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Research papers

Chemical soil surveys at the Bremer Site (Dakota county, Minnesota, USA): Measuring phosphorous content of sediment by portable XRF and ICP-OES

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A B S T R A C T

The Bremer Site lies along the shores of Spring Lake in southeastern Minnesota, and excavations in the 1950s uncovered evidence of Woodland and Mississippian occupation phases. In 2011, a new program of systematic survey and excavation began to better understand cultural patterning and diachronic changes at the Bremer Site. The investigations came to include microarcheaological methods, including sediment micromorphology and soil chemistry. No element has received more archaeological attention than P, which can reflect human and animal waste, organic refuse, burials, and ash. There has been interest in integrating soil chemistry into the workflow of fieldwork and in the potential of portable analytical instruments to yield data within the timeframe of an excavation season. The last few years have seen the rise and proliferation of portable XRF (pXRF) instruments in archaeological studies. The newest generation of pXRF instruments is able to quantify P at low concentrations, and our focus here is developing effective methods to do so in archaeological soils and sediments. Using sediments from the Bremer Site, we evaluate two preparation techniques in order to find which one best balances analytical quality and preparation time. To analyze as many specimens as possible during an excavation season, it is desirable to identify adequate preparation methods as well as the smallest number of analyses needed to attain reasonable confidence levels. Regarding repeatability, we show that, to attain P values with a standard error better than 10% or 20% of the mean, one or two analyses per specimen are sufficient in a majority of cases. Regarding reproducibility, we compare the pXRF data to two independent ICP-OES datasets. Ultimately, these tests aid in establishing methods that enable archaeologically significant pXRF analyses of soil P concentrations, even when working far from an analytical laboratory.

1. Introduction

Along the shores of Spring Lake in southeastern Minnesota, a variety of archaeological sites, including open-air habitations, mounds, and caves, lie on terraces of the Mississippi River, reflecting Early Woodland through Oneota occupations. One of them is the Bremer Site (21DK06; Fig. 1a), prominently situated over an area that, prior to dam construction in the 1930s, was a wetland with diverse faunal and floral resources. Based on excavations led by the Science Museum of Minnesota during the 1950s, this multi-component site was inhabited for at least a millennium, spanning from ~100 CE (Middle Woodland) to ~1400 CE (Oneota). Paleoindian (11,200–10,500 BCE) and Archaic components

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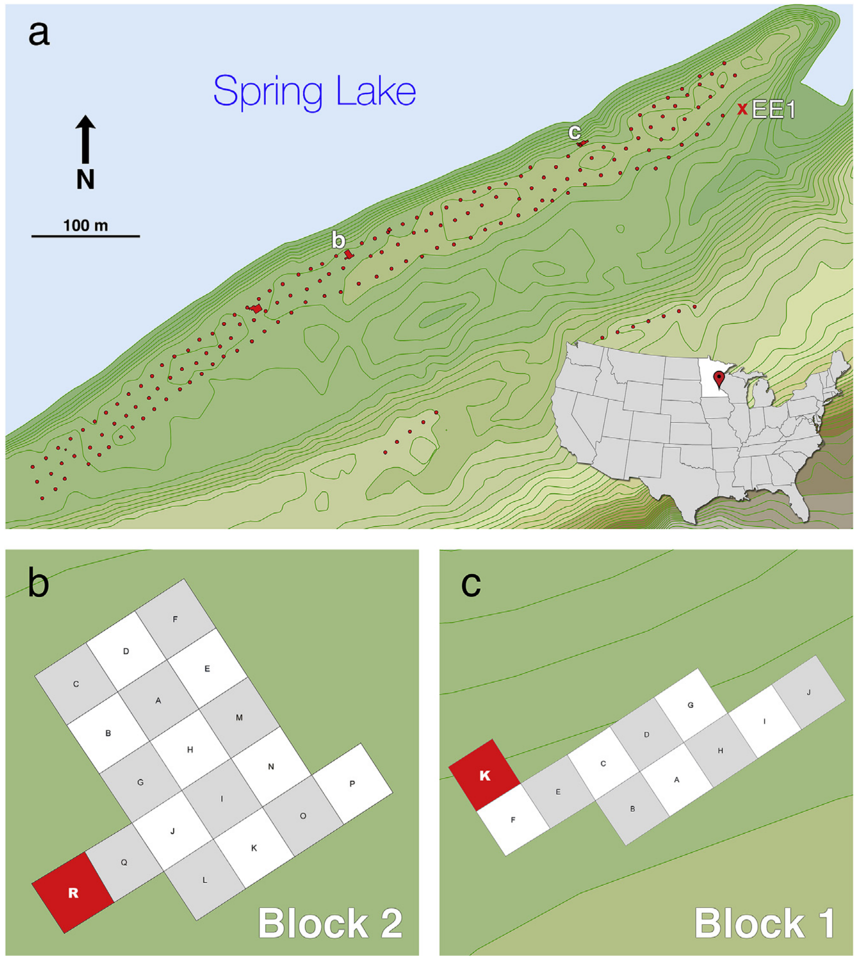

Fig. 1. Locations and layout of (a) the Bremer Site in southeastern Minnesota along the shore of Spring Lake and the 2011–2014 excavations (red) of the University of Minnesota and Science Museum of Minnesota, (b) Unit R (red square) of Excavation Block 2, and (c) Unit K (red square) of Excavation Block 1. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

(10,500–500 BCE) also occur. Five decades later, in 2011, the Science Museum partnered with the University of Minnesota to start a new program of systematic survey, excavation, and data collection to better understand cultural patterning and diachronic changes at the Bremer Site, including settlement size, resource use, occupation duration and seasonality, and the organization of space. The new excavations revealed impediments to addressing such research issues, including the lack of faunal and macrobotanical preservation (except decomposed charcoal) and poorly preserved features. Thus, investigations of the site came to include microarcheaological methods (*sensu* Weiner, 2010), and excavations during the 2014 season focused on sampling for a variety of soil and sediment micromorphological and chemical studies.

Human activities enrich, deplete, or redistribute a variety of elements in soils, and patterns within these chemical traces can be detected today and reveal behaviors in the past (e.g., Cook and Heizer, 1965; Woods, 1982; Middleton and Price, 1996; Scudder, 1996; Entwistle et al., 1998, 2000; Wells, 2004). The advantages of elemental signals in archaeological soils include the capacity to recognize activity areas, to examine the use of space within a site, and to observe other spatially expressed behaviors (e.g., Middleton and Price, 1996; Middleton, 2004; Oonk et al., 2009; Abrahams et al., 2010; Vyncke et al., 2011). No element has received more attention in such studies as an archaeologically significant indicator than phosphorous (P; see Holliday and Gartner, 2007: Tables 1 and 2). Anthropogenic sources of P include human and animal waste, organic refuse, burials, and ash from fires (Provan, 1971; Proudfoot, 1976; Eidt, 1984). Bethell and Máté (1989:9) summarize the relationships between humans and P in soils:

> Human activities can strongly redistribute P in soils. Plants take up P from the soil. They can be eaten by animals or harvested. The animals themselves can be moved or 'harvested'; they can be enfolded, concentrating P in a particular area. Dung residues can be collected and used as manures … on the other hand, they may be used as a fuel, as a walling material, or ignored … As part of the produce of an economic system, P is very mobile; its importance lies in the strong fixative powers of the soil. When P enters the soil system, it is relatively immobile compared to other elements concentrated by the activities of humans.

In short, P is a stable, sensitive, and ubiquitous indicator of human activities. Thus, the literature on P in archaeological soils is extensive. Holliday and Gartner's (2007) useful review summarizes the sources of anthropogenic P, the chemistry and dynamics of P in soils, the history of archaeological P studies, the techniques for measuring P in soils, and the applications of such analyses.

There has been interest in incorporating soil chemistry into the workflow of archaeological fieldwork, sometimes with the goal of making it standard practice for surveys and excavations (e.g.,