ASSESSING THE USE OF SOIL PHOSPHATE ANALYSIS AS AN ARCHAEOLOGICAL PROSPECTION TOOL AT THE AMES SITE (40FY7), FAYETTE COUNTY TENNESSEE

by

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ii

Abstract

Archaeologists are increasingly integrating multiple survey techniques to reduce errors and biases in attempts to locate archaeological deposits and to gain a better understanding of people's use of space. This study assesses the utility of soil phosphate analysis as an archaeological prospecting tool in western Tennessee through soil analyses at the Ames site (40FY7) in Fayette County, Tennessee. The spatial distribution of available phosphorus and percent loss on ignition are compared to mapped magnetometry data over two areas with confirmed archaeological deposits. The loss on ignition results did not visually correspond with areas of archaeological activity; however, the spatial distribution of available phosphorus corresponds with archaeological activity at Ames. Further post hoc statistical analyses indicate significant differences in phosphorus values between areas with archaeological activity and areas without archaeological activity. This study demonstrates the utility of soil phosphate analysis as a tool for locating archaeological deposits at the Ames site.

Table of Contents

Cha	Chapter	
List	of Tables	vi
List	of Figures	vii
1.	Introduction	1
	Archaeological Survey	2
	Trends in Tillage Practices	4
	Geoarchaeological Survey Methods	5
	Remote Sensing	6
	Geophysical Methods	6
	Geochemical Methods	9
	Research Questions	10
	Hypotheses	11
2.	Environmental and Cultural Background	14
	Environmental Setting	14
	Soils of Fayette County, Tennessee	15
	Cultural Background	16
	Previous Research at Ames	20
3.	Soil Phosphate Analysis in Archaeology	22
	Extraction Techniques	24
	Total vs. Available Phosphorus	25
	Measurement Techniques	26
4.	Methods and Analysis	28
	Magnetometry Surveys	28
	Soil Sampling	30
	Soil Sample Processing	32
	Soil Phosphate Analysis	34
	Loss on Ignition	36
	Mapping Results	37
5.	Results and Discussion	39
	Physical Characteristics	39
	Soil Phosphorus Spatial Distribution	42
	Phosphorus Results and Prior Research	48
	Loss on Ignition	56
	Statistical Analyses	57

6.	Conclusions and Future Directions	63
Refere	ences Cited	68
Appen	ıdix	82

List of Tables

Table		Page
1.	Factors Affecting Discovery Probabilities from Schiffer et al. 1978:4-10.	3
2.	Cultural and Natural Processes Responsible for Magnetic Variations from Kvamme 2006:214-221.	8
3.	Descriptive Statistics and Normality Test Results.	58
4.	Descriptive Statistics for Soil Phosphorus Concentrations (mg/kg).	59
5.	Descriptive Statistics for LOI550 (% weight loss).	60
6.	Descriptive Statistics by Location within Study Area.	60
7.	Descriptive Statistics for Phosphorus Concentrations Comparing Archaeological and Non-Archaeological Area Membership.	61
8.	Results of DSCF Test.	62

List of Figures

Figu	ire	Page
1.	Tillage trends for major crops in Tennessee from 2007-2017, data from USDA, NASS 2012, 2014, 2016, 2018.	5
2.	Ames site (40FY7) in regional archaeological and ecological context.	15
3.	Ames community plan based on magnetometry, shovel tests, and ground-truthing excavations (Mickelson 2019:Figure 8). Red points indicate location of soil samples collected for this study.	21
4.	Red box indicates area surveyed in March 2018 using a fluxgate gradiometer. The surveyed area is located in a field east of the Ames town which was enclosed by the labeled palisade wall.	29
5.	Results from the March 2018 magnetometry survey. Green box indicates a magnetic signature of a Mississippian-period structure excavated in May 2019. The red boxes indicate two additional potential archaeological magnetic signatures.	30
6.	Sampling design for this study compared to artifacts collected during surface surveys, mound locations in northwest corner, and magnetometry survey area. Locations of soil samples collected for present study are indicated by black points and artifacts are indicated by green x's.	32
7.	Standard curve based on absorbance values of known phosphate concentrations.	35
8.	Munsell color distribution of soil samples.	40
9.	Distribution of ped diameter range.	41
10.	Distribution of estimated organic debris in samples.	42
11.	Phosphorus concentrations compared to magnetometry data. The higher phosphorus concentrations are located within the boundary of the Ames town represented by the palisade wall visible in the magnetometry data.	43

12.	Soil phosphorus distribution compared to artifacts collected during surface surveys. The majority of surface artifacts are located within the palisade near the center of the Mississippian settlement between AD 1050-1290. The eastern field has fewer surface artifacts because the area was not systematically surveyed and is now a no-till field.	44
13.	Soil phosphorus distribution compared to archaeological remains confirmed by "positive" ground-truthing excavations (outlined in green).	45
14.	Phosphorus distribution with different classification methods and 7 classes. Natural breaks (Jenks) (top left); quantile (top right); manual interval (bottom right); geometric interval (bottom left).	46
15.	Phosphorus distribution with different classification methods and 10 classes. Natural breaks (Jenks) (top left); quantile (top right); manual interval (bottom right); geometric interval (bottom left).	47
16.	Areas of relatively higher phosphorus concentrations (mg/kg) designated as Area-A, Area-B, and Area-C.	48
17.	Phosphorus distribution highlighting Area-B compared to ground- truthing excavation units. Again, no samples were collected from within excavation unit boundaries.	50
18.	Phosphorus distribution compared to magnetometry data of the eastern field. The labeled signature was excavated in May 2019. There has been no further testing of the additional signatures outlined by red boxes.	52
19.	Eight 20 m grid squares surveyed in April 2019 indicated by red box.	53
20.	Magnetometry data processed as one composite compared to phosphorus distribution. Previously discussed signatures are enclosed in red boxes. This area appears to be highly susceptible to lightning strikes as observed in the magnetometry data.	54
21.	Magnetic signatures characteristic of Late Prehistoric habitation. Red boxes indicate signatures previously discussed in the text. Green boxes indicate signatures revealed after the prediction was made based on phosphorus distribution.	55

- 22. Magnetometry data from a field approximately 5 miles southeast of the study area (left) and the April 2019 survey of the eastern eight 20 m grid squares (right). Red arrows are pointing to the northeast corner of magnetic signatures of similar size and shape. The signatures in the right image are highlighted by green boxes in Figure 21.
- 23. Side-by-side comparison of LOI550 (left) and phosphorus concentrations (right) highlighting the inverse relationship between the two variables.

1. Introduction

Mississippian societies are considered the first agricultural societies in eastern North America, occupying much of the Southeast and Midwest United States from AD 1000-1600. These early farming societies required land suitable for agricultural production, so it is no surprise to find that many Mississippian archaeological sites are located on modern agricultural fields. Tillage practices of modern farmers aid archaeological surveys intended to locate archaeological deposits; however, there has been a recent shift to no-till practices in response to increasing soil erosion. Consequently, surface survey techniques commonly utilized to locate archaeological deposits are no longer tenable. Therefore, alternative methods of archaeological prospection must be utilized or developed. Two approaches to improve the probability of detecting archaeological deposits are integrating multiple survey techniques and adopting survey methods and instrumentation from the field of Earth sciences.

The purpose of this research is to assess the utility of soil phosphate analysis for detecting archaeological deposits in western Tennessee by conducting soil surveys at the Ames site (40FY7). The Ames site is an Early-Middle Mississippian town dated to approximately AD 1050 – 1290 (Mickelson 2019). Due to the extremely low artifact densities on the ground surface, the settlement model of Ames was initially proposed as a vacant center, where people dispersed in hamlets and farmsteads across the landscape only occupy a mound center for specific occasions (Mainfort 1992:204; Mickelson 2008; Peterson 1979). This hypothesis was refuted in 2011 after additional surveys of surface artifact collection, shovel-test pits, and magnetometry revealed subsurface evidence of a palisade wall and remains of residential areas (Goddard 2011; Mickelson and Goddard 2011). Issues concerned with the probability of discovery are well established at Ames and other Mississippian sites in the west Tennessee

interior uplands and geophysical surveys were shown to be an effective tool in locating archaeological deposits (Goddard 2011; Mickelson and Goddard 2011). This study sought to test if the same can be said for geochemical surveys, specifically soil phosphate and loss on ignition (LOI).

The remainder of this chapter provides an overview of how survey methods from Earth sciences are integrated into archaeological prospection surveys to reduce problems concerned with visibility and obtrusiveness (see Table 1). First, a brief description of archaeological survey is provided. Factors influencing the probability of discovery of archaeological deposits (i.e., the likelihood that archaeological deposits are revealed) are explained to demonstrate awareness of these issues among the archaeological community. Following the discussion of influencing factors is a brief description of current trends in agricultural practices showing the relationship between such trends and the influencing factors in archaeological surveys. Once the relationship between influencing factors and current agricultural trends is established, a review of the three major types of Earth sciences survey methods are discussed to demonstrate their role in increasing the discovery probability in archaeological prospection surveys. Finally, the research questions and hypotheses are discussed.

Archaeological Survey

Archaeological survey is one of the primary tools utilized for recovering archaeological data (Banning 2002; Dancey 1981). Schiffer and colleagues (1978:2) define archaeological survey as "the application of a set of techniques for varying the discovery probabilities of archaeological materials in order to estimate parameters of the regional archaeological record." In the decades since Schiffer and colleague's, as well as other publications (e.g., Ammerman 1981; McManamon 1984; Plog et al. 1978; Schiffer et al. 1978; Shott 1987), archaeological

survey has become more conceptually refined by the intended goal of the survey (Banning 2002; Dunnell and Dancey 1983). Prospection surveys are performed with the goal of discovering particular kinds of archaeological remains (Banning 2002:133). Prospection surveys are differentiated from statistical surveys, which are employed in parameter estimation, and from what Banning (2002:28) refers to as "structural" surveys, which are employed to examine the spatial distribution of particular archaeological remains.

Archaeologists must take under consideration many factors that have the potential to impact the probability (i.e., likelihood) of detecting archaeological remains from a survey utilizing a given technique. Five commonly discussed factors are presented in Table 1, and include visibility, abundance, obtrusiveness, clustering, and accessibility (Banning 2002; Schiffer et al. 1978:4). The two factors most related to the probability of detecting archaeological remains are visibility and obtrusiveness (Banning 2002; McManamon 1984; Schiffer et al. 1978).

Factor	Definition
Abundance	The frequency or prevalence of a site or artifact type in the study area.
Accessibility	The constraints on observer mobility or effort to reach a particular place.
Clustering	The degree to which archaeological materials are spatially aggregated.
Obtrusiveness	The probability that particular archaeological materials can be discovered by a specific technique.
Visibility	The extent to which an observer can detect the presence of archaeological materials at or below a given place.

Table 1. Factors Affecting Discovery Probabilities from Schiffer et al. 1978:4-10.

Trends in Tillage Practices

Tillage is defined as the mechanical manipulation of soil for seedbed preparation on agricultural fields (Lal et al. 2007; Reicosky and Allmaras 2003). The mixing of the soil brings artifacts to the surface providing greater visibility for a surveyor; however, agricultural practices in the United States are changing rapidly due to consequences of climate change (e.g., drought) and technological advances. No-till fields are increasing in acreage as a response to the need for soil conservation (Figure 1). In Tennessee, the percentage of total acres planted with major crops (i.e., corn, cotton, soybeans, winter wheat) in no-till fields increased by 11.6% between 2007 and 2017 (United States Department of Agriculture, National Agricultural Statistic Service [USDA, NASS] 2012, 2014, 2016, 2018). During this same period, the percentage of total acres planted with major crops using conventional tillage decreased by 60%, accounting for only 4.4% of total acres planted in 2017. In 2017, no-till fields accounted for 78% of total acres planted with majors crops in Tennessee, increasing to an estimated 80% in 2018 (USDA, NASS 2018).



Figure 1. Tillage trends for major crops in Tennessee from 2007 - 2017, data from USDA, NASS 2012, 2014, 2016, 2018.

Geoarchaeological Survey Methods

Human activity greatly influences the natural biological, chemical, and mechanical processes of soil. Soils that have been chemically or physically altered by human activity are called anthrosols (Wells et al. 2000:449). Survey methods from the field of Earth sciences are increasingly utilized in archaeological prospection because they reduce issues concerned with visibility and obtrusiveness while detecting and measuring natural and cultural land modifications. The following provides a brief review of each of the major types of survey methods from the field of Earth sciences utilized in archaeological prospection, including remote sensing, geophysical survey methods, and geochemical survey methods.

Remote Sensing

Remote sensing has proved to be a powerful tool in advancing archaeological research by providing useful data from areas inaccessible to the researcher. Remote sensing is defined by Kvamme (2008:65) as "techniques that acquire information about a subject through indirect means." Remote sensing employs two different types of instruments. Active remote sensing instruments measure responses of specific properties to energy transmitted through the surface and subsurface of the Earth and passive remote sensing instruments measure natural variations in properties detectable on the surface (Kvamme 2008:65). Remote sensing techniques coupled with a Geographic Information System (GIS) are particularly beneficial in archaeological surveys as the surveyor acquires as much information as possible about the study area, such as topography or vegetation, leading to a more informed survey design.

Geophysical Methods

Geophysical methods measure variations in the physical properties of subsurface soils and rocks otherwise invisible to the surveyor (Kvamme 2003; Lockhart and Green 2006; Weymouth 1986). The first geophysical survey at an archaeological site was an electrical resistivity survey performed by Richard Atkinson in 1946 (Clark 1990:11). About a decade later, Martin Aitken and Edward Hall tested their prototype magnetometer with success (Clark 1990:17). The foundation for the field of archaeogeophysics was in place by 1960 and since the 1980s, significant technological advances in instrumentation and the development of Geographic Information Systems has led to the proliferation of the use of geophysical methods for locating and understanding archaeological sites today (Clark 1990; Kvamme 2003:436). Two geophysical techniques commonly used in archaeology include ground-penetrating radar (GPR) and magnetometry; however, only magnetometry was used in this study.

Magnetometry. Magnetometry is a passive remote sensing technique which measures variations of the strength of the Earth's magnetic field (Dalan 2006; Kvamme 2006). Magnetometers measure the strength of both remanent magnetism (i.e., magnetism that exists in the absence of a magnetic field) and induced magnetism (i.e., magnetism that exists only in the presence of a magnetic field) (Dalan 2006; Kvamme 2006:207-208). Magnetometry data are collected relatively quickly with high spatial resolution, resulting in plan-view maps of the spatial variation of magnetism in subsurface deposits.

The strength of the magnetic field is measured in nanoteslas (nT). Although the strength of the Earth's magnetic field ranges from 30,000 nT at the magnetic equator to 60,000 nT at the magnetic poles, the strength of magnetism of interest at prehistoric archaeological sites in west Tennessee ranges from \pm 5 nT (Kvamme 2006:208-209). Magnetic variations at archaeological sites are caused by natural and cultural processes. Examples of natural processes with the potential to cause magnetic variations include natural soil changes, animal burrows, and rising bedrock (Kvamme 2008). Seven cultural processes and three natural processes responsible for magnetic variations, discussed at length by Kvamme (2006:214-221), are listed in Table 2.

Cultural Processes	Natural Processes
Human occupation exacerbate magnetic enrichment of surface soils.	Differences in magnetic susceptibilities exist between various materials, deposits, and soils.
People make constructions and artifacts composed of fired materials.	Topsoil becomes magnetically enriched due to physical and chemical processes.
People create fires.	Firing increases soil magnetism.
Human constructions accumulate topsoil.	
Human constructions remove topsoil.	
People import stone and other materials for construction.	
People make iron artifacts.	

Table 2. Cultural and Natural Processes Responsible for Magnetic Variations from
Kvamme 2006:214-221.

Many behaviors involved with living in a particular area for some time often leads to the creation of geometric features across the landscape (e.g., Mississippian period rectangular residential structures). Magnetometry surveys are particularly productive because the interpretive approach is pattern recognition of contrasts, highlighting archaeological features when the features are significantly different from the surrounding soil matrix (Kvamme 2008:66). Interpreting the results of magnetometry surveys requires the researcher to be informed about the potential archaeological features encountered for the particular survey area (e.g., size of different features, potential settlement layout) as well as the principles concerning how the magnetometer sensors respond to various archaeological deposits (Kvamme 2008:67).

Geochemical Methods

Geochemistry is broadly defined as "the study of the chemical composition of the Earth and other planets, chemical processes, cycles, reactions that govern the composition of rocks and soils, and temporal and spatial changes in these controlling factors" (Hannigan 2007:1). Geochemical methods commonly employed in archaeology include radioisotope dating techniques, multi-element analysis, and loss on ignition. Archaeologists employ various geochemical methods to answer different questions concerned with human behavior and artifacts. Examples of different applications of geochemical methods in archaeology include provenance studies in which multi-element analyses determine the sources of raw material (e.g., obsidian), organic residue analyses in which multi-element analyses determine the foods contained in ceramic vessels, and activity area studies in which multi-element analyses determine various signatures of different types of behavior through systematic soil surveys (Cook et al. 2006; Glascock et al. 1998; McGovern and Hall 2016).

Loss on Ignition. Loss on ignition (LOI) is a method for estimating the organic and carbonate content in sediments (Ball 1964; Dean 1974; Heiri et al. 2001:101). Organic matter is oxidized to ash and carbon dioxide when heated to 550°C. Carbon dioxide is then evolved from carbonate, leaving only oxide when heated at 1000°C (Heiri et al. 2001:101). Weighing a sample before and after heating provides the information to determine the percent loss on ignition. The application of this method is increasing in archaeology due to low costs and minimal direct supervision necessary (Entwistle et al. 1998, 2000). The decision to include LOI in this study was to examine the interpretive relationship between soil phosphate and the enrichment of organic material. The results of LOI compared to those of soil phosphate analysis

can assist in determining whether low soil phosphorus concentrations are the result of low organic material or a different chemical interaction.

Research Questions

The west Tennessee interior uplands are subjected to at least two conditions that negatively impact the probability of discovery for archaeological surveys. The first condition is acidic soil, which adversely impacts the preservation of artifacts and organic material. The second condition is the area's geology, which consists of few naturally occurring lithic sources in the form of rock outcrops or cobbles. Moreover, the increasing trend toward no-till agricultural fields means that surface survey techniques in such circumstances are not viable. With such trends in agricultural practices leading to greater probability one will encounter issues concerned with visibility and obtrusiveness, and in turn, reducing the probability of discovering particular archaeological remains, the following question drives this study. Are there alternative survey techniques for locating archaeological deposits on agricultural fields in western Tennessee and reduce the impact of issues concerned with visibility and obtrusiveness?

Although soil phosphate analysis has been established as a successful prospection tool in archaeology, the utility of the specific method chosen for a given survey cannot necessarily be generalized to areas with different soil conditions. For example, a soil extraction method used in alkaline soils may not necessarily produce the same results if extracting from acidic soils (Crowther 1997; Eidt 1973; Holliday and Gartner 2007; Middleton 2004; Sjoberg 1976). To assess the utility of soil phosphate analysis as an archaeological prospection tool, one must test a specific method of extraction and measurement on local soils. For this reason, the following question is important in assessing the technique. Can the soil phosphate method of Mehlich-2

extraction measured by molybdate colorimetry detect archaeological deposits within the soils of the west Tennessee interior uplands?

Hypotheses

With consideration to the two questions driving this study, three hypotheses have been formulated. The null hypothesis (H₀) is there is no relationship between archaeological deposits and the soil matrix within which the deposits are located. The null hypothesis would be supported if there is no discernable spatial patterning of phosphorus and LOI. The first hypothesis (H₁) is that archaeological deposits in the west Tennessee interior uplands can be identified through the systematic mapping of soil phosphorus concentrations across the landscape. The second hypothesis (H₂) is that archaeological deposits in the west Tennessee interior uplands can be identified through the systematic mapping of LOI results across the landscape. The third hypothesis (H₃) is that soil phosphate analysis alone can locate archaeological deposits in the west Tennessee interior uplands.

Hypothesis H_1 is evaluated based on the spatial relationship of phosphorus concentrations and magnetic signatures observed through magnetometry surveys. The phosphorus concentrations are compared to results of magnetometry surveys due to the high accuracy and spatial precision of magnetometry surveys. Kvamme (2006:205) argues magnetometry to be "one of the most productive prospecting methods in archaeology." Magnetometry has been established as a highly accurate prospecting tool at Ames, therefore, assessing the utilization of an additional technique requires the comparison for considering future use of the additional technique. Supporting evidence for H_1 is marked differences in phosphorus concentrations in areas near documented magnetic signatures characteristic of Mississippian structures as they are very obtrusive in magnetometry surveys. H_1 is rejected if there is no observable spatial

patterning of phosphorus concentrations in proximity to known archaeological deposits detected through magnetometry. Evidence used to evaluate H_2 is the same as H_1 but comparing the magnetometry data to LOI results.

Hypothesis H_3 is evaluated based on the relationships between the variables examined. The evaluation of H_3 is dependent on the support or rejection of H_1 . The LOI results are included in the evaluation of H_3 only if H_2 is supported. Supporting evidence is in the form of accurate predictions for the presence of magnetic signatures from magnetometry surveys completed after soil phosphorus concentrations are known, as well as a correlation between the spatial distribution of phosphorus concentrations, LOI results given H_2 is supported, and magnetic intensity discussed in the analysis section. H_3 is rejected if blind predictions are inaccurate.

It is important to emphasize that correlations between soil phosphorus concentrations, organic matter, and magnetic signals are not necessary for evaluating the validity of soil phosphate analysis as a technique because the results of the three techniques may be revealing signatures produced from different human behaviors. Correlations are, however, necessary to support hypothesis H₃ because the question at-hand is if soil phosphate analysis could identify archaeological deposits without other survey methods. For this to be supported, the spatial distribution of phosphorus concentrations must reveal at least some signatures of behaviors revealed by alternative geophysical and geochemical survey techniques. Additionally, since the goal of this study is to assess the technique as an archaeological prospection tool, the major goal is to locate archaeological deposits, not to investigate settlement structure or activity areas.

The following paragraph provides an outline for the remainder of the thesis. Chapter 2 provides the environmental and cultural background, including the ecological setting and local

soils of west Tennessee as well as a review of Mississippian culture. Since the goal of this study is to assess the use of soil phosphate analysis, Chapter 3 provides the background information necessary for understanding the use of soil phosphate analysis in archaeological research. Background information including the history of the use of the technique, the connection between soil phosphate and archaeological deposits, and the various techniques of extraction and measurement involved with the method will be discussed. Chapter 4 discusses the methods and analyses for this study, followed by the results and discussion presented in Chapter 5. Finally, the conclusions and future directions are discussed in Chapter 6.

2. Environmental and Cultural Background

The following chapter provides a review of the environmental and cultural background for this study. The environmental setting of west Tennessee is reviewed, followed by a discussion of the soils of Fayette County. A review of the Mississippian culture discusses the sociopolitical organization, subsistence strategies, settlement system and patterns, and ideology. Reviewing the environmental and cultural settings for this study connects the archaeological deposits found at the study area to the people who produced the deposits and the landscape upon which those people lived.

Environmental Setting

West Tennessee is defined as the region of the state situated between the Mississippi River to the west and the Tennessee River to the east. Ames Plantation is located on the border of two ecoregions in west Tennessee—Mississippi Valley Loess Plains and Northern Hilly Gulf Coastal Plains, as shown in Figure 2 (Griffith et al. 1998). The Loess Plains ecoregion to the west is characterized by "gently rolling, irregular plains, 250-500 feet in elevation, with loess up to 50 feet thick" (Griffith et al. 1998). The productive agricultural industry in west Tennessee is a result of the flat bottomlands and floodplains consisting of loess and alluvial silt in the Loess Plains ecoregion. The eastern half of the region is comprised of the Northern Hilly Gulf Coastal Plains, which "contain several north-south trending bands of sand and clay formations…and more rolling topography and more relief than the Loess Plains" (Griffith et al. 1998). The natural vegetation type for both the Loess Plains and Northern Hilly Gulf Coastal Plains is oakhickory forest; however, the vegetation type grades "into oak-hickory-pine to the south" of the Northern Hilly Gulf Coastal Plains and "most of the forest cover has been removed for cropland" (Griffith et al. 1998).



Figure 2. Ames site (40FY7) in regional archaeological and ecological context.

Soils of Fayette County, Tennessee

Local soil classifications are typically grouped by soil series and soil types according to the soil profiles examined. Flowers (1964:1) explains, "except for different texture in the surface layer, all the soils of one series have major horizons that are similar in thickness, arrangement, and other important characteristics." Flowers (1964:1) continues that within a soil series, soils with different textures are further divided into soil types. Mapping classified soils results in general patterns among major and minor soil series and types, referred to as "soil associations" (Flowers 1964:2). Four soil associations are found in Fayette County, Tennessee: Grenada-Memphis-Loring association, Lexington-Ruston association, Waverly-Falaya-Collins association, and Loring-Memphis-Lexington-Ruston association (Flowers 1964).

Since Ames Plantation is located on the border of two ecoregions, it is difficult to place soils at Ames into only one of the four previously mentioned soil associations. Most of the cultivated land at Ames consists of Memphis and Loring soil series. The Memphis series is a thermic Typic Hapludalf (Soil Survey Staff 2018). The Loring series is a thermic Oxyaquic Fragiudalf (Soil Survey Staff 2013). The Memphis and Loring series have many shared characteristics. Both series were formed in loess deposits on nearly level to very steep sloping uplands and terraces and textures include silt, silt loam, and silt clay loam. Both are moderately to well drained, and both have moderately acidic through very strongly acidic A, E (where present), and B horizons (Soil Survey Staff 2013, 2018).

Cultural Background

Mississippian is a term referring to one of the major archaeological groups sharing the same set of adaptive cultural characteristics in eastern North America during the Late Prehistoric period (AD 1000-1500). The Mississippian period in the Central Mississippi Valley (CMV), which extends from the confluence of the Ohio River as the northern boundary to the Arkansas River, is divided into the Early (AD 1000-1150), Middle (AD 1150-1300), and Late (AD 1300-1450) periods (Cobb and Butler 2002:627; McNutt 1996:xi). Mississippians occupied the floodplains and adjacent uplands of the Mississippi River and its tributaries from Wisconsin to

Mississippi, across much of the Midwest, Midsouth, and Southeast United States. The section below provides a discussion on the social organization, subsistence strategies, settlement system, and ideology of Mississippians in the CMV.

Cultural Overview. The Mississippian cultural period is one of increasing socio-cultural complexity demonstrated in the archaeological record through large quantities of elaborately decorated shell-tempered pottery, large planned communities featuring mounds, plazas, and defensive features (e.g., palisades), and changes in projectile point forms to the smaller triangular Madison and stemmed to corner-notched forms (Lewis 1996; Smith 1996). Holmes (1886) first used the term "Mississippian" in referring to the shell-tempered pottery often indicative of the period and later described the shared characteristics marking the period recognizable in the archaeological record within the CMV region, including shell-tempered pottery, earthen mounds, and wattle-and-daub architecture (Holmes 1914:424-428). Research over the past century has resulted in a more refined conceptualization of Mississippians highlighting regional variation in the expression of shared cultural traits (Beck 2003; Scarry 1993; Wilson 2017). This refinement is exemplified in Kidder's (1998:124) description of Mississippian groups as "a fluid, regionally distinct, and particularistic group of settlements incorporated through local historical contexts and linked through shared cultural tendencies and widely diffused technological innovations."

Subsistence. Mississippians are considered the first prehistoric population to be primarily dependent on a maize agricultural subsistence system in eastern North America (Griffin 1967; Lewis 1996). Mississippians cultivated many domesticated plants including maize, beans, pepo gourds and squash, bottle gourds, sunflower, sumpweed, and other species first domesticated in the earlier Archaic and Woodland periods (Fritz 1990; Scarry 1993). Fishing and hunting wild game such as white-tailed deer, raccoons, waterfowl, turkeys, and turtles, and gathering wild

plants were also important (Kreisa 1987; Kruger 1985). Plants commonly gathered include goosefoot, maygrass, hickory nuts, walnuts, pigweed, smartweed, and American lotus (Delcourt and Delcourt 2004; Edging 1985; Fritz 1990; Woodward 1987).

Social Organization. Mississippian society is considered a kin-based, ranked society that included a class ruling over a local or regional community, as well as warriors, religious practitioners, craft specialists, and agriculturalists (Milner 2004; Nass and Yerkes 1995). Archaeological evidence for hierarchical social organization includes the orientation and accompanying objects in mound burials when compared to non-mound burials, remains of charnel houses and objects depicting ancestors, and the spatial distribution of objects made of non-local materials (Milner 2004). Extended families of higher-ranking lineages would live in a specific area and build earthen mounds to express and legitimize their higher status. Milner (2004:126) also notes that "building onto earlier platforms also established a connection with the past that legitimized the positions of the living."

Settlement System. Defining an overall settlement system for Mississippian society is a challenge for archaeologists due to the regional variability of Mississippian societies and changes within each region over time. Williams (1995:133) suggests the basic settlement system for Mississippian society is a two-tier system based on the presence or absence of farmsteads. Certain Mississippian polities (e.g., Cahokia, Etowah, Moundville) may have had up to a four-tier system, including a large paramount political center, smaller mound-and-plaza towns, hamlets, and farmsteads dispersed across the regional landscape (Phillips et al. 1951). However, Williams (1995:133) points out that the large regional political centers "are the exception rather than the rule."

Ideology. Various aspects of Mississippian ideology have been reconstructed based on iconographic and archaeological data, as well as folklore and ethnohistoric accounts from tribes across the Midwest, Great Plains, and Southeastern United States (e.g., Creek, Omaha, Osage). The Mississippian cosmic universe consists of a horizontal quadripartite plane with special emphasis on the cardinal directions and a vertical tripartite plane consisting of the upper, middle, and lower realms connected by the axis mundi (Reilly 2004). The belief system has many shared elements with earlier temporal periods with earthen mounds being the most visible; however, a suite of elements within the belief system first appeared during this time associated with the intensification of agricultural production.

Much of the archaeological evidence for the connection between many ideological elements and the increase in agricultural production comes from the "architectural grammar" of Mississippian communities and iconographic motifs depicting a female deity associated with fertility and agriculture, referred to as Corn Mother by many archaeologists (Hall 2004; Lewis et al. 1998; Reilly 2004). One of the most distinct characteristics of Mississippians is the mound-and-plaza complex (Lewis and Stout 1998:228). Over the course of 100 years, large, planned settlements similar in spatial layout, architecture, and the alignment of architectural features with various celestial events appear across the Midwest and Southeast United States. These planned communities included earthen mounds, a plaza surrounded by clusters of residential structures, and defensive features (e.g., palisades) (Lewis et al. 1998; Milner 2004). Many architectural features were aligned with the cardinal directions and solstices.

Previous Research at Ames

The information known about the Ames site has been uncovered through the research efforts of Mickelson (e.g., Mickelson 2008, 2019; Mickelson and Goddard 2011) of the University of Memphis and his students (e.g., Cross 2016; Goddard 2011; Guidry 2013) over the past decade. The Ames site was first recorded in the 1960s by Morse, Graham, and Polhemus (Mickelson 2008). In addition to the documentation from Morse and colleagues, Smith (1969) also investigated the Ames site (Mickelson 2008:201). Guthe's test excavations in 1972 produced "virtually nothing" (Peterson 1979:28). As part of the archaeological survey of the Wolf River Watershed contracted by the USDA Soil Conservation Service, Peterson and other Memphis State (University of Memphis) archaeologists also found very few artifacts from four test excavation units. Issues of obtrusiveness and visibility, which led to the general conclusion that Ames was a vacant ceremonial center, is echoed by Peterson (1979:28),

Our tests and collections, in the absence of checks from past collections, place no late or Mississippian settlements in the vicinity. Therefore, one of the few previously recognized problems in the Wolf drainage—the finding of the population that built and supported the mound center at 40FY7—has not been clarified (in fact, it has been made worse) by this study.

Based on the ceramics collected by Peterson (1979) and surface surveys in the 1980s and 1990s, Mainfort suggested the Ames site was occupied during the Middle Woodland period, emphasizing similarities between Ames and Pinson (Mainfort and Kwas 1985; Mainfort 1986, 1992; Mickelson 2008). In light of new evidence and radiocarbon dates, we now know Ames is one of several Mississippian period towns in the west Tennessee interior uplands occupied from approximately AD 1000 – 1300 (Mickelson 2019). The crucial new evidence was the result of

magnetometry surveys conducted by Goddard and Mickelson (Goddard 2011; Mickelson and Goddard 2011). Goddard's (2011) research consisted of 22 shovel test pits, magnetometry surveys across the open area south of the mounds, and 10 ground-truthing excavation units. Figure 3 shows the community plan at Ames inferred by Mickelson (2019:Figure 8) after combining the evidence from surface collections, magnetometry, and ground-truthing excavations.



Figure 3. Ames community plan based on magnetometry, shovel tests, and ground-truthing excavations (Mickelson 2019:Figure 8). Red points indicate location of soil samples collected for this study.

3. Soil Phosphate Analysis in Archaeology

The following chapter is a summary of the use of soil phosphate analysis and begins with a brief description of the history of the technique and its use in archaeological research. The reasoning behind using the technique and the implications are also explained in the context of the chemistry of soil phosphorus. Following the discussion on the chemistry of soil phosphorus, an explanation of the four techniques for extracting phosphorus and the two most common techniques for measuring soil phosphorus in archaeology is provided to demonstrate the use of the specific extraction and measurement technique used in this study.

The utility of soil phosphate analysis for detecting human occupation has been refined through archaeological case studies and experimentation over the last two decades (Holliday and Gartner 2007; Nolan 2014; Nolan and Redmond 2015; Parnell et al. 2001; Parnell et al. 2002; Roos and Nolan 2012; Terry et al. 2000; Wells et al. 2000). The connection between the enrichment of phosphate and archaeological remains was first established by Olof Arrhenius in the 1920s (Hjulstrom and Isaksson 2009). Once a relationship between phosphate levels and archaeological remains was established, the application of soil phosphate analysis in archaeology was primarily for site prospection and delineation of site boundaries (Middleton 2004; Middleton et al. 2010). To this day, phosphorus is the most commonly studied element in archaeology due to the early establishment of the archaeological connection, as well as a number of unique chemical characteristics (Holliday and Gartner 2007; Holliday et al. 2010; Wilson et al. 2008).

Phosphorus has been a relatively consistent indicator of human occupation due to a number of unique chemical characteristics. Phosphorus appears in soil as various forms of orthophosphate (i.e., $H_2PO_4^-$, HPO_4^{2-} , PO_4^{3-}) (Eidt 1977:1327). The source of inorganic phosphate is apatite which enters the soil during its formation due to weathering of the parent

material (Roos and Nolan 2012:25). Organic phosphate is abundant in all living organisms as phosphorus is one component of DNA (Eidt 1977:1327). As organic material decays, mineralization gradually converts organic phosphate into inorganic phosphate (Eidt 1977:1328; Holliday and Gartner 2007). When phosphate enters the soil, the molecule becomes "fixed" as it is adsorbed on the surfaces of clay minerals or bound with iron or aluminum in acidic soils and calcium in calcareous soils to form Fe-, Al-, or Ca- phosphate minerals (Bethell and Máté 1989; Holliday and Gartner 2007:344; Proudfoot 1976; Roos and Nolan 2012:25).

The two characteristics above have advantageous implications for archaeological surveys. Elevated phosphate concentrations are often associated with the accumulation of organic material, which occurs with human occupation and varies with the duration of occupation. Spatial distributions of elevated and depleted phosphate may reveal the structure of an occupied settlement and the behavior of the people within the settlement. Phosphate is mechanically stable moving very little vertically and horizontally from the place of deposition due to its fixation on the fine fraction (< 2mm) of the soil (Crowther 1997:99; Roos and Nolan 2012; Sjoberg 1976). Additionally, phosphorus is not susceptible to removal by normal oxidation, reduction, or leaching processes, as are other common elements analyzed as indicators of human activity (e.g., nitrogen, calcium) (Carr 1982; Eidt 1977:1327). These implications are particularly advantageous for archaeologists working in areas where surface surveys are untenable because the samples collected for soil phosphate analysis come from subsurface soils, in which soil phosphate is more stable than it is in surface soils (Sjoberg 1976).

Soil phosphate studies in archaeology have been largely influenced by the work of Robert Eidt (1973, 1977). In the decades since Eidt's work, there has been a proliferation of phosphate analysis methodology resulting in over 30 different methods currently employed in archaeology

and many more when disciplines outside of archaeology are considered (Holliday and Gartner 2007, Roos and Nolan 2012). When choosing a specific analytical method, many factors concerning the local soil conditions must be considered. Factors affecting phosphate analysis due to their impact on the detection of phosphate in the soil include organic matter, soil pH, soil moisture, particle size, and time (Holliday and Gartner 2007). For example, the phosphorus binding capacity of iron and aluminum compounds and soil pH have an inverse relationship (Holliday and Gartner 2007:306). Knowledge of the soil at the site in question is the most important interpretive tool because the method chosen must take these factors into consideration to reduce their potential influence on results and to help explain the patterns shown in the results.

Extraction Techniques

There are various techniques for the two steps of phosphate analysis, extraction of the phosphorus from the soil and measurement of the phosphorus concentration (Holliday and Gartner 2007). The archaeological approaches used for extraction and measurement of soil phosphorus, sometimes conceived of in combination, are often divided into four categories: portable field techniques (e.g., spot- or ring-test), chemical digestion of a soil sample for total phosphorus analysis, phosphorus fractionation, and extraction of available phosphorus (Bethell and Máté 1989; Holliday and Gartner 2007; Terry et al. 2000). Each has advantages and disadvantages that impact the method's applicability to archaeological research questions, although the research questions being asked heavily influences which method is chosen.

The spot-test was originally designed to be conducted in the field and reveal results immediately, both of which are advantageous qualities for archaeology (Terry et al. 2000). However, the results are subjective and may vary due to external factors, such as temperature, as well as human error (Eidt 1977; Terry et al. 2000). The methods involving chemical digestion

for total phosphorus and phosphorus fractionation both require intensive laboratory work, which would presumably entail more cost (Terry et al. 2000). While there are issues with each of these methods, there have been successful applications of each method in archaeological prospection studies (e.g., Ahler 1973; Bjelajac et al. 1996; Leonardi et al. 1999; Parnell et al. 2002).

Total vs. Available Phosphorus

The fourth method, involving the extraction of available phosphorus, is becoming more popular for many reasons, including increased cost efficiency and advancements in instrumentation (Holliday and Gartner 2007; Roos and Nolan 2012; Terry et al. 2000). This method involves the extraction of phosphorus with a dilute acid solution and measurement of the phosphorus concentration with the aid of a colorimeter (Terry et al. 2000). This technique is often used in Mayan studies in Central America (Dahlin et al. 2007; Dahlin et al. 2010; Eberl et al. 2012; Hutson et al. 2007; Hutson et al. 2009; Parnell et al. 2001; Parnell et al. 2002a; Parnell et al. 2002b; Terry et al. 2000; Terry et al. 2004; Wells et al. 2000). This method was employed by Nolan and his colleagues in the Midwestern United States (Nolan 2014; Nolan and Redmond 2015; Roos and Nolan 2012). While this method is increasing in popularity, there is some debate as to the utility of measuring available phosphorus compared to total phosphorus in geoarchaeological research (Bethell and Máté 1989; Holliday and Gartner 2007).

A major argument against the extraction and measurement of available phosphorus in its most basic sense concerns the issue of proxy data. Critics of the technique argue that available phosphorus represents only roughly 1-3% of total phosphorus (Bethell and Máté 1989; Holliday and Gartner 2007:313) and that the technique does not necessarily measure anthropogenic inputs of phosphorus (Holliday and Gartner 2007). Although these criticisms are valid, Terry and colleagues (2000:153) have asserted that while available phosphorus is not always proportional

to total phosphorus—for archaeological prospection and activity area research—the relative spatial patterning of phosphate concentrations is important, rather than the absolute concentration. Additionally, Terry and colleagues (2000) and Parnell and colleagues (2002a) found available phosphorus to be more sensitive to human inputs than total phosphorus, which suggests that high levels of phosphorus in the parent materials of the soil will produce analysis results in which the total phosphorus overwhelms signatures of human activity in the soil (Holliday and Gartner 2007:314).

Measurement Techniques

There are two main techniques used in archaeology for measuring soil phosphate concentrations. The first technique is colorimetry, which is the "use of the absorption of visible radiation by solutions to quantitatively measure the concentration of the absorbing species" (Pollard et al. 2007:70). In the Mehlich-2 procedure employed by Roos and Nolan (2012) and Terry and colleagues (2000), the phosphate is extracted in solution, which turns blue during a reaction after an ascorbic acid reagent is added. This blue colored solution can be quantitatively measured with a calibrated colorimeter. The intensity of the color is linearly proportional to the concentration of phosphate in the solution (Pollard et al. 2007:86-87). This technique is becoming increasingly common with the increasing affordability of portable colorimeters, allowing archaeologists to quantitatively measure phosphorus concentrations in the field (Holliday and Gartner 2007; Terry et al. 2000).

The second main technique to measure soil phosphate is inductively coupled plasma spectroscopy (ICP). ICP is a form of atomic spectroscopy, where the soil sample, digested in a strong acid, is converted into a gaseous state and heated with a plasma torch (Malainey 2011:447-448). The thermal energy causes the ionized atoms to become excited and, when the

excited ionized atoms return to their ground state, an emission of light is measurable with a spectrometer. The spectra of light emitted is unique to each individual atom as well as the element concentration (Holliday and Gartner 2007:312; Malainey 2011:447). This method is often used when multiple elements are analyzed in a single procedure. Holliday and Gartner (2007:312) caution that the accuracy of measuring phosphorus is partially dependent on the capacity of the reagent to free the phosphate molecules during the chemical digestion of the sample.

Since there are many different soil phosphate methods, it has become somewhat common in recent years to compare different methods on the same site in question to not only reduce errors for the study, but also to evaluate the accuracy of certain methods in particular contexts. For example, to test the applicability of the Mehlich-2 procedure (Mehlich 1978), Terry and colleagues (2000:157) statistically compared four different methods: the Mehlich-2 procedure, modified for their site in question; the Olsen bicarbonate (Olsen and Sommers 1982) extraction; the Eidt ring-test rating (Eidt 1977); and the perchloric acid digestion (Olsen and Sommers 1982) method for total phosphorus determination. Their analyses concluded that the Mehlich-2 extraction procedure was the most viable of the four methods (Terry et al. 2000). Based on the findings of Terry and colleagues (2000) and others (e.g., Holliday and Gartner 2007; Wilson et al. 2006), comparative studies of phosphate extraction methods are important and necessary because as analytical methods continue to advance, the validity of such methods must be tested in various environments and contexts to be applicable in archaeology.
4. Methods and Analysis

The following chapter consists of the methods and analyses utilized in this study. Following a brief description of the magnetometry survey conducted, the procedure for soil sampling and soil processing are described to illustrate the steps prior to performing the soil phosphate and loss on ignition analyses. The procedure for the soil phosphate analysis is then described followed by the procedure for loss on ignition. Detailed step-by-step procedures for each stage of the study are included as an Appendix.

Magnetometry Surveys

The field to the east of the Ames town site was surveyed in March 2018 using a Bartington 601-2 dual fluxgate gradiometer (Figure 4). Magnetometry data were collected from eleven 20 m grid squares at a 0.50 m transect interval with four readings per meter along each transect. The survey revealed many potential archaeological signatures with the most prominent signature being a rectangular feature characteristic of a Mississippian residential structure, shown in the center of Figure 5. Two additional potential archaeological signatures shown in Figure 5 are located approximately 20 meters south of the rectangular signature and approximately 40 meters northeast of the rectangular signature.

28



Figure 4. Red box indicates area surveyed in March 2018 using a fluxgate gradiometer. The surveyed area is located in a field east of the Ames town which was enclosed by the labeled palisade wall.



Figure 5. Results from the March 2018 magnetometry survey. Green box indicates a magnetic signature of a Mississippian-period structure excavated in May 2019. The red boxes indicate two additional potential archaeological magnetic signatures.

Soil Sampling

The sampling design for this study is a combination of a North-South transect across the town site with samples spaced 20 meters apart, two East-West transects spaced 40 meters apart with samples placed 20 meters apart on each transect, and a regular 20 x 20-meter grid in the nearby field where magnetometry surveys were performed (Figure 6). The locations of the

sample points are positioned on the same grid as the 20 x 20-meter magnetometry surveys for the area. Samples were extracted using a bucket auger, brushing off excess soil between every use. The auger was marked with a permanent marker at the 5, 10, and 15-centimeter depths for vertical control. The top 5 centimeters of each sample was first extracted and set aside. The next 10 centimeters (i.e., 5 - 15 centimeters below the surface) was extracted for analysis. The extracted sample was then wrapped in aluminum foil, placed into a labeled plastic bag, and stored in a cooler before being transported to the laboratory at the end of the day. Once at the laboratory, the samples were stored in a refrigerator until they were processed and analyzed.



Figure 6. Sampling design for this study compared to artifacts collected during surface surveys, mound locations in northwest corner, and magnetometry survey area. Locations of soil samples collected for present study are indicated by black points and artifacts are indicated by green x's.

Soil Sample Processing

Prior to performing laboratory analyses, the soil samples were processed. The first step in processing the soil samples was to record the morphological characteristics for each sample. Soil texture—the proportions of clay, silt, and sand—were determined qualitatively by visual inspection of grain size, shear test, and test of stickiness and plasticity. Soil color was determined using a Munsell Color Chart. Soil structure was determined by recording the shape and size of peds, which are natural aggregates of major particles and considered the basic unit of soil structure (Schaetzl and Anderson 2005). Finally, the proportion of organic debris in the sample was estimated by visual inspection of organic debris (e.g., roots, grass) and observing the reaction when 10% dilute HCl is placed on the sample to estimate the presence and degree of reaction with calcium carbonate.

Once the morphological characteristics were recorded, the samples were quartered to achieve a representative subsample on which the analyses were performed. Each sample was placed on a sheet of aluminum foil in the shape of a mound. After mixing the sample with a clean soil knife, the mound was divided into four quarters. The target mass of the subsample was 40-60 grams. If the mass of the chosen quarter was greater than 60-70 grams, the quarter was divided into quarters as well. If dividing the subsample into quarters resulted in too small of a subsample, the subsample was divided in half to obtain the subsample. The subsample was then placed in a 250 mL beaker by moving the final subsample to the edge of the foil, tearing the foil to ensure the entire subsample was placed into the beaker. The mass of the empty beaker and mass of the beaker including the subsample were recorded to calculate the mass of the wet soil sample. The beakers were then loosely covered with aluminum foil in a fashion to allow moisture out while reducing the risk for contamination while in the drying oven. The beakers were left to dry overnight, between 15 - 20 hours, in the drying oven set to approximately 102-105°C. This drying procedure is necessary for reporting soil properties on a dry-weight basis (Sheppard and Addison 2006). The dried samples and beakers were weighed for the dry sample mass and the samples were ground and sieved through a 250-micron mesh. The sieved samples were weighed, placed in a glass sample bottle, and stored in the refrigerator.

33

Soil Phosphate Analysis

The soil phosphate analysis method chosen for this study uses the Mehlich-2 Soil Extractant Solution to extract available phosphate, and molybdate colorimetry to measure the phosphorus concentration (Holliday and Gartner 2007; Mehlich 1978; Parnell et al. 2001). Terry and colleagues (2000:162) suggest the Mehlich-2 extraction procedure, "is a viable method for archaeologists who need to measure phosphorus accurately in the field...we believe that it would be suitable for archaeological prospecting and mapping in both equatorial, tropical, and midlatitude environments, where the soil pH is acidic to mildly alkaline." This suggestion was made after conducting experiments finding that phosphorus concentrations of samples subjected to the Mehlich-2 method performed directly in the field were within 7% agreement compared to the same samples subjected to the same method in a soil chemistry laboratory. The geographic range of use in archaeological contexts (e.g., Birch 2016; Parnell et al. 2002a; Roos and Nolan 2012) and potential for performing the analysis directly in the field are two reasons for choosing to assess this specific method for the local soil conditions of west Tennessee interior uplands.

For this study, 2 g of the sieved soil sample was measured with an analytical balance and placed into a 50 mL glass sample bottle. Next, 25 mL of 1:6 dilute Mehlich-2 solution was added to the sample and shaken in a fixed-speed reciprocal shaker for 10 minutes. After 10 minutes, the shaken sample solution was filtered through Whatman 44 ashless filter paper. Once filtered, 5 mL of filtered sample solution was diluted to 50 mL with deionized water. One PhosVer3 reagent pillow was added to 10 mL of the diluted solution and shaken by hand for 1 minute. After 1 minute, the solution sat for 4 minutes to allow for color development. After 4 minutes, the sample was poured into the appropriate sample cell and absorbance was measured

34

using a Hach DR2400 Spectrophotometer at 880 nm. Measurements on each sample were repeated four times.

Since absorbance values were measured using the spectrophotometer, further calculations were required to find the concentrations of soil phosphorus (mg/kg). Standards of orthophosphate (PO₄³⁻) were created in March 2019. Standard concentrations of 0.10 mg/L, 0.30 mg/L, 0.60 mg/L, 0.90 mg/L, and 1.20 mg/L were measured with the spectrophotometer and absorbance values were recorded. The absorbance values recorded from the standard concentrations were graphed in Microsoft Excel to produce a standard curve by adding a 3rd order polynomial trendline, shown in Figure 7. The cubic polynomial equation of the trendline was then used to determine the unknown concentrations (mg/L) from the recorded mean absorbance value for each sample.



Figure 7. Standard curve based on absorbance values of known phosphate concentrations.

The phosphate concentrations (mg/L) for each sample were converted to concentrations (mg/kg) using the following formula,

$$PO_4^{3-}$$
 (mg/kg) = ((C (mg/L) * V (L) * DF) / W (kg))

where C is the concentration calculated using the standard curve, V is the volume of the undiluted sample (i.e., volume of solution in which the sample was digested), DF is the dilution factor, and W is the weight of the sample. The dilution factor is the result of the volume of the diluted sample solution (mL) divided by the volume of aliquot taken for dilution (mL).

The concentrations of phosphate (mg/kg) were then converted to concentrations of phosphorus (mg/kg) by dividing each phosphate concentration by 3.06 (i.e., based on atomic weight) (Dabkowski and White 2016). The phosphate compound weighs 95 atomic units (i.e., sum weight of four oxygen atoms and one phosphorus atom), which is 3.06 times heavier than a phosphorus atom weighing 31 atomic units. The final result from the preceding series of calculations is the concentration of soil phosphorus (mg P/kg soil) for each sample.

Loss on Ignition

For this study, 5 g of soil from each sample was measured and placed in a porcelain crucible. The initial mass of the sample was measured and recorded, as well as the mass of the empty crucible and accompanying lid. The samples were heated in a muffle furnace at a temperature of 360°C for two hours. After two hours, the furnace was turned off and allowed to cool for about 10-15 minutes. After about 15 minutes, the crucibles were taken out one at a time, weighed, and placed back into the furnace. Once the mass was recorded for each crucible, the furnace was turned on and set to 550°C. After the crucibles were heated for three hours at

550°C, the samples were again removed one at a time, weighed, and placed on a tray overnight to cool.

The variables recorded during the analysis include the mass of the empty crucible (CRCBM), initial sample mass (SSIM), the mass of the crucible and sample after the first round of heating at 360°C (SSCMAFH), and the mass of the crucible and sample after the second round of heating at 550°C (SSCMASH). The percent loss on ignition at each temperature (LOI₃₆₀ and LOI₅₅₀) for each sample were calculated using the following formulae:

$$LOI_{360} = ((DW_{105} - DW_{360}) / DW_{105}) * 100$$

$$LOI_{550} = ((DW_{105} - DW_{550}) / DW_{105}) * 100$$

where the SSIM was designated as DW_{105} (i.e., dry-weight at 105°C) and the CRCBM was subtracted from SSCMAFH and SSCMASH for each sample to determine the mass of each sample, designated as DW_{360} and DW_{550} , respectively.

Mapping Results

Raw data generated from this study were imported into ArcMap 10.5.1 to explore the data via mapping. Maps were created by classifying data from the whole study area and by classifying data from the town site and the eastern side field separately to investigate correlation and relationships. Separating the data into even smaller sample sizes was warranted because the town site has not been plowed in approximately seven years, while soybeans grown in the eastern side field was harvested within two months of collecting soil samples.

Four classification schemes were assessed to be sure the maps accurately represent the spatial distribution of phosphorus concentrations and LOI results. Natural Breaks classification,

based on the Jenks Natural Breaks algorithm, was assessed first because the histogram indicated multiple large breaks in the distribution. Geometric interval was then assessed because the raw geochemical data were not normally distributed. Frye (2007) suggests the method "works reasonably well with data not normally distributed." For the manual classification method, class breaks were inserted at the background mean (i.e., mean of lowest 25% samples), increasing in intervals of five background standard deviations (i.e., standard deviation of lowest 25% of samples multiplied by 5). The fourth method assessed was quantile, which is often used with ranked data, classified so each class contains equal number of features (Law and Collins 2013:253-254). Maps were created using each classification method with 5 classes, 7 classes, and 10 classes.

5. Results and Discussion

The following chapter provides the results of the study and a discussion of the archaeological implications at Ames. First presented are frequency distributions of the physical characteristics for the soil samples. Next, the spatial distribution of the soil phosphorus concentrations is presented and discussed in the context of prior research at the Ames town. This discussion is divided into separate areas to highlight converging evidence. The LOI results are then briefly discussed, followed by the results of statistical analyses performed to understand the relationships between the results of the soil analyses and prior research at Ames.

Physical Characteristics

The 53 soil samples were relatively uniform in each physical characteristic recorded. The soil texture for all 53 samples are within the range classified as silt loam. Of the 53 samples, 28 were recorded as dark yellowish brown and 19 were brown (Figure 8). Calcium carbonate was not present in any of the 53 samples. The only ped shape observed in all 53 samples was subangular blocky and in all but one sample, the peds were less than 20 mm in diameter (Figure 9). All 53 samples contained less than 5% organic debris, 49 samples had less than 2%, and 35 samples had less than 0.50% organic debris (Figure 10). When the two sampling locations (i.e., the town site and the eastern field) are taken into consideration, all 21 samples from the eastern field contained less than 0.50% organic debris. This is most likely explained by the harvesting of soybeans which took place within two months of sample collection.

39



Figure 8. Munsell color distribution of soil samples.



Figure 9. Distribution of ped diameter range.



Figure 10. Distribution of estimated organic debris in samples.

Soil Phosphorus Spatial Distribution

When compared to the magnetometry data (Figure 11), artifacts collected from surface surveys (Figure 12), and the community layout of Ames inferred from ground-truthing excavations (Figure 13), the phosphorus concentrations across the study area are generally well correlated with suspected and confirmed archaeological remains. The quantile classification method appears to best reflect the relationship between phosphorus distribution and Mississippian activity at Ames. All maps presenting the results were created using the quantile classification method and 7 classes. It is important to note that none of the samples were collected within the boundaries of a previous excavation, although the preceding maps show there were. The sample points for the soil analyses were mapped based on the magnetometry grid due to technical issues with the GPS unit and the points do not necessarily represent the exact location of the sample points (within 2-5 meters).



Figure 11. Phosphorus concentrations compared to magnetometry data. The higher phosphorus concentrations are located within the boundary of the Ames town represented by the palisade wall visible in the magnetometry data.



Figure 12. Soil phosphorus distribution compared to artifacts collected during surface surveys. The majority of surface artifacts are located within the palisade near the center of the Mississippian settlement between AD 1050-1290. The eastern field has fewer surface artifacts because the area was not systematically surveyed and is now a no-till field.



Figure 13. Soil phosphorus distribution compared to archaeological remains confirmed by "positive" ground-truthing excavations (outlined in green).

The maps created to assess the different classification methods are presented in Figure 14 and Figure 15, distinguished by setting 7 and 10 classes respectively. The eight maps created show a patterned distribution where three sub-areas within the study area have relatively higher phosphorus concentrations compared to the areas to the south and in the center of the study area. The sub-areas with higher phosphorus concentrations are designated as Area-A, Area-B, and Area-C for clarity when discussing the relationships between the results and previous research at Ames (Figure 16).



Figure 14. Phosphorus distribution with different classification methods and 7 classes. Natural breaks (Jenks) (top left); quantile (top right); manual interval (bottom right); geometric interval (bottom left).



Figure 15. Phosphorus distribution using different classification methods and 10 classes. Natural breaks (Jenks) (top left); quantile (top right); manual interval (bottom right); geometric interval (bottom left).



Figure 16. Areas of relatively higher phosphorus concentrations (mg/kg) designated as Area-A, Area-B, and Area-C.

Phosphorus Results and Prior Research

Area-A. Samples with high phosphorus concentrations within Area-A were located across a Mississippian-period mound (i.e., Mound C in Figure 16) and public plaza area. The mound sampled was also the location of an antebellum house. The phosphorus concentrations were expected to be high in this general region due to the association with the Late Prehistoric and Historic human activity. Of the six samples assigned to Area-A, five are among the highest

six concentrations in the study. For statistical testing, the five northernmost samples in Area-A were omitted for being extreme outliers in the distribution.

Area-B. Previous ground-truthing excavations of the magnetic signatures between 2009 and 2016 confirmed the presence of an east-west line of Mississippian structures featuring multiple sequential periods of construction, as well as other structures, pit features, and palisades (Cross 2016; Goddard 2011; Guidry 2013; Mickelson and Goddard 2011). These excavations included three block excavation units located within Area-B (Figure 17). The possibility of phosphorus enrichment due to previous excavations was considered, especially since the units within Area-B were open for over a year. However, when taking radiocarbon dates from multiple samples into consideration, a more plausible explanation for the higher phosphorus concentrations may be the activity of Mississippians living at Ames.



Figure 17. Phosphorus distribution highlighting Area-B compared to ground-truthing excavation units. Again, no samples were collected from within excavation unit boundaries.

Radiocarbon dates from the wall trenches excavated within Area-B indicate the structure was one of the earliest Mississippian structures built and was reconstructed twice throughout the ~250-year period of Mississippian occupation at Ames (AD 1050-1290) (Guidry 2013). The block units also revealed a palisade north of the structure which was also excavated and dated to approximately AD 1050 (Guidry 2013). The higher concentrations enclosed in Area-B are located east of Mound A and south of Mound B. Additional ground-truthing excavation units on the eastern portion of the town site confirmed the presence of a wall-trenched structure (i.e.,

excavation unit outside the northeast corner of Area-B in Figure 17) and the presence of a large pit feature (i.e., excavation unit located outside the palisade to the east in Figure 17), neither of which yielded high phosphorus concentrations. A radiocarbon date for the large pit feature indicates the feature dates between AD 1220 - 1280 (Guidry 2013).

Area-C. Area-C encompasses the northeast portion of the study area in what is referred to as the eastern or east field. Figure 18 presents the phosphorus distribution compared to magnetometry data. The magnetic signature in the center of Figure 18 was excavated in May 2019. Preliminary interpretations from the excavation considers the magnetic signature to be three superimposed Mississippian-period structures. Phosphorus concentrations around this structure are higher than areas with no recorded cultural features, but not as high as expected given the intensity of the magnetic signature.



Figure 18. Phosphorus distribution compared to magnetometry data of the eastern field. The labeled signature was excavated in May 2019. There has been no further testing of the additional signatures outlined by red boxes.

The eight 20 m grid squares of magnetometry data missing from the east end of the eastern field in the preceding figures were surveyed in April 2019 (Figure 19). This area was surveyed after the soil samples were analyzed so predictions concerning the magnetometry data could be made prior to the survey. The samples in this portion of the study area are some of the highest in phosphorus, which led to the prediction that there would be evidence of Mississippian habitation in the magnetometry data. After processing the more recent magnetometry data and examining the results for patterns characteristic of Late Prehistoric habitation, many potential archaeological signatures stand out.



Figure 19. Eight 20 m grid squares surveyed in April 2019 indicated by red box.

The magnetometry data for the eastern field was processed as one composite in TerraSurveyor (Figure 20). The April 2019 survey revealed at least one additional signature characteristic of Late Prehistoric habitation (Figure 21). This interpretation is based on archaeological context, such as recorded dimensions of Mississippian structures and comparisons between multiple surveys with similar signatures. Figure 22 shows the magnetic signature revealed by the April 2019 survey compared to a magnetic signature revealed by a May 2019 survey on a different field at Ames.



Figure 20. Magnetometry data processed as one composite compared to phosphorus distribution. Previously discussed signatures are enclosed in red boxes. This area appears to be highly susceptible to lightning strikes as observed in the magnetometry data.



Figure 21. Magnetic signatures characteristic of Late Prehistoric habitation. Red boxes indicate signatures previously discussed in the text. Green boxes indicate signatures revealed after the prediction was made based on phosphorus distribution.



Figure 22. Magnetometry data from a field approximately 5 miles southeast of the study area (left) and the April 2019 survey of the eastern eight 20 m grid squares (right). Red arrows are pointing to the northeast corner of magnetic signatures of similar size and shape. The signatures in the right image are highlighted by green boxes in Figure 21.

Loss on Ignition

The first step of the LOI procedure was modified from what would be deemed to be normal protocol. During the soil processing stage of the study, the samples were oven-dried overnight at 105°C, crushed and sieved, and stored in a refrigerator in a glass sample bottle until soil analyses could be performed. While performing the LOI procedure, the samples analyzed were not dried at 105°C immediately prior to heating the samples at 360°C and 550°C, thus the sample's weight was not standardized for calculating the percent loss on ignition. This modification has the potential to introduce errors in the results. However, because the sieved samples were stored in a refrigerator and there was no observable moisture in the initial sample prior to ignition, the results of LOI₅₅₀ were still analyzed and mapped. Interestingly, the spatial distribution of LOI₅₅₀ reflects the inverse relationship with the phosphorus concentrations and the three areas of known archaeological activity (Figure 23).



Figure 23. Side-by-side comparison of LOI_{550} (left) and phosphorus concentrations (right) highlighting the inverse relationship between the two variables.

Statistical Analyses

The phosphorus concentrations and LOI₅₅₀ (i.e., % weight loss) were subjected to exploratory data analysis to investigate the relationships between the two variables, as well as between different groups of which membership was assigned to specific samples. First, phosphorus concentrations and LOI₅₅₀ were subjected to the Shapiro-Wilk normality test (*W*). Both variables were not normally distributed (p < .01). Only phosphorus was normal after performing log transformations on both variables. As normality could not be assumed, nonparametric analyses were chosen to examine the relationship between these two variables. After removing the five northernmost samples from Area-A, due to the presence of several extreme outliers, Spearman's rank-order correlation was performed to examine the relationship between phosphorus and LOI₅₅₀. Results revealed a significant inverse relationship between phosphorus and LOI₅₅₀, rs(48) = -0.44, p = 0.002. Select descriptive statistics and the normality test results are presented in Table 3.

Variable	N	Mean	SD	Skew	Kurtosis	Shapiro-Wilk	
v arrable						W	р
Soil-P	53	23.56	31.76	4.85	28.67	0.516	<.0001
Soil-P (Log)	53	2.81	0.86	0.21	0.60	0.970	0.193*
Soil-P	48	16.87	11.14	0.90	1.10	0.933	0.009
Soil-P (Log)	48	2.66	0.72	-0.50	0.62	0.948	0.034
LOI ₅₅₀	53	5.24	0.59	1.18	2.35	0.923	0.002
LOI550 (Log)	53	1.83	0.09	0.85	1.16	0.951	0.030
LOI ₅₅₀	48	5.16	0.47	0.30	-0.91	0.959	0.093
LOI550 (Log)	48	1.82	0.08	0.18	-0.96	0.964	0.142*

Table 3. Descriptive Statistics and Normality Test Results.

Abbreviations: Soil-P = soil phosphorus concentration (mg/kg); LOI_{550} = loss on ignition (% weight loss); (Log) = log transformed variable; N = sample size; SD = standard deviation; IQ = interquartile; W = Shapiro-Wilk statistic; *p* = statistical significance value

*p > 0.1

Descriptive statistics are presented in greater detail for both phosphorus and LOI550 in the tables below. Table 4 presents descriptive statistics for phosphorus concentrations and Table 5 presents the descriptive statistics for LOI550 across the study area. The samples were then grouped by location within the study area, distinguishing the Ames town site from the eastern field. Table 6 presents the descriptive statistics for both variables when grouped by location within the study area.

	Concentrations (mg/kg).				
	Samples in Study Area	Samples in Study Area (Log)	Samples in Study Area	Samples in Study Area (Log)	
Ν	53	53	^a 48	^a 48	
Mean	23.56	2.81	16.87	2.66	
Median	17.88	2.94	16.67	2.87	
Std. Deviation	31.76	0.86	11.14	0.72	
IQ Range	17.55	1.05	15.18	1.10	
Minimum	2.16	1.15	2.16	1.15	
Maximum	219.77	5.40	52.97	3.99	
Range	217.60	4.25	50.80	2.84	
Skewness	4.85	0.21	0.90	-0.50	
Kurtosis	28.67	0.60	1.10	0.62	

Table 4. Descriptive Statistics for Soil Phosphorus

^a Outliers omitted

	Samples in Study Area	Samples in Study Area (Log)	Samples in Study Area	Samples in Study Area (Log)
Ν	53	53	^a 48	^a 48
Mean	5.24	1.83	5.16	1.82
Median	5.18	1.82	5.12	1.81
Std. Deviation	0.59	0.09	0.47	0.08
IQ Range	0.79	0.13	0.73	0.12
Minimum	4.34	1.68	4.34	1.68
Maximum	7.37	2.12	6.14	1.97
Range	3.02	0.45	1.80	0.29
Skewness	1.18	0.85	0.30	0.18
Kurtosis	2.35	1.16	-0.91	-0.96

Table 5. Descriptive Statistics for LOI $_{550}$ (% weight loss).

^a Outliers omitted

Table 6. Descriptive St	tatistics by L	location within	n Study Area.
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	Town Site		East Field	
	Soil-P	LOI ₅₅₀	Soil-P	LOI ₅₅₀
Ν	32	32	21	21
Mean	25.83	5.39	20.09	5.01
Median	14.85	5.29	20.27	4.92
Std. Deviation	40.01	0.65	10.96	0.39
IQ Range	21.70	0.96	11.12	0.49
Minimum	2.16	4.41	2.41	4.34
Maximum	219.77	7.37	52.97	5.79
Range	217.61	2.96	50.56	1.45

The areas with relatively higher phosphorus concentrations discussed in the preceding sections (i.e., Area-A, Area-B, Area-C) were grouped as such based on the phosphorus values. The three areas correspond to confirmed and suspected archaeological activity at Ames. Further statistical testing was performed to better understand the differences between the samples inside and outside of these assigned areas associated with archaeological activity and higher phosphorus values. Table 7 presents descriptive statistics for phosphorus values comparing samples assigned to archaeological and non-archaeological areas, as well as breaking down the archaeological areas into the three specific groups.

	Samples Assigned to Archaeological Areas	Samples Assigned to Non- Archaeological Areas	Samples Assigned to Area-A	Samples Assigned to Area-B	Samples Assigned to Area-C
N	26	27	6	9	11
Mean	37.58	10.05	80.13	23.83	25.62
Median	26.54	8.80	58.01	22.06	25.31
Std. Deviation	40.73	5.96	71.57	6.77	11.70
IQ Range	13.85	10.47	41.15	8.02	7.69
Minimum	4.56	2.16	19.51	12.47	4.56
Maximum	219.77	20.70	219.77	34.88	52.97
Range	215.21	18.54	200.26	22.41	48.41

 Table 7. Descriptive Statistics for Phosphorus Concentrations Comparing Archaeological and Non-Archaeological Area Membership.

A Kruskal-Wallis test was performed to examine the relationships between the three areas and non-area. Results indicate there are statistically significant differences between the areas ($\chi_2 = 32.48$; df = 3; p < 0.001). Following the significant results of the Kruskal-Wallis test, the Dwass-Steel-Critchlow-Fligner test (DSCF) was used to make post hoc comparisons between the areas. Table 8 presents the results of the DSCF test. Samples within the archaeological areas are significantly higher than samples not in one of the three areas indicated by the significant p-values (i.e., p < 0.05) for the first three comparisons in Table 8. This was expected due to the high phosphorus values being the basis for grouping the samples into areas. The DSCF test found that Area-A (i.e., mound and plaza area) is marginally higher than Area-B (i.e., town residential area). Finally, the DSCF test found Area-B and Area-C (i.e., eastern field) to be essentially equal. This is an important finding because portions of Area-B were subjected to extensive excavations prior to collecting soil samples while Area-C was not. The two areas being equal may represent the same explanation for the enrichment of available phosphorus—Late Prehistoric habitation areas.

Areas Compared	Wilcoxon Z	DSCF Value	р
Non-Area vs. Area-A	-3.6874	5.2148	0.0013
Non-Area vs. Area-B	-3.9638	5.6057	0.0004
Non-Area vs. Area-C	-3.9107	5.5306	0.0005
Area-A vs. Area-B	2.357	3.3333	0.0856
Area-A vs. Area-C	2.2111	3.1269	0.1202
Area-B vs. Area-C	-0.038	0.0537	1

Table 8. Results of DSCF Test.

6. Conclusions and Future Directions

This study sought to assess if archaeological deposits in areas with known issues of visibility and obtrusiveness could be located using soil phosphate analysis. The Mehlich-2 available phosphate extraction measured by molybdate colorimetry was assessed for the local soil conditions common throughout the west Tennessee interior uplands. This specific method has the potential to be performed directly in the field and is cost-effective compared to more specialized laboratory methods (e.g., ICP); however, this study was carried out in a laboratory setting for the purposes of establishing the utility of the technique. The following chapter discusses the results of the study in relation to the initial hypotheses and implications for future research.

The first hypothesis (H_1) tested was that archaeological deposits could be located based on the systematic mapping of available phosphorus concentrations across the landscape. While the sampling design for the study resulted in too low of power to perform spatial statistical testing or interpolation, the spatial distribution of the concentrations mapped using multiple different classification schemes detected three distinct areas of relatively higher phosphorus concentrations. These three areas correspond with known archaeological activity. The spatial patterns also show decreasing phosphorus levels with distance from the center of the Mississippian-period town. In the eastern field, the phosphorus distribution is higher in areas with known archaeological signatures. Therefore, the results of this study support H_1 that archaeological deposits in west Tennessee can be located through mapping available phosphorus concentrations.

The second hypothesis (H₂) concerned with locating archaeological deposits through mapping the LOI results is rejected for a couple of reasons. The first reason is due to the

63
assumption associated with the use of LOI in archaeology. LOI is used as a proxy measure for organic matter and/or carbon content. When the distribution of LOI results are interpreted for archaeological prospection, a commonly employed heuristic is that a signature of archaeological deposits is the accumulation of organic material, hence higher LOI values likely indicate archaeological deposits. Despite making a minor modification in the LOI procedure, the resulting LOI₅₅₀ range (4.34 – 7.35 %), coupled with the lack of calcium carbonate, indicate the soil matrix of the study area contains a small proportion of organic material. At Ames, areas with higher LOI₅₅₀ did not correspond to areas with archaeological activity, the spatial patterns of LOI₅₅₀ are less discernable than phosphorus, and phosphorus and LOI₅₅₀ have a statistically significant inverse relationship. These results may accurately reflect the archaeological signatures created by different behaviors of people living at Ames or the LOI modification may have noticeably impacted the results. Future analyses could determine the impact the timing of heating samples at 105°C prior to LOI has on the results.

Based on the support of H₁ and rejection of H₂, evaluating the third hypothesis (H₃) (i.e., soil phosphate analysis alone can locate archaeological deposits) was done so through the prediction of evidence in magnetometry data after phosphorus concentrations were known. As discussed in the preceding chapter, the prediction that there would be evidence for Late Prehistoric habitation based on the high phosphorus concentrations was found to be accurate, although ground-truthing excavations have not yet occurred. Without the excavations confirming the presence of Late Prehistoric habitation as predicted, the results of the Kruskal-Wallis and Dwass-Steel-Critchlow-Fligner (DSCF) tests provide converging evidence supporting the presence of Late Prehistoric habitation in the area. The DSCF test found the phosphorus concentrations from Area-B and Area-C to be statistically equal despite differences in modern

land use including portions of Area-B being subjected to extensive excavations. Based in this evidence, H_3 is supported at this time.

Assessing the future use of a technique of any kind requires specific criteria be met to meet the needs of the specific research questions at hand. If soil phosphate analysis was the first survey at Ames to detect the presence of archaeological deposits, the spatial distribution resulting from the current sampling design would have narrowed further surveys to the three areas with archaeological deposits revealed through magnetometry and past excavations, regardless of the presence of mounds at the site. However, at the chosen spatial resolution with 20-40 m sampling intervals, excavations at that scale would not be feasible and little would be known about the archaeological settlement without further surveys. Since the major research objective at Ames is to investigate the regional settlement system during the Mississippian period (AD 1000 - 1600), the desired survey techniques must meet the needs for locating archaeological deposits as well as examining the settlements of various sizes across the landscape. Therefore, magnetometry surveys will continue to be the preferred survey method at Ames due to the spatial resolution and precision desired and the rapid data collection possible with the use of a fluxgate gradiometer. Although this is the case for Ames, the use of soil phosphate analysis for prospection would be viable if magnetometry were not available.

There are two directions for future research based on the findings of this study. The first direction is to examine the spatial distribution of phosphorus concentrations across the study area measuring the different fractions of phosphorus. The current study measured available phosphorus because there have been case studies and experiments suggesting that available phosphorus is likely a better indicator of the variability caused by human activity (Parnell et al. 2002; Terry et al. 2000). Experiments have also shown a strong positive correlation between

65

total phosphorus and Mehlich-2 extracted available phosphorus (Holliday and Gartner 2007; Parnell et al. 2002; Terry et al. 2000). Future research could be carried out to examine the spatial variability of different phosphorus fractions (i.e., organic, inorganic, total) across the study area by analyzing the soil samples with a portable X-ray Fluorescence (pXRF) instrument. Additionally, using the pXRF provides the opportunity to examine the spatial patterns of multiple elements in a single analysis.

The second direction for future research based on the findings of this study is to conduct experiments to assess the effects of different modifications throughout the procedure. This study followed laboratory-based standard practices for soil analyses with the understanding the technique has been modified in the past to be directly used in the field (Terry et al. 2000). Archaeological research is often constricted by feasibility and should be taken into consideration when assessing survey techniques (Banning 2002). Experiments could be carried out to determine whether certain steps in the procedure are necessary or can be modified with little impact on the results. Two examples of modifications that could be tested include different drying procedures, where the same sample could be subjected to oven-drying at 105°C overnight, drying inside a climate-controlled building but not in an oven overnight, and drying outside overnight, as well as the use of glass or plastic supplies for the analyses.

In conclusion, this study has successfully demonstrated the use of the soil phosphate method of Mehlich-2 available phosphate extraction procedure measured by molybdate colorimetry for locating archaeological deposits in soil conditions common throughout the west Tennessee interior uplands (e.g., soil pH ranging from moderately acidic to very strongly acidic, silt to silt loam texture). The spatial distribution of phosphorus concentrations detected three areas of relatively higher phosphorus concentrations that also correspond to three separate areas

66

of archaeological activity at Ames. When examined with magnetometry data, the higher phosphorus concentrations are located near the center of the Mississippian-period town and decrease with distance moving away from the center of the town. Additionally, the phosphorus concentrations near the confirmed residential area at the Ames town are statistically the same as the concentrations in the eastern field within the study area, possibly indicating a second Late Prehistoric residential area.

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Appendix

Weighing Wet Subsamples

1. Dump soil sample into a mound on a piece of aluminum foil on the table.

2. Divide the sample into quarters.

3. Set 3 of the quarters aside.

4. Divide the one remaining quarter into quarters.

5. Set 3 of the quarters aside.

6. Weigh the empty beaker (250 mL) in which the subsample will be dumped and tare the scale after recording the weight of the empty beaker.

7. Dump the remaining quarter into the beaker.

8. Record the weight of the subsample in the beaker.

* if there are a lot of chunks in the subsample, weigh the subsample and then break the chucks up with the straight knife thing and then weigh it again. Record both weights and make a note that it was broken up

* The final mass of the subsamples should be between 40 - 60 grams

9. Once the weight is recorded, loosely place aluminum foil over beaker and make a "hood" so moisture can escape and sediment cannot enter

* At some point when you are about half done or can handle the heat, turn the drying oven to 105° C

10. Once aluminum foil is over the sample, place the sample into the drying oven.

* The oven must be checked once and a while for the first few hours to ensure the temperature of the oven does not exceed 105° C. The optimal temperature is between $100 - 105^{\circ}$

11. Once all of the samples are in the oven, the oven will remain on overnight (12-24 hours)

12. First thing the next morning, turn oven off, but do not open the oven door

13. About 30-40 minutes later, take each sample out one at a time, and record the mass of the sample with the beaker without foil

14. Place foil tightly on the beaker and place the beaker in the refrigerator until ready to grind and sieve

Washing Crucibles

1. Wash crucibles and lids with deionized water - wearing nitrile gloves

- *** crucibles and lids should **not** be touched with bare hands once cleaned
- 2. Wrap drying oven shelf with aluminum foil
- 3. Turn oven on to 105°C

4. Once crucibles and lids are washed, place crucibles and lids on tray wrapped in aluminum foil with tongs to transport from sink to oven

5. Place lids face down on oven shelf with tongs and crucibles upside down leaning on lid to allow air flow underneath crucible

6. Once crucibles and lids are in oven and oven reaches 105°C, set timer for 2 hours.

7. After 2 hours, turn oven off and allow 15 minutes to cool, but do not open the oven.

8. After 15 minutes, place crucibles and lids into the desiccator until ready for use.

Grinding and Sieving Samples

1. Place sample into mortar.

2. Using the pestle, crush the larger grains on the side of the mortar

* Should be more of a crushing (short, quick taps) than grinding (smearing)

3. Attach the catch to the bottom of the sieve

4. Once the sample appears to be the same consistency, dump the sample into the sieve and shake gently to spread sample across whole mesh surface area

5. When it appears nothing more will fall through mesh, dump coarse grains back into the mortar and repeat the crushing

* you want to ensure that you crush as much as you can, but recognize when to stop when

you are only crushing the coarse sand or organic material

6. Place sample back into sieve and shake gently again

7. The remains of the coarse grains are then wrapped in aluminum foil and labeled as such

8. Weigh and record the empty sample bottle in which the sieved sample will be dumped, then tare the weight

9. Place the fine fraction (what is in the catch) onto a piece of foil to funnel into the sample bottle

10. Weigh the sample bottle with the sample in it and record.

11. Place sample bottle in fridge when finished

Loss on Ignition (LOI)

1. If oven is dirty, brush out the oven

2. Turn oven on and set temperature to 360°C

3. Place silver hose connected to oven out the window

4. Record the sample number (**SMPL**) and crucible number that is on the lid (**CRCB**) and measure and record the mass of the empty crucible and lid (**CRCBM**)

* Crucible and lid must remain together because the numbers may differ but the lid is the crucible number recorded

* Always record the crucible number and sample number

5. Tare the mass of the crucible and lid

6. Add 5 grams of sample to crucible with metal pick and record the mass (SSIM)

7. Place the lid on the crucible, set the crucible on metal tray for transport, and wipe off tongs with Kim wipe

8. Wash metal pick with deionized water and dry with Kim wipe

9. Repeat steps 4-7 for the same sample to result in two crucibles per sample (duplicate measurements) and repeat step 8 between each sample

10. Once 3 samples are weighed and recorded (6 crucibles), transport the crucibles to be placed in the oven

* The safest maximum number of crucibles that can be in the oven at one time is 6 crucibles

11. Once the oven has reached 360°C, place heat resistant glove on right hand

12. With the tongs, take the lid off the crucible

13. With the tongs, place crucible in oven

14. With tongs, place lid on top of crucible in oven

15. Repeat steps 12-14 with the heat resistant glove on while closing the oven door (cracked) between each crucible

16. Once the crucibles are in the oven, close the door.

17. Once the oven has reached 360°C after placing the crucibles in the oven, set the timer for 2 hours.

18. After two hours, turn the oven off, crack the oven door when cooling for $\sim 10 - 15$ minutes

19. Once the oven has cooled, take the lid off of one crucible with the tongs and take out of the oven and place onto the transport tray

20. Then, with the tongs, take the crucible out of the oven and place on the transport tray

21. Bring the transport tray to the balance and place the crucible and lid on the balance

22. Weigh and record the mass of the crucible and lid (**SSCMAFH**). Record the first stable weight because the mass will continue to increase as moisture from the air comes in contact with the sample

23. Place the lid and crucible back onto the transport tray with the tongs

24. Repeat steps 12-14 with heat resistant glove on to return crucible into the oven.

25. Once crucible is returned to the oven, repeat steps 19-24 until all crucibles have been weighed and returned to the oven.

26. Once all of the crucibles have been weighed and returned to the oven, turn oven back on and set the temperature to 550°C

27. Once the oven temperature reaches 550°C, set a timer for 3 hours

28. After three hours, turn the oven off, crack the oven door when cooling for $\sim 10 - 15$ minutes 29. Repeat steps 19-22 for each crucible, but when finished recording the mass for each crucible (**SSCMASH**), the crucible can be set to the side until cooled enough to dump and wash.

Laboratory Pre-Analysis Preparation

1. Wipe counters and glassware with kimwipes to prevent contamination

- 2. Fill plastic squeeze bottle with deionized water
- 3. Fill up large beaker with deionized water
- 4. Turn Hach colorimeter on, press "Single Wavelength" and make sure "abs 880nm" is selected
- 5. Prepare diluted Mehlich-2 Soil Extractant solution
- 6. Gather and set up glassware required for the number of samples to be analyzed

7. Set up timers for each analysis step (shaking in agitator, shaking by hand, color development, sample from start to finish)

8. Set up recording sheets at the balance (weight of sample, weight of paper after transfer) and colorimeter (measures for up to four readings for a single sample)

Prepare Mehlich-2 Soil Extractant

1. Under the hood, measure 25 mL of Mehlich-2 Soil Extractant Concentrate with a graduated cylinder.

- 2. Pour the 25 mL Mehlich II into a 250 mL beaker.
- 3. Bring the solution in the beaker to 150 mL by adding deionized water.
- 4. Stir solution with glass wand.

Soil Phosphate Analysis

- 1. Weigh 2 grams of each soil sample on precision balance using weighing paper and record weight (only handle weighing paper with tongs)
 - * close both sides of the balance and wait for measure to become stable
- 2. Transfer sample to 50 mL bottle

3. After sample is transferred to the 50 mL bottle, reweigh the weighing paper and record the weight to determine the loss from transferring

- 4. Add 25 mL of dilute Mehlich-2 solution to sample
- 5. Shake 50 mL bottle in agitator for 10 minutes
- 6. After 10 minutes, filter sample through quantitative ashless filter paper (Whatman 44)
 - * fold filter paper into quarters and pull apart so one side of the filter "funnel" has one layer of paper and the other side has three layers, and then place filter paper in funnel
 - * it will take about 13 minutes for the solution to filter through the paper

7. Extract 1 mL of the filtered solution and dispense to a beaker, then dilute the vial to 10 mL by adding deionized water

- 8. Place a PhosVer3 reagent in the beaker
- 9. Shake for 1 minute by hand
- 10. Allow 4 minutes for color development
- 11. Measure color development four times with spectrophotometer

Measure Color Development with Spectrophotometer

1. Wipe colorimeter vials of the same glass number to make sure there is no dust or anything on the glass

2. Fill 1 vial with deionized water and PhosVer3 pillow as the 'blank'

3. Place the blank into the colorimeter (the diamond on the vial must match with the yellow triangle hazard symbol) and press 'zero'

4. Once extracted sample is ready to be measured, wipe the glass to make sure there are no fingerprints or anything sticky

- 5. Place vial with sample in colorimeter just like the blank and press 'read'
- 6. Wait for ~ 10-30 seconds or up to 1 minute and press read again for a second measure
- 7. Repeat steps 3-6 for each sample