as an alternative to phosphate fractionation (Weihrauch *et al.*, 2020).

In the Czech Republic phosphate analysis has a long history of use for analysing cemeteries and possible burials. Early research focused on using soil P to aid in identifying graves with poor or no bone preservation (Pelikán, 1954), including the identification of elevated P in cremation burials (Págo 1963, cited in Petřík *et al.*, 2012). More recently, phosphate analysis was combined with the Brongers method of identifying wood remains at a Bell Beaker site in Moravia (Hlavica *et al.*, 2011). At the Bronze Age Únětice site of Prague 9 – Miškovice site, phosphate anomalies in the form of burials aided in identifying grave-pits as a third burial form (along with inhumations and cenotaphs); cenotaphs lacked any P enrichment (Ernée and Majer, 2009). Similar results were obtained from Bell Beaker burials at several sites in the Znojmo district in Moravia (Petřík *et al.*, 2012). In that study, researchers also identified potential complications arising from various formation processes, including the influence of burial in or on wood, which apparently produced a lower signature than bodies placed directly on soil, and elevated P values associated with a secondary intrusion of organic sediments.

Phosphate testing is most effective when used as one component of an integrated multi-proxy approach to regional prospection and site investigations. In Hungary, soil P is used along with magnetic survey, magnetic susceptibility, and surface collection to identify vertical and horizontal limits of Neolithic and Copper Age settlements, and the extent of settlement and activity areas within micro-regions (Gyucha *et al.*, 2015; Parkinson *et al.*, 2010; Salisbury *et al.*, 2013; Sarris *et al.*, 2004). Phosphorus was used as one proxy in a multi-proxy reconstruction of functional spaces at a Late Bronze Age farmstead in Poland (Markiewicz and Rembisz-Lubiejewska, 2016).

3. Multi-element soil chemistry

Lutz (1951) recognized in the 1950s that elements other than P could be useful in archaeological contexts. Multi-element geochemistry provides more detailed information about what people did and where they did things, because human activities alter all the chemical and physical

properties of soils. Within archaeology, multi-element analyses have increasingly been applied for identifying different activity areas in connection with settlements, craft production, and marketplaces (Coronel *et al.*, 2015; Holliday *et al.*, 2010; Salisbury, 2017). The elements most often found to be associated with human settlements are P, K, Ca, Mn, Cu, Zn, Sr, Ba and Pb, but elements such as Mg, Rb, Cs and Th have also proven useful in some instances (Entwistle *et al.*, 2000; Oonk *et al.*, 2009a; Wilson *et al.*, 2008; 2009). Archaeological applications of multi-element soil chemistry have typically used ICP-MS; portable X-ray fluorescence is now being adopted and adapted (Coronel *et al.*, 2014; Gauss *et al.*, 2013; Šmejda *et al.*, 2017).

Much of what we know about the relationship between these elements and human activities comes from ethnographic studies, where human behaviour is observed and analyses conducted to see how this behaviour affects the soil chemistry. Ethnoarchaeological studies working with indigenous people in their households in small, rural villages have been done in Central America, including Oaxaca (Middleton and Price, 1996), several other areas in Mexico (Barba and Ortiz, 1992; Barba *et al.*, 1996), Guatemala (Fernández *et al.*, 2002; Terry *et al.*, 2004), and Honduras (Wells and Urban, 2002). These studies found connections between specific domestic activities, such as cooking, storage and crafting, and specific chemical elements, compounds, and soil properties. There are also a few examples from arctic and subarctic regions (Butler *et al.*, 2018; Knudson and Frink, 2010), but few from temperate Europe. This means that these studies occur on soil types that do not include loess, and focus on a specific set of input materials, some of which, like maize, did not exist in prehistoric and early historic Europe. Carbon (C) and Nitrogen (N) are also important elements to consider, especially in terms of depletion due to agriculture, or enrichment through fertilization, but these elements are too light to be analysed using either ICP-MS or XRF, so their investigation requires other instrumentation to be employed. A key point here is the gap in our knowledge base, which can only be closed by experimental and ethnoarchaeological research in Central Europe.

Multi-element work is almost exclusively restricted to excavation contexts, where we already have a potential archaeological interpretation and want confirmation, or when we try to interpret “empty” places (Terry *et al.*, 2015). Analysis of a paleosol from beneath a Bell Beaker (c. 2500–2200 BC) burial mound in Moravia indicated that the burial location had not been used for habitation or production activities (Hejzman *et al.*, 2013b). In part, identification of the paleosol was based on levels of lead and cadmium lower than surrounding soils. In rare occasions, multi-element chemistry has been used to interpret unexcavated areas. For example, Principal Components of multi-element data from Late Neolithic and Early Copper Age farmsteads in eastern Hungary were interpreted to delineate potential activity zones, and compared to identify similarities in the use of space (Salisbury, 2013; 2016).

Multi-element soil chemistry has also been used to determine archaeological context as a primary factor for the appearance of cropmarks. In a series of papers, Hejcman and colleagues demonstrated that the chemical composition of ancient pit fills, subsoils, surface soils and vegetation was directly enhanced by the presence of ancient human activities (Gojda and Hejcman, 2012; Hejcman *et al.*, 2011; Hejcman and Smrž, 2010; Hejcman *et al.*, 2013a). In particular, the analysis of ashed vegetation from assumed archaeological features (positive cropmarks) and “background” vegetation enabled a straightforward correlation between anthropogenic chemical enrichment and the effectiveness of aerial archaeology (Gojda and Hejcman, 2012).

3.1 Extraction processes

By comparing the relative concentrations and combinations of elements, as well as other soil components (*e.g.* pH, soil organic content, and magnetic susceptibility), activity patterns can be identified and examined (Pető *et al.*, 2015; Pető *et al.*, 2019; Salisbury, 2013). We use relative concentrations because many variables affect elemental levels in soils, and we are specifically looking for anthropogenic inputs (Wells *et al.*, 2000; Wells *et al.*, 2007). This focus on anthropogenic inputs has also resulted in broad disparities in analytical methods.

These disparities lead to a second knowledge gap, one that has been noted several times (Oonk *et al.*, 2009b; Pastor *et al.*, 2016; Wilson *et al.*, 2006). Despite advances in sample preparation and analytical methods, we still lack standardized protocols, or a fundamental agreement on how sediment characteristics and laboratory condition influence our extraction methods and subsequent results. In addition, meta-studies investigating the efficacy of methods and comparability of results are largely lacking (but see Lubos *et al.*, 2016). The type of extraction used will highly influence the results. Americanist archaeologists frequently rely on a weak-acid extraction that is intended to extract only the anthropogenic signature (Middleton, 2004; Salisbury, 2016; Wells, 2004). In the experience of this author, geologists are horrified by this approach, arguing that total extraction using strong acids at high temperature is the only acceptable method. Both can be made to sound like reasonable arguments, but each might be inappropriate for archaeology. Geoarchaeologists have developed alternatives that give quasi-total extraction. One example using HNO_3 is set out in Wilson (2008). Mehlich 3 extractant is widely used in the Czech Republic, and has been recommended as an international standard for archaeological soil chemistry (Hejcman *et al.*, 2013b).

Attempts at standardization are unlikely to be a perfect solution, because the extractions should be based partly on methodological consistency and comparability, but also on regional soils, environmental conditions, and the nature of the elements themselves (Pastor *et al.*, 2016; Wilson *et al.*, 2006). Therefore, reliability and comparability will be better served by consistent standardization in sample handling and preparation, documentation of methods and protocols, and presentation of results.

3.2 Portable XRF – a new state-of-the-art

Portable, handheld, energy dispersive X-ray Fluorescence spectroscopy (pXRF) is comparatively inexpensive, non-destructive, and enables rapid acquisition of large datasets, and therefore is rapidly being adopted for a range of archaeological applications (Holcomb and Karkanas, 2019; Michałowski *et al.*, 2020; Riebe, 2019; Vianello and Tykot, 2017). Technological innovations are solving many of the problems confronted by early adopters, such as the inability to measure P and other light elements, and the interference of silicon with P (Coronel *et al.*, 2014). Handheld XRFs are now being widely used for soil analyses, in Central Europe and elsewhere (Dreslerová *et al.*, 2020; Horák *et al.*, 2018; Lubos *et al.*, 2016; Šmejda *et al.*, 2017). In addition, pXRF returns total elemental composition, making it comparable to total and quasi-total chemical extractions.

At the fortified Early Bronze Age settlement of Fidvár in southwest Slovakia, pXRF analysis of samples from an Early Bronze Age house, the site centre, a potential metal workshop, and the fortification ditch indicated P enrichment in the ditch and low levels in the house. Calcium and strontium varied within house samples, and were again higher in the ditch. No geochemical evidence for metalworking activities were found (Gauss *et al.*, 2013). Using a similar methodology, 74 samples were analysed from a Neolithic Linear Pottery Culture house and associated ditch near Vrábce in Slovakia. Results indicated the need to consider post-depositional processes, in this case in bio-cycling in particular, for accurate interpretations (Dreibrodt *et al.*, 2017).

Medieval settlements have also received attention. At the abandoned medieval village of Lovětín near Třešť in western Moravia, general household waste was likely spread on agricultural plots, based on elevated levels of Mn, Sr, and K. Corresponding low levels of P were interpreted as P depletion due to ineffective fertilization; widespread use of manuring was not evident. Nevertheless, the authors cautioned that the detection limits of their instrument constrained measurements of P and Ca (Horák *et al.*, 2018).

Portable XRF has also been applied to cemetery research. In a Late Bronze Age and Early Iron Age example, pXRF was used to determine that urn cenotaphs – burial urns with no macroscopic bone remains inside – never contained bones (Pankowská *et al.*, 2018). Fill of urns without bones had lower levels of P, Ca, Mn, Zn, Pb and V when compared to samples from urns containing visible bone fragments.

Effective application of portable X-ray fluorescence analysis in the field, to establish multi-element chemical analyses as a standard approach in archaeological fieldwork, requires a workflow optimized for field conditions. The optimal *ex situ* methodology, wherein sediment samples are oven dried, milled to c. 20 microns, homogenized, and pressed into pellets or disks, clearly remove many factors that influence measurements, such as sunlight, soil moisture content, and measuring a single large particle in un-sieved, un-homogenized sediments. However, transportation, storage, and laboratory processing remove the advantages of a portable, affordable instrument that provides results in

one day. Conversely, *in situ* methods that take full advantage of portability and speed by directly measuring sediments at the surface (Šmejda *et al.*, 2017; Šmejda *et al.*, 2018) do not always produce reliable and replicable data on the elemental composition of a sampled context (Goff *et al.*, 2020). Nevertheless, in field analyses will be conducted, because of the obvious advantages. Therefore, establishing a protocol for in field testing, particularly for consistent sample preparation, is essential (Frahm *et al.*, 2016; Goff *et al.*, 2020). The best alternative for in field analysis is sampling, air-drying, sieving through 2 mm mesh, crushing and homogenizing by hand using ceramic mortars and pestles, and packing into plastic sample cups covered with thin polypropylene or mylar film (*cf.* Dreibrodt *et al.*, 2017; Goff *et al.*, 2020).

Unlike the situation of various extraction methods for ICP-OES/MS, serious attention has been given to the question of how pXRF results compare to ICP-based analyses (Frahm *et al.*, 2016; Gauss *et al.*, 2013; Lubos *et al.*, 2016). For the most part, results are comparable when total or quasi-total extraction methods are used. The primary purpose of archaeological soil chemistry is to establish patterns of activities rather than absolute elemental values. Therefore, the most important comparison is of the spatial patterning of element enrichment and depletion, which is less frequently reported. The study by Lubos and colleagues (2016) is an important exception to many critiques; comparison of several strong acid and weak acid extractions with pXRF indicates high correlation of results using weak HCL, strong HNO₃, Aqua Regia (all ICP-OES), and pXRF.

Moreover, the accuracy and reproducibility of measurements depend on instrument calibration, availability of appropriate material standards, and regular measurement of blanks (*e.g.* SiO₂ blanks). Three sets of calibrations for soil analysis are available from major pXRF models. Although each company uses different names for these, they can be grouped as an empirical mode (requiring known samples), fundamental parameters (FP), and Compton normalization. The latter two come preinstalled; recent models often include a combined fundamental-Compton mode. International soil and sediment geological standards (GBW 7411, NIST 2780, NCS 73308, TILL-4, and USGS SdAR-M2) might be problematic, particularly in areas of redeposited loess, such as the Great Hungarian Plain. For example, TILL-4 is a sample of till taken in New Brunswick, Canada; NIST 2780 is hard rock mine waste. Correction of reported pXRF data with local calibration samples can resolve these issues (Goff *et al.*, 2020), but local calibration samples must be developed for each region and geology.

4. Future directions

Following the third-science revolution (Kintigh *et al.*, 2014; Kristiansen, 2014), issues of mobility and migration, increasing complexity in social and settlement organization, human-environmental interactions, economic sustainability,

and cultural and environmental resilience are becoming increasingly relevant for archaeology. In spite of the unremarkable fact that most human activities generate measurable traces in sediments, plasters and other surfaces, these papers do not specifically mention the role of geoarchaeology. In addition to the continued development of multi-element sediment geochemistry, biogeochemistry is providing new insights into questions about mobility, domestication, land use, anthropogenic impacts, and socio-political interactions. This section will briefly examine some recent developments in these arenas, with contributions from geoarchaeology and soil science.

Biogeochemistry is a highly inter-disciplinary concentration on cycles of chemical elements and their isotopic ratios, and natural or anthropogenic organic compounds such as proteins, lipids, carbohydrates, and nucleic acids. Organic molecules from biological sources can be preserved in soils and sediments, and serve as markers of anthropogenic activity, although the preservation of these biomarkers is highly dependent on the pedological conditions (Bull *et al.*, 2000; Bull *et al.*, 2002; Killops and Killops, 2005). Soil biomarkers have been used, albeit sporadically, to aid reconstructions of palaeoenvironmental conditions, cultivation and manuring, and other human activities at multiple analytical scales (Bethell *et al.*, 1994; Hjulström and Isaksson, 2009; Prost *et al.*, 2017; Simpson *et al.*, 1999).

Faecal biomarkers, in particular 5β-stanol lipids, provide data on pastoral practices and land-use in France (Zocatelli *et al.*, 2017), animal husbandry and uses for dung in Anatolia (Portillo *et al.*, 2020), and plants as a significant component of Neanderthal diet in Spain (Sistiaga *et al.*, 2014). Faecal biomarkers can now be used to distinguish between different animal species (Harrault *et al.*, 2019; Prost *et al.*, 2017), significantly increasing their usefulness for research on early domestication and animal husbandry.

Lipid biomarkers from plants, in the form of plant sterols and *n*-alkanes from leaf waxes, are chemically inert, persist in sediments for thousands of years or more, and provide direct evidence of vegetation types (Patalano *et al.*, 2020). Sterols produce chemical signatures specific to different plant types, and are used to infer palaeovegetation changes, such as shifts from grasses to trees and shrubs, or lacustrine to terrestrial species (Schatz *et al.*, 2011; Schwark *et al.*, 2002; Zech *et al.*, 2010). Analyses of carbon and hydrogen isotopes in these compounds are also used to infer palaeoclimate variability (Patalano *et al.*, 2020; Schirrmacher *et al.*, 2019).

4.1 Isotope biogeochemistry

One widely recognized biogeochemical application in archaeology is the analysis of isotopic ratios of strontium, carbon, oxygen, nitrogen, sulphur and other elements in bones and teeth from humans and animals, and the relationship between these and depositional environments. These methods are now routinely used for reconstructing diet, climate, mobility, and environmental changes indicative of anthropogenic modifications to subsistence and habitation strategies (Balasse *et al.*, 2017; Chazin *et al.*, 2019; Demény

et al., 2019; Giblin and Yerkes, 2016; Makarewicz and Sealy, 2015). For example, stable strontium, oxygen, and carbon isotopes provided evidence for different subgroups of the massacred Neolithic community at Talheim (Bentley *et al.*, 2008). Strontium ratios from Late Neolithic and Copper Age human dental enamel indicate greater variability during the Copper Age on the Great Hungarian Plain, suggesting a wider geographical range of food acquisition (Giblin *et al.*, 2013).

Biochemistry of ancient bone, as in the examples above, typically includes a geological component from the depositional environment. An isotopic study from Czech La Tène cemeteries illustrates the problem of differentiating land-use practices and local geological variability from human mobility when interpreting strontium variability (Scheeres *et al.*, 2014). This highlights the need to map regional baseline isoscapes, or isotopic landscapes, from proxies appropriate for ancient conditions (Bataille *et al.*, 2020; Snoeck *et al.*, 2020).

Sediment biogeochemistry has unrealized potential for reconstructing past human activities and ecosystems (D'Anjou *et al.*, 2012; Vranová *et al.*, 2015; Vranová *et al.*, 2012). A study of $\delta^{15}\text{N}$ ratios of samples taken from two long-standing agricultural experiments on the impact of manuring on agricultural yields demonstrates that manuring significantly raises nitrogen values in both grains and chaff (Bogaard *et al.*, 2007). Sodium, chlorine, nitrate, and nitrate-nitrogen isotope values from waste layers at Aşıklı Höyük, a Neolithic tell in central Turkey, were used to calculate increasing numbers of caprines (Abell *et al.*, 2019).

4.2 Sedimentary ancient DNA

Ancient human and environmental DNA preserved in palaeosoils can provide evidence for human presence, species identification, and changes in ecological diversity. Sedimentary aDNA (or *seDaDNA*) can contribute to multi-proxy interpretations, but are especially useful when physical remains are not preserved (Brunson and Reich, 2019; Epp *et al.*, 2019; Thomsen and Willerslev, 2015). For example, a study of lake sediments from Poland contrasted human-specific bacterial DNA, a marker of human faecal material, and pollen counts; results revealed direct correlation of human presence and vegetation changes (Madeja, 2015). Although most recent research has been conducted on lake sediment samples from cores (Giguët-Covex *et al.*, 2014; Madeja, 2015; Parducci *et al.*, 2017), sediment samples can be taken directly from secure archaeological contexts (Hebsgaard *et al.*, 2009; Nejman *et al.*, 2020; Slon *et al.*, 2017).

4.3 Proteomics from sediments

A relatively new area of research in archaeology is proteomics. Proteomics involves the extraction, sequencing, and analysis of proteins that form proteomes, and the identification of species based on the weight of specific proteins; ancient proteomics, or palaeoproteomics, involves the extraction and analysis of proteins from archaeological remains. The

application of proteomic methods in archaeology is most developed in the analysis of human and animal bones (Brown *et al.*, 2016; Lanigan *et al.*, 2020), dental calculus (Charlton *et al.*, 2019), and ceramics (Shevchenko *et al.*, 2018). Archaeological and environmental proteomes can also be extracted from soil; preliminary results were promising, but this method is in the early stages of development (Oonk *et al.*, 2012).

5. Summary: Bringing it all together with multi-proxy approaches

As we move towards the middle of the 21st century, archaeology departments need to become more interdisciplinary and more attuned to the information stored in the sediment archive: ancient human DNA, other ancient DNA, ancient fats, carbohydrates, and stomach acids, the microbial environment, and changing soil conditions. Bio-geoarchaeology can address new questions, or bring new methods to acquire data that was previously unavailable. Accessing these data requires an acceptance that anthropogenic sediments are archaeological remains.

A couple of points concerning the future development of archaeological soil chemistry must be considered. One is that the latest analytical methods cross disciplinary boundaries and push the current limits of archaeological soil chemistry. Geoarchaeologists will need to integrate knowledge of these methods into their toolkit, without losing their existing expertise. Furthermore, existing methods and protocols may require modification for archaeological contexts, to accommodate the effects of formation processes and the vagaries of human activities. Potential rewards make these efforts worthwhile. Converging lines of evidence from multi-element soil chemistry, magnetic susceptibility, and soil biomarkers will provide greater interpretive power for settlement and activity areas research, whilst also producing complementary evidence for zooarchaeology and other environmental analyses (*e.g.* Dreslerová *et al.*, 2020; Lauer *et al.*, 2014).

This leads to a second point, which is that our older methods of sampling for inorganic soil chemistry are inadequate for current analytical capabilities. One immediate methodological aim in archaeological soil chemistry should be to establish new and standardized sampling and storage methods for biomarkers, in particular those collected directly from archaeological contexts.

The need for site prospection employing primarily soil phosphate analysis is likely to remain, particularly in woodlands or other conditions that limit geophysics and surface collection. Unfortunately, this approach is rarely used today, despite its obvious application to filling gaps in our survey areas. Portable XRF provides a tool for in field geochemical analysis of geological samples, including soils, that can be done as a prospection method or as biogeochemistry samples are collected. Further, pXRF fits within the budget of most research programmes. In

these respects, pXRF is both the new state-of-the-art and a method for the future. However, comparisons between institutes or field projects should be undertaken to assess results using standard calibrations, and determine whether correction factors can be developed for general use or need to be established for each individual device. This represents a second essential aim for geoarchaeology. In addition, we need an effective universal protocol for collecting and processing sediment samples in the field, so as not to lose the advantages of speed and portability.

Geoarchaeologists need to be able to collect appropriate samples, and aid in data interpretation, allowing laboratory scientists to develop analytical protocols for sediment bimolecular studies. The combination of biomarkers with geoarchaeological methods such as soil phosphates, magnetic susceptibility, micro-remains, and thin-section analysis will open new frontiers in our understanding of the human past.

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