University of Reading

Archival approaches to environment and lifeways: origins of sedentary agriculture at Neolithic Abu Hureyra, Syria, ~8600-7000 cal. BC

PhD Archaeology

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Declaration of Original Authorship

I confirm that this is my own work and the use of other material from other sources has been properly and fully acknowledged.

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Abstract

The development of agriculture ~10,000 years ago in SW Asia, was a fundamental shift in human economy. During the pre-pottery Neolithic B (PPNB), ~8700-6500 cal. BC, plant and animal domestication spread and intensified, alongside technology such as plaster manufacturing. However, whether socio-economic changes were regional, local or site-specific, and how environment related to human resource selection is debated. This research aims to provide new evidence for human-plant-resource use at a site-specific level, using the environmental archaeological archives from PPNB Abu Hureyra, Middle Euphrates, Syria, an ~11ha, long-lived farming settlement.

The taphonomy of different plant proxies means some plant types and parts are over or under-represented in the archaeobotanical record. This research analyses phytoliths which preserve under different conditions to charred plant macro-fossils, previously studied at Abu Hureyra, to provide new perspectives on plant-uses. Phytoliths indicated a variety of vegetation was used consistently as the site expanded from ~8ha to ~11ha, indicating that the ecotonal location between steppe grassland and moist valley bottom facilitated resilient, sustainable resource management.

Plants enter the archaeological record through a range of potential pathways. Therefore, this study evaluates whether some plant remains were deposited by animal dung through the integrated analysis of faecal spherulites and GC-MS to detect faecal biomarkers. Faecal spherulites were identified in ~80% of occupation residues from Abu Hureyra, including a hearth base, indicating the use of dung fuel, corroborated by faecal biomarkers detected by GC-MS. Faecal spherulites were also identified in construction materials, particularly gypsum plasters, indicating significant quantities of dung were used to maintain and expand the built environment. Analysis of the plaster components show remarkable continuity in the manufacturing process across phases of occupation. However, variations in the elemental composition of floor plasters suggest minor intra-site differences, possibly owing to fluctuations in resource availability and/or preferences at a household level.

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Chapter 1. Research Context and Rationale

1.1 Introduction

The emergence of sedentary agricultural societies and lifeways in South-West Asia is one of the most significant periods of change in human history (Bar-Yosef, 2017; Kuijt and Goring-Morris 2002). The domestication of plants and animals marks a transformation in human-environment interactions which had been based on hunting and gathering for over 200,000 years (Jones et al., 2021). The study of early agricultural settlements provides insights into human responses, adaptations and resilience to changing environmental conditions, and the way that humans have shaped the modern world (Goldberg and Macphail, 2006, p. 65; Roberts et al., 2018; Jones et al., 2019). Today, current food systems must undergo another significant transformation to provide food security for everybody around the world and promote environmental restoration (Firbank et al., 2018; Poore and Nemecek, 2018). However, clarifying exactly how people in the past managed environmental resources and responded to or were resilient to changes in the environment remains debated (Roberts et al., 2011; 2018; Jones et al., 2019) and likely developed independently in different regions of SW Asia (Fuller et al., 2011), with new research increasingly highlighting the individual site specific lifeways (Kabukcu et al., 2021). Therefore it is important to understand human-environment relationships at a site-specific level. This research, therefore, aims to provide new insights into plant-use and resource management at one of the earliest large scale agricultural societies in the world, pre-pottery Neolithic B (PPNB) Abu Hureyra, Syria (~8600-6000 cal. BC).

This chapter introduces the PhD thesis by providing an overview of key themes in this research which includes; the environmental and cultural context for the development of early agricultural societies in SW Asia (section 1.1.1) and the challenges and potential associated with studying how people in the past used plants and other resources, with a focus on animal dung and gypsum (1.1.2). Section 1.1.3 outlines the significance and rationale for selection of the key case study in this research, the Neolithic settlement of Abu Hureyra, Syria. Section 1.2 then introduces the key research questions, aims and objectives, which focus on identifying plant-use as early agricultural societies developed, and critically examining potential depositional pathways through which plant material becomes preserved in the archaeological record and assessing how we can use environmental archaeological archives to answer current questions in archaeology. Section 1.3 provides a contextual overview of the development of the Neolithic and farming in SW Asia and highlights key theories and frameworks which are referred to throughout this thesis. Section 1.4 highlights both the challenges and importance of working with environmental archaeological archives. Finally, section 1.5 summarises this chapter and provides an outline of the thesis content and structure.

1.1.1 The development of agriculture in Southwest Asia

The development of agricultural societies in South-West Asia marked a significant shift in human economy, which laid the foundation on which modern society and food systems are based. Over the past decade it has become widely accepted that plant and animal domestication developed independently in different regions of SW Asia (Fuller et al., 2011; 2012; Arranz-Otaegui et al., 2016), as opposed to spreading out from a "core area" (Abbo et al., 2010; Abbo and Gopher, 2017). In a move away from frameworks which attempt to explain the development of agriculture, through "push" and "pull" factors such as climate change, population pressure or increased social complexity, increasingly more nuanced theories attempt to account for the complex symbiotic relationship humans have with plants, animals, and the environment (Hodder 2006; Price and Bar-Yosef, 2011; Zeder, 2012; Jones et al., 2021). Unique cultural identities and traditions are reflected in plant and animal selection at a site or community level (Kabukcu et al., 2021) and small-scale experimentation and innovation at a local and household level played an important role in community wide resilience and adaptation strategies at Çatalhöyük (Bogaard et al., 2017). This research therefore aims to find out if the longevity of other sites of a comparable scale shared a similar style of community or had their own unique ways of interacting with the local environment, plants and animals. New palaeoenvironmental data sets globally and in SW Asia, and syntheses of existing data have provided further insights into human responses (or non-responses) to environmental change (Roberts et al., 2018; Jones et al., 2019). However, the relationship between changes in human ecology with climate and associated environmental change remains uncertain. This study therefore integrates the evidence for the growth and intensification of agricultural society with the most up to date climate records for SW Asia to provide more information about how the development of agriculture may have corresponded to environmental changes at a site-specific level.

1.1.2 Understanding plant-use in the Neolithic

Archaeological plant remains are vital media through which to understand past human ecology and environment associated with the transition from foraging to farming and development of early farming practices, as they inform on a range of themes including consumption practices, fuel-use and construction materials and practices. Understanding the depositional context and taphonomic processes affecting plant assemblages is essential for the analysis and interpretation of the wider significance of an assemblage (van der Veen, 2007; Matthews, 2010; Portillo et al., 2012). In the semi-arid regions of Southwest Asia, archaeological plant macro-fossils (e.g. seeds) are most commonly preserved by charring as opposed to waterlogging. Charred plant macro-fossils are formed under a restricted set of conditions and only represent those plants which have been burned, intentionally or not, and therefore over and under-represent some plant types and economic functions which is examined in detail in section 2.1. Phytoliths, microscopic plant silica bodies, are present in a far broader set of both burnt and non-burnt archaeological contexts, and therefore have the potential to provide information on a wider spectrum of plants which may not be represented in the charred plant macrofossil record (section 2.2). This research analyses phytoliths, as part of a multi-proxy approach to identify past plant use and the types of vegetation and environments utilised by early farming communities.

Plants enter the archaeological record through a multitude of different pathways (van der Veen, 2007; Matthews, 2010; Wallace and Charles, 2013). It is important to understand not only what plants were used but also how they were used and entered the archaeological record. This research therefore investigates the potential depositional pathways of plant remains to better understand what archaeobotanical assemblages represent (e.g. food, fuel, construction) with a specific focus on dung. Animal dung burnt as fuel represents a potential depositional pathway for charred plant macrofossils (Miller 1984; Miller and Smart 1984), which it is now well established survive digestion (Charles 1998; Wallace and Charles 2013; Valamoti 2013). Furthermore, plant remains such as phytoliths, which do not require burning for preservation are often preserved in non-burnt dung deposits in SW Asia (Matthews 2005; Shillito et al., 2013a; 2013b; 2013c), which may be difficult to detect during excavation (Shillito et al., 2011). Therefore, this research aims to assess whether dung is a potential depositional pathways for plant remains at Abu Hureyra through the analysis of faecal spherulites, microscopic calcitic particles formed in the guts of animals, most commonly ruminants, which provide an indicator of dung in the archaeological record (Brochier 1992; Canti, 1997; 1998; 1999), combined with gas chromatography and mass spectrometry (GC-MS) to detect organic compounds which are faecal biomarkers (Bull 2002; 2005).

1.1.3 Introduction to case study rationale

Tell Abu Hureyra is situated in the Middle Euphrates Valley, N. Syria; a key region for understanding the development of sedentary farming villages due to the density and complexity of sites and networks in this region (Cauvin, 2000; Moore et al., 2000; Molist, 2006) (Chapter 3.1). Sites in this region document the transition from mobile hunter-gatherers to sedentary farmers and hold key evidence on the early management and domestication of plants (Willcox, 2005; Willcox et al., 2008). Located on the boundary of several ecozones, occupants of Tell Abu Hureyra would have had access to a variety of resources (Chapter 3).

Abu Hureyra was occupied during the Epi-Palaeolithic (~11,000 cal. BC) as a small, sedentary, hunter-gatherer community, with archaeobotanical evidence argued to represent the world's earliest pre-domestication cultivation (Hillman et al., 2001), which has since been contested (Nesbitt, 2002, p. 119; Colledge and Conolly, 2010, p. 135). The site later (~8600 cal. BC) developed into a substantial sedentary agricultural village, up to ~11ha, occupied for more than 2000 years and spanned the transition from the mid to late pre-pottery Neolithic B (PPNB hereafter) (Moore et al., 2000). The PPNB is a significant period to understand human resource management, as substantial settlements emerged across SW Asia, such as Çatalhöyük, Ain Ghazel, Jericho and Abu Hureyra which required previously unprecedented quantities of resources to sustain increasing populations and the built environment. This research, therefore, aims to identify plaster manufacturing practices at Abu Hureyra through integrated microfossil and geochemical analysis of floor plasters, to inform on variations over time and in different spaces.

As one of the first excavations to employ wide scale, systematic flotation, combined with favourable preservation conditions, the site yielded significant quantities of charred plant material (Moore et al., 2000), particularly for the Epipalaeolithic phases where charred plant macro-fossils are rare in the region (Arranz-Otaegui et al., 2017). Following the construction of the Tabqa dam in the mid-1970s, the site was flooded and is no longer accessible. Fortunately, a wealth of material was recovered and archived by Professor Andrew Moore and colleagues during rescue excavations in 1972/3. These archival collections provide the opportunity to continue research into early farming communities and lifeways at this key site and region. The research presented here investigates the potential contribution of archival environmental archaeological material such as this to current research and to archival practice more widely, as demonstrated in a recent study by Smith et al., (2022).

1.2 Aims and Objectives

The development of agricultural societies was a key period in human history and changed the dynamics and evolutionary paths of plants and animals which most of the world rely on today (Watkins, 2010; Scott, 2017; Jones et al., 2021). However, the extent to which these significant changes were responses to external factors, intentional selection by people or a more complex symbiosis between humans, plants, animals and the environment remains contested (Smith, 2007; 2011; Zeder, 2012; Stiner and Kuhn, 2016; Bar-Yosef, 2017; Roberts et al., 2018; Jones et al., 2021). The ways in which people managed environmental resources and whether changes in human economy and culture occurred at a regional, local, site specific or even household level also continues to be debated (Fuller et al., 2012; Bogaard et al., 2017; Wallace et al., 2019; Kabukcu et al., 2021; Weide et al., 2022).

This research, therefore, aims to provide new insights into the development of sedentary agriculture at Abu Hureyra, through a multi-disciplinary, integrated study of plant-use and resource management. This research also aims to employ and evaluate methodological approaches that can be used with environmental archaeological archives, with a focus on Epipalaeolithic and Neolithic sites in SW Asia where new excavations are not possible, through the case study of Abu Hureyra, Syria. Key questions addressed in this research are:

1. In what ways do plant-use and resource management change during the development of agriculture at PPNB Abu Hureyra?
2. Was animal dung a potential depositional pathway for plant remains at Abu Hureyra?
3. What are some of the most effective ways of using the environmental archaeological archives from Abu Hureyra to inform on current questions in archaeological research?

The specific aims and objectives in this research are to;

Aim 1. Assess what plants were used and how plant-use changes over time and for different purposes during the development of agriculture at Abu Hureyra. The objectives are to:

i. analyse phytoliths, microscopic plant silica bodies, which represent different vegetation types (e.g. woody plants, grasses, reeds, sedges), and parts of the plant (e.g. wood bark, wood leaves, grass stems, grass husks) from a range of time periods and materials at Abu Hureyra.
ii. integrate new phytolith analysis with previously analysed charred macrofossil (Hillman, 2000; de Moulins, 2000) records to provide new perspectives on plant-use, as phytoliths are formed and preserved under different conditions to charred plant macro-fossils (Piperno, 2006).

Aim 2. Identify whether animal dung was a component of mixed occupation residues, fuel and construction materials at Abu Hureyra, and could have contributed to the charred macro-fossil assemblage, as suggested by Miller (1996) for the Epipaleolithic phase of the site. The objectives are to:

i. identify and quantify faecal spherulites, microscopic calcitic particles, which form in the guts of animals, mostly ruminants, and are excreted, providing an indication of dung in archaeological deposits (Brochier et al., 1992; Canti, 1997) in occupation resides and construction material.

ii. identify faecal biomarkers by Gas Chromatography and Mass Spectrometry (GC-MS) analysis of occupation residues and plaster floors fragments as 5β-stanols and bile acids can be used to determine whether deposits are of faecal origin; elevated 5β -stigmastanol and coprostanol are indicative of ruminant (herbivore) and omnivore faeces respectively (Eneroth et al., 1964; Bethell et al., 1994; Evershed et al., 1997) and are confirmed through the identification of bile acids (Elhmmalli et al., 1997).

Aim 3. Identify plaster manufacturing practices at Abu Hureyra to inform on technological developments and resource-use. The specific objectives are to:

i. identify the elemental composition of floor plaster fragments and occupation residues by pXRF to identify variations over time and in difference spaces.
ii. assess variations in vegetation types and plant parts used in construction material, identified through phytolith analysis (e.g. Tsartsidou et al., 2009; Portillo et al., 2012).
iii. identify whether dung is a component of construction material by analysing faecal spherulites and identifying dung biomarkers through GC-MS, the presence of which could indicate the deposition of plant remains through dung.

Aim 4. Compare and evaluate methodologies for detecting dung in the archaeological record. The objectives are to:

i. conduct a methodological comparison of different methods to quantify faecal spherulites to assess comparability and the interpretative value of faecal spherulite concentrations in archaeological deposits
ii. test the relationship between the positive identification of dung spherulites and faecal biomarkers identified by GC-MS
iii. identify the elemental concentrations (by pXRF) associated with the detection of dung

Aim 5. Demonstrate the ways in which archaeological environmental archives can be used for addressing current questions in archaeology. The objective is to:

i. conduct the analyses (aims 1-4) on material from Abu Hureyra in order to evaluate the strengths and challenges of working with archival environmental samples

The original aims and objectives of this research were significantly impacted and modified and owing to restrictions caused by the Covid-19 pandemic as outlined in the Covid-19 impact statement which accompanies this thesis.

1.3 The development of farming in Southwest Asia: theories and frameworks

This research primarily focuses on the expansion and changing economies and lifeways of established Neolithic agricultural settlements during the mid to late pre-pottery Neolithic B (PPNB) (8700-7000 cal. BC), rather than the shift from hunting-gathering to farming. However, since the "origins" of agriculture have received so much academic attention, the wealth of theories and frameworks used to discuss how agriculture came about provide a logical starting point for understanding the trajectories on which early farming villages evolved and agricultural practices intensified.

This section discusses the development of farming in SW Asia and critically reviews the theories and frameworks which have been utilised to examine the development and expansion of agriculture in SW Asia. The theories and frameworks critically discussed in this section form the basis from which to evaluate the original data generated in this research to provide new insights into the complex interactions between humans, plants, animals and the environment, which laid the foundations for the societies and food systems of today.

1.3.1 Contentions and debates in the emergence and spread of agricultures

The timing and locations of the domestication of different plants and animals continues to be refined by new evidence. The chrono-cultural periods discussed and referenced in this research broadly follow those outlined in Asouti and Fuller (2013) (Table 1.1), although in some cases regional variations and exceptions are also considered. It is now widely accepted that agriculture developed independently and in diverse ways in different regions of SW Asia (Fuller et al., 2011; 2012; Arranz-Otaegui et al., 2016), although some sustain arguments that plant domestication occurred in a "core area" from where it spread out (Abbo et al., 2010; Abbo and Gopher, 2017). The reasons why agriculture developed also remain contested, in particular the extent to which the domestication of plants and animals was the result of intentional human selection or part of a co-evolutionary process (Zeder, 2015).

The relationship between climate change and subsequent changes in vegetation distribution and availability with significant changes in human economy are also unresolved (Bar-Yosef, 2017; Roberts et al., 2018, Jones et al., 2019). Climate events are referenced below and discussed in more detail in section 3.2. Demographic changes have also been argued to drive or facilitate the development of agricultural societies (Cohen, 1977; 2009), but disentangling cause from consequence is complex. Human cultures and changing social structures are also argued to play an important role in emergence of sedentary societies and agricultural practices (Kuijt and Goring-Morris, 2002; Hayden, 2009; Watkins, 2010). These arguments are evaluated and integrated below, as they are not mutually exclusive, and likely were all factors in a complex web of human, plant, animal, environment interactions (e.g. Hodder 2012) which led to the formation of sedentary agricultural societies, and the subsequent development and growth of agricultural practices thereafter, which is the focus of this research.

Table 1.1 Chronological framework for the chrono-cultural horizons discussed in this research. Table adapted from Asouti and Fuller (2012, p. 150, Table 1)

Cultural period	Chronology (years cal. BC)	Key Attributes
Late Epipalaeolithic	~12,000-10,000	• Often small settlements, semi-sedentary? "base camps" • hunting-gathering
Pre-pottery Neolithic A (PPNA)	~10,500-8700	• PPNA semi-sedentary? settlements and communal structures • hunting-gathering, pre-domestication cultivation
Early PPNB	~8700-8200	• Increased sedentism and permanent dwellings • hunting-gathering, pre-domestication/mixed cultivation, first domesticated "crop packages"
Middle PPNB	~8200-7500	• Increased community size, substantial rectilinear mudbrick architecture across SW Asia (particularly in N. Levant) • diverse subsistence practices – continued hunting and gathering of wild plants and animals. Increasing evidence for managed and domesticated plants and animals
Late PPNB/PPNC	~7500-6500	• Increased settlement size across SW Asia • expansion of agricultural practices including the more widespread adoption of domesticated crops and completion of domestication process • caprine herding, increased spread of domestic cattle and pig

1.3.2 Climate and Environment

Human selection of economic resources can be both intentional and habituated, both of which are argued to have contributed to domestication (Zohary, 2004). A change in climate and its impact on vegetation has long been argued to have been a factor which encouraged humans to start intensifying their management of the environment through cultivation, eventually leading to the domestication of plants and animals (Moore and Hillman, 1992; Bar-Yosef, 2011). The warmer, wetter conditions during the Bølling-Allerød, ~12,750 – 10,750 cal. BC (~14,700 – 12,700 cal. BP) (Alley et al., 1993; Weaver et al., 2003) likely resulted in more abundant resources, for example the expansion of steppe grasslands, parklands and forests which could have encouraged and enabled hunter-gatherers to become increasingly sedentary (Bar-Yosef and Belfer-Cohen, 1989; Bar-Yosef and Meadow, 1995). The subsequent onset of the colder, drier conditions of the Younger Dryas (~11,050 – 9550 cal. BC, ~13,000 – 11,500 cal. BP) which likely depleted stands of wild cereals, and created a strain on resources, has been argued to have increased mobility (Byrd, 2005) and also to have been the catalyst which stimulated cultivation (Hillman et al., 2001).

Evidence from Abu Hureyra has been previously interpreted as suggesting that climate deterioration, which led to the reduced availability of resources during the Epipalaeolithic phase of the site encouraged humans to start managing the plants in the local environment more intensively, which led to cultivation (Moore et al., 2000). The argument for cultivation during the Epipalaeolithic at Abu Hureyra was largely constructed based on an increase in the proportions of small-seeded legumes and grasses, interpreted as potential arable weeds, as well as the presence of a low number of domestic-type grains, following the onset of the cooler dryer conditions of the Younger Dryas (Hillman et al., 2001). However, a reassessment of the archaeobotanical assemblage argues the variety of plants is consistent with a broadening of the diet in response to deteriorating conditions, and that the domestic type grains could also be present in wild populations (Nesbitt, 2002; Colledge and Conolly, 2010). Naomi Miller also argued that at least some of the charred seeds from Abu Hureyra could have been deposited by animal dung burnt as fuel (Miller 1996). The evidence for pre-domestication cultivation has also been more recently called into question at PPNA sites, including those in the Middle Euphrates (Weide et al., 2022).

It has been argued that agriculture was not possible before the start of the more stable climate conditions at the start of the Holocene (Richerson et al., 2001; Bettinger et al., 2009). However, whether agriculture was possible or not before the Holocene does not explain why people shifted towards more sedentary agricultural systems. Especially since the foundations for sedentary agriculture are well documented before the start of the Holocene, with increased evidence for sedentism, and a sense of place and ritual (Schmidt, 2011; Dietrich et al., 2013). This study, therefore, focuses on the ways in which agricultural practices expanded at a site-specific level, and assesses whether lower impact and local changes environment within the Middle Euphrates Valley and immediately accessible environs (section 3.2) influenced the trajectory of human socio-economic practices at Abu Hureyra following the establishment of a sedentary agricultural community.

1.3.3 Frameworks for understanding human responses to resource availability

A range of frameworks have been developed or “borrowed” from other disciplines to help understand the circumstances under which agriculture developed (Smith, 2007; 2015; 2016; Zeder, 2008; 2012; 2015). This section evaluates some of the key frameworks which are relevant to this research and applied to help interpret the data generated by this study.

The Diet Breadth Model (DBM) suggests that high-return resources are prioritised and favoured, and therefore when those resources become depleted, subsistence patterns are likely to shift towards a broader spectrum of lower ranked resources (Winterhalder and Kennett, 2006). The Broad-Spectrum Revolution (BSR), initially proposed by Flannery (1969), suggested that late-Pleistocene hunter-gatherers broadened their diets as a response to the decrease in higher order resources (Stiner et al., 2000). At the Neolithic site of Aşıklı Höyük, the transition to farming, specifically caprine management, is associated with a rapid decline in the meat consumption of a variety of wild animals and small game (Stiner et al., 2014; Itahashi et al., 2021). In contrast, at Abu Hureyra, prior to the widespread adoption of caprine management, wild gazelle were the dominant meat source, and there is little evidence in the zooarchaeological record that inhabitants regularly exploited small mammals, fish and birds, despite their likely abundance (Legge and Rowley-Conwy 2000). Traditionally, the domestication of cereals and legumes during the Neolithic has been associated with a narrowing of the diet (Moore et al., 2000; Weiss et al., 2004; Savard et al., 2006), though increasingly this interpretation is being called into question (Wallace et al., 2021).

Although, while DBM provides a framework to understand the context in which lowed ranked resources were incorporated into human diet, it has been argued that it does not explain why domestication came about as a result of that transition (Smith, 2015). Furthermore, initial domestication has often occurred in resource rich areas as a result of intentional human enhancement (Smith, 2011), and therefore, diversification and innovation need not occur as a response to resource scarcity, but can happen in zones of resource abundance, which enables increased experimentation and human engineering of the environment to promote greater productivity (Zeder, 2012). This research, therefore, explores potential depositional pathways for the plant remains recovered from Abu Hureyra, with a focus on animal dung burnt as fuel, as identified at comparable large Neolithic settlements (Fairbairn et al., 2005; Matthews 2005). A better understanding of whether plant remains represent human food, animal fodder or fuel enables more robust interpretations of the broadening and narrowing of plant-use through different phases of occupation and environmental conditions (Miller 1996).

Niche construction theory (NCT) refers to the ability of organisms to influence evolution of their own and other species through modification of their environment (Laland and O'Brien, 2010). NCT provides an evolutionary and behavioural context with which to understand the development of agriculture, arguing that humans modified plants and animals to the extent that they incurred morphological and genetic changes to become domesticated (Smith, 2007, p. 195). Within a niche construction framework, it is proposed that the amelioration of environmental conditions at the start of the Holocene enabled the development and expansion of the agriculture communities and villages of the Neolithic (Zeder, 2012). Smith (2016) argues that NCT provides a better fit to understanding domestication than standard evolutionary theory (SET), whereby species adapt in response to environmental change. It is important to understand the symbiosis between people, plants, animals and their collective influence on the environment in the past to assess current environmental thresholds and predict future environmental responses. This research, therefore, assesses plant-use during the development of agriculture at Abu Hureyra as a key site for understanding early farming economies and lifeways.

1.3.4 Demographic changes

Demographic changes have been argued to be a factor that may have stimulated the changes in human economy that led to agriculture, as an imbalance between human populations and their choice of resources and commitment to work can lead to a depletion of resources which can encourage changes in habit (Cohen, 1977; 2009). Bettinger et al. (2009), however, argue that while demographic changes may be important, they do not answer questions about when, why or how agriculture began. A demographic increase preceding sedentism and agriculture could have created more overlaps and connections between groups which stimulated cultural transmission of ideas and technology (Shennan, 2001). Demographic changes likely also played an important role in the growth and expansion of later PPNB Neolithic settlements, and possibly the increased adoption of agricultural practices, whether as a cause or effect.

During the mid-late PPNB, many farming settlements across SW Asia increased in settlement size and capacity. Neolithic Abu Hureyra, for example, has two major phases of occupation, period 2A (~8600-7300 cal. BC) and period 2B (7300-6200 cal. BC) which are defined by the shift from wild gazelle to managed caprines (discussed in Chapter 3), and an increase in settlement size which was estimated to have grown from 8ha to 11ha (Moore et al., 2000). However, it remains unclear whether the foundational agricultural systems in place during period 2A, based on some domesticated cereals and low numbers of managed caprines, facilitated the expansion of the settlement, or whether the increased pressure on resources led to an intensification of managed resources. This research, therefore, employs environmental archaeological methods, which are widely utilised in the study of early agricultural societies in SW Asia, but prior to this study, not yet employed on material from Abu Hureyra, to identify changes in economy, resource-use and management between periods 2A and 2B to clarify the extent to which economy and lifeways were sustained or changed during the development and growth of Abu Hureyra.

1.3.5 Human intentions: cultural and social perspectives on economic changes

Human societal and technological development is intrinsically linked with symbolism, culture and belief systems (Boivin, 2000; Hodder 2012). Social developments are argued to have been a factor that encouraged the domestication of plants and animals and culminated in the birth of agricultural societies. Watkins (2010) suggests that an increased sense of place, religion and ancestry encouraged sedentism. While Haydon (2009) argues that the social role of feasting led to the intensification of food production. During the Palaeolithic, human brains evolved to enable people to live and cooperate in larger groups (Gowlett et al., 2012). In support of these arguments, sites such as Göbekli Tepe demonstrate that people were able to coordinate and cooperate in large groups prior to the development of agriculture and also provides evidence of feasting (Schmidt, 2011; Dietrich et al., 2012; 2013). The PPNB is characterised by social and cultural factors, which are discussed below and cannot be separated from the economic decisions made by inhabitants. Plant and animal selection from earlier sites has been shown to reflect the unique cultural identity at a site-specific level (Kabukcu et al., 2021). This research, therefore, provides new evidence for changes in plant-use at a site-specific level (Aim 1), and crucially, through archaeological and environmental archives (Aim 5), at Abu Hureyra, which is no longer accessible.

1.3.6 The emergence, definition and expansion of the Pre-Pottery Neolithic B (PPNB)

The PPNB is characterised broadly by lithic technology, rectangular architecture, "skull cult" rituals and the establishment of different forms of agricultural societies across SW Asia (Asouti, 2006, p. 90 and references therein). During the PPNB, substantial settlements, often termed "mega sites" became more widespread throughout the region. Although establishing contemporaneity between different areas of a site and thereby size and population estimates are complex and remain widely debated (e.g. Kuijt, 2000; Asouti, 2013; Bernardini and Schachner, 2018). However, methods are being increasingly refined to address this issue (Birch-Chapman and Jenkins 2019). Although, as highlighted by Akkermans and Schwartz (2003, p. 58), whilst large settlements are frequently seen as characteristics of the PPNB, they tend to be the exception, as most people across SW Asia would have continued to live in smaller, possibly more mobile settlements.

The appearance and increase in numbers of large settlements, however, demonstrates significant investment in the built environment during the PPNB. Substantial quantities of raw materials were required to maintain settlements, particularly for the manufacturing of plasters which were used widely in SW Asia during the PPNB (e.g. Clark 2012). While there is evidence for plaster manufacturing pre-agriculture, from ~ 12,050 cal. BC (~14,000 cal. BP) at Hayonim Cave (Kingery et al., 1988; Chu et al., 2008), the quantities used for construction during the PPNB for construction, vessels, decoration and covering burials, were unprecedented (e.g. Moore et al., 2000; Molist, 2012).

Large PPNB settlements would have required a considerable increase in raw material acquisition, energy (i.e fuel) and labour requirements (discussed further in Chapter 8).

The PPNB is of especial significance because many of its defining features persist in the world today providing examples of the sustainable use of plants, materials and ideas. For example, the rectilinear buildings constructed at Neolithic Abu Hureyra from ~8600 cal. BC, were built on the same alignment as in the modern village of Abu Hureyra, using locally sourced materials such as mudbrick and reeds (Moore et al., p. 263). It has been argued that the PPNB lifeways and associated traits started in the Middle Euphrates region (Cauvin, 2000), placing Abu Hureyra in the heartland of this significant shift in human culture, economy and lifeways.

In light of new excavations across wider regions of SW Asia, more polycentric and protracted models of key developments are now more widely accepted (Ibáñez et al., 2018). The rise of larger, more sedentary settlements required increased levels of inter-personal and group interactions, with more complex strategies and systems to manage resource distribution and ownership. The *Koine* model proposed by Cauvin, argues for a shared Neolithic culture across the Levant, based on similar cultural practices and beliefs, which may manifest at different scales of identity, i.e. regional, settlement or household (Cauvin, 1994; 2000). Bar-Yosef and Belfer-Cohen proposed a model whereby economic and social interaction spheres along the Levantine corridor account for similarities in materials and culture (Bar-Yosef and Belfer-Cohen, 1989). Asouti (2006) suggests that a focus on local socio-economic contexts of group interactions and population movement, which focuses on social organisation and exchange, is a more useful framework through which to understand the PPNB. Borrell and Molist (2014) investigate the social interaction between sites in the Euphrates which share common features and technology, but also demonstrate that there was not a homogenous regional culture, evidenced by inter-site variation in material culture and mortuary practices (Borrell and Molist, 2014, p. 226).

1.3.7 Integrated explanations and panarchy theory

The development of Neolithic lifeways was likely a complex process which encompassed a diverse set of interactions at different levels and with numerous agents, rather than an adaptive response (Asouti, 2006, pp. 119–120). Panarchy theory predicts that ecological uncertainty leads to innovation and that flexibility is more limited by complex social obligations (Gunderson and Holling, 2001; Holling, 2001). Through a panarchy framework, the longevity of the Neolithic site of Çatalhöyük, Anatolia, has been attributed to 1) its diverse crop base, which promoted innovation, 2) the modular social structure which allowed small-scale experimentation and innovation and 3) the agglomerated social morphology, which facilitated the scaling up of developments across the wider community

(Bogaard et al., 2017). Panarchy theory and its application at Çatalhöyük, which has been intensively and rigorously excavated over more than two decades, provides a useful comparison to understand the socio-economic dynamics of Abu Hureyra, which, although set in a different region, shared many of the characteristics which have been argued to have contributed to the longevity of the site. This research assesses the extent to which environmental changes or uncertainty may have affected the inhabitants of Abu Hureyra through integrated analysis of climate records, and site-specific uses of plant resources to provide new insights into successful strategies which contributed to the site's longevity of over 2000 years.

Different plants and animals were managed and eventually domesticated in multiple regions across SW Asia (Fuller et al., 2012) and interactions between different groups which were developing at different rates, with their own cultures, facilitated the common Neolithic trajectory (Ibáñez et al., 2018). Increasing numbers of case studies demonstrate that economic choices; the selection of plant and animal resources are site-specific, rather than determined entirely by local resource availability, which is interpreted as reflecting distinctive cultures and traditions (Kabukcu et al., 2021). This research, therefore, provides new data to clarify in more detail choices made by the inhabitants of Abu Hureyra related to plant-use and fuel selection (Aims 1, 2 and 3).

1.4 Archival challenges in archaeology

The nature of archaeological field excavations is that destruction of the archaeology is often inevitable to understand the nature and formation of the site. Archaeological excavations often produce significant quantities of artefacts, ecofacts and environmental samples, a subset of which are recovered and retained for further analyses.

Archaeological archives provide a valuable medium through which to investigate current questions in archaeological research. Archival material is also important as new methods are developed because previously analysed material can be revisited, reassessed and new analytical techniques applied to address current questions in archaeological research, particularly in light of new evidence which shapes debates. However, in England, the storage of excavated material is a major challenge facing archaeology. In 2012 there was an estimated national total of around 9000 undepositable archaeological archives, and of 150 surveyed museums, less than a quarter could provide detailed information about the collections they held, and only around a third had a designated archaeological curator employed (Edwards, 2013).

This section, therefore, evaluates some of the challenges currently facing archaeology, specifically the recovery and long-term curation of environmental archaeological material. Examples are provided to illustrate the value of environmental and archaeological archives, as well as examples of other challenges facing access and accessibility of archaeology.

SW Asia has some of the world's earliest evidence for farming and is a key region for understanding the initial management and domestication of plants and animals (sections 1.1, 1.3). At Abu Hureyra, the charred plant assemblage from the Epipalaeolithic period of occupation at Abu Hureyra has been interpreted as evidence for pre-domestication cultivation, including the world's earliest domestication of rye (Hillman et al., 2001), however, a subsequent reassessment of the material, has reinterpreted the same assemblage as a broadening of the diet in response to climate deterioration and its impact on local vegetation and resource availability (Colledge and Conolly, 2010). New syntheses of a number of archaeobotanical datasets have concluded that there is no evidence for a narrowing of human diet as agriculture developed across SW Asia (Wallace et al., 2021), contrary to previous interpretations that Palaeolithic hunter-gatherers exploited a broad spectrum of plant resources which narrowed with the development of domestic plants and animals (Willcox et al., 2008). This research identifies plant-use and resource management at Abu Hureyra, through the analysis of techniques which have not previously been employed on material from the site (section 1.1).

Access to many key regions for new excavations has been restricted. The COVID-19 pandemic restricted domestic and international fieldwork opportunities for many archaeologists and projects worldwide. The civil war in Syria has prevented new excavations and projects working in the region, while the relative stability of Jordan has led to sustained and relatively intensive archaeological investigations in comparison with some surrounding countries. Current evidence suggests agricultural practices developed independently in different parts of SW Asia (Fuller et al., 2011), and that pathways towards domestication were often highly localised and reflected site-specific cultural preferences (Kabukcu et al., 2021) rather than dictated by the availability of local resources. Therefore, it is crucial to understand the development of agricultural societies in a range of different regions and at a site-specific level, to fully understand resource acquisition strategies and strengthen current interpretative models and frameworks.

The research conducted in this PhD is primarily based on environmental archaeological archival material from Neolithic Abu Hureyra in Syria, which was flooded following the construction of the Tabqa Dam, and therefore, there is no possibility of further excavations. Dam construction possesses a severe threat to cultural heritage and archaeology (Cunliffe et al., 2012). The Euphrates is the second longest river in the Middle East and North Africa and has the highest number of dams in this region which has caused around 880km^2 out of 2800km^2 of the floodplain to be submerged

(Marchetti et al., 2019, p. 21). When the Tabqa dam was constructed, an archaeological survey covered the entire area which was predicted to become flooded, which identified 47 sites which are now submerged under Lake Assad (Marchetti et al., 2019, p. 19, Table 1).

Due to the significance of Abu Hureyra and its imminent destruction, a wealth of environmental samples and artefacts were recovered and are now housed in museums and institutions around the UK (Moore et al., 2000, p. 547). This research provides a case study to evaluate and demonstrate the value of environmental archaeological archival material to continue research in key regions and sites which are no longer accessible. This research also endeavours to highlight some of the challenges associated with working with archival samples in order to make recommendations for the best practices in the recovery and storage of archaeological material in current and future excavations.

1.5 Summary and overview of thesis structure

This chapter has provided a critique of current theories and frameworks in the study of early agriculture and sedentism, an outline of methodological challenges arising and has laid out the aims and objectives of this research. Chapter 2 provides an in-depth critique and rationale for the methodological selection and laboratory processes chosen in order to meet the aims and objectives of this research. Chapter 3 places the key case study, Abu Hureyra, into a regional and environmental context, and highlights the relevant previous research which is integrated with new data in this thesis. The materials selected for each methodological approach are summarised and justified in Chapter 4, which also describes the analytical techniques, methods and protocols employed in this study.

Some of the key results of this research have been written up and presented in the style of journal articles (Chapter 5, 7 and 8), and therefore contain their own introduction, method and discussion sections, but have been formatted to be consistent with the rest of the thesis and cross-refer to other chapters, figures and tables where useful.

Chapter 5, “A quantitative comparison of methods for analysing faecal spherulites”, has been written in the style of a journal article and provides a foundation for the method development (Aim 4i) and new insights into detection of animal dung and early animal management through spherulite analyses conducted in this research (Aim 2i, Aim 3iii).

Chapter 6 presents the results of investigations into the taphonomy of phytoliths and spherulites and evaluates cost effective rapid screening methods to identify material with high concentrations of phytoliths and potentially dung.

Chapter 7, “New perspectives on plant-use at Neolithic Abu Hureyra, Syria: an integrated phytolith and spherulite study” is written as a journal article and is currently under review at *Vegetation History and Archaeobotany*. This paper presents some of the key results of the phytolith and spherulite analysis and provides new insights into the types and parts of plants used at Abu Hureyra (Aim 1i), assesses the potential role of dung as a depositional pathway for plant remains (Aim 2i), and integrates the new data with the charred plant macro-fossil assemblage previously analysed at Abu Hureyra (Aim 1ii).

Chapter 8, “Insights into resource management and technological development through microbotanical and geoarchaeological characterisation of floor plasters from Neolithic Abu Hureyra, Syria, 8600-6000 cal. BC” is under review at *Quaternary International*. This paper characterises floor plasters through the integrated analysis of phytoliths, faecal spherulites and pXRF to identify construction manufacturing practices and technological changes (Aim 3i, Aim 3ii, Aim 3iii and Aim 4iii).

Chapter 9 presents the results, interpretation, and discussion of the detection of faecal biomarkers by GC-MS in plasters and occupation residue sediments to identify whether dung was a potential depositional pathway for plant remains, to inform on the use of secondary animal products and compare methodologies for the detection of dung in the archaeological record (Aim 2ii, Aim 3iii, Aim 4i, Aim 4ii and Aim 4iii).

Chapter 10 integrates the results presented in Chapter 5 to 9, with excavation reports and other material analysis from Abu Hureyra, to add new information about specific contexts, spaces and phases at Abu Hureyra. Material from the key case study, Abu Hureyra, is the focal point of the analyses reported in Chapters 5 to 8, and therefore provide a case study for the potential of archival archaeological and environmental materials to address current archaeological questions (Aims 5i), which is further evaluated in the integrated discussion (Chapter 11) (Aim 5ii).

Chapter 11 evaluates the results of this research within a regional environmental and archaeological framework and reflects on the contribution of the new data presented here to contribute to current debates in archaeology, and the specific aims of this research, which have been outlined in Chapters 1 and 2. Finally the conclusion (Chapter 12) summarises the main findings of the thesis and recommends further research which could build on the work presented in this thesis.

Chapter 2. Methodological rationale and critical evaluation of techniques

This chapter provides a rationale for the selection and integration of methodological approaches applied in this research. The strengths and weaknesses of each method for assessing past human-environment interactions are critically evaluated, and potential challenges are highlighted, alongside proposed mitigation strategies. A running theme in this chapter is the examination and development of ways in which the selected methodologies complement one another and are integrated to provide a better understanding of the interconnected themes of culture, society, diet and resource management as people shifted towards more sedentary agricultural lifeways in the Neolithic.

This chapter first discusses key issues in archaeobotany (section 2.1), with specific reference to charred macrofossils which are a key proxy to understand domestication and changing human lifeways in the Neolithic. In section 2.2, the rationale for the selection of phytoliths in this study is evaluated. The chapter then discusses and evaluates methods employed in this research to investigate the potential depositional pathways of plant remains analysed previously and in this study (section 2.3), with a focus on the significance of dung in the archaeological record (section 2.3.1). The application of micromorphology for understanding taphonomy and the economic and social significance of plant remains in the archaeological record, as well as its potential to identify dung, which was an initial aim of this research (see Covid-19 impact statement), is discussed in section 2.3.2. Methods for the detection of dung applied to this research are evaluated, focussing on faecal spherulite analysis (section 2.2.3) and GC-MS for the identification of dung biomarkers (section 2.3.4). Finally, this chapter evaluates the application of elemental analysis through portable X-Ray fluorescence (pXRF) (2.4) as a screening method and to inform more broadly on uses of materials within the site and wider environmental resource management choices.

2.1 Problems and potential in archaeobotany

Archaeobotany is a key approach through which to better understand how humans interacted with their environment and available resources in the past with the potential to inform on a range of environmental, economic, social and cultural themes (Hastorf, 2016). Past plant proxies commonly analysed in archaeological investigations include plant macrofossils (charred wood/seeds/chaff, water-logged and desiccated), phytoliths, pollen and starch (Pearsall, 2015). Each of these represent diverse plant species, materials and parts, as well as plant uses, related activities and depositional events, and are subject to a range of different taphonomic and preservation conditions, all of which are crucial to consider when interpreting an archaeological assemblage (e.g. van der Veen, 2007; Matthews, 2010). This section focuses on the problems and potential of the past plant proxies most relevant in this study, that is, charred plant macrofossils and phytoliths.

Charred plant macrofossils, which encompass seeds, chaff, glume bases, charcoal and charred food stuff, are commonly recovered from SW Asia archaeological sites and are vital for understanding early cereal domestication as well as wild plant use (section 1.3). Charred plant macrofossils are also particularly relevant to this study, they have been studied extensively from Abu Hureyra, the key case study in this research, section 1.1.3, Chapter 3 (de Moulins, 2000; Hillman et al., 2001; Colledge and Conolly, 2010). However, charred plant macrofossils only represent a small fraction of the plant uses and functions within a site, summarised in Table 2.1.

Carbonised plant remains only represent those that have been burnt at low temperatures, <400-500°C (Boardman and Jones, 1990; Matthews, 2010, 2020; Colledge and Conolly, 2014), and have been shown to represent less than 20% of an assemblage of desiccated plant remains (van der Veen, 2007, p. 977). Agricultural by-products from cereal processing, weed seeds and plants used for fuel are generally overrepresented (Hillman, 1981). Some plant types or parts are underrepresented or absent from charred assemblages. These include cereal chaff, an important by-product to identify processing strategies, which burns quickly (Boardman and Jones, 1990, p. 7). Leaves, roots and tubers, often roasted by direct heat (Colledge, 1991; Colledge and Conolly, 2014, p. 194) and oily seeds which may explode, are also less likely to survive than seeds with lower oil content (Wright, 2003, p. 578). Dry seeds with lower densities are most combustible and therefore more likely to survive than fresh seeds with higher densities (Colledge and Conolly, 2014, p. 194).

The way in which plants are burnt also affect their survival, with plant remains more likely to survive in non-oxidising conditions (Boardman and Jones 1990, Wright 2003, p. 578, Märkle and Rösch 2008, p. 259). The most widely utilised method of recovering charred plant remains from archaeological deposits is by flotation which causes plant remains to lose their primary context and associations with other organic remains and artefacts. This study adopts a multi-proxy approach to contribute new information to the archaeobotanical record from Abu Hureyra by studying phytoliths, which are preserved in non-burnt contexts and can withstand high burning temperatures (section 2.2), to identify plant types and parts that may not be represented in the charred macro-fossil assemblage.

Table 2.1 Depositional pathways for charred and non-charred plant remains in the archaeological record (compiled from: Matthews, 2010; van der Veen 2007; Colledge, 1991, Hillman 1981; Hillman, 2000; Miller, 1984; Wallace and Charles, 2013; Colledge and Conolly, 2014)

Depositional pathways of charred plant macro-fossils	Plants usually under-represented in charred assemblages
• Cereal processing such as drying or parching (Hillman, 2000, p. 354), though parching may not be necessary: (Hillman, 1981) • Cooking: accidental spillages into the fire • Waste from crops used as fuel and tinder (Hillman, 1981; Miller, 1984) • Animal dung burnt as fuel (Miller, 1984; Wallace and Charles, 2013) • Waste disposal: burning as reduction of noisomeness or general waste disposal e.g. craft waste & debris from construction • Accidental (or deliberate) burning of a building (e.g. at Çatalhöyük) • Ritual offerings • Wild plant pathways (e.g. seeds blowing into fire or dropped by birds)	**Not burnt:** • Raw seeds and fruits consumed • Construction materials, e.g. roofing or tempering in mudbricks • Artefacts, e.g. reeds for weaving basket and cultural uses of plants for decorative or ritual purposes (e.g. grave lining) **Plants which usually don't survive burning** • Straw and plants/parts which burn off quickly and oily seeds which explode and are destroyed when heated. • Roots, tubers or legumes may be heated directly, leaving only residues which cannot be identified (Colledge, 1991) • Seeds and chaff burnt at low temperatures and not sufficiently carbonized for preservation or high temperatures (<500°) (Boardman and Jones, 1990; Matthews, 2010)

2.2 Phytoliths as a tool for investigating past plant use and resource exploitation in archaeology

Phytoliths are microscopic bodies of silica (SiO_2nH_2O), formed when silica is absorbed in a soluble state (H_4SiO_4), by plants through groundwater, then deposited in cell interiors and the in-fillings of cell walls, where it solidifies (Piperno, 2006, p. 5). Once the plant dies and the organic matter decays, the silica is deposited into soil or sediment in various forms, often replicating the cells in which it was formed. As largely minerogenic rather than organic, phytoliths are often more robust and durable than other plant remains found in the archaeological record, surviving under a broader set of preservation conditions, for example, pH ranges between 2 and 8.2 (Weiner, 2010, p. 175). Phytoliths retain identifiable features in temperatures of over ~850° (Elbaum et al., 2003; Cabanes and Shahack-Gross, 2015; Portillo et al., 2017a; Portillo et al., 2020b), and some morphotypes can remain identifiable in temperatures exceeding 1000°C (Wu et al., 2012). However, phytoliths also have limitations as a past plant proxy which are discussed below.

Phytolith analyses have a broad range of applications including as an archaeobotanical tool for understanding past plant-use (Rosen, 2005; Jenkins and Rosen, 2007; Nadel et al., 2013; Portillo et al., 2014; Portillo et al., 2020a) and in palaeoecological studies to provide information about past environment and climate. Phytoliths are often deposited as single cells, however, these cells are also found in anatomical articulation, creating multi-celled phytoliths (also referred to as silica skeletons), which often provide more information about the type of plant and anatomical origin from which the phytolith is derived, within Middle Eastern archaeological sites where multi-cells are frequently preserved and numerous reference collections exist for comparison (e.g. Rosen, 1992). Angiosperms, or flowering plants, are divided into two major groups; monocotyledons (hereafter monocots) and eudicots, formerly dicotyledons, which differ structurally (key characteristics summarised in Table 2.2). Monocotyledon plants evolved from dicotyledon plants, and therefore, in botany, the term dicotyledon also encompasses monocot plants. A smaller subset of flowering plants, now classified as eudicots includes all the woody phytolith producing plants expected in this study. Therefore, in this thesis, the term dicot, which is used as both an abbreviation of dicotyledon and eudicot, is used to refer to woody angiosperms, as current phytolith studies continue to use both terms to discuss woody plant remains (e.g. An and Xie, 2022, Neumann et al. 2019).

Table 2.2 Typical structural characteristics of monocotyledon plants versus eudicot plants

	Embryos	Leaf venation	Stems	Roots	Flowers
Monocots	1 cotyledon	Veins parallel	Complex vascular bundle arrangement	Fibrous roots	Floral parts in multiples of 3
Eudicots	2 cotyledons	Veins netlike	Vascular bundles in ring arrangement	Taproot	Floral parts in multiples of 4/5

Monocot plants represent grasses, including cereals, reeds, sedges and palms, whilst dicots are woody plants including many common garden flowering plants, herbaceous shrubs and trees (non-coniferous). It is worth noting that while the main focus of this study differentiates monocot from dicot phytoliths, coniferous woody plants also produce phytoliths which may be morphologically similar to dicot derived phytoliths (Hodson, 2016).

Phytoliths are produced in both monocots and dicot (and coniferous) plant types, however, monocots have been shown to produce up to 20 times more phytoliths than dicots (Albert et al., 2003, p. 470; Tsartsidou et al., 2007), which leads to the under-representation of woody plants in the phytolith record. An initial aim of this study was to mitigate this issue through the analysis of wood charcoal from the same samples. However, this was not possible due to the COVID-19 pandemic (see COIVID statement).

A key advantage of phytoliths over other plant microfossils such as starch and pollen, is that some distinct morphologies represent the parts of plant from which they are derived, including grass stems, leaves and inflorescences, and dicot leaves from dicot wood and bark (Albert et al., 2003; Tsartsidou et al., 2007, p. 1270), Appendix 4. Within archaeology, the proportions of phytoliths derived from different plant parts are used to inform on processing activities (Harvey and Fuller, 2005; Portillo et al., 2017b) and uses of space (Tsartsidou et al., 2009). Unlike pollen, phytoliths are not usually airborne and tend to provide a more localised signal of the plants present in a specific deposit and have been used to determine variations in spatial functions in archaeological and ethnographic sites (Tsartsidou et al., 2007; 2009; Portillo et al., 2012). However, within archaeological contexts it is possible for intrusive modern plants to decay, depositing phytoliths which may become mixed with the archaeological samples. This issue should be addressed at a macro-scale during excavation and sampling, where invasive modern plant roots and bioturbation should be noted and considered as an additional potential depositional pathway for some phytoliths. Another means of further investigating the depositional pathway of phytoliths is at a microscale, through the analysis of micromorphological thin sections, where phytoliths are visible in their primary context and indicators of disturbance or intrusive material may be identified where they are not apparent at a macro scale (discussed further in section 2.3).

Phytoliths are highly durable, particularly as they are predominantly inorganic and therefore more resistant to destructive processes than other plant materials, Table 2.3, (Piperno, 2006, p. 5; Tsartsidou et al., 2007; Rosen, 2008; Pearsall, 2015). They can withstand high temperature burning and are frequently preserved following exposure to temperatures exceeding 850°C (Elbaum et al., 2003; Portillo et al., 2020b), and have been shown to retain morphologically identifiable characteristics in temperature exceeding 1000°C. Some phytoliths begin to undergo morphological changes from 600°C (Brochier, 2002) which can provide an indicator of exposure to heat. Discolouration, for example, opaque browning sometimes occurs following heating (Parr, 2006). Other signs of heat exposure include surface bubbling or “melting”. When phytoliths can no longer be identified due to surface bubbling and distortion, they are classified as “melted phytoliths” and, in this study, expressed as a percentage of the total phytolith assemblage.

Phytoliths can also be preserved without the need for burning, waterlogging or desiccation on which macro-fossils are dependent, and survive well in a broad spectrum of soil types from pH 2 to 8.2, but may dissolve in more alkaline conditions, more than pH 8.2 or in very strong acids (Matthews, 2010; Cabanes et al., 2011; Cabanes and Shahack-Gross, 2015). Phytoliths therefore provide a different, often more diverse, representation of plant materials, types and parts, and thereby, plant-use within an archaeological site (Hillman, 1981). As demonstrated in a wealth of case studies, integrated archaeobotanical data sets have the potential to inform on a wealth of research themes to contribute to a better understanding of the development of agriculture in SW Asia (Asouti et al., 2020; Elliott et al., 2020; García-Suárez et al., 2018; Jenkins and Rosen, 2007; Matthews et al., 2020; Portillo et al., 2009, 2010, 2013, 2014, 2019, 2020; Power et al., 2014; Ramsey et al., 2016, 2017, 2018; Ramsey and Rosen, 2016; Rosen, 2005; Shillito and Elliott, 2013; Shillito and Matthews, 2013). This study compares proportions of plant types and parts represented at Abu Hureyra in the charred macro-fossil record (de Moulins, 2000) with those in the phytolith record analysed in this study, to provide new perspectives into continuity and change in plant exploitation during the emergence and development of agriculture and sedentism.

Table 2.3 Comparison of the representation of charred plant macro-fossils versus phytoliths in the archaeological record

Charred plant macro-fossils	Phytoliths
Requires restricted low temperature burning <400-500°	Preserved both without burning and in high temperatures of over 850° (see discussion in text)
Over representation of agricultural by-products (Hillman, 1981), weed seeds and plants used as fuel	Over representation of monocot plants which produce up to 20 times more phytoliths than dicots (Albert et al., 2003) and dissolution of more fragile morphologies (e.g. sedges), as well as agricultural by-products such as chaff and 'straw' which may dominate some phytolith assemblages
Under representation of chaff (burns quickly), roots and tubers (roasted by direct heating) and oily seeds (may explode)	Lower taxonomic resolution than charred seeds, but able to differentiate between plant parts (grass stem/leaf or inflorescence, dicot wood/bark or leaf)
Disaggregation, disarticulation and loss of context when recovered by flotation	Disarticulation, disaggregation and loss of context when extracted from sediments for quantification and morphotype analysis

Phytoliths recovered from archaeological sediments are often disarticulated, either through pre depositional activities such as grinding (e.g. Portillo et al., 2017b), taphonomic processes during deposition, including bioturbation and through the processing of sediments to extract phytoliths in the laboratory (e.g. ashing, Jenkins, 2009). Disarticulated phytoliths, or single cells, are usually identifiable to a lower taxonomical resolution than articulated, multi-celled phytoliths in the Middle East, which is the focus in this study. The use of morphometrics, which can be applied to provide a higher taxonomic classification to phytoliths (e.g. Ball et al., 2016) is problematic in groups of single celled phytoliths in mixed archaeological deposits, which may represent numerous plant remains. Within a single deposit, even those sampled at a high resolution, for example, subsampled from micromorphological thin sections (Vrydaghs et al., 2016), the hundreds, thousands, or millions of phytoliths likely represent different plant uses, activities and depositional events. This issue is further accentuated by the issue of redundancy, discussed above, whereby the same phytolith morphotype is produced by a variety of vegetation types, and must be considered when interpreting phytolith assemblages.

Phytoliths are most commonly only identified to the order or family, rather than genus or species level, thus providing a lower taxonomic resolution compared with charred macro-fossils. The following section 2.2.1 provides and more detailed outline of the specific types of vegetation identifiable in the phytolith record. This study uses phytoliths as a tool for investigating plant-use to inform on plant types and parts either not represented or under-represented in the published charred macro-fossil assemblage (de Moulins, 2000, pp. 399–416; Hillman, 2000, pp. 341–348). The macro and micro plant analyses are integrated to evaluate interpretations about plant-use, human economy and lifeways at Abu Hureyra in Chapter 7, 10 and 11.

2.2.1 Phytolith vegetation and ecozone indicators

Phytoliths are identified either as single cells or joined together in anatomical connection, known as multicell phytoliths or silica skeletons. The term silica skeleton is more frequently used to refer to large phytolith bodies, often with some anatomical or taxonomic significance (Rosen, 1992). Therefore, the term multicell is used in this study, here defined as three of more phytoliths, as phytoliths joined tended to contain low number of single phytoliths (generally between 3 and 10 single cells). Phytolith morphologies vary in different types of vegetation and therefore can often provide an indicator of the plant in which it was formed, and are particularly effective at differentiating between monocots, particularly grasses (Poaceae family) and dicots (woody/herbaceous plants and shrubs) (Table 2.4). The key phytolith morphotypes expected to be identified in this study and the vegetation to which are they attributed are outlined in Table 2.4 and examples of some of the key phytoliths morphotypes identified in this study are provided in Figure 2.1 and 2.2.

Table 2.4 Key phytolith morphotypes identified in this study with vegetation attributions based on reference material and archaeological examples

Phytolith morphotype	Attribution and reference
Single cells (following ICPN 2.0 where possible)	
Phytoliths usually produced by monocots	
Acute bulbosus	Mostly grasses (Poaceae) (Kaplan et al., 1992)
Papillate	Usually inflorescence bract of grasses (Poaceae) (Ball et al., 1996; 1999; Piperno, 2006)
Bulliform flabellate	Commonly associated with reed leaves (Sangster and Parry, 1969; Chen et al., 2020), also produced in leaves of other grasses (Poaceae) and sedges (Cyperaceae)
Bulliform blocky	Commonly produced in leaves of sedges (Cyperaceae) and grasses (Poaceae)
Blocky	Dicot wood (Albert et al., 2003)
Elongate entire - Cylindrical - Parallelepipedal (thick) - Tabular (thin)	Mostly monocots. Produced in wide variety of plant types, particularly grasses (Poaceae) leaves/culms (Twiss et al., 1969; Albert et al., 1999; 2003)
Elongate sinuate	Mostly monocots, commonly in grass leaves (Albert et al., 2003; Gallego and Distel, 2004)
Elongate dentate	Monocots, often in grass inflorescences and leaves, also associated with reed culms (Ramsey et al., 2016)
Elongate dendritic	Grass inflorescences, especially Pooid grass husks (Rosen 1992)
Sedge cones (c.f. hat-shaped)	Conical phytoliths from Cyperaceae (Ollendorf, 1992)
Grass silica short cell phytoliths	Diagnostic of grasses, produced in all parts of the plant (Twiss et al., 1969)
GSSCP Saddle	Poaceae, most commonly Chloridoideae sub family. Occasional occurrence in C_3 and C_4 PACMAD grasses reeds (Twiss et al., 1969)
GSSCP Bilobate	Poaceae, commonly associated with Panicoid grasses (Twiss et al., 1969). Also occur in Pooideae grasses, especially Stipeae tribe (Fredlund and Tieszen, 1994; Strömberg, 2004; Piperno, 2006)
GSSCP Polylobate	Poaceae, usually from Panicoideae and other PACMAD grasses (Twiss et al., 1969)
GSSCP Cross	Poaceae, mostly Panicoideae and other PACMAD grasses (Twiss et al., 1969) (also Bambusoideae and Oryzoideae, not expected in this study)
GSSCP Crenate	Poaceae, abundant in Pooideae sub family (Brown, 1984)
GSSCP Rondel	Poaceae, produced in most grass sub families (Mulholland, 1989) but especially abundant and often associated with Pooideae (Twiss et al., 1969)
GSSCP Trapezoid	Poaceae, mostly Pooideae (Brown, 1984; Mulholland, 1989
Stomata	Usually monocot leaves, also some dicot leaves
Spheroid echinate	Monocots, especially Arecaceae (e.g. Piperno, 2006; Morcote-Ríos et al., 2016)
Phytoliths usually produced by dicots	
Acute bulbosus – dicot type	Produced in dicot leaves but rarely able to distinguish from Acute bulbosus produced in grasses
Blocky	Dicot wood/bark (Albert et al., 2003; Tsartsidou et al., 2007)
Ellipsoid (psilate/rugose)	Dicot wood/bark (Albert et al., 2003)
Spheroid psilate/rugose	Dicot wood/bark (Albert et al., 2003)
Platelet	Dicot leaves (Albert et al., 2003)
Polyhedral	Dicot leaves (Bozarth, 1992)
Tracheary	Dicot leaves (Bozarth, 1992; Strömberg, 2004)
Irregular (psilate/rugose)	Dicot wood/bark (Albert et al., 2003)

Honeycomb	Dicot leaves (Bozarth, 1992)
MC Polyhedral	Dicot leaves (Bozarth, 1992)
MC Jigsaw	Dicot leaves (Bozarth, 1992)
Multi-cell forms expected in this study	
Stacked bulliforms	Grass leaves. Often associated with reeds or moister environments (Sangster and Parry 1969; Bremond et al., 2005)
Phragmites (reed) culm	Elongate dentate cells connects with narrow or "pinched" GSSCP rondel (sometimes saddle) (Ryan, 2011)
Phragmites (reed) leaf	Elongate dentate cells with small frequent stomata (and short cells) (Ryan 2011)
Cyperaceae (sedge)	Sedge cones
Poaceae (grass) husk	Often Pooid grasses. Elongate dendritics with GSSCP rondels and papillate – usually not possible to distinguish wild from domestic
Triticum sp. (wheat) husk	MC elongate dendritic with irregular erratic waves (Rosen, 1992)
Hordeum sp. (barley) husk	MC elongate dendritic thick squarish, regular wave pattern (Rosen, 1992)
Lolium sp. (rye)	Rounded, long cell waves with regular amplitude (Rosen, 1992)
Avena sp. (oat)	Lobed long cell waves and irregular papillate (Rosen 1992).

Key: PACMAD is a clade of grasses which include families; Aristidoideae, Panicoideae, Chloridoideae, Danthonioideae, Arundinoideae, Micrairoideae, MC = multicell phytolith (here defined as three or more single cell phytoliths in anatomical connection), GSSCP (Grass silica short cell phytoliths) = phytoliths which form in short cells in the epidermis of Poaceae

A challenge relating to the production and interpretation of phytolith assemblages is in multiplicity, whereby, a range of phytolith morphologies are produced by a single taxon, and in redundancy, whereby, the same morphotypes are produced in many taxa, often representing a diverse range of vegetation types (Hart, 2016, p. 27; Vrydaghs et al., 2016, p. 80, Table 2.4). In this research, 'redundant' phytolith morphotypes are assigned to monocots, dicot leaves or dicot wood/bark for quantification based on the proportion in which experimental and reference collection work have shown them to be produced in reference studies from SW Asian material (Albert et al., 2003).

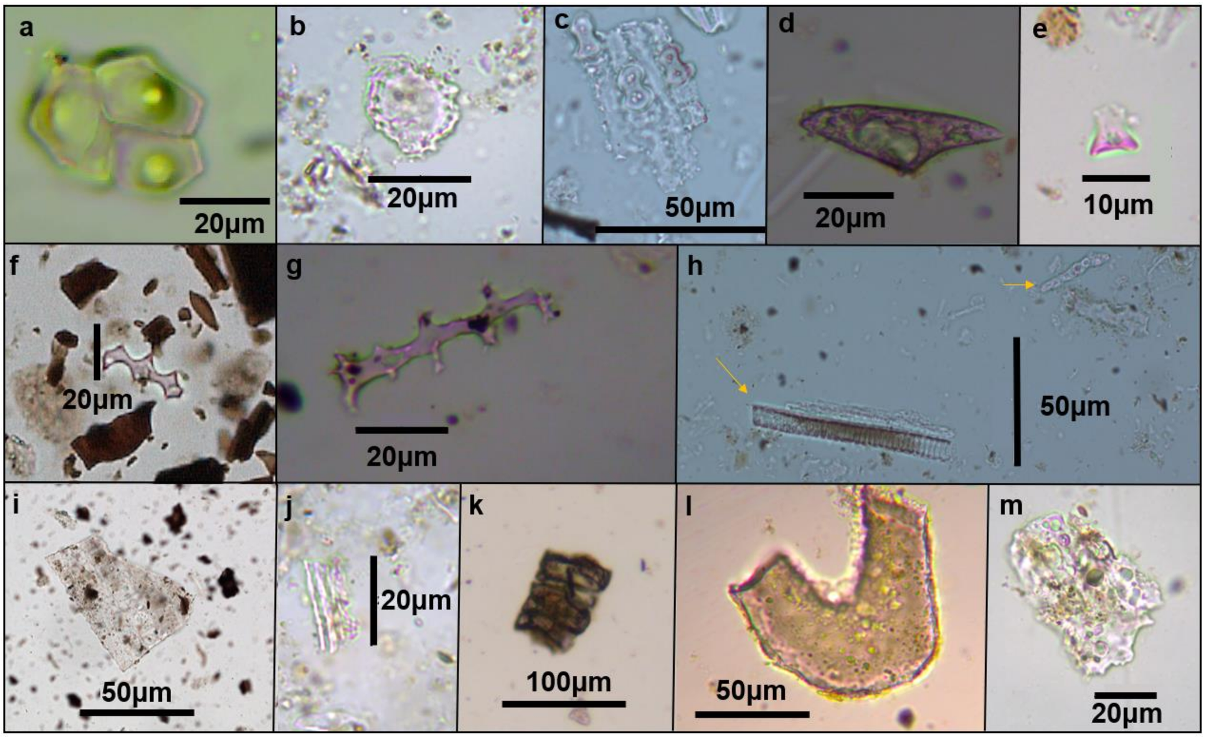


Figure 2.1 Photomicrographs of some of the key phytolith morphotypes referred to in this study showing a) "hat shape cones" from Cyperaceae family, Carex *sp. (UoR reference collection) ; b) papillate; c) MC dendritic with SC bilobates; d) acute bulbosus "prickle"; e GSSCP rondel; f) elongate dentate; g) elongate dendritic; h) left arrow: tracheary, right arrow c.f. MC "hat shape"; i) platelet; j) MC elongate entire (partially weathered); k) MC bulliform, c.f. reed leaf; l: "weathered" bulliform showing signs of both chemical and physical degradation; m) indet melted phytolith*

Grass silica short cell phytoliths (GSSCP) are formed in the Poaceae family, and their presence is therefore highly diagnostic of grasses (Twiss et al., 1969; Brown, 1984). GSSCP are most commonly associated with Pooid grasses, and in this study, as demonstrated in the charred macrofossil record, . Bilobates are most commonly formed in Panicoid grasses (Twiss et al., 1969), however do also form in other grass sub families, and in some cases can be distinguished based on their three-dimensional shape and morphology (Neumann et al., 2019). Bilobates are occasionally produced in Pooid grasses, particularly in *Stipa* sp., however, tend to be more trapezoidal in cross section compared with bilobates from in Panicoids which are more flat and symmetrical (Fredlund and Tieszen, 1994; Strömberg, 2004, p. 258, fig. 4h). Bilobates formed in the Aristidoideae and Arundinoideae families on the other hand, tend to have longer, more slender shafts, with convex or "saddle" like lobes in contrast to the larger, straight or semi-rounded lobes and short, wider shafts typical of bilobates which form in Panicoid grasses. This study therefore typically assigns bilobates to the Panicoid grasses, except where the morphology suggests they are derived from another grass family, or in some instances where both Rondels and bilobates are observed in anatomical connection, suggesting both types are derived from Pooid grasses.

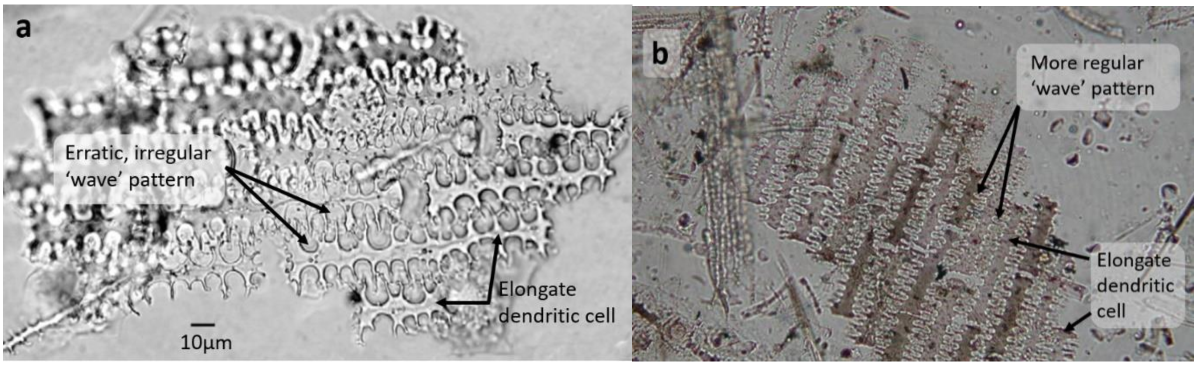


Figure 2.2 Key 'wave' pattern features of wheat and barley (Rosen 1992). Pictures showing a) a diagnostic multicell from the husk of Triticum aestivum *(adapted from: Rosen 2008, p. 1821, fig. 5) and b)* Hordeum Vulgare *husk (adapted from: Fuller 2007 [Online]*

At Neolithic sites in SW Asia, the wave pattern produced by dendritic multi-cells can sometimes be used to differentiate between *Hordeum* (Barley) and *Triticum* (Wheat) (Rosen, 1992; Table 2.4), illustrated in Figure 2.2. There are also several distinct multicellular forms which can be attributed to ecologically important categories of vegetation, for example, reed stems and leaves (e.g. Ryan, 2011; Ramsey et al., 2017; Table 2.4, Figure 2.1, Figure 2.2).

2.2.2 Critical evaluation and comparison of phytolith extraction protocols

Phytolith extraction protocols aim to recover an assemblage of phytolith morphotypes in a concentration that is representative of the original sediment from which they are extracted and can be carried out in a cost and time effective manner (Madella et al., 1998). This section critically evaluates some of the key factors to consider for each stage of phytolith extraction procedure. Numerous methodologies have been developed for analysing and quantifying phytolith assemblages. Methodological variation in the extraction of phytoliths are discussed below, but tend to include the removal of organic matter, the disaggregation and separation of clays of minerals and mounting on a microscope slide in an appropriate medium.

The methods applied to achieve the stages of phytolith extraction may be adapted depending on the deposit type, phytolith concentration, phytolith preservation and research questions and objectives and are discussed further below. Key factors affecting the selection of a phytolith extraction methodology and examples of how a methodology may be adapted are highlighted in Table 2.5.

Table 2.5 Key factors which may influence selection of phytolith extraction methodology and examples of alterations which may be made

Factor and example	Example of methodological adaptation
Sediment type • Clays	More vigorous methods for disarticulation of sediment aggregates, for example through sonication (Katz et al., 2010; Lombardo et al., 2016)
Phytolith concentration • Deposits with high concentrations of phytoliths	Deposits such as 'spodograms' with very high concentrations of phytoliths may require little to no processing to obtain a representative count of diagnostic morphotypes (Piperno, 2006, p. 98)
Research question/specific characteristic • Investigating burning through the identification of the refractive index of phytoliths (Elbaum et al., 2003) • Investigating water availability through number and size of multi-celled phytoliths (Rosen and Weiner, 1994; Jenkins et al., 2016)	May select 'wet extraction' (Piperno, 2006) instead of 'dry ashing' (Rosen, 2005) which has been shown to alter the refractive index (Jones and Handreck, 1967; Parr et al., 2001) Requires careful consideration of 'wet' versus 'dry' ashing method, as this has been shown to alter the concentration/proportion of multicelled phytoliths (Jenkins, 2009; Shillito, 2011). Sonication may also break down multicellular phytoliths (Katz et al., 2010, p. 1558)

Removal of organic matter

Most standard phytolith extraction protocols include a stage to remove organic matter which may obscure phytoliths and inhibit counting. This is achieved through either 'dry ashing', where the sediment is combusted usually at ~500° (Rosen, 2005), or 'wet ashing', also referred to as 'acid extraction', where the organic matter is dissolved by acid (Piperno, 2006, p. 90). Experimental work has shown differences in the proportions of multi-celled phytoliths when comparing these techniques (Jenkins, 2009; Shillito, 2011), although an earlier study comparing 'wet' and 'dry' ashing techniques found no statistical differences between the phytolith morphotype assemblage (Parr et al., 2001). The presence, proportion and size of multi-celled phytoliths has been used as an indicator of water availability in arid to semi-arid regions (Rosen and Weiner, 1994; Jenkins et al., 2016). This information has then been used to make inferences about Neolithic economic strategies and sedentism, such as the seasonal fission-fusion of Çatalhöyük's Neolithic population hypothesis (Roberts and Rosen 2009). Previous studies have also shown that the application of the dry ashing technique has caused phytoliths to shrink (Jones and Milne, 1963) thereby impacting size and any inferences regarding water availability. In addition, they have shown that dry ashing may also cause phytoliths to become warped or alter their refractive index (Jones and Handreck, 1967; Parr et al., 2001) which has implications for studies which use the refractive index to indicate burning temperature (Elbaum et al., 2003).

Disaggregation and separation of clays and minerals

Sonication has been shown to improve the removal of clay and organic matter by breaking down aggregates and reduces the processing time and use of more toxic or dangerous chemical for the same purpose (Lombardo et al., 2016). However, sonication has also been associated with the breakdown of multicell phytoliths which may have implication in both identification of plant type and part as well as in studies using multicell phytoliths as a proxy for water availability (Rosen and Weiner, 1994; Jenkins et al., 2016) discussed above. Furthermore, multicell phytoliths often provide a higher taxonomic resolution than single cells, particularly in SW Asia, for example to differentiate between cereal taxa (Table 2.4; Rosen 1992). Samples are often centrifuged to separate clays and minerals (e.g. Katz et al., 2010) and when conducted correctly, are effective at doing so without losing phytoliths (Pearsall, 2015) and is more time-efficient than manual fractionation which may need repeating up to 7 or 8 times, often over a number of days (Piperno 2006, p. 90, fig. 5.1).

Mounting media

A further consideration is the medium in which extracted phytoliths are mounted onto a microscope slide. Phytoliths have a refractive index of ~1.42 and should therefore ideally be mounted in agents with refractive indexes of between 1.51 and 1.54, which include permount, Canada basalm, histoclad, Entellan New Merck, benzyl benzoate, glycerine and clove oil (Piperno, 2006). Whilst the refractive index and visibility of phytoliths in each agent is important, other factors which must be considered include, cost, shelf life and toxicity. Other properties, such as whether it remains liquid or solidifies after mounting are also important as phytoliths are 3-dimensional, and liquid mounting media (or slow setting mounting medias) enable the rotation of phytoliths which often provides clarification for morphotype designation. Organic compounds, such as clove oil or benzyl benzoate are inexpensive, have a long shelf life and are non-toxic making them advantageous choices. They remain liquid after mounting which is an advantage as it allows the rotation of the phytoliths allowing them to be viewed in three dimensions and improving accuracy in identification of individual morphotypes. However, these slides are non-permanent, making it difficult to reanalyse samples and are especially inappropriate for creating reference collection material. The primary method employed in this study for quantitative and morphological phytolith analysis involves mounting the phytoliths on the slide in sodium polytungstate (SPT), the heavy liquid used to separate phytoliths from clays and minerals in the centrifuge, the advantages and disadvantages of which are discussed further in Chapter 4, section 4.2.

Quantification

Within phytolith studies, much discussion exists over how phytoliths are quantified and recorded (e.g. Zurro, 2018) and as with extraction procedures, selection of an appropriate method depends on phytolith concentrations and research questions. This study follows an adapted version of the Katz *et al.* (2010) quantification methodology which has an overall error of around 30% (Katz et al., 2010, p. 1561), described in full in section.4.2.1. This is comparable with the average errors using the widely employed Albert et al. (1999) method, however, is much less labour intensive to obtain comparable results.

2.3 Identifying depositional pathways and plant function in the archaeological record

A major challenge in the interpretation of archaeobotanical assemblages is disentangling the complex diversity of plant uses and taphonomic processes which may have contributed to a single deposit or context. Charred assemblages may be derived from burning of plant processing waste, craft making waste, fuel, contamination of fuel and accidental burning by spillage during cooking, for example. Furthermore, charred macrofossils are most commonly recovered by flotation, where samples are usually processed by context as identified during excavation at the macroscale. During flotation, plant remains become mixed, losing their context and inhibiting the precision to which their past uses may be interpreted, creating ambiguity as to whether they represent food, waste, fuel, or animal diet deposited by dung burnt as fuel (e.g. Miller, 1996; Hillman et al., 1997). The rise of geoarchaeological approaches such as micromorphology highlight the importance of identifying and understanding microstratigraphic contexts, which often reveal a range of activity types, micro-contexts, depositional events, formation processes and post-depositional alterations within a single macro-scale unit (Matthews, 2010; Shillito, 2011; Banerjea et al., 2015; García-Suárez et al., 2018).

2.3.1 The significance of dung in the archaeological record

Plant remnants, including seeds and phytoliths are also preserved in human and animal coprolites within archaeological contexts (Miller, 1984; Matthews, 2010; Portillo and García-Suárez, 2021; Shillito et al., 2011; 2020; Valamoti, 2013; Wallace and Charles, 2013). One of the major depositional pathways for plant remains is through dung deposited by humans or animals (Miller 1984; Spengler et al., 2018; Portillo and García-Suárez 2021). Dung is of especial importance in the archaeological record as plant remains preserved in coprolites can provide direct evidence of human or animal diet and has been argued as a potential depositional pathway for some of the small-seeded grasses and legumes present in the Abu Hureyra Epipalaeolithic charred plant assemblage (Miller, 1996; Hillman et al., 1997). Animal dung has been a popular choice of fuel from prehistoric times (Miller and Smart, 1984; Matthews, 2005; Portillo et al., 2014; Smith et al., 2019; Spengler, 2018) to the present day (Miller 1984, Reddy 1998, Gur-Arieh et al. 2013, Portillo, Belarte, et al. 2017). It is, therefore, possible that some of the phytolith and charred plant material in archaeological contexts originates from animal dung burnt as fuel.

Dung is often difficult to identify during excavation and may appear as amorphous organic material (Shillito et al., 2011) which requires the employment of specialised and targeted techniques for its detection (for overview of methods for dung identification see Shahack-Gross 2011, p. 206 table 1). Dung disintegrates during flotation resulting in the mixing of plant remains derived from dung with those from other depositional activities (Matthews, 2010). This is problematic for the interpretation of plant remains, as illustrated by the case study of Abu Hureyra, addressed in this research, discussed above. Understanding fuel choices is important as it may also inform on resource availability as the use of dung fuel has been associated with deforestation or woodland retreat (Miller, 1984; Anderson and Ertug-Yaras, 1998). However, dung may also be the preferred fuel choice for specific activities for its long, regular burning properties (Zapata et al., 2003).

2.3.1.1 Case Study to illustrate potential implications of dung for archaeobotanical interpretations

The excavations at Abu Hureyra in 1972 and 1973 represent one of the first research projects to employ large scale, systematic flotation (Moore et al., 1975), and set a new benchmark for archaeological practice that resulted in one of the most significant archaeobotanical assemblage of charred remains from the SW Asian Epipalaeolithic to date. However, in the absence of micro-contextual information, the interpretation of the charred plant assemblage from the Epipaleolithic phase of the site has been contested. Initially, the increase in small-seeded grasses after the onset of the Younger Dryas were argued to represent a broadening of the diet in response to a deterioration in the local resource base (Flannery, 1969; Hillman et al., 1997), also supported by a more recent review of the assemblage (Colledge and Conolly, 2010). It has also been suggested that the increase in small-seeded grasses and legumes during this period represent weeds of agriculture, and are therefore evidence of some of the world's earliest cultivation (Hillman et al., 2001). This argument was supported by the identification of some cereal grains with morphologically domestic traits. However, as some domestic-like seeds do appear within natural populations, and the presence of weed seeds can also be interpreted as a broadening of the diet, this evidence for cultivation in the Epipalaeolithic has largely been discredited (Nesbitt, 2002; Colledge and Conolly, 2010). A third interpretation of the Epipalaeolithic charred plant macro-fossil assemblage is that some of the small-seeded grasses were deposited by animal dung burnt as fuel (Miller, 1996b), as observed at Ali Kosh

(Miller and Smart, 1984) and more recently discovered at other Late Natufian sites (Arranz- Otaegui et al., 2017). These three interpretations have vastly different implications for the overall understanding of human ecology and shifting relationships with the environment in the face of a change in local resources and responses to climate change (see discussion of theories and frameworks, section 1.3).

2.3.2 The application of micromorphology to disentangle plant use in the archaeology

Micromorphology, the integrated, microscopic study of the arrangement and nature of the components of soils and sediments in thin-section (Courty et al., 1989) was first applied to better understand archaeological site formation processes by Cornwall in 1958. Within archaeology, micromorphology contributes information on site formation processes, the depositional history of stratigraphic units, use of space and past human activities, and is becoming an ever increasingly integral tool within archaeological excavations (e.g. Matthews, 1995; 2005; 2010; Matthews and Farid, 1996; Matthews et al., 1996; 2013; Roe, 2007; Shillito et al., 2013b; Banerjea et al., 2015). Understanding the ways in which contexts, features and deposits relate to each other is essential in archaeological excavations at the macro-scale, while micromorphology enables these key connections to be made at a microscale. Post depositional alterations and disturbances of deposits are often detectable through micromorphology where they are invisible or unclear during excavation. This enables more precise interpretations about the nature of a deposit and allows the identification of possible contamination. For example, through micromorphology, the reworking of material by earthworms, modern roots or burrowing is often identified (Matthews et al., 2020). Another key advantage to analysing archaeological material in micromorphological thin sections is the ability to identify components in their primary context. For example, coprolites can be identified through micromorphology (Shillito et al., 2011) where their identification may allow differentiation between plants consumed as food and those burnt as fuel. Through the application of traditional techniques, such as flotation for charred macro-botanical remains and extraction of phytoliths within a laboratory, these components are no longer visible in their original context.

This research had aimed to analyse micromorphological thin sections from fire installations and a midden deposit at the Neolithic site of Bestansur, Iraqi Kurdistan, to identify fuel use, and compare plant-use between contemporary sites situated in different environments. However, this research was not completed because of time restrictions caused by the Covid-19 university closures (see Covid-19 impact statement).

2.3.3 Identifying faecal spherulites in the archaeological sediments

One of the arguably most straightforward mediums to identify whether dung may be present in archaeological sediments is through the study of faecal spherulites (Brochier et al., 1992; Canti, 1997; 1998; 1999). Dung spherulites are made up of radially crystallized monohydrocalcite, and form in the guts of animals, most commonly ruminants, and are excreted, providing a good indicator of the presence of animal dung and, by proxy, animals, in the archaeological record (Canti, 1997; 1998; 1999). Dung spherulite analysis is particularly powerful when combined with other techniques such as micromorphology (section 2.4.2) and GC-MS analysis for dung biomarkers (section 2.3.4) (e.g. Shillito et al., 2011). In comparison to techniques such as micromorphology and GC-MS which are both time consuming and costly means through which to identify dung in archaeological sediments, the analysis of faecal spherulites in spot samples of sediments, where preserved, is relatively time and cost effective to identify. At the most basic level, approximately 1mg of sediment can be mounted onto a microscope slide in a non-toxic medium such as clove oil, cover slipped and scanned for spherulites under a polarising microscope in cross polarised light (XPL) at between x200 and x400 magnification. Specific methodologies for identifying and quantifying faecal spherulites are critically evaluated in this research in Chapter 5.

Identification features

Faecal spherulites are most easily identified in crossed polarised light (XPL) where they are usually between 5 and 20µm in diameter and characterised by bright interference colours, low order white to first order red and second order blue and a fixed extinction cross (Canti, 1998, p. 437). Faecal spherulites are pseudo uniaxial negative, which can be tested using the λ plate, where low order white colour changes to blue and yellow in opposite quadrants. Whilst Faecal spherulites provide a rapid assessment of the probable presence of ruminant dung, where preservation conditions are favourable, but are not a definitive indicator as some spherulites may be other microfossils such as coccoliths which have a similar appearance (Canti, 1999). This study mitigates the potential misidentification of faecal spherulites through rigorous comparison with modern reference dung samples.

Preservation and taphonomy

Faecal spherulites are best preserved in alkaline soils with a pH of more than 7, and rarely preserved where the pH is below 6 (Canti, 1999). A pilot investigation of 16 sediment samples from Abu Hureyra, representing a range of context and deposit types show all samples to have a pH favourable to the preservation of spherulites (Chapter 6, Appendix 4). However, it is important to note that soil pH may fluctuate over time and the conditions may not have always been appropriate for faecal spherulite preservation. Furthermore, the relatively arid conditions at Abu Hureyra, mean spherulites are less susceptible to dissolution, which may even occur over time in distilled water. Bioturbation can severely inhibit the preservation of faecal spherulites which may not survive digestion through the guts of micro-organisms (Canti, 1999, p. 256).

Faecal spherulites are able to survive combustion temperatures of more than 800°C in reducing conditions (Canti and Nicosia, 2018), and therefore have the potential to provide information on the use of animal dung burnt as fuel, a contentious debate in archaeology since first suggested as a potential origins for parts of the charred macro-botanical assemblage at Ali Kosh by Naomi Miller in 1984 (Miller, 1984) and subsequently during the Epipalaeolithic period of occupation at Abu Hureyra (Miller, 1996). To provide further information on impact of combustion on phytoliths and spherulites, including formation of darkened spherulites, this research contributed to an experimental combustion of modern dung material (cow and ovicaprine) collected from the modern village of Bestansur in Iraqi Kurdistan. This research has been published as a paper (Portillo et al., 2020b). This research builds on previous work on faecal spherulite taphonomy through a comparison of methodologies for analysing and quantifying faecal spherulites, presented in Chapter 5. Darkened spherulites are recorded in all material analysed for spherulites to provide new information about burning conditions and temperatures in both archaeological and ethnographic samples (Chapters 5, 7, 8).

Faecal spherulites, however, do not require burning for preservation and may be present in other archaeological contexts such as middens or animal pens, providing information about early animal management and a proxy for identifying penning areas within an archaeological site (Matthews et al., 1996; Portillo et al., 2009; Matthews, 2010). This is of especial importance for better understanding early animal management, prior to the manifestation of morphologically domestic traits in animals which may take up to 1000 years to appear depending on anthropogenic management strategies and the environment (Zeder, 2008).

Increasingly, studies integrating spherulite analysis are especially effective at understanding use of space, identifying fuel types and better understanding animal management and plant-use at archaeological sites (e.g. Gur-Arieh et al., 2014; Portillo et al., 2014; 2020a; portillo and García-Suárez, 2021; García-Suárez et al., 2018; Smith et al., 2019). In this research, all sediment samples are scanned for faecal spherulites in order to ascertain if dung was a likely depositional pathway both for the plant remains identified in this study and those previously analysed (de Moulins, 2000; Hillman, 2000; Hillman et al., 2001). Furthermore, this research aims to use the identification of the presence or absence and quantification of faecal spherulites as a rapid screening method to inform on the selection of a set of samples which are also tested for dung biomarkers through GC-MS to provide more robust evidence of the presence of dung and compare methodologies (section 2.3.4).

This study therefore applies spherulite analyses in multi-scalar approach to establish in particular, whether a small increase in small seeded grasses and legumes towards the end of the PPNB occupation of Abu Hureyra (de Moulins, 2000) may have been deposited by animal dung burnt as fuel in order to contribute to current debates on whether the importance of gathering wild crops (Bogaard et al., 2017; Arranz-Otaegui et al., 2018) was sustained after the domestication of the Neolithic “founder crops” (Weiss and Zohary, 2011) (section 1.3) and to inform on animal management and dung use more widely.

Common methods used in ethnographic and archaeological research to identify and quantify faecal spherulites is critically evaluated in Chapter 5, and therefore summarised only briefly here and in Table 2.6. The most common method involves mounting a known quantity of material (e.g. dung, sediment or ash) onto a microscope slide, counting a set number of spherulites or fields of view and calculating the number of spherulites on the slide and thereafter the number of spherulites per gram of sediment (after Canti, 1999). More recently, a methodology has been developed which involves concentrating spherulites onto a microscope slide for quantification following an adapted method of the Katz *et al*. (2010) phytolith rapid extraction procedure, which was first employed by an experimental study on cooking installations (Gur-Arieh et al., 2013).

Table 2.6 Comparison of a) 'non-processed' smear slide method and b) 'extraction' method for quantifying faecal spherulites

Advantages	Disadvantages
Non-processed, smear slide methodology after Canti (1999)	
• Rapid (6+ samples mounted in ~ 30 mins) • Cost effective and uses minimal equipment • It is possible to simultaneously identify other materials (e.g. microcharcoal, phytoliths in PPL and mineral components in PPL & XPL) • Slides can be made permanent when mounted using agents such as Entellen New Merck	• Spherulites may be bound within organic matrices/aggregates • Spherulites may be masked by other materials • Slides are non-permanent when made using clove oil or other liquid mounting agents preventing longer-term storage and transport • Spherulites less clear in high viscosity mounting agents (compared with SPT – see below)
Examples of studies employing this methodology: Portillo et al., 2012, 2014, Portillo et al., 2017a; 2017b, Canti and Nicosia 2018, García-Suárez et al., 2018	
Extraction methodology after Katz et al. (2010); adapted by Gur-Arieh et al. (2013)	
• Methodology concentrates spherulites • Spherulites relatively homogenously distributed on slide • Quantification more precise • Easily integrated with phytolith quantification following same method • Low viscosity heavy liquid solution promotes visibility of spherulites	• Less cost efficient and requires more laboratory equipment (see Chapter 4.3) • More time consuming • Calcitic spherulites dissolve in SPT mounting agent (pH ~ 4.5) therefore must be analysed within one hour following extraction.
Example of studies employing this methodology: Gur-Arieh et al., 2013; 2014; Dunseth et al., 2018	

As with phytolith extraction protocols, the selection of an appropriate methodology must consider the research question and deposit type. It is possible to mitigate some of the disadvantages of each method highlighted in Table 2.6. For example, although SPT dissolves spherulites because of its acidic pH, studies have shown there to be an hour before SPT begins reacting with calcite (Skipp and Brownfield, 1993), which provides sufficient time to extract and count approximately two samples (more or fewer depending on the abundance of spherulites, the total number required to be counted for the purpose of the specific research question and the experience of the person counting the spherulites). An average error of approximately 30% has been reported for the quantification of dung spherulites following the adapted Katz et al. (2010) methodology (Gur-Arieh et al., 2013, p. 4336). There has not, however, been a study to date which specifically measures the rates in which spherulites are dissolved following this method and the external factors (such as air temperature or sediment type) which may affect the rate of dissolution.

As it is possible to calculate the total number of spherulites per gram of sediment for both methodologies, it should be possible to compare concentrations of faecal spherulites across different studies, even if they have employed different methodologies. However, to date, there have been no studies directly comparing these two methods for quantifying spherulites which limits the comparisons which can be made across different sites and research areas. It is particularly important to understand whether both methodologies are replicable in that they produce statistically similar total numbers of spherulites per gram of sediment to compare spherulite concentrations across different archaeological sites and to use ethnographic and experimental studies to draw conclusions about archaeological samples (Gur-Arieh et al., 2013, Portillo et al., 2017a, Canti and Nicosia, 2018).

This research therefore aims to compare the concentrations of faecal spherulites per gram of sediment following the two key methodologies described, in modern dung samples, and subsequently also archaeological samples where faecal spherulites have been identified, the results of which are presented in the style of a short research article in Chapter 5. This study calculates whether the estimated number of faecal spherulites per gram of sediment are sufficiently similar between the two methodologies to enable the integration of spherulite studies across different sites and regions.

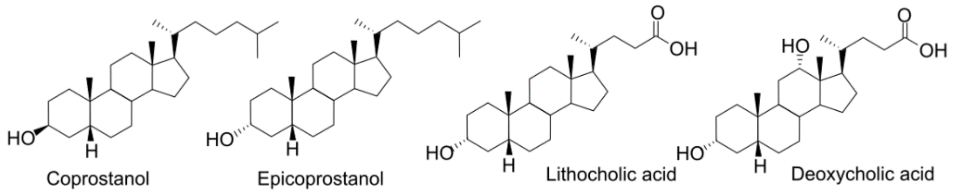
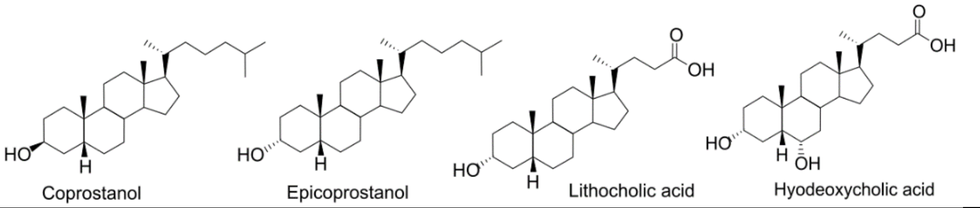
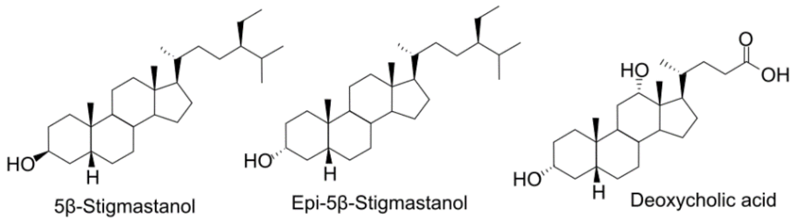
2.3.4 Gas Chromatography and Mass Spectrometry (GC-MS) for the detection of dung

To further identify the potential presence of the faeces excreted by humans and animals, or where spherulites have not survived, this study detects faecal biomarkers by GC-MS in archaeological sediments and plaster floors from Neolithic Abu Hureyra. As relatively stable compound classes, 5β-stanols and bile acids are also hydrophobic and thus they are not readily leached from the stratigraphic unit from which they are subsequently extracted (Evershed et al., 1997; Evershed, 2008). Lipids are also chemically stable and have been identified in rocks millions of years old (Brocks et al., 2005). Faecal biomarkers are subject to different taphonomic processes compared with spherulites (section 2.3.3), and therefore may identify sediments or materials where faecal matter is present, but spherulites are not. The aim of combining both techniques for identifying dung in a range of materials and sample types (sections 4.1.2 and 4.1.4) is to comprehensively assess the likely presence of dung, and to mitigate, to an extent, against preservation issues which may affect one of the dung proxies. Secondly, this study compares and evaluates the identification of dung spherulites and faecal biomarkers as methods for assessing whether there is a faecal component to archaeological sediments which may either have been a depositional pathway for plant remains and may inform more widely on the use of secondary animal products.

The 5β-stanol class of compounds originates from the microbial reduction of cholesterol and Δ^5-unsaturated phytosterols, which include campesterol, stigmasterol and sitosterol, which occurs in the guts of higher animals (e.g. mammals). When cholesterol and other related compounds are reduced in the natural environment, they usually form 5α-stanols, and therefore elevated levels of 5β-stanols compared with control samples may be indicative of faecal material (Evershed et al., 2001, p. 338). Bile acids are hydroxylated steroids which are produced in the guts of mammals, in varying quantities which enable the distinction between some species (Simpson et al., 1999) (Figure 2.3).

The identification of faeces through biomarker detection was first developed in the Biogeochemistry Research Centre at the University of Bristol (Evershed and Bethell 1996, Elhmmali et al. 1997, Bull et al. 1999, 2002). A successful pilot study at Neolithic Çatalhöyük identified faeces through this biomarker approach (Bull et al., 2005), demonstrating the application of feacal biomarker analysis to deposits of a similar type and age to those from Neolithic Abu Hureyra. 5β -stanols and bile acids indicate faecal origin whilst elevated 5β -stigmastanol and coprostanol are indicative of ruminant (herbivore) and omnivore faeces respectively (Eneroth et al., 1964; Bethell et al., 1994; Evershed et al., 1997) and are confirmed through the identification of bile acids, Figure 2.3 (Elhmmali et al., 1997). The following ratio is used to calculate the origin of faecal matter; (Coprostanol + epicoprostanol) / (5β-stigmastanol + epi-5β-stigmastanol) whereby >1 = human, carnivore or porcine and <1 = herbivore. Human, carnivore or porcine faeces can then be differentiated by the presence of different bile acids (Figure 2.3).

Figure 2.3 Dominant faecal sterol and bile acids for humans and omnivores.

Species	Dominant faecal sterol and bile acids
Omnivore – human 	
Omnivore – pig 	
Herbivore 	

A potential interpretative challenge is to unpick whether biomarkers from plaster floors represent construction material and/or activity residues. Equally, in this and other case-studies many sediments were sampled in bulk from contexts which included construction debris, and therefore faecal biomarkers could represent dung used in construction rather than burnt as fuel, or otherwise deposited in refuse areas or animal throughways. Elevated faecal biomarkers in plasters may confirm dung used in construction, while in occupation soil could suggest livestock roamed within those spaces or midden deposits for the disposal of human/animal waste. Where faecal biomarkers are elevated in paired plasters and occupation residues, but absent from other material, this would indicate an area where faecal input is more likely and can be considered as a potential depositional pathway for plant remains.

Organic compounds, including faecal sterols, have been shown to be destroyed by firing, even at relatively low temperatures of 400°C (Reber et al., 2019), and therefore they might not be detected in floor plasters which are likely to have been fired, although it depends at which stage dung is added to the plasters. The presence or absence of any organic residues, therefore, also inform on the production, construction and maintenance of plaster floors. High temperature burning can cause the degradation of faecal sterols (Matthews et al., 2020, p. 277); an issue for detecting if burnt animal dung contributed to the archaeobotanical assemblage at Abu Hureyra. However, a background faecal component of herbivore origin has been confirmed by GC/MS in a fire installation at Bestansur (Elliott et al., 2020, pp. 389–390), demonstrating the potential of this method for identifying dung fuel.

This study further compares and evaluates different methods for the identification of dung in the archaeological record by testing the relationship between the presence/absence of faecal spherulites and faecal biomarkers and identifying, by pXRF, elemental compositions associated with the presence of dung.

2.4 Geochemical characterisation of archaeological sediments and materials

Elevations of specific elements or clusters of elements can provide indicators for activities, spatial function and materials (Rondelli et al., 2014; e.g. Jenkins et al., 2017; Vos et al., 2018). Phosphorous (P) is widespread in bones, plant materials, faeces and ashes, and as it is also relatively stable in most soils, provides a good indicator of anthropogenic activity in the archaeological record (Holliday and Gartner, 2007). However, because such a wide range of materials produce P, further analysis is required to determine its origin, which may be mixed. Furthermore, concentrations of P can be affected by soil processes and pH, and direct soil measurements usually have large error margins due to the heterogeneity of the materials (Entwistle et al., 2000; Oonk et al., 2009; Canti and Huisman, 2015). Similar issues are encountered for most elements used to infer information from archaeological materials and deposits and comparative ethnoarchaeological research (e.g. Jenkins et al., 2017; Vos et al., 2018). For example, Calcite (Ca), is often the dominating element for all activity and construction categories analysed, and identifying specific uses of space in the archaeological record through pXRF remains a challenge (Jenkins et al., 2017; Middleton et al., 2005).

This research aims to identify patterns of elemental enrichments across different areas of Abu Hureyra and different deposit types and materials from those spaces, by pXRF. Multivariate statistics, e.g. PCA analysis, are conducted to identify spatial and temporal variations in elemental composition of archaeological sediments and plasters. Differences in elemental concentrations between plasters from different parts of the site could suggest household level autonomy in the selection of specific recipes, as suggested at Çatalhöyük in mudbrick recipes (Love 2012). The analysis of material from different phases of occupation investigates continuity and changes in uses of space and manufacturing practices. Despite the limitations of pXRF analysis to identify specific activities or plaster recipes, the identification of variations of elemental composition aims to provides new insights into resource use, technological innovations and management during the mid-late PPNB at Abu Hureyra (Aim 4).

2.5 Summary of Methodological rationale

The analysis of plant remains provides important evidence to understand changes in human economy as societies shifted from mobile hunter-gatherer groups to sedentary farmers. Plant proxies provide different information about plants at varying taxonomic resolutions. They are also subject to different preservation conditions, taphonomic processes and recovery and analytical techniques. Studying just one past plant proxy provides a fragmented overview of past plant exploitation, with some plant types and parts either over or under-represented. Although all archaeological study of past plant remains are fragmentary by nature, adopting a multiproxy approach goes some way to mitigating this challenge and provides a more representative overview of plant use on a site.

In order to investigate plant-use and human-plant-environment interactions this study analyses phytoliths, preserved under different conditions to charred plant macro-fossils to provide new information about plant-use and vegetation exploitation (Aim 1i and Aim 1ii, section 1.2). Archaeological plant remains represent a diverse range of economic functions and depositional pathways and it is important to untangle these to better understand human-plant-environment interactions. Specifically, this study investigates potential depositional pathways of plant remains by identifying if faecal spherulites are present in all samples to assess whether plant remains could have been deposited by animal dung burnt as fuel (Aim 2i, section 1.2). Further GC-MS analysis of selected samples to detect biomarkers of animal dung provides further confirmation as to whether dung is present in ashy samples and as a component of plaster floors and could therefore have been a fuel which contributed to burnt plant assemblages or mixed in with large deposits through construction debris (Aim 2ii).

Chapter 3 Case Study: Abu Hureyra, Syria

Abu Hureyra is significant for its size, an estimated ~11ha, and longevity, with rich evidence for human occupation, spanning several changes in human economy (8600-6000 cal. BC). Initially occupied as an Epipalaeolithic hunter-gather settlement (11,200-9800 cal. BC), the site later developed into one of the world's largest and earliest farming villages. This chapter firstly highlights the significance of Abu Hureyra and justifies its selection as a case study (section 3.1) to achieve the key aims and objectives of this research to provide new insights into human selection of plants and resource management over key periods of change in human economy (section 1.2). The next section (3.2) examines the geographical, environmental and climatic context for Abu Hureyra as these are an important baseline to understand how people interacted with the environment and managed local resources. Section 3.3 provides a brief overview of the archaeological context of Abu Hureyra, focussing on other Neolithic sites in the Euphrates Valley, which are drawn upon in the discussion (Chapter 11), to identify which resource acquisition strategies were shared regionally or varied at a site-specific level. Section 3.4 is a critical evaluation of the chronology of Abu Hureyra which was initially written in preparation for making an application for an additional set of radiocarbon dates, which was not possible due to Covid-19 related delays. However, this section provides an important baseline and framework for comparing cultural and economic strategies at different sites, and to evaluate the role of climate events and environmental change on the settlement. Finally, section 3.5 discusses diet, economy and lifeways at Abu Hureyra, which are integrated with the new data analysed in this study in the discussion in Chapters 7 and 11.

3.1 Rationale for selection of Abu Hureyra as a case study

Abu Hureyra is especially significant for its longevity, as the site was occupied during several key periods of human innovation and development beginning as an Epipalaeolithic hunter-gatherer settlement from ~11,000 cal. BC, also referred to as Abu Hureyra 1 (Table 3.1). The initial, moderately large (~8ha) Neolithic farming settlement had a mixed economy based on both domestic and wild plants and animals (Period 2A, ~8600-7200 cal. BC). As the settlement expanded to an estimated ~11ha, agricultural practices intensified and the economy became more focused on managed and domesticated plants and animals (Period 2B, ~7200-6000 cal. BC). Abu Hureyra, therefore, provides an opportunity to investigate changes in economy and human-environment relationships as agriculture intensified, at a site-specific level.

Table 3.1 Key periods and phases with quality checked radiocarbon dates and key cultural and economic attributes for Abu Hureyra. Dates and contextual information from Moore et al. *(2000, p. 528-529). Dates calibrated in OxCal 4.3, using calibration curve IntCal13 (Reimer* et al. *2013). Database of radiocarbon dates available in Appendix 2A and dates specific to Trench phases in Appendix 2B.*

Period and date	Dates for trench specific phases in this study	Key attributes at Abu Hureyra
Neolithic 2C 8150-7950 cal. BP 6200-6000 cal. BC	**Trench B** phase 10: 7250$^{+/-}$ 600 (OxTL 196a) Trench E, Phase 8: 5350$^{+/-}$400 (Ox TL 196j)	• ~7ha mudbrick house • Cereal and pulse agriculture • Sheep, goat, cattle and pig husbandry
Neolithic 2B 9250 – 8150 cal. BP 7300 – 6200 cal. BC	**Trench E** Phase 4: 9246$^{+/-}$224 (OxA-1267) Phase 5: 9278$^{+/-}$243 (OxA-2168) Phase 6: 8918$^{+/-}$298 (BM-1724R) Phase 7: n.d **Trench G** Phase 1: n.d Phase 2: 8745$^{+/-}$261 (OxA-1931) Phase 3: 9291$^{+/-}$201 (OxA-1227)	• ~11ha mudbrick houses • Cereal and pulse agriculture • Sheep, goat, cattle and pig husbandry
Period 2A to 2B transition occurred 7465 – 7175 cal. BC (at 95.4% probability)		
Neolithic 2A 10,550 – 9250 cal. BP 8600 – 7300 cal. BC	**Trench B** Phase 7: 9021$^{+/-}$ 203 (BM-1424) Phase 8: n.d Phase 9: 8156$^{+/-}$ 218 (OxA-1232)*date rejected **Trench D** Phase 4: 9438$^{+/-}$298 (OxA-878)*date rejected as material is bone	• ~8ha mudbrick houses • Cereal and pulse agriculture • Sheep and goat husbandry
Hiatus 11,750 – 10,750 cal. BP 9800 – 8600 cal. BC	**Trench G** phase 0/1 (subsoil): 10,997$^{+/-}$240 (OxA-1228)	• Argued continued occupation (based on 1x radiocarbon date)
Epi-Palaeolithic 1B/C 12,800 – 11,750 cal. BP 10,850 – 9800 cal. BC	**Trench E** Phase 2: 12,279$^{+/-}$437	• Timber and reed huts • ?Cultivation • Plant gathering • Gazelle hunting
Epi-Palaeolithic 1A 13,150 – 12,800 cal. BP 11,200 – 10850 cal. BC	**Trench E** Phase 1: 13,146$^{+/-}$123 (?OxA-882/OxA-172)	• Pit dwellings • Plant gathering • Gazelle hunting

3.1.1 Epi-Palaeolithic

Abu Hureyra has been argued to hold some of the earliest evidence for cultivation in the world, although archaeobotanical data from Ohalo 2, suggests a dietary shift to grains may have occurred as far back as 23,000 years cal. BP (c. 21,000 years cal. BC), based on the presence of morphologically wild wheat and barley and other grasses (Weiss et al. 2004). However, in the absence of morphologically domesticated seeds, which can take between 500 and 1000 years to manifest in a plant population (Zeder 2005), evidence for pre-domestication cultivation is in many cases debated, particularly owing to more recent methodological developments (Weide et al., 2022). The site excavators and archaeobotanist, Hillman et al. (2001), argued pre-domestication cultivation occurred at Abu Hureyra based on an increase in small-seeded grasses during the Epi-Palaeolithic occupation, interpreted as weeds of agriculture, as they include species such as *Stipa*, which have been shown to occur in statistically significantly higher proportions alongside cultivation (Fairbairn et al., 2002). More recently, the increase in small-seeded grasses has been reinterpreted as a broadening of the plant-based diet to compensate for the retreat of habitats which supported edible, large seeded annual plants, abundant in the charred macro-fossil record at the start of occupation at Abu Hureyra which preceded the onset of the colder, drier conditions of the Younger Dryas (Colledge and Conolly 2010). Miller (1996), however, argued that some of the charred seeds included in the Epi-Palaeolithic assemblage could represent dung burnt from fuel. Miller's evidence is based on the comparative case study of Ali Kosh in Iran (Miller 1984) as well contextual considerations. Recent research has identified the presence of dung at Epipalaeolithic Abu Hureyra through the analysis of faecal spherulites in flotation residues, including from a hearth base, and therefore argued to represent the use of dung as fuel (Smith et al., 2022). The main focus of this research is on the development of the Neolithic, however, phytoliths and dung spherulites are also analysed from three Epipalaeolithic occupation residues, to build on the evidence reported by Smith et al. (2022). Phytolith analysis has also provided key evidence for Epi-Palaeolithic plant use at Ohalo 2, where comparisons between charred macrofossil and phytolith assemblages have been compared (Ramsey et al., 2017), which provides a baseline for the similar comparisons conducted in this study at Abu Hureyra (Aim 1).

3.1.2 Neolithic

During the Neolithic, charred macro-fossil assemblage is argued to demonstrate an increase in the reliance of domestic cereals and legumes, largely based on high proportions of arable weeds. However, de Moulins (2000) also notes evidence in the assemblage, a possible sustained reliance on wild, gathered foods. The continued exploitation of gathered foods complements evidence from Çatalhöyük, where it has been argued gathered plants remained an important component of the diet and resource base (Bogaard et al., 2017; 2021). However, the depositional context of charred macro-remains needs further consideration, particularly in light of new research on flotation residues from the Neolithic occupation layers at Abu Hureyra which have identified the presence of animal dung. Isotopic studies at Çatalhöyük have shown cattle and sheep were herded in environments with different compositions of C_3 and C_4 grasses (Pearson et al., 2007). If similar practices were followed during the Neolithic at Abu Hureyra, at least some of the charred macro-fossils previously analysed (de Moulins 2000), as well as microfossil phytolith evidence in this research, could represent seeds from dung burnt as fuel. Therefore, this research will investigate signals for dung to ascertain whether at least some plant remains tentatively interpreted as reliance on gathered products, may have been deposited by animal dung burnt as fuel. This research builds on the identification of faecal spherulites at Abu Hureyra by analysing a larger number of samples representing more diverse context and deposit types and with a focus on different spaces within the site.

3.2 Abu Hureyra location, environmental setting and climate

3.2.1 Location

Tell Abu Hureyra is located in Northern Syria in the Middle Euphrate's valley, 35.866 °N and 38.400 °E, ~ 130km east of the modern city of Aleppo (Figure 3.1). The site was built on a well-drained terrace above the floodplain on the South bank of the river Euphrates, in an area which is predominately calcareous with a chalk substrate on the south bank of the river, which would have provided a dependable water supply (Moore et al., 2000, p. 28). The site is now flooded and lies under Lake Assad, following the completion of the Tabqa Dam in 1974, preventing the possibility for further excavations. Situated on the border of several ecozones, the inhabitants of Abu Hureyra would have had advantageous access to a broad resource base which included riverine forest, woodland steppe, stands of wild cereals (wheats, ryes and feather grasses) and park woodland.

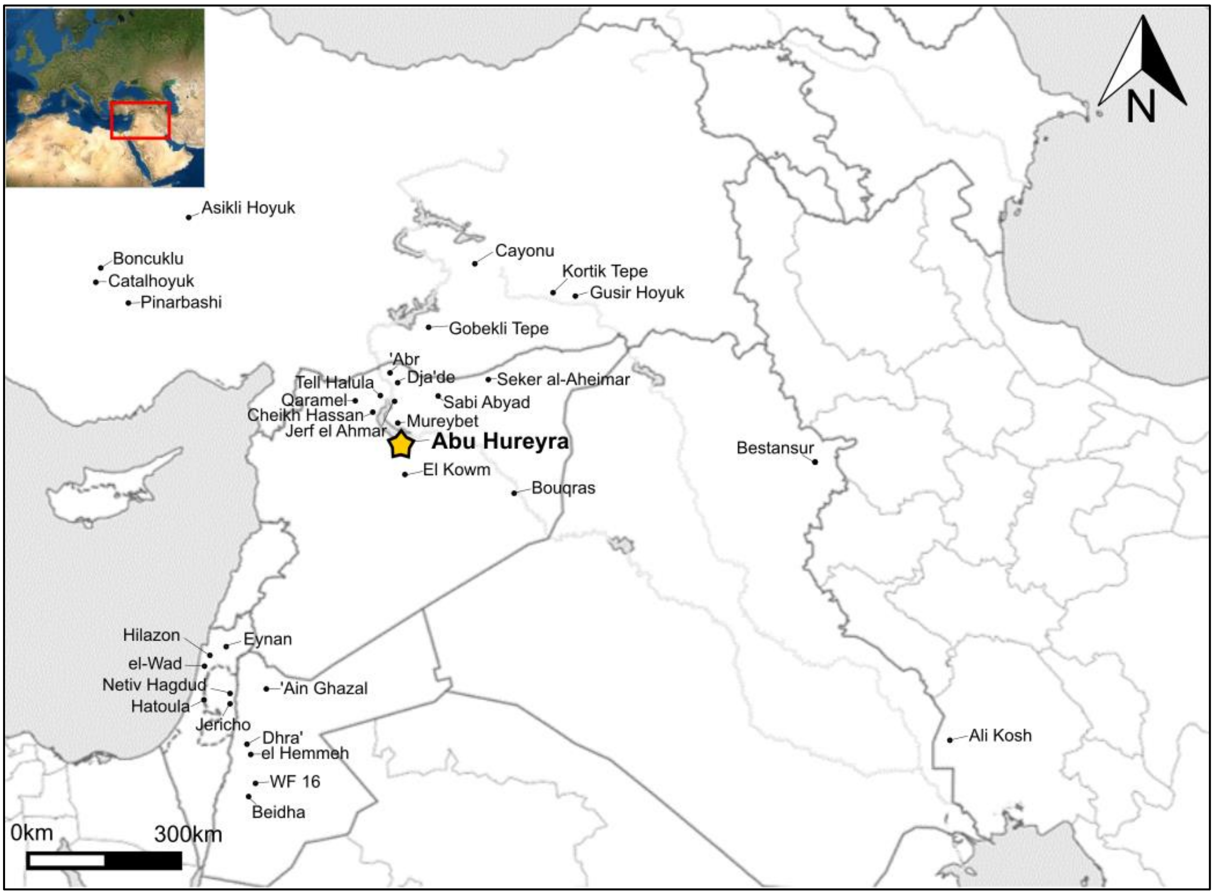


Figure 3.1 Location map of Abu Hureyra and other key sites discussed in this research

3.2.2 Present day climate and environment

Changes in climate are well documented in Syria in the period focussed on in this study, Late Pleistocene-Early Holocene, (e.g. Roberts et al., 2018 and references therein; Jones et al., 2019; section 3.2.3) to present (e.g. Trigo et al., 2010). Climate change and its effect on precipitation in the present day is important to understand because boundaries between vegetation zones are highly sensitive to shifts in precipitation (e.g. Goodfriend and Magaritz, 1988), which thereby effect vegetation availability, particularly as sedentism increased during the Neolithic. At a local level, Syria’s climate is heavily influenced by the country’s topography, which has remained relatively unchanged since the Late Pleistocene to the present day. Therefore, modern climate systems such as rainfall distributions can provide valuable analogies to better understand past climate regimes and changes that are documented in palaeoclimate records.

The former Tell of Abu Hureyra, now submerged under Lake Assad, lies to the East of the Jabal an Nusayreyah mountain range and on the border of two climate zones. According to the *Köppen–Geiger* climate classification, the area to the north is characterised by a warm temperate climate with hot and dry summers, whilst the area to the south is defined as arid, hot steppe/desert (Kottek et al., 2006). The weather station in the modern city of Deir ez-Zur, ~165km south east of the former site of Abu Hureyra, recorded average daytime temperatures of over 40.9°C during the hottest month of July and 12.9°C in the coldest month, January, over the past 20 years (Eglitis, 2022).

Precipitation in this region is usually between 200mm and 400mm per annum (Beck et al., 2005). Rainfall is most frequent during the winter months between December and February with an average of between 5 and 7 days of rain, while rain is rare during the summer months between June and September (Eglitis, 2022). However, this area was highly affected by the well-documented drought which hit Syria between 2007 and 2011, and more recent years have also seen significantly lower than expected average annual rainfalls (Trigo et al., 2010; GPCC, 2020).

Today, dense stands of woodland are largely absent from northern Syria (Deckers, 2016) and natural vegetation in the region is mostly made up of degraded steppe, as a result of deforestation, intensive grazing and cultivation (Moore et al., 2000, p. 49; Willcox, 1996). Gordon Hillman and colleagues mapped the potential distribution of vegetation under modern climate conditions, in the absence of cultivation, deforestation and grazing, based on extensive ecological surveys of the environs surrounding Abu Hureyra (Moore et al., 2000, p. 49). Under current day climatic conditions, Abu Hureyra would sit adjacent to the riverine forest running through the Euphrates valley, in an area of moist to medium dry steppe extending southwards, with Terebinth-Almond woodland steppe in close proximity less than 10km north of the site (Moore et al., 2000, p. 50, fig. 3.7).

3.2.3 Past Climate (summary of climatic events affecting AH 1 and 2)

The last glacial-interglacial climatic transition and subsequent fluctuations in climatic conditions, summarised in Table 3.2, are well documented globally (e.g. Hoek and Bos, 2007). Stable isotope records indicate, within the limits of dating records, that climate change events occurred across southwest Asia at a similar temporal scale, and are further substantiated by regional vegetation changes shown in pollen and charcoal records (Roberts et al., 2018 and references therein).

Table 3.2 Global climate events and key attributes in southwest Asia

Climate Event	**Dates (cal. Years BP/BC)**	**Key Attributes**	**Key reference**
Bølling-Allerød	14,600-13,000 cal. BP 12,650-11,050 cal. BC	Warmer, wetter	Alley et al., 1993; Weaver et al. 2003
Younger Dryas	13,000/12,800-11,700 cal. BP 11,050/12,850-9750 cal. BC	Colder, drier	van der Plicht et al., 2004; Miebach et al., 2016
Holocene	11,700 cal. BP onwards 9750 cal. BC onwards	Warm, relatively stable	Walker et al., 2008
9.2	9,250 cal. BP 7300 cal. BC	Brief cold event	Fleitmann et al., 2008; Flohr et al. 2016
8.2	8200 cal. BP 6250 cal. BC	Increased aridity	Bar-Matthews et al, 1999; 2003

As discussed in Chapter 1.3, the apparent synchronicity between climate change events and significant shifts in human economy in the Late Pleistocene to Early Holocene (Figure 3.2), from mobile hunting and gathering to sedentary farming, has led to a number of theories attributing this shift in human economy to climatic changes (Moore and Hillman, 1992; Enzel et al., 2003; Rosen, 2010; Bar-Yosef, 2011; 2017). There are no climate or palaeoenvironmental proxy records (e.g. lake cores, cave speleothems) for the Middle Euphrates region to reconstruct the local environment of Abu Hureyra. Pollen records from Lake Ghab in NW Syria and Lake Huleh in Israel provide an indication of the broader regional vegetation history in the Levant (Bottema and van Zeist, 1981; Barach and Bottema, 1991; 1999), however, have large margin of chronological uncertainty as they as subject to significant old carbon distortions due to the marine reservoir effect (Meadows 2005). A combination of: a) high resolution global records (e.g. GISP: Hoek and Bos, 2007), b) regional records from across southwest Asia (Roberts et al., 2018 and references therein; Jones et al., 2019), c) on-site archaeobotanical evidence and d) synthesis of these records with modern climatic patterns and vegetation distributions in the region, make it possible to build up a relatively comprehensive reconstruction of the local environment for the Middle Euphrates region.

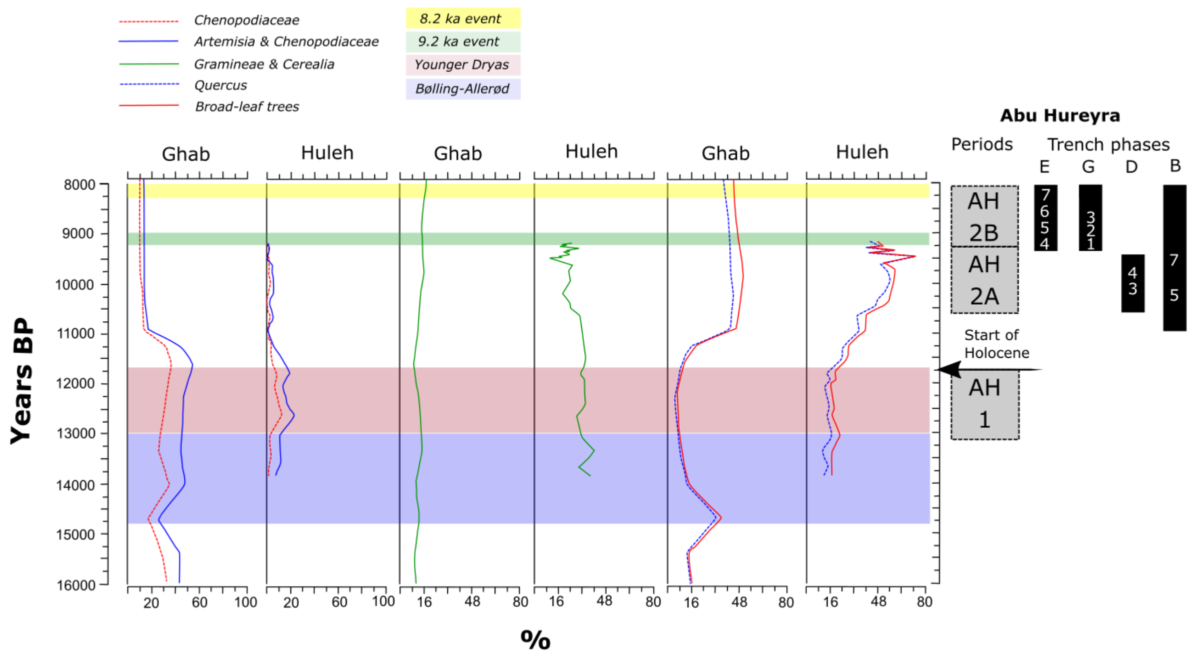


Figure 3.2 Schematic diagram showing changes in vegetation represented by pollen from Lakes Ghab and Huleh, and corresponding archaeological periods and phases of occupation included in this study at Abu Hureyra. Adapted from Roberts et al., 2018, pp. 53-55, fig. 4. Data from: Bottema and van Zeist, 1981; Baruch and Bottema, 1991; 1999; Meadows, 1995; Moore et al., 2000

The climatic events of most significance for southwest Asia and the period of occupation for Abu Hureyra are discussed in this section and the subsequent vegetation changes which would have affected the resource base of Abu Hureyra's inhabitants in the following section. The impact of climate and environmental change remains contentious and increasingly urgent to assess in the present day. This review enables the analysis conducted in this research to be framed within the regional environmental context in order to investigate relationships between humans and their environment to assess the impact of environmental change.

The last glacial maximum (LGM) occurred between 26,500 cal. BP (24,550 cal. BC) to 19,000 – 20,000 cal. BP (17,050 – 18,050 cal. BC), with cool and relatively dry conditions, after which deglaciation began because of increased northern summer insolation (Clark et al., 2009). Following the LGM, isotopic records from southwest Asia consistently show a shift towards more negative $\delta^{18}O$ values, which indicates and increase in moisture availability (Bar-Matthews et al., 1997; Roberts et al., 2001; Stevens et al., 2001; Wick et al., 2003; Bar-Matthews and Ayalon, 2011; Kwiecien et al., 2014; Cheng et al., 2015).

Temperatures, precipitation and CO_2 levels increased during the Bølling-Allerød, c. 14,600 and 13,000 cal. BP (12,050 – 11,050 cal. BC) (Alley et al., 1993; Weaver et al., 2003), which encouraged the expansion of woodland-steppe and annual grasses. Bayesian analysis of quality checked radiocarbon dates model, with 91% probability, that Abu Hureyra was initially occupied towards the end of the Bølling-Allerød (Colledge and Conolly, 2010, p. 127) when local resources were at peak abundance.

The Younger Dryas (hereafter YD), c. 13,000-12,800 cal. BP (11,050 – 10,850 cal. BC) to 11.7ka (9750 cal. BC), is a globally recognised event of colder, drier conditions in southwest Asia (van der Plicht et al., 2004). The YD had a significant impact on the landscape and vegetation distributions of southwest Asia, and caused a large-scale die-back of vegetation, culminating in a more restricted resource base (Hillman, 2000; Miebach et al., 2016, discussed in 3.1.2.4 below). The amelioration of the climate is well documented at the start of the Early Holocene from c. 9750 cal. BC (11,700 cal. BP), where warmer, wetter, and crucially, more stable conditions prevailed, enabling the woodland and stands of wild cereals to expand and recolonise some of the areas of their former distribution (Roberts et al., 2018 and references therein).

The start of the Neolithic occupation of Abu Hureyra is difficult to determine due to a paucity of secure context radiocarbon dates, but the available evidence suggests it began c. 10,000 cal. BP/8000 cal. BC (see section 3.1.4) when the climate is characterised by warm and relatively stable conditions. A brief cold event has been identified in SW Asia, c. 9250 cal. BP (7300 cal. BC), during the Neolithic occupation of Abu Hureyra (Fleitmann et al., 2008), hereafter referred to as the "9.2ka" event. At a regional scale, this appears to have had little impact on settlement distribution patterns (Flohr et al., 2016). Within the limitations of chronological precision which include uncertainties spanning several hundred years, this event could be considered synchronous with the shift from periods 2A and 2B at Abu Hureyra. This transition is characterised by an increased reliance on domesticated and managed plants and animals accompanied by an increase in settlement size (de Moulins, 2000; Hillman, 2000; Legge and Rowley-Conwy, 2000).

The end of the settlement of Abu Hureyra also remains ambiguous due to a paucity of suitably dated material from the layers assigned to the final period of the site. The final phase of Abu Hureyra, period 2C, is published as occurring c. 8200 to 8000 cal. BP (6200-6000 cal. BC), which coincides with a well-documented global climate event which occurred at ~8200 cal. BP (6250 cal. BC), referred to hereafter as the "8.2ka" event. The impact of the 8.2ka event is recorded in southwest Asia by well dated, high resolution speleothem records from Soreq cave in the Southern Levant which indicates a period of drier conditions in the region (Bar-Matthews et al., 1999; 2003). The 8.2ka event is significant as it has been cited as a cause of collapse and abandonment of Neolithic societies and a driving factor of the subsequent spread of Neolithic cultures west into Europe (Weninger et al., 2006; 2014). However, a subsequent study provides evidence that Neolithic communities were in fact resilient to this event at a regional scale (Flohr et al., 2016). Abu Hureyra, therefore, provides an important opportunity to assess the impact of both the 9.2 and 8.2ka events at a local, site-specific level, within the limitations of the chronological resolution (discussed in section 3.1.4).

In summary, there were several climate change events during the occupation of Abu Hureyra, each of which altered the vegetative landscape and resource base available to inhabitants of the site. Significant shifts in human economy and lifeways are also evident during the occupation of Abu Hureyra, and well documented across southeast Asia. This research therefore investigates human responses, adaptions and resilience to changes in climate and environment at a site-specific level to assess human, plant and animal adaptive strategies and resilience to environmental changes.

3.2.4 Vegetation and resource availability during the occupation of Abu Hureyra

The global climatic fluctuations discussed above would have had a significant impact on the vegetation and landscapes of southwest Asia (Moore et al., 2000, p.74; Figure 3.3). This section evaluates how the local environment and available resources changed throughout the occupation of Abu Hureyra by synthesising the influence of climatic alterations based on regional (southwest Asian records) with localised site-specific indicators of changes in resource-use patterns.

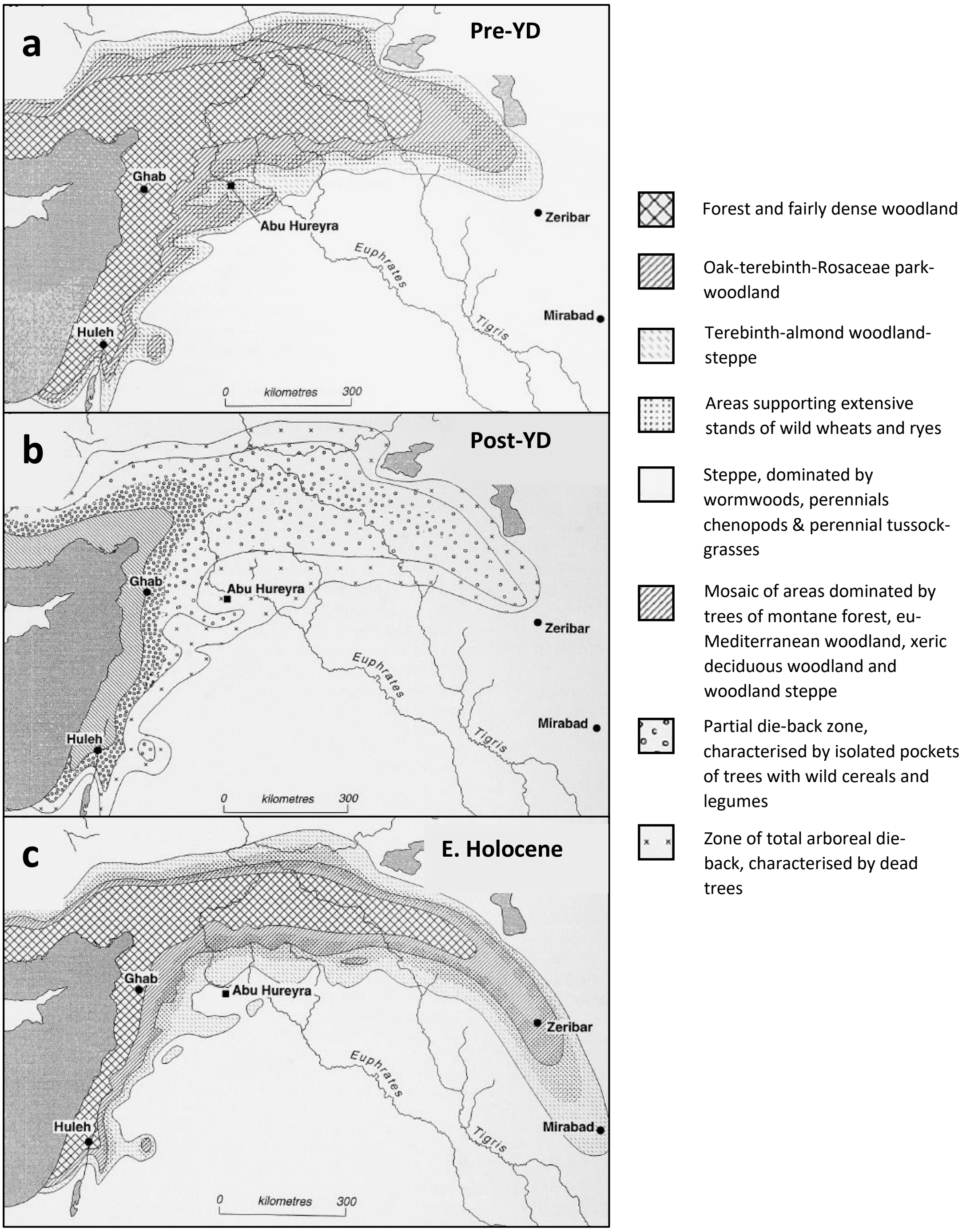


Figure 3.3 Models of vegetation distribution showing a) likely vegetation prior to the start of the YD. b) At the end of the YD and c) during the Holocene. Adapted from Moore et al. *(2000, p.78-80, fig. 3.18)*

Palaeoecological records from SW Asia are relatively consistent in showing changes in temperature, precipitation and evaporation rates which would have shifted vegetation zones (for a detailed evaluation of proxies from SW Asia see Roberts et al., 2018). Significantly, areas of refugia for plants, animals and humans existed, including the Middle Euphrates Valley, which it has been argued enabled the continuity of socio-economic lifeways during unfavourable, cold, dry, conditions (Moore et al. 2000) and facilitated a more rapid response to the new opportunities presented by climatic amelioration compared with areas which saw a break in settlement (Zeder, 2012; Roberts et al., 2018).

A detailed analysis of the likely vegetation distribution patterns in SW Asia, with specific reference to the vicinity of Abu Hureyra are provided by Moore et al. (2000, p.76-84), based on palaeoecological records from Lakes Ghab, Huleh, Zeribar and Mirabad (Figure 3.1) and therefore this section does not recite this information. However, since the publication of *Village on the Euphrates* (Moore et al., 2000), a number of new palaeoenvironmental records from the region have been analysed, for example, Lake Neor, NW Iran (Sharifi et al., 2015) and Lake Iznik in northwestern Turkey (Miebach et al., 2016). Furthermore, the temporal resolution of older records has been significantly improved, for example for Lake Zeribar (Hutchinson and Cowgill 1963; Van Zeist and Wright, 1963; Stevens et al., 2001; Wasylikowa et al., 2006).

3.2.4.1 Vegetation following LGM

Following the LGM, c. 19,000-20,000 cal. BP (17,050 – 18,050 cal. BC), woodland steppe and annual grasses expanded, with wild cereals a significant feature in open, oak-dominated woodland. Meanwhile, the steppe would have been dominated by perennial shrublets and tussock grasses, particularly feather grasses such as *Stipa* sp. (Moore et al., 2000, pp. 77–78). Park woodland could have expanded to within 10-15km of Abu Hureyra, with some trees even closer, and therefore the stands of wild cereals which grew in drier subzones of park-woodland and moister subzones of woodland-steppe would have been accessible, possibly even growing within sight of Abu Hureyra (Moore et al., 2000, p. 81).

Regional records indicate that the climate and environment would have likely been highly favourable for human habitation (Bar-Matthews et al., 1999; Miebach et al., 2016), with soil likely very fertile and suitable for dry farming (Goodfriend and Magaritz, 1988), however, the conditions were far less stable than during the subsequent Holocene.

3.2.4.2 Younger Dryas

As no palaeoenvironmental records exist for the Middle Euphrates region, archaeobotanical evidence from Abu Hureyra provides important evidence for local vegetation distributions and resource availability and changes. Charred seeds and wood charcoal evidence from the start of the occupation at Epipalaeolithic Abu Hureyra suggest the region was characterised by forest-steppe vegetation made up of *Pistacia atlantica* trees and grasses, punctuated by sporadic oak trees (Hillman, 2000; Roitel and Willcox, 2000).

Pollen analysis from cores from Lakes Ghab, Zeribar and Huleh indicate forest retreat and increasing aridity (van Zeist and Bottema, 1981; 1991; Baruch and Bottema, 1991; 1999), reflecting the onset and development of the Younger Dryas (Figure 3.3), although the chronological uncertainties associated with the Ghab and Huleh cores present a challenge to ascertain how rapidly vegetation changes occurred (Meadows, 2005). Discrepancies between the lowland Ghab and Huleh records compared with the highland Zeribar lake records could be attributed to both the geographical setting and chronological resolution of the records. There are also local indications reflecting similar patterns at Abu Hureyra 1, phase 2, where forest retreat and increasing proportions of arid tolerant plant, are identified in the charred archaeobotanical and wood charcoal record, in contrast to more drought sensitive plant which go into decline (Hillman, 2000; Roitel and Willcox, 2000, p. 545; Hillman et al., 2001, p. 386). Initially, wild cereals which likely grew on the ecotone between park-woodland and woodland steppe continued to be well utilised, however, from phase 2 of Abu Hureyra, there is a notable decline in their occurrence, indicating the retreat of local stands (Moore et al., 2000, p. 81). At both Abu Hureyra and in pollen cores from Lake Huleh in the S. Levant (Baruch and Bottema, 1991; 1999), there is some evidence that park woodland resources were still accessible and utilised, for example through the continued, albeit diminished presence of oak charcoal at Abu Hureyra (Roitel and Willcox, 2000, p. 545). This likely suggests these regions as "refugia", where vegetation was able to withstand the unfavourable conditions of the Younger Dryas for a longer period compared with more marginal areas.

The increasingly heavy exploitation of feather grasses attests the first stages of vegetation retreat, as they would have been most prevalent in the dry subzone of woodland steppe, and would therefore have become more locally available to Abu Hureyra (Hillman, 2000, p. 346, fig. 12.7; Moore et al., 2000, p. 82). Following a peak in feather grasses, these plants also diminish in the archaeobotanical record, interpreted by Moore *et al*. (2000, p. 83) as illustrating the transition from woodland steppe to moist steppe and then dry steppe, where feather grasses grow at much lower densities.

Nutlets of club-rush and knotgrasses, which grew on the moist valley bottom, were the last plant type to decline following the deterioration of climatic conditions during the Younger Dryas (Hillman, 2000, pp. 347–348, fig. 12.7). These plants were to an extent buffered from the aridity as they depended on the Euphrates' River regime which relied on snow melt from the Anatolian Plateau (Moore et al., 2000, p. 83). Furthermore, it can take between 1 and 10 years for large rivers to respond to meteorological drought conditions and can be significantly longer for regional scale ground water to dry up (Jones et al., 2019, p.5).

3.2.4.3 Conditions during the emergence of Abu Hureyra 2

From the end of the Younger Dryas, c. 11,700 cal. BP/9,700 cal. BC (van der Plicht et al., 2004), pollen records attest the expansion of woodland, although unlikely reaching the full extent of the area they previously covered prior to the start of the Younger Dryas (Moore et al., 2000, p. 84). For example, pollen records from Lake Iznik maintain consistently high quantities of *Quercis* pollen since the Lateglacial, providing evidence for the significant presence of oaks in the landscape of NW Turkey. The Lake Ghab pollen sequence indicates a slow retreat of woodland after the initial expansion at the start of the Holocene (van Zeist and Bottema, 1981), which is also apparent in the pollen record from Lake Huleh (Baruch and Bottema, 1991; 1999). Integrated analyses of wood charcoal, pollen and modern vegetation data from SW Asia have demonstrated the change from biodiverse grasslands to oak-dominated parklands during the Early Holocene which was driven by human activities such as sheep herding and woodland management practices (Asouti and Kabukcu, 2014). However, following the appearance of agricultural societies and expansion of Neolithic settlements, it becomes increasingly complex to decipher whether natural (e.g. climate driven), anthropogenic (e.g. woodland clearance) or complex interactions between both (Roberts et al., 2011) are the driving contributors to these vegetation trends.

A decline in in *Artemisia* and chenopods is represented in several pollen records during the Early Holocene, having prevailed in the arid conditions of the Younger Dryas (Roberts et al., 2018, p. 56). Terebinth (*Pistacia*) parkland is represented in pollen assemblages from Lake Van (Wick et al., 2003), Lake Zeribar (van Zeist and Bottema, 1991), Al Jourd (Cheddadi and Khater, 2016) and Eski Acıgöl (Roberts et al., 2001), despite being a low pollen producer, and also in archaeological charcoal records and was likely a significant part of semi-arid, Early Holocene landscapes (Roberts et al., 2018, p. 56).

3.3 Archaeological context and developments in the Euphrates

This section discusses the emergence of Neolithic farming in the Euphrates River Valley (Table 3.3), with a particular focus on the Middle Euphrates, but also drawing on themes which emerge from sites in the Upper and Lower Euphrates, to contextualise the primary case study in this research, Abu Hureyra.

Table 3.3 Key Epipalaeolithic - Neolithic sites in the Middle Euphrates Valley, and surrounding environment (compiled using data from (Cauvin, 1980; van Zeist and Bakker-Heeres, 1986; Stordeur, 1999; Moore et al., 2000; Akkermans and Schwartz, 2003; Molist et al., 2006; Willcox et al., 2008; 2009)

Site	Date (cal. years BP)	Period	Key references
Abu Hureyra 1	13,250 – 12,750	Late Natufian	(Moore et al., 2000)
Mureybet 1	12,500 – 12,000	Late Natufian	(van Zeist and Bakker-Heeres, 1986)
Mureybet 2	12,000 – 11,500	Khiamian	(van Zeist and Bakker-Heeres, 1986)
Qaramel	12,000 – 11,500	Khiamian	(Willcox et al., 2008)
Tell 'Abr	11,500 – 11,200	PPNA	(Willcox et al., 2008)
Mureybet 3	11,500 – 11,200	PPNA	(van Zeist and Bakker-Heeres, 1986)
Jerf el Ahmar 1	11,500 – 11,200	PPNA	(Willcox et al., 2008)
Jerf el Ahmar 2	11,200 – 11,000	PPNA	(Willcox et al., 2008)
Sheikh Hassan		PPNA/EPPNB	(Cauvin, 1980; Stordeur, 1999)
Dja'de	11,000 – 10,300	Early PPNB	(Willcox et al., 2008)
Abu Hureyra 2	10,000 – 9300	Middle PPNB	(Moore et al., 2000)
Halula	9800 – 9300	Middle PPNB	(Molist et al., 2006)
Bouqras 11-8	9400 – 8200	Middle/Late PPNB	(Akkermans and Schwartz, 2003)

The Middle Euphrates is occupied following the Younger Dryas by sites characteristic of the PPNA such as Jerf el Ahmar and Tell'Abr (Table 2), which have evidence of cereal cultivation (Willcox et al., 2008), and architectural features interpreted as communal storage features (Willcox, 2002; Willcox and Stordeur, 2012). In the Southern Levant, PPNA sites such as Wadi Faynan, Jordan, share similar, large communal structures (Mithen et al., 2011; Colledge and Conolly, 2018; Colledge et al., 2018), though alongside shared features, there is a huge diversity of building types, which may have been used for ritual and domestic functions which likely not mutually exclusive (Finlayson et al., 2011). Mureybet was initially occupied during the Younger Dryas, but later levels are PPNA, at which time there is also increased evidence for cereal cultivation (van Zeist and Bakker-Heeres, 1986; Willcox et al., 2009). The Middle Euphrates is well established during the PPNA as a centre for the pre-domestication cultivation of cereals, based on increased proportions of agricultural weeds seeds (Colledge, 1998; Willcox et al., 2008; Willcox and Stordeur, 2012). However, this identification of pre-domestication cultivation is problematic as weeds have been shown to characterise both arable fields and grasslands (Weide et al., 2021). More recently, based on traits related to soil disturbance, researchers have employed a functional ecology model which suggested that weed flora identified at Jerf el Ahmar were most likely not arable, while at Early PPNB Dja'de, there is some evidence that a low number of the plants may have been cultivated (Weide et al., 2022).

Few mid-late PPNB sites, contemporary with Abu Hureyra, have been identified close-by, despite extensive archaeological surveys of the region preceding the construction of the Tabqa dam (Wilkinson and Moore, 1978). Within the Middle Euphrates, sites contemporary with Abu Hureyra include Tell Halula (Molist et al., 2006; Molist, 2013), located c. 70km north up the Euphrates from Abu Hureyra and Bouqras (Akkermans and Schwartz, 2003), a little further away to the south (Figure

3.1). Neolithic Abu Hureyra shares specific lithic technology such as the off-set bi-directional strategy which was used to produce high quantities of bi-directional blades with Tell Halula and Bouqras (Borrell and Molist, 2014), although other cultural practices vary between sites within the same region. Moore et al. (2000, p. 494) conclude that in terms of subsistence, Abu Hureyra was largely self-sufficient and there is little evidence for high levels of exchange networks or trade with other contemporary sites in the region.

Contemporary PPNB sites from outside the Middle Euphrates provide the opportunity to identify practices which are shared across the wider region, more locally in the Middle Euphrates Valley, and at a site-specific level. Aşıklı Höyük and Çatalhöyük are substantial PPNB sites in Anatolia (Figure 3.1), which share architectural, cultural and economic traits with Abu Hureyra, and are therefore provide a useful comparison in this study.

3.4 The chronology of Abu Hureyra

This section critically evaluates some of the key challenges associated with radiocarbon dating, focussing specifically on those most relevant to SW Asian Epipalaeolithic and Neolithic sites. An understanding of the potential and limitations of Abu Hureyra's chronology and phasing is essential to a) investigate relationships between shifts in plant-use and specific environmental changes (section 3.5) and b) facilitate comparison between data analysed in this study with other case studies at a local and regional level (section 3.3). Firstly, the technical challenges associated with radiocarbon dating which effect Abu Hureyra are discussed, followed by a critical assessment of specific materials most often recovered for radiocarbon dating from SW Asian Neolithic sites, and finally, stratigraphic and taphonomic considerations are evaluated. This section then critically evaluates the chronology of Abu Hureyra by analysing and quality checking each radiocarbon date to assess their reliability and provide an accurate temporal framework in which to discuss the material and associated contexts analysed within this study. An initial aim of this study was to obtain new dates to provide a higher resolution chronology for Abu Hureyra, however, this was not possible during the Covid-19 pandemic (see Covid-19 impact statement).

3.4.1 Chronological challenges and considerations

Reliable radiocarbon contexts from Neolithic sequences in SW Asia tend to be limited, primarily because the taphonomy of charred plant remains is often ambiguous and environmental conditions are not ideal for the preservation of bone collagen (Zazzo and Saliège, 2011; Jacobsson, 2019).

Technical challenges

Carbon may be introduced from multiple sources during deposition, therefore it is key to only use samples where the original carbon can be isolated which is achieved through pre-treatment to prevent secondary carbon contamination (Brock et al., 2010, p. 104, table 1). This is a relatively well established and standardised procedure for charred plant-remains, making them technically reliable candidates for dating. Charred plant remains (seeds and charcoal) make up 53% of the Abu Hureyra dating series (Figure 4a) and all of the 'accepted' dates are on these materials (Figure 4b). The carbon source of dated humic/fulvic material on the other hand is usually difficult to ascertain meaning these dates are often not representative of the context from which they are derived (13% of the Abu Hureyra dated assemblage, Figure 4a) and make up 15% of the 'rejected' Abu Hureyra dates (Figure 4c).

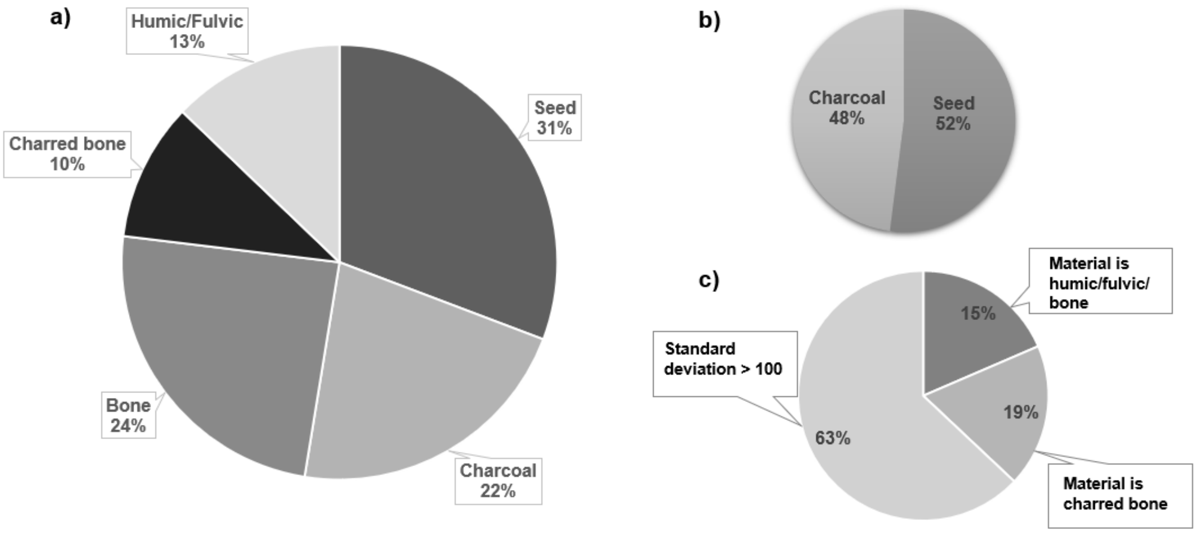


Figure 3.4 a) Proportions of different materials included in the 71 radiocarbon dates from Abu Hureyra. b) Proportions of materials for 'accepted' radiocarbon dates. c) Reasons for rejection of Abu Hureyra dates n.b. some samples rejected for more than one criteria. For detailed breakdown see Appendix 2

Charred plant taphonomy

Charred plant remains of short-lived species such as seeds are preferential for dating, and of especial importance in research concerned with identifying the manifestation of morphological domestication traits in plants. From a technical perspective, contaminants are relatively easily removed through well-established standard acid-alkali-acid pre-treatments (Brock et al., 2010). However, the taphonomy of charred plant remains is often ambiguous, with factors such as bioturbation meaning they may not be representative of the deposit from which they are recovered. This issue is particularly important at Abu Hureyra, where domestic type cereal grains were recovered from Epipalaeolithic contexts, nine out of thirty-six (25%) of which were obviously intrusive and returned dates consistent with the Neolithic occupation of the site. This highlights the need for caution regarding the stratigraphic integrity and security of material recovered from the Epipalaeolithic phase of the site, particularly as frequent animal burrows were observed during excavation (Moore et al. 2000). During the Neolithic period at Abu Hureyra, some material could have moved between phases, or represent secondary deposition, and therefore not be representative of the context or assigned phase from which it was recovered. Approaches such as micromorphology (discussed in section 2.4.2) may help with some of the issues of plant taphonomy discussed above and has great potential to aid understanding of links and associations between contexts and stratigraphic units. However, archival material where approaches such as micromorphology were not implemented must be considered within the limitation of the published knowledge of the stratigraphic and contextual integrity of each sample. For this reason, this research selects material for analysis from different phases of the site, but also from different trenches which represent different periods of occupation (Chapter 4).

Challenges and potential of dating wood and charcoal

Wood and charcoal are one of the most reliable materials for radiocarbon dating and require little pre-treatment. However, the dating of wood charcoal is problematic as it may have been recovered from deadwood or recycled from other uses on a site, for example, parts of a building, and therefore does not directly date the context in which it is associated. Furthermore, the 'old wood effect' (Schiffer, 1986) whereby long-lived species such as Juniper (*Juniperus*) or Oak (*Quercus*), may survive for several hundred years before becoming burnt and preserved in an archaeological context, can make a context, feature or even whole site appear older than it actually is. This issue is discussed in detail in relation to PPNA Wadi Faynan, Jordan (Schiffer 1986, Mithen and Finlayson 2007, p. 460) but has also been mitigated to a degree by quantifying the "old wood effect" through Bayesian modelling (Wicks et al., 2016).

Twenty-two percent of the radiocarbon dates for Abu Hureyra were obtained from charcoal (Figure 3.4a), although the species is not identified in the published record of dated material (Moore et al., 2000), and therefore it cannot be discounted that at least some of the charcoal dates may be several hundred years older than the context from which they were recovered. However, in a study of 3,115 fragments from Epipalaeolithic contexts, Abu Hureyra, is generally dominated by the Salicaceae, including Euphrates poplar (*Populus euphratica*) and willow (*Salix*) (Roitel and Willcox, 2000, p. 545). These tend to be relatively short-lived species with lifespans of ~50 years and rarely exceeding 100 years. Furthermore, although long lived species such as oak, are identified in the previously studied Abu Hureyra charcoal assemblage, the fragments were very small (Roitel and Willcox, 2000, p. 545), and therefore, perhaps less likely to have been selected for dating, particularly as in the early 90s when the material was dated, much higher quantities of charcoal were required than for radiocarbon dating today. Therefore, the old wood effect may have less impact on the overall chronology of Abu Hureyra compared with sites where long-lived species are more prolific (Austin, 2007; Mithen and Finlayson, 2007a).

Stratigraphic ambiguity and contemporaneity

Aligning stratigraphic units between different trenches and areas of excavation can be complex, particularly where several hundred years may exist between them. At Göbekli Tepe, for example, it has been tentatively suggested that the enclosures of Level III are not necessarily contemporary and may have been active hundreds of years apart (Dietrich et al., 2013, pp. 40–41), which has implications for interpretations of the engravings.

Abu Hureyra has been interpreted by the original excavators as one of the earliest and largest PPNB "megasites", however, it has also been argued that so called "megasites" may represent a shifting of smaller settlements (Hole, 2000). The contemporaneity between different areas of a site is important to establish as it also has implications for population estimates and subsequent assessments on carrying capacity on the local environment and management of resources (Birch-Chapman and Jenkins, 2019). That is why an initial aim of this study was to obtain a new series of radiocarbon dates corresponding to specific contexts analysed in this research, and for phases which had not been previously dated, to strengthen temporal associations between trenches. However, as new dates were not possible to obtain due to the Covid-19 pandemic (Covid-19 impact statement), this study focuses on comparing plant-exploitation between the relatively well-defined Neolithic periods 2A and 2B of Abu Hureyra (Table 3.1, Figure 3.5).

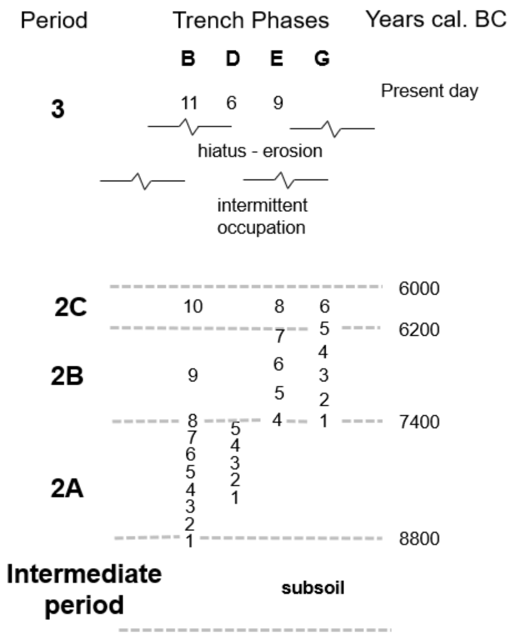


Figure 3.5 Schematic diagram showing the temporal relationship between phases of trenches at Abu Hureyra. Adapted from: Moore et al. *(2000; p. 257, fig. 8.75)*

3.4.2 Quality Checking Criteria

Abu Hureyra has a substantial set of seventy-one radiocarbon dates (Moore et al., 2000, p. 527) and the chronology of the site benefitted from the introduction of Accelerator Mass Spectrometry dating, allowing small samples of material to be dated. However, since the time at which the samples were processed in the late 80s and early 90s, standards and precision of radiocarbon dating has progressed substantially. This study applies a set of quality control criteria to the dates from Abu Hureyra, and other sites discussed within this research. The quality control criteria are based on those applied by (Flohr et al. 2016, pp. 27–28, Jacobsson 2019, p. 11, table 1) and summarised with their justification in Table 3.4.

Table 3.4 Quality checking of radiocarbon dates and justification for rejection criteria

Quality checked constraint	Reason for applying rejection criteria
Standard deviation of >100	Margins of error are larger after calibration and therefore may not be precise enough to address the research questions posed in this study and to synthesise human economic changes with environmental changes
Material is humic/fulvic	May contain secondary carbon contamination therefore providing an inaccurate representation of the archaeological context from which it is derived
Material is charred bone	Proteins used for dating usually destroyed during burning and diagenetic changes may occur contaminating sample

This study also addresses some of the issues highlighted in the above section by quality checking dates using a combination of the criteria applied by Flohr et al. (2016, pp. 27-28) and Jacobsson

(2019, p.9, table 1). This study rejects dates where the standard deviation (1σ) is more than 100 ^{14}C years, as after calibration, this margin of error is larger than is useful to answer the research questions addressed in this study. This study also rejects material which was dated on bone, which is subject to diagenetic changes (Hedges and Law, 1989), and was dated prior to the development of the Lanting method (Lanting et al., 2001), as well as on humic and fulvic fractions which likely contain carbon contamination. The dates from Abu Hureyra have all been calibrated using the calibration curve, IntCal13 (Reimer et al., 2013).

3.4.3 Dating Abu Hureyra 1: Epipalaeolithic

As discussed above, some of the short-lived species which were dated directly from Epipalaeolithic contexts in Trench E, returned dates consistent with the later Neolithic occupation of the site (Figure 3.5, Figure 3.6), and it is therefore, critical to acknowledge in this and other studies, that at least some of the material and plant remains may not be representative of the context from which they were sampled and assigned to.

For the Epipalaeolithic occupation of Abu Hureyra, there are 15 quality-checked, non-intrusive radiocarbon dates, excluding humic/fulvic dates (Figure 3.6). Bayesian analysis by other researchers indicate occupation of the site from 13,146$^{+/-}$123 cal. BP (11, 196 cal. BC) with a 91% probability that the site was inhabited prior to the onset of the Younger Dryas (Buck et al., 1999; Colledge and Conolly, 2010, p. 127). The same model indicates that occupation continued until 11,981$^{+/-}$217 cal. BP (10,031 cal. BC) with only a 16% probability that occupation extended into the pre-Boreal period (Buck et al., 1999; Colledge and Conolly, 2010, p. 127). This assessment of the radiocarbon dates for Abu Hureyra 1 indicates a total occupation of 1231$^{+/-}$265 years (at 1σ) which is a long sequence of occupation for the relatively shallow (~1 metre) occupation deposits associated with this period at the site.

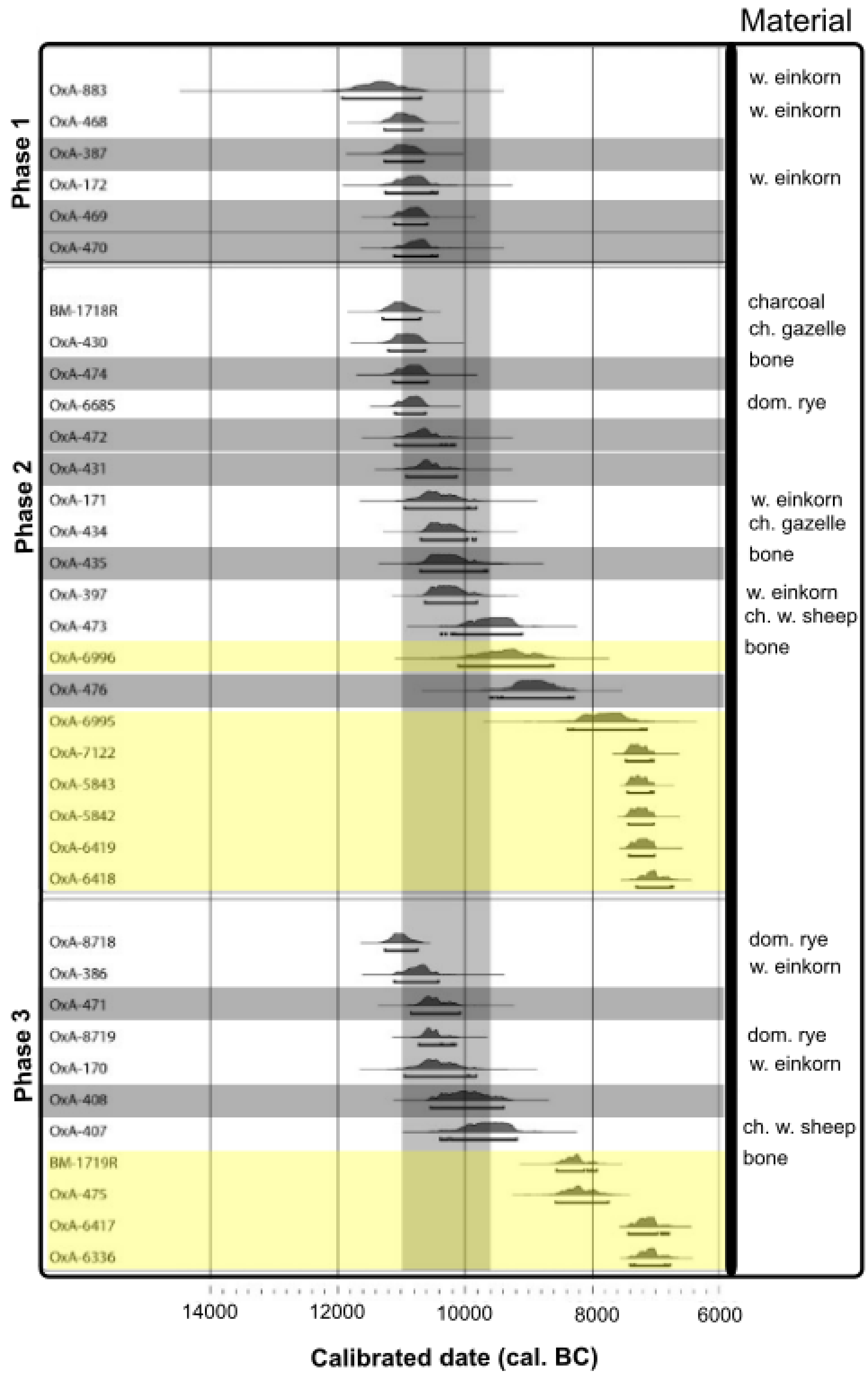


Figure 3.6 Calibrated radiocarbon dates ordered by phase for Epipalaeolithic Abu Hureyra. The horizontal grey bar shows the Younger Dryas. Grey banded dates identify dates on humic/fulvic fractions discounted by Moore et al. (2000). Yellow banded dates were deemed intrusive from Neolithic levels. Material type: w=wild, dom=domestic, ch=charred (all einkorn and rye are charred). Material type from Moore et al., 2000, p. 527-528, Appendix 1, Table A1. Adapted from: Colledge and Conolly (2010, p. 128, fig. 2). Model created by Colledge and Conolly, 2010 in OxCal v4.1.6 (Bronk Ramsey, 2010) and r5 Atmospheric data from Reimer et al., (2009).

3.4.4 Dating Abu Hureyra 2: The Neolithic

Despite suggestions that Abu Hureyra was continuously occupied from the Epipalaeoliothic into the Neolithic, this is supported by only one date (Trench G – OxA-1228) and there has been no evidence which is convincingly associated with the PPNA period. Therefore, this study treats Abu Hureyra 1 (Epipalaeolithic) and Abu Hureyra 2 (PPNB/C) as two periods of occupation, likely separated by a gap in occupation.

When constrained by a strict technical and contextual assessment as defined by Jacobsson (2019, p.11, table 1), only one of the Neolithic Abu Hureyra radiocarbon determinations passes all of the criteria (OxA-2169). It was obtained from charred seed (*Triticum monococcum/hordeum*) recovered from a firepit in Trench B, which qualifies as a secure context, representative of the context from which it was sampled and is assigned to period 2A (Table 3.1, Figure 3.5, Appendix 2A, 2B). However, by accepting, with caution, dates which only need comply with a more relaxed contextual criteria, whereby the sample can be reasonably associated with the context and there are no stratigraphic complications, there are ten dates associated with the Neolithic occupation of Abu Hureyra, all obtained from charred plant material (Jacobsson 2019, p. 11 table 2) which are modelled in Figure 3.7. Most of the dates proposed for trench phases which are included in this study, meet both the technical and relaxed contextual criteria (Table 3.1). However, there is not insufficient material from Trench B to securely identify the start of the Neolithic settlement of Abu Hureyra 2, although it likely began during the mid-PPNB. While some studies cite that Abu Hureyra was occupied from 8800 cal. BC (e.g. Styring et al., 2016), when referring to the periods of occupation in more general terms, this study uses 8600 cal. BC as a start date, but acknowledges that it is not possible to know exactly when the Neolithic occupation of the site started.

The dates obtained for Trench D, period 2A, phase 4 were all dated on charred bones material which is deemed unreliable as they were submitted prior to the development of the Lanting method (Lanting et al., 2001) and also discounted in the studies by Flohr (2016, p. 27) and Jacobsson (2019, SI, table 1). This study analysed material from Trench D, phases 3 and 4 (Table 3.1) which can be securely assigned to this period, despite the lack of quality checked radiocarbon dates, as the faunal records, dominated by wild gazelle, are consistent with this period of the site.

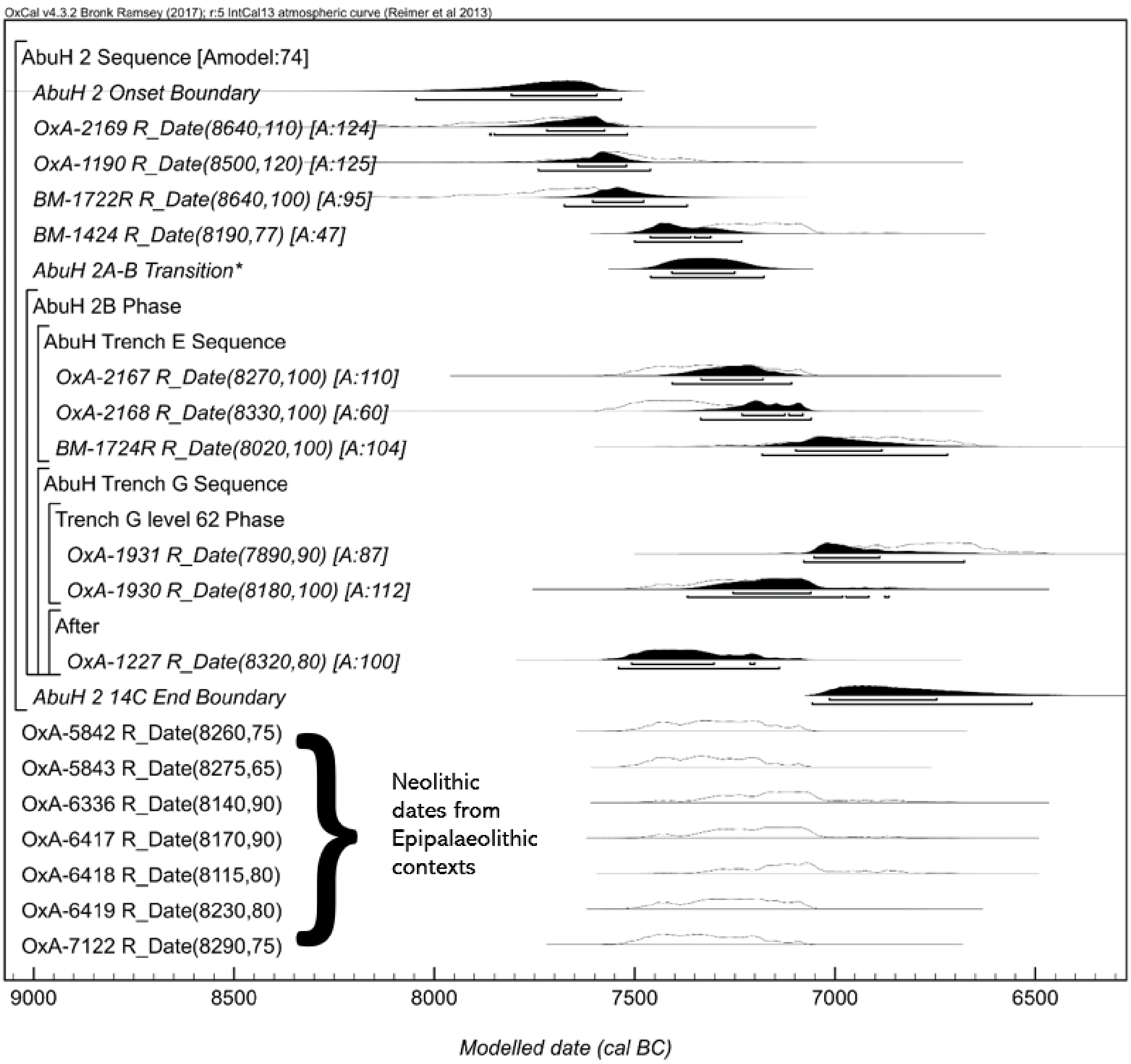


Figure 3.7 Modelled quality checked radiocarbon dates from Neolithic Abu Hureyra, calibrated using OxCal 4.3, calibration curve IntCal13 (Bronk Ramsey, 2009; Reimer et al., 2013). *Adapted from: Jacobsson (2019; supplementary figure s2).*

The quality checked and calibrated radiocarbon dates for Abu Hureyra, referred to within this study have a minimum error margin of 260 years ($^{+/-}$130 years at 2σ) up to a maximum error margin of 758 years ($^{+/-}$379 years). The chronological resolution overall for the site is relatively low which limits and calls for caution when identifying links between climatic events and corresponding environmental changes, with subtle changes in human lifeways at a site-specific level. However, this is somewhat compensated for in more general terms by the relatively large dataset of dates consistent with the mid-late PPNB (Appendix 2A).

Neolithic Abu Hureyra was divided into two key phases by the excavators, Period 2A and 2B, where there is a clear shift in the exploitation of plants and animals as well as a significant expansion of the settlement, Table 3.1, (Moore et al., 2000, pp. 251–252, 269–271). Recent reassessment and modelling of the available and quality checked ^{14}C dates, also indicate a division between periods 2A and 2B which occurred between 7465-7175 cal. BC at 95.4% accuracy (or 7410-7250 at 68.2% accuracy) (Jacobsson, 2017, p. 1683; 2019). In more general terms, when referring to periods of occupation at Abu Hureyra, this study uses the approximate midpoint, 7300 cal. BC as the start of Period 2B (Table 3.1). This temporal focus of this study is on identifying differences in plant-use between the relatively well-defined periods 2A and 2B as this will inform on changing human-plant-animal-environment interactions as the Neolithic economy developed.

The generally accepted date for the latest Neolithic period, 2C, at Abu Hureyra (Figure 3.5, Table 3.1) is based on one radiocarbon date from Trench B (OxA-1232). However, the changes in settlement pattern characteristic of the shift from period 2B to 2C are also evident in Trenches D and E. This study refers to period 2C in more general terms as 6200-6000 cal. BC, however, due the paucity of material dated from this period and severe erosion of parts of the mound, it is not possible to calculate a more precise end date for the site.

3.5 Diet, economy and lifeways at Abu Hureyra

The Neolithic settlement of Abu Hureyra is characterised by densely packed rectilinear mudbrick buildings, with polished plaster floors, and plaster was also used for vessels and storage containers (Moore et al., 2000, p. 256). The transition between periods 2A and 2B is marked by significant settlement growth (~8ha-~11ha), although in terms of material culture, the periods are quite similar. Period 2C marks a significant shift in the composition of the settlement, whereby spaces increase between buildings, often with large pits of burnt debris between them and a further increase in the exploitation of cattle and pig (Moore et al., 2000, p. 258). This section reviews the evidence from the zooarchaeological record, charred remains and other archaeological evidence for continuity and change in animal and plant management.

Six hundred and fifty samples were floated from the Neolithic phase of the site, of which ninety-four samples represented different phases and contexts from Trenches B, D, E and G were analysed by de Moulins (2000, p. 399). Animal bones were also systematically recovered during excavation and the faunal sequence published based on bones from Trenches B, D and E (Legge and Rowley-Conwy, 2000). The archaeobotanical and zooarchaeological evidence from Abu Hureyra 2, summarised in Table 3.6, have been interpreted as demonstrating an increased shift towards managed and domesticated plants and animals.

Table 3.6 Data from de Moulins (2000, pp. 399-416); Legge and Rowley-Conwy (2000, p. 423-472)

Site period, trench and phase	**Summary of plant remains**	**Summary of animals**
Period 2A Trench D, phases 1-5 Trench B, phases 1-7	• Relative uniformity between samples • Dominated by weed seeds (high numbers of small-seeded legumes) • Cereal grains ~10% of most assemblages • Low amounts of chaff	• Abundant gazelle • Caprines present, less ubiquitous than gazelle • Low frequencies of onager and cattle • Sporadic occurrence of pigs and fallow deer • Low numbers of hare and fox (declined since AH 1)
Period 2B Trench B, phases 8 and 9	• Slight increase in cereal representation • Low amounts of chaff • Weed seeds include large numbers of small-seeded legumes and wall-barley • Half of samples contained brome grass • Decrease in numbers of samples containing weed seeds between phases 8 and 9	• Characterised by reversal in proportions of gazelle to caprines • Large mammals dominated by caprines • Decline in gazelle • Decline in proportion of onager • Pig and deer become very rare • Increase in cattle (5-7% of identified bones)
Period 2B Trench E, phases 4-6	• Large numbers of weed seeds • Relatively low numbers of cereals • Gradual reduction in small-seeded legumes (especially after phase 6) • Some samples contained large numbers of Euphrates knotgrass and club rush • Reed and straw impressions in mudbrick fragments	
Period 2C Trench B, phase 10 Trench E, phase 8	• Cultivated barley and other cereal dominant in assemblage • Lower proportions of weed seeds	

The charred plant assemblage is characterised high numbers of weed seeds, and the presence of some domesticated type cereal. From the earliest PPNB phases of occupation, domesticated cereals including rye, wheat (einkorn and emmer) and barley (two and six rowed) are present, alongside pulses such as lentils, peas and vetches (de Moulins, 2000). In Trench B, there is a slight increase in cereal representation in period 2B, compared with period 2A, accompanied by a decrease in the numbers of samples containing weed seeds. In Period 2B, Trench E, the numbers of weed seeds continue to dominate the assemblage, though cereals are relatively low in number, there is also a gradual reduction in small-seeded legumes. By period 2C, cultivated barley and other cereals dominate the charred archaeobotanical assemblages and there are lower proportions of weed seeds (de Moulins, 2000).

In the zooarchaeological assemblage period 2A is dominated by gazelle, with low numbers of caprines, onager and cattle as well as the sporadic presence of pig and fallow deer. The change to period 2B is defined by the reversal in the exploitation of gazelle to caprine, whereby caprines dominate the period 2B mammal assemblage. There is also a decline in the representation of onager in period 2B, while pig and fallow deer become rare with an increase in cattle exploitation. The switch from dependence on gazelle to caprines defines the shift from period 2A to 2B (Moore et al., 2000, p. 257), however, the assemblages which demonstrate this reversal are recovered from different trenches (Trench B and Trench E) which are situated on different parts of the mound. It is therefore possible that the transition between periods could represent different uses of spaces, perhaps where the processing of different animals took place in different areas. Based on the lack of morphological change in sheep/goat bones throughout the Neolithic occupation of the site, which spanned over 2000 years, Legge and Rowley-Conway (2000), conclude that the caprine population was likely already domesticated at the start of period 2A. Low numbers were initially kept during the period 2A occupation and protein intake was largely based on wild animal meat, mostly gazelle. In period 2B, the exploitation of gazelle dramatically decreases which Legge and Rowley-Conwy (2000) attribute to increased pressure on the population as a result of the growth of the settlement.

3.6 Summary and applications of chapter

This chapter has provided the context and rational for the key case study in this research, Neolithic Abu Hureyra, Syria. In particular, the site's location and environmental reconstructions (section 3.2) will be drawn on throughout the thesis to discuss and evaluate resource management at Abu Hureyra. The critique of the chronology of Abu Hureyra (Section 3.4) also provides a baseline to assess how the inhabitants may or may not have responded to changes in climate and environment, as well as more robustly identifying changes in plant use and resource management between Periods and phases of occupation across the site. The archaeobotanical and zooarchaeological data from Neolithic Abu Hureyra, is referred to throughout this thesis, particularly in the integrated interpretation and discussion chapters (Chapters 10 and 11). The following chapter, provides more specific information about the excavation strategies and samples analysed in this research.

Chapter 4. Materials and Methods

4.1 Materials

This chapter firstly describes the sampling strategy at Abu Hureyra and the rationale for specific materials selected for analysis in this research. This chapter then provides a summary of the samples analysed for each technique with reference to the more detailed sample and context information available in the Appendix.

4.1.1 Abu Hureyra sampling strategy

All materials analysed from Abu Hureyra were recovered by Prof. Andrew Moore and colleagues during the excavations in in 1972 and 1973. Seven trenches were excavated which were located in key areas across the mound (Figure 4.1). The excavation was part of a rescue mission, prior to the construction of the Tabqa dam, which would flood the site, which now lies under Lake Assad. A wealth of material was excavated from the site and distributed to institutions and museums across Asia, N. America and Europe for further study (Moore et al., 2000, pp. 547–548, Appendix 7). Professor Andrew Moore kindly donated a substantial archive of archaeological environmental samples and materials from the site for scientific analyses to different institutions which have been recorded on a database (Appendix 1).

The UoR archive includes over 100 bulk sediment samples, which mostly represent ashy occupation residues with frequent charcoal inclusions, as most were collected for potential future radiocarbon dating. There are also over 100 fragments of floor plaster, fragments of white ware vessels, plaster with reed impressions and other miscellaneous plaster. Other materials include bitumen, yellow and red ochre, burned clay, clay balls, haematite, malachite and gypsum fragments.

This research examined phytoliths and faecal spherulites from bulk sediment samples and also from plaster floor fragments (4.1.2). The elemental composition of sediments and plaster fragments was determined by pXRF (4.1.3). Based on the results of the phytolith and spherulite analysis, a sub-set of 50 sediment and plaster floor fragments were selected for further analysis by GC-MS (4.1.4). The justification for the selection of specific samples is provided in the corresponding sections below.

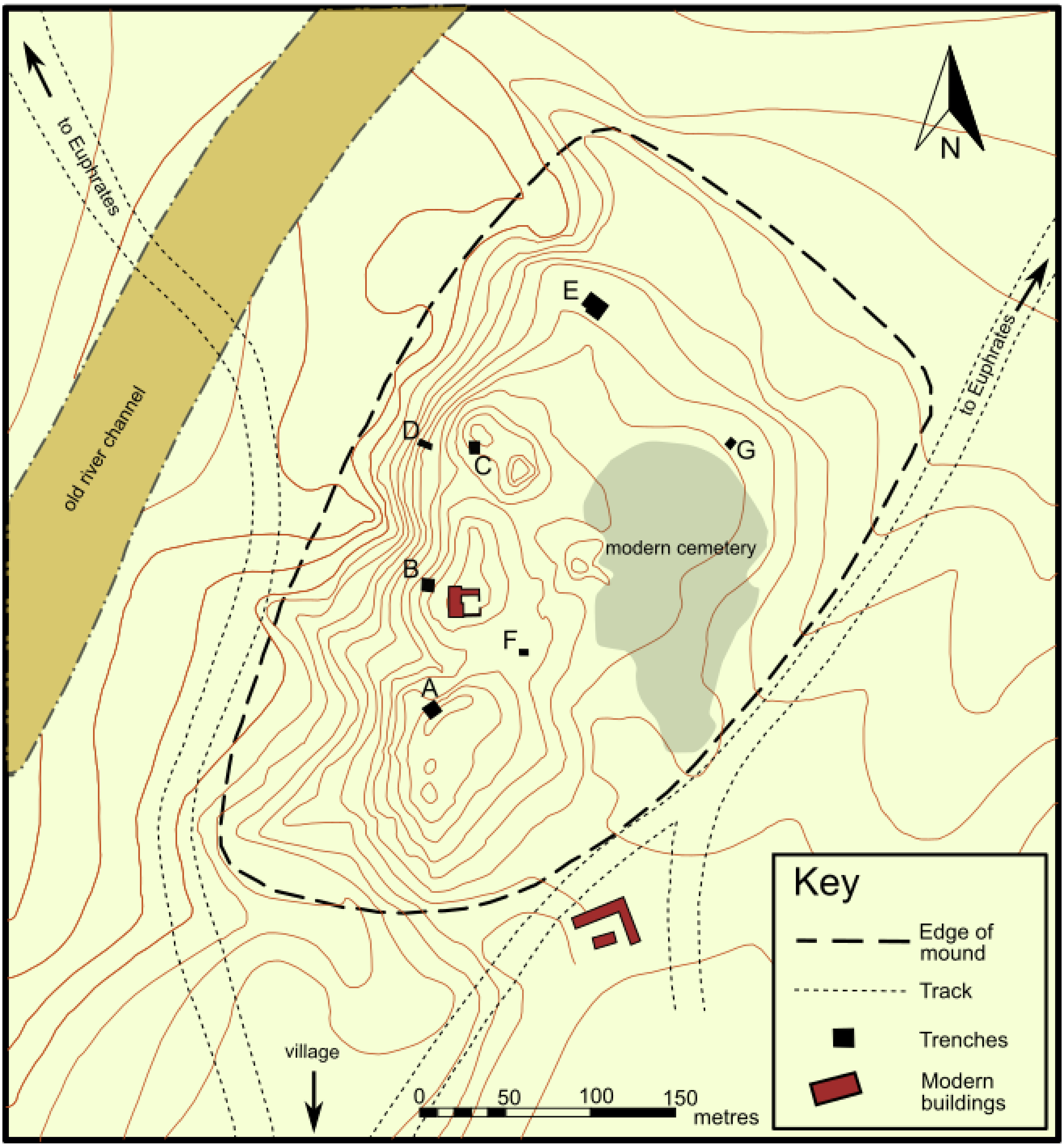


Figure 4.1 Contour plan of the Abu Hureyra mound, including the locations of trenches. Adapted from Moore et al. (2000, p. 257, fig. 8.75).

4.1.2 Materials selected for phytolith and spherulite analyses

Bulk sediment samples

In addition to the material collected for flotation, over 100 bulk sediment samples were also recovered. The bulk sediments were primarily collected to include charcoal for potential further radiocarbon dating and ranged in size from a few grams to almost a kilogram of material. Most sediments available for analyses were ashy occupation residues with frequent charcoal inclusions.

The similar nature of all of the bulk sediments selected for analysis provided a good basis for comparing different areas of the site and changes over time. As most of the samples contained charred material and ash, the samples were also appropriate to compare with the charred macrofossil record, as it could be assumed that both represented spaces and plants which had been burned.

Eighty-eight bulk samples were analysed for phytoliths and faecal spherulites, which represented all of the bulk sediments which were available, feasible for analysis (sufficient material) and aligned to the research questions (Table 4.1). Material for the study in this research was selected to represent different spaces within in the site, Trenches A, B, C, D, E and G, and where available, both internal and external spaces and different material types including ashy and unburnt (Table 4.1, Figure 4.1). Material was also selected to cover different periods of occupation from the site, which include three samples from the earlier Epipalaeolithic occupation for comparative purposes. As there are only three extant sediment samples from the Epipalaeolithic phase of the site, a full comparison with the Neolithic was not feasible. That is why an initial aim of the study was to identify phytoliths from ground stone tools which are from both Epipalaeolithic and Neolithic contexts to enable a comparison between these two periods. As ground stone tools from Abu Hureyra are held in museums (Ashmolean, British Museum) this aspect of the research was unable to take place because of Covid-19 restrictions (see Covid-19 impact statement).

Most samples were selected on the basis that the context from which they had been recovered had been assigned to period 2A or 2B, to enable a comparison of the plant economy between the two periods, which is important as it spans the intensification of agriculture and unprecedented growth of the settlement. Extensive contextual analysis has been published about the site, most of which is available in Moore et al. (2000) and on the Abu Hureyra online database (https://www.rit.edu/academicaffairs/abuhureyra/statement.php). Fifty of the available bulk sediment samples available for analysis were recovered from Trenches A and C, where the accompanying contextual information has not yet been fully published, however, Prof. Andrew Moore has kindly provided a provisional phasing for Trenches A and C. Therefore, the material can be assigned to periods 2A and 2B, and has been incorporated to provide a greater sample size through which to analyse changes in phytolith and spherulite concentrations between the two periods. Plans for key areas of the site discussed in this analysis are provided below and annotated with key sample numbers where possible (Figures 4.1-4.6). Contextual information including space types and deposit types with sample descriptions are available for all bulk sample material in Appendix 1A.

Table 4.1 Summary of periods and phase of occupation residue sediment samples selected for integrated phytolith and spherulite analysis (see Table 3.1 and Figure 3.5 for associated dates and attributes)

Trench	Period (and phase if available)	Space and deposit types	Total samples analysed
E	1A, phase 1	Pit fill – occupation residues	2
E	1C, phase 3	Pit fill – occupation residues	1
B	2A, phase 5	Internal occupation residues	1
B	2A, phase 7 (Fig 4.2)	Internal occupation residues	1
D	2A, phase 4 (Fig 4.3)	external activity area occupation residues (11) external pit fill with *Bos* skull (2)	13
C	2A	Unknown	19
A	2B	Unknown	12
C	2B	Unknown	12
E	2B, phase 5 (Fig 4.4)	External occupation residues (9) External hearth base (1) Internal occupation residues (2)	12
E	2B, phase 6	External occupation residues (3) External pit fill (1) Internal occupation residues (2)	6
E	2B, phase 7	External occupation residues	3
F	2B	Unknown – poor phytoliths, not included	1
G	2B, phase 1	External fire spot	1
G	2B, phase 2 (Fig 4.5)	Internal occupation residues	2
Trench G	2B, phase 3 (Fig 4.6)	External occupation residues	2
Total number of samples analysed for integrated phytolith and faecal spherulite analysis: 88			

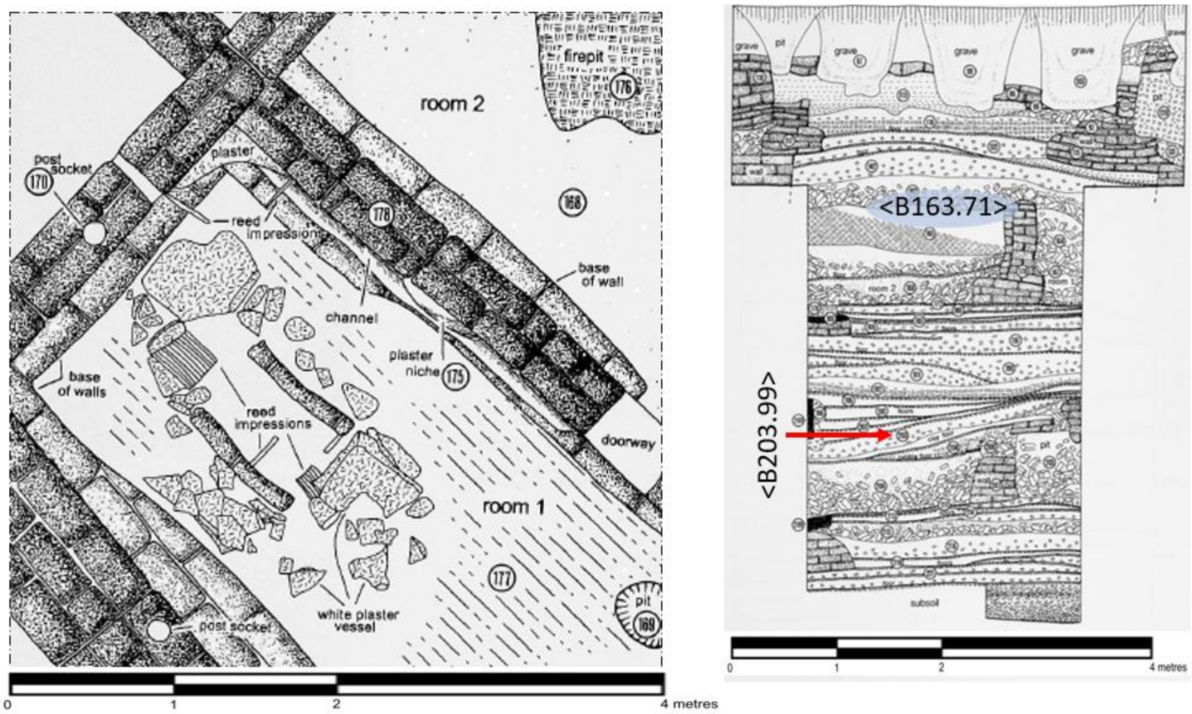


Figure 4.2 Plan of Trench B, phase 7 (adapted from: Moore et al. 2000, pp. 191, 199, fig. 8.11, 8.2)

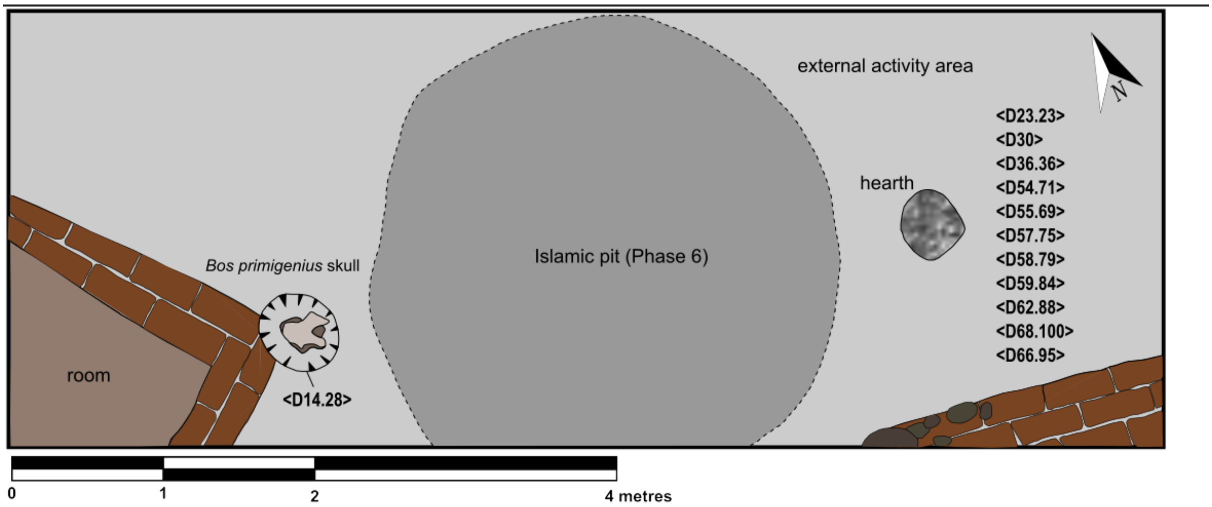


Figure 4.3 Plan of Trench D, phase 4 (adapted from Moore et al. 2000, p. 216, fig. 8.33). Approximate locations of samples in bold. All occupation residues analysed from Trench D, phase 4 (except for D14.28) represent successive layers of occupation in the external activity areas. Samples are ordered in stratigraphic sequence as dug (youngest at top).

Figure 4.4 Plan of Trench E, phase 5 (adapted from Moore et al. 2000, p. 233, fig. 8.51). Sample locations are approximate and where applicable, ordered in the stratigraphic sequence as dug (youngest at top).

Figure 4.5 Plan of Trench G, phase 2 (adapted from Moore et al. 2000, p. 247, fig. 8.65). Approximate sampling location of material analysed in this study shown in bold.

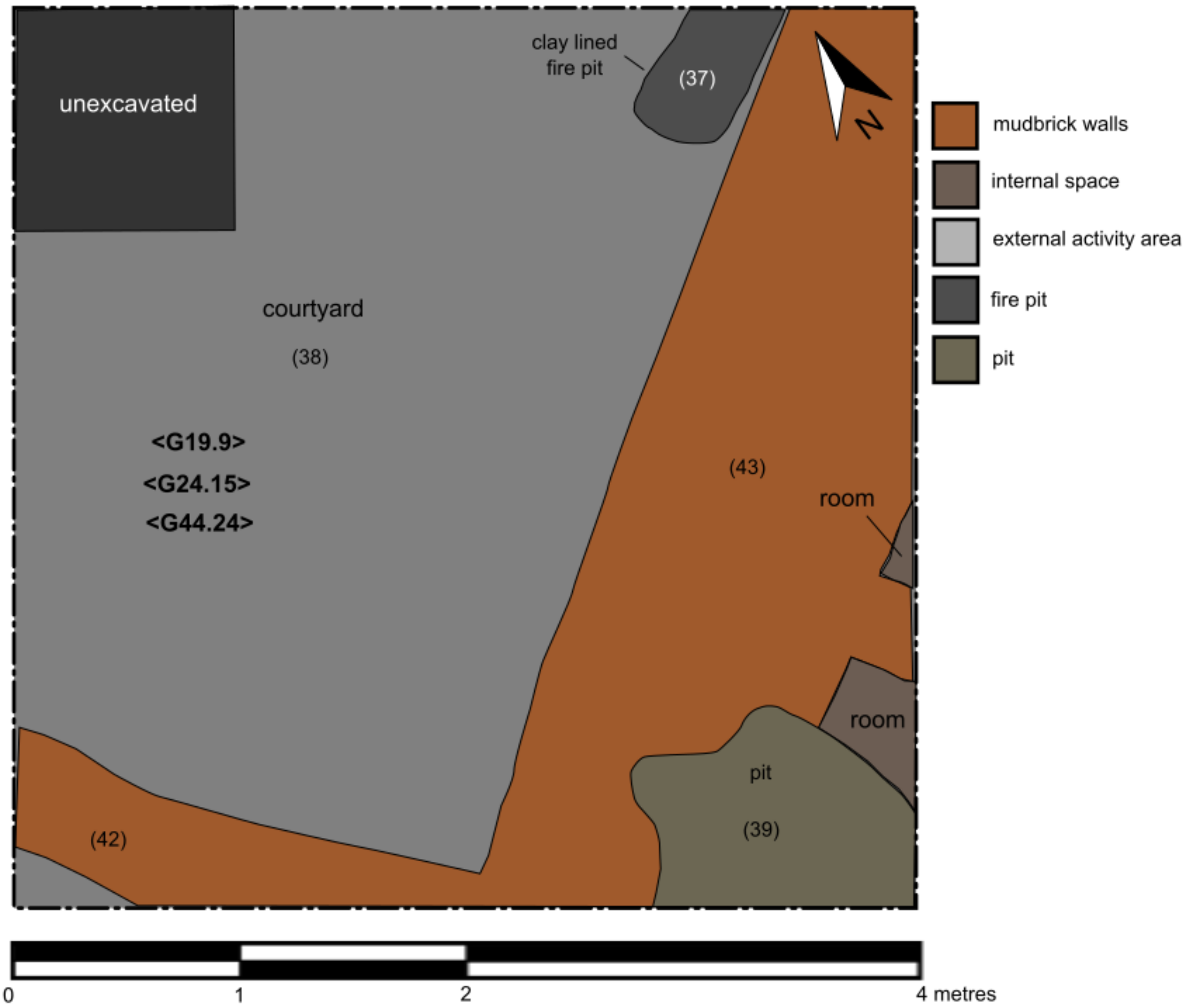


Figure 4.6 Plan of Trench G, phase 3 (adapted from Moore et al. 2000, p. 247, fig. 8.65). Approximate sampling location of material analysed in this study shown in bold.

Plaster fragments

Plaster was a major component of the built environment at Abu Hureyra and used to construct internal floors, plaster walls and make storage vessels and niches within the buildings. Phytoliths and spherulites were also analysed from six gypsum plaster floor fragments to provide information about the plant types used in construction and to assess whether animal dung was used as a building material. Contextual and sample information for all plaster fragments are available in Appendix 1B.

Table 4.2 Summary of the periods and phases of plaster fragments selected for phytolith and spherulite analysis

Trench	Period and phase	Area recovered from	Number of fragments
Trench D	2A, phase 3	Internal in situ (1) External – secondary deposition (1)	2
Trench D	2A, phase 4	External – secondary deposition (1)	1
Trench E	2B, phase 7	External – secondary deposition (1)	1
Trench B	2C, phase 10	External – secondary deposition (1)	2
Total number of plaster fragments analysed for phytoliths and spherulites	**6**		

4.1.3 Materials for pXRF

All material which was analysed for phytoliths and spherulites including bulk samples of occupation residues was also analysed by pXRF to enable associations between the elemental composition of samples and concentrations of phytoliths and spherulites. A larger set of floor plaster fragments (n=17, Table 4.3) were selected for pXRF analysis, compared with those analysed for phytoliths and spherulites. The floor plasters were selected to represent different trenches (B, D and E) and different time periods (2A, 2B and 2C), as well as those which appeared morphologically similar and different, to assess changes and consistencies in building materials over time. Further analysis was conducted on a range of different materials including yellow and red ochre, bitumen, gypsum crystals and other types of plaster, including white ware "vaiselle blanche" and fragments of plaster with reed impressions. Descriptions of all the material analysed by pXRF are available in Appendix 1B.

Table 4.3. Summary of floor plaster fragments analysed by pXRF

Trench	Period, phase	Area recovered from	Number analysed
Trench E	1A	External – secondary deposition (probably intrusive from Neolithic layers)	1
Trench D	2A, phase 3	Internal in situ (1) External – secondary deposition (1)	2
Trench D	2A, phase 4	External – secondary deposition (1)	1
Trench B	2B, phase 9	External – secondary deposition (1)	1
Trench E	2B, phase 7	External – secondary deposition (2)	2
Trench E	2C, phase 8	External – secondary deposition (2)	2
Trench B	2C, phase 10	External – secondary deposition (2)	5
Trench D	3, phase 6	External – secondary deposition (3)	3
Total number of floor plaster fragments analysed by pXRF	**17**		

Table 4.4 Summary of non-floor plaster materials analysed by pXRF

Material	Context recovered from	Number analysed
Wall plaster	Internal ashy occupation soil with floor surfaces (1) External brown silty wash and occupation debris (1)	2
Burned plaster	Grey ashy occupation soil, stones	1
White plaster with reed impressions	Internal fill (2) External occupation debris (1)	3
White ware vessel "vaiselle blanch"	Internal fill/occupation debris (7)	7
Gypsum plaster with reed impression and red paint	Internal room fill/occupation debris (1)	1
Burned clay object	Occupation soil from surface cleaning	1
Hardened clay balls	Occupation soil over pit complexes	1
Gypsum	External occupation residues	4
Ochre (red and yellow)	External occupation surfaces	4
Haematite	Ashy occupation soil in pit	1
Bitumen/carbonised material	Internal occupation residues	8
Total number of "other" (non-floor plaster or sediment) analysed by pXRF	**33**	

4.1.4 Materials for GC-MS

Archaeological sediments and floor plaster fragments were selected for GC-MS analysis to detect whether faecal biomarkers are present and ascertain whether dung was a potential depositional pathway for plant remains. In addition, one fragment of white ware vessel, a piece of gypsum plaster with reed impressions (not floor) and a small (<50mm^3) clay ball, detailed in Table 4.5, were analysed for comparative purposes. Analyses focused on occupation soils and plaster floor fragments from external areas, associated with intensive domestic activity and refuse deposition (n=20) (Moore et al. 2000, p. 268). This analysis targeted material from Neolithic periods 2A and 2B to enable comparison between these periods as the PPNB developed. Sediments were selected where spherulites had been identified (in varying concentrations, n=15) and where no spherulites had been identified (n=8), but where a faecal component was suspected based on contextual data and other analyses. Additional occupation soil and plasters representing different spaces (internal/features, n=3) and time periods (Epipalaeolithic and Neolithic period 2C, Fig. 1) and controls from sediments with no suspected faecal component (n=2) were also analysed for comparison. Where possible, plaster floors were paired with occupation soil from the same spaces to help distinguish between biomarkers which represent construction material and activity residues.

Table 4.5 Samples selected for analysis by GC-MS for the detection of dung biomarkers

Samples **Total n = 45**	**Context and deposit type**	**Material type**
Trench E, period 1, phase 1 and 3 N = 3	Pit fills – occupation soil	Sediment – brown silty, slightly ashy
Trench D, period 2A, phase 4 N = 7	External activity area - occupation soil	Sediment – grey to very dark grey, ashy
Trench D, period 2A, phase 3 and 4 N = 8	External activity area (and 1 x internal) - occupation surfaces	Plaster fragments – floor plasters with reddish or dark smooth surface.
Trench E, period 2B, phase 5 and 6 N = 4	Internal occupation soil	Sediment – brown to dark grey, ashy
Trench E, period 2B, phases 5, 6, 7 N = 8	External activity areas – occupation soil	Sediment – grey to dark greyish brown, ashy
Trench E, period 2B, phase 7 N = 3	External activity areas - occupation surfaces	Plaster fragments – smooth, dark surfaces
Trench B, period 2B, phase 9 N= 3	External activity areas – occupation surfaces	Plaster fragments – smooth dark coloured surfaces
Trench B, period 2C, phase 10 N = 3	Internal - occupation surfaces	Plaster fragments – smooth, dark surfaces
Trench B, period 2C, phase 10 N = 3	External activity areas and pit fill of occupation residues and surfaces	Plaster fragments – smooth, dark surfaces
Trench B, Period 2A, phase 8 N = 1	Internal - Neolithic burial, mudbrick and plaster collapse	Gypsum plaster with reed impression and red paint
Trench E, Period 1, phase 1 N = 1	Occupation soil over pit complexes 1 and 2	Hardened clay balls
Trench B, Period 2C, phase 10 N = 1	Internal, mudbrick collapse and plaster	White ware vessel
Total number of sediments analysed by GC-MS: N = 22 **Total number of floor plaster fragments by GC-MS: N = 20** **Total number of other plaster types and clay analysed by GC-MS: N = 3**		

4.2 Methods

4.2.1 Phytolith analysis

4.2.1.1 Rational for methodology selection

A range of protocols have been applied to extract phytoliths for analysis (e.g. Albert et al., 1999; Rosen, 2005; Piperno, 2006; Katz et al., 2010; Pearsall, 2015), often selected based on the sediment type, deposit type, abundance of phytoliths and specific research questions. The Katz et al. (2010) methodology has been selected for this research because a) the chemicals used are less toxic and relatively safe and inexpensive compared with other procedures for phytolith extraction, b) phytoliths are distributed homogenously on the slide, compared with phytolith procedures that use a mounting agent to mix phytoliths extracted in powdered form and c) the sample preparation process can be completed rapidly (~6 samples in less than an hour). Furthermore, a pilot study carried out on a similar set of samples from Abu Hureyra, as part of my Maters thesis, showed the Katz et al. (2010) method to be effective for analysing phytoliths from the site. Conducting further research for my PhD following the same methodology enabled a more accurate comparison between samples previously studied and samples analysed as part of this study.

A disadvantage of the Katz et al. (2010) method is the low pH of the SPT which dissolves the phytoliths and crystallises on the slide within 3-6 hours, meaning slides are not permanent and must be counted immediately following extraction. This issue was mitigated in this study as the remaining supernatant was diluted with de-ionised water, vortexed and centrifuged for five minutes at 5000rpm, leaving pellets at the bottom. Excess water was removed and the process repeated, twice or until the liquid was clear after centrifuging. The pellets are dried at 50°C for ~24hours, after which they were archived or mounted using Entellan New Merck for further morphological identification and micrographs.

Phytoliths were extracted from occupation residue sediments and plaster fragments to identify different types of vegetation (woody, grasses, wetland reeds and sedges) to inform on plant-use and resource management as agriculture developed at Abu Hureyra.

4.2.1.2 Phytolith Extraction and quantification

Phytoliths were extracted following the rapid extraction method of Katz et al. (2010). Sediment samples were sieved to remove fractions more than 0.5mm and combusted at 500°C for ~90 minutes in a muffle furnace to remove organic material. An aliquot of ~40mg was weighed into a 0.5ml conical plastic centrifuge tube. 50µl of 6NHCl was added to dissolve carbonates, followed by 450µl of Sodium Polytungstate (SPT) ($Na_6(H_2W_{12}WO_{40})H_2O$) with a density of 2.4g/ml to concentrate the phytoliths. The solution was sonicated for 5 minutes then centrifuged for 5 minutes at 5000

RPM. Microscope slides were mounted with 50µl of the supernatant using a 24mm x 24mm coverslip, representing 10% of the total number of phytoliths per gram of sediment which enabled quantitative comparisons between samples. The phytoliths were therefore mounted in the SPT for quantification.

A minimum of 200 phytoliths with diagnostic morphologies were counted in each sample where possible, using a Leica DMEP optical microscope at x200 magnification and x400 for further morphological identification. Digital images were recorded using a Leica DFC420 camera and DMPL optical microscope. Phytoliths which could not be identified because of surface pitting and etching caused by dissolution were recorded as 'weathered morphotypes' which are expressed as a % of the total phytolith assemblage for each sample. Three or more conjoined cells were counted as multi-cells and the individual cell morphologies were noted to identify the plant type or part it originated from. Individual cells within a multicellular structure were not counted, as quite often the state of preservation was not sufficient to ensure consistency, however, the approximate number of individual cells was noted.

Phytolith morphologies were identified using standard published literature (Twiss et al., 1969; Brown, 1984; Mulholland and Rapp, 1992; Rosen, 1992; Piperno, 2006), the Phytcore online reference collection (http://www.phytcore.org) (Albert et al., 2016) and the University of Reading phytolith reference collection. Nomenclature used within this study followed the International Code for Phytolith Nomenclature 1.0 and 2.0 (Madella et al., 2005; Neumann et al., 2019a; 2019b) where possible and appropriate, particularly for geometric morphologies. Modern reference studies (Albert et al., 2008; Tsartsidou et al., 2008; 2009; Portillo et al., 2014; 2017; Neumann et al., 2019a; 2019b and references therein; Chen et al., 2020) were referred to for the interpretation of phytolith morphologies.

4.2.1.2 Phytolith extraction from other materials (plaster vessels, mudbrick and plasters)

A mini spatula was used to scrape material from the surface of plaster vessels, mudbrick and plaster samples. The material was collected on clean foil and if required, gently crushed using a sterilised pestle and mortar which was thoroughly washed and rinsed with deionised water, then dried between samples to prevent contamination. Phytoliths were then extracted from the loose material following the procedure outlined above. For the plaster samples, the sieving and ashing steps were not required as particle size was already small and samples were in low organic content which did not obscure the phytolith identification or counting.

4.2.2 Spherulite analysis

Faecal spherulites were identified and analysed as a rapid method to establish if there was a faecal component in the archaeological sediments and plasters analysed in this study (Aim 2). Faecal spherulites were identified and quantified following a method based on Canti (1999a). Approximately 1mg of dried sediment was weighed onto a 25x75mm microscope slide and thoroughly mixed with clove oil and distributed as evenly as possibly using a new glass pipette for each sample to avoid contamination. This method was rapid and can be conducted with a very minimal lab set up and using no harmful chemicals. A disadvantage of this method, however, is that as the sediment is not at all processed, other materials, such as minerals, charcoal and sediment aggregates sometimes obscured spherulites. Spherulites were counted on an optical microscope DMEP at x400 magnification in crossed polarised light (XPL). Five transects of the slide were counted for each sample. The following equation calculates the number of spherulites per gram of sediment:

a)

$$\left(\frac{no.\ faecal\ spherulites\ counted}{area\ counted}\right) \times area\ of\ the\ slide = no. spherulites\ on\ slide$$

b)

$$\frac{no. of\ spherulites\ per\ slide}{initial\ weight\ (g)} = \boldsymbol{number\ of\ faecal\ spherulites\ per\ gram\ of\ sediment}$$

This research also conducted a pilot study to compare spherulite quantification methodologies. Faecal spherulites were quantified in four modern ruminant dung and animal penning dung associated samples. The second method extracted faecal spherulites and followed the method outlined by Gur-Arieh et al. (2013). This method is based on the rapid phytolith exaction methodology described by Katz et al. (2010) but excluded the hydrochloric acid step which would dissolve any spherulites. The advantage of this method is the more homogenous distribution of the sediment across the slide, disaggregation of clays that spherulites could be bound up in, and separation of spherulites from heavy particles. In addition, following the same method for extracting and quantifying both phytoliths and spherulites makes it easier to integrate the results of both analyses.

Spherulites were identified by size, the presence of a fixed cross of extinction and colour; low order white become blue/yellow in opposite quadrants when using the λ plate. The faecal spherulites were compared with modern reference material from cow, sheep and goat dung to aid identification. Spherulite concentrations were compared with ethnoarchaeological datasets which follow similar quantitative approaches (Tsartsidou et al., 2008; Gur-Arieh et al., 2013; Portillo et al., 2014; 2017; 2020).

4.2.3 Portable Hand-Held X-Ray Fluorescence (pXRF)

pXRF was conducted to characterise sediments and materials to compare elemental compositions between materials to inform on resource selection and technological developments over time, as well as provide new insights into different uses of space.

Sediments were sieved at 2mm, and readings were taken in the plastic sample bag. In an initial assessment, different parts of plaster fragments were analysed to assess the variation between plaster floor surfaces compared with the plaster matrix. Readings were also taken from several parts of white plaster fragments with reed impressions, for example in the reed impressions and on the surface (detailed in Appendix 1B). All readings were taken three times to identify any erroneous or anomalous readings. pXRF was conducted using a Thermo Fisher Niton Goldd+ XL3t pXRF Analyser. Material was analysed in the Cu/Zn Mining mode for four minutes per sample (30 seconds for main, 90 seconds for low, 30 seconds for high and 90 seconds for light elements). A sample of Camberley sand was used as a standard. Readings from the Camberley sands reference material were taken at the start of every batch of samples, and also approximately every thirty readings.

4.2.4 Data Exploration and Statistical analyses

Principal Component Analysis was conducted in IBM SPSS Statistics for Windows, version 25.0 (IBM Corp Armonk, USA) on the elemental composition data acquired by pXRF analysis. The pXRF analysis determined the concentrations of thirty-nine elements. The data was checked for large error readings (>10%) which were removed from the data set. Elements with a high proportion of the results below the limit of detection (< LOD) were excluded from the analysis. Where concentrations of some elements were below the limit of detection in samples, the value was replaced with the corresponding lower limit of detection which is provided by the analyser as the error reading value.

Principal Component Analysis (PCA) was conducted with orthogonal rotation (Varimax). KMO verified the sampling adequacy. Bartlett's test of sphericity indicated whether correlations between items were sufficiently large for PCA. Eigenvalues of >1 were included in the analyses. Scatter plots were generated to visually assess the data and highlight any groupings or clusters.

4.2.5 pH determination

pH was measured for a subset of sixteen sediment samples, which were selected to represent different trenches, time periods and deposit and material types to provide an overview of general preservation conditions across the site. C. 10g of air-dried sediment was sieved at 2mm and weighed into a 50ml centrifuge tube. 25ml of ultra-pure water was added using an automatic dispenser. The tube was then capped and placed on an end over shaker working at 20-30rpm for 15 minutes. The pH meter was calibrated with pH 7.00 and 9.22 buffers. The pH electrode was placed into the soil suspension, and the pH reading was taken after 30 seconds. The electrode was cleaned with ultra-pure water between samples to prevent contamination.

4.2.6 Methods of preparation and analysis of faecal biomarkers in sediments using GC/MS

GC-MS analysis was conducted to detect if faecal biomarkers were present in any of the samples. The primary aim of the GC-MS analysis was to assess whether dung was a component of any of the samples, and if present, which species it was produced by (human, pig, ruminant) to inform on the depositional pathways of plants remains identified in this study, consumption/foddering practices and resources used for construction. A secondary aim was to compare methods for identifying dung between the identification of dung through faecal spherulite analysis and the detection of dung by GC-MS.

Given the anticipated low abundances of faecal biomarkers, the GC-MS analyses of sterols and bile acids was performed using gas chromatography-high resolution mass spectrometry (GC-HRMS) using the GC/Q-TOFMS, to take advantage of its higher sensitivity and selectivity (Shillito et al., 2020). Materials were analysed by GC-HRMS following a microwave extraction protocol as outlined in Shillito et al., 2020, with sterol and bile acid fractions isolated, enabling identification of faecal inputs (Elhmmali et al., 1997; Bull et al., 1999).

Both sediments and plasters were crushed using a pestle and mortar, where required, and approximately 1g of material was weighed into a microwave extraction test tube. 50µL of hyocholic acid (0.1 mg ml^{-1}) and 50µL of preg-5-en-3β-ol (0.1 mg ml^{-1}) solution are added as internal standards. The lipids were microwave-assisted solvent extracted using 10mL of DCM:CH_3OH (2:1 v/v), heated to 70°C for 10 minutes, then held at 70°C for 10 minutes and then left to cool for 20 minutes. The material was centrifuged at 1700 rpm for 3 minutes and the supernatant was decanted into a 28mL vial, three times, then dried under nitrogen to obtain the total lipid extract (TLE).

The TLE was saponified by adding 2mL of sodium hydroxide in CH_3OH (5M) and heated at 120°C for one hour. When the samples had cooled to room temperature, 5mL of DCM (dichloromethane) extracted H_2O were added, and acidified to pH 3-4 using hydrochloric acid (6M) in an ice bath. The saponified TLE was extracted with 5mL of chloroform ($CHCl_3$) was added three times to extract the saponified TLE into a 28mL vial and the excess liquid was dried under N_2.

To separate the acid and neutral fractions, the saponified TLE was dissolved in 1mL of DCM:2-propanol (2:1 v/v) and an aminopropyl column was preconditioned with 6mL of DCM:2-propanol (2:1 v/v). The neutral fraction was eluted with 6mL of DCM:2-propanol into a 7mL vial and the acid fraction was eluted with 9mL of 3% acetic acid in methanol (v/v), also into a 7mL vial, and both dried under N_2.

The ketone/wax ester fraction and alcohol fraction were eluted from the neutral fraction by column chromatography. The neutral fraction was dissolved in 1mL of DCM and a dried activated silica gel 60 column was preconditioned with DCM. Ketones/wax esters were eluted with DCM and the alcohol fraction was eluted with DCM:CH_3OH (1:1), then both fractions were dried under N_2. The acid fraction was methylated using 200µL of BF_3-methanol as a reagent at 70°C for two hours. The methyl esters were extracted using 2mL of $CHCl_3$ threes times, then dried down under N_2.

The monocarboxylic FAMEs and bile acids fractions were eluted by column chromatography. A preconditioned silica column was loaded with 6mL of DCM:hexane (2:1 v/v). Monocarboxylic FAMEs were eluted with 6mL of DCM:hexane (2:1 v/v), and the hydroxy carboxylic acid methyl esters and bile acids were eluted with 9mL of DCM:CH_3OH (2:1 v/v). The bile acids were transferred into two 3.5mL vials, so that half of the material could be analysed and the other half archived.

The alcohol fraction was derivatised using 100µL of HMDS:TMCS:Pyridine (3:1:9) at 70°C for one hour, then dissolved in 100µL of hexane for the initial GC analysis, which was then adjusted if the material required further dilution or concentration. The bile acid fraction was derivatised using 100µL of HMDS:TMCS:Pyridine (3:1:9) at 70°C for 12 hours (or overnight) and was then dissolved in 50µL of hexane for analysis using the GC and GC-MS.

4.2.6.1 Instrumental analysis

An HP 5890 Series II gas chromatograph was used for the initial screening of biomarkers. 1µL of the trimethylsilylated sterols and methylated and trimethylsilylated bile acids were injected with an on-column injector. A fused silica capillary column (50m x 0.32mm) coated with a 100% dimethylpolysiloxane nonpolar stationary phase (HP-1, 0.17µm; Agilent) was fitted in the GC. One sterol standard was also analysed on the GC for each batch of the sterol fractions to assess whether the concentrations needed adjusting. The temperature program on the GC for the analyses of the sterol fraction, held at 50°C for 2 minutes, then a gradient increase to 200°C at 10°C min^{-1}, followed by ramping to 300°C for 20 minutes at 3°C min^{-1}, and lastly an isothermal at 300°C for 20 minutes. The carrier gas was helium, which was set to a constant flow of 2.0ml min^{-1}. Column effluent was monitored using the flame ionization detector (FID), which was kept at a constant temperature of 300°C. DataApex Clarity (version 2.6.6.226) was used to obtain the data. The GC temperature program for the analyses of the bile acid fraction held at 40°C for 1 minute, then a gradient increase to 230°C at 20°C min $^{-1}$, followed by 300°C at 2°C min^{-1}, and finished with an isothermal at 300°C for 20 minutes. The carrier gas and data acquisition were the same as for the sterol fraction.

An Agilent 7890-7200B GC/Q-TOFMS was used for the GC-MS analyses of sterols and bile acids. A 7693 autosampler injected 1µl of each sample with a multimode inlet which was set to track the temperature of the oven. Samples were loaded onto a 50m x 0.32 mm (Agilent) fused silica capillary column, which was coated with a 100% dimethylpolysiloxane nonpolar stationary phase (HP-1, 0.17µm; Agilent. The same temperature programs were used as for the initial GC screening for both bile acid and sterol fraction, as was the carrier gas (Helium) and flow. The GC transfer line was set to 320°C, the ion source at 230°C and the quadrupole at 150°C. The MS acquired in the range of mass to charge ratio (*m/z*) 50 to 1050 with a scan rate of 0.2Hz in extended dynamic range mode.

4.2.6.2 Data analysis

The program Qualitative Analysis of MassHunter Acquisition data (B.07.00) was used to visualise and analyse the data. The likely origin of faecal material is determined following the criteria originally outlined by Bull et al. (2002, p. 652, fig. 4, Figure 4.7). The peak areas were measured, and three ratios were calculated to assess the input of faecal material (Table 4.6). Ratio 1 identified the presence of faecal material whereby a value of more than 0.7 is indicative of faecal pollution (Grimalt et al., 1990). Ratio 2 was calculated to account for diagenetic changes which might transform coprostanol to epicoprostanol (Bull et al., 2002). Ratio 3 was calculated to identify the source of faecal material whereby >1 = human, carnivore or porcine and <1 = herbivore. Human, carnivore or porcine faeces can then be differentiated by the presence of different bile acids.

Table 4.6 Ratios used to calculate the presence of faecal material, account for diagenetic changes and identify the source of faecal material

Ratio 1	$\frac{coprostanol}{coprostanol + 5\alpha - cholestanol}$
Ratio 2	$\frac{coprostanol + epicoprostanol}{coprostanol + epicoprostanol + 5\alpha - cholestanol}$
Ratio 3	$\frac{coprostanol + epicoprostanol}{5\beta - stigmastanol + 5\beta - epistigmastanol}$

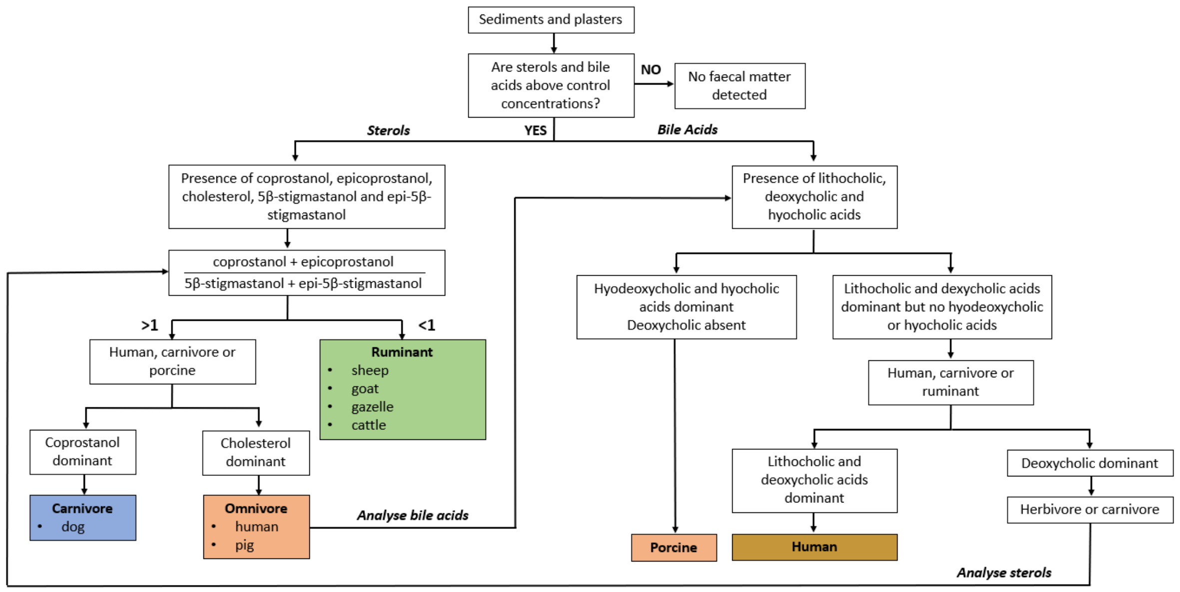


Figure 4.7 Flow diagram summarising the criteria used to identify the source of lipid faecal elements using lipid biomarkers and animals most likely represented at Abu Hureyra. Adapted from: Bull et al. (2002, p. 652, fig. 4) and Shillito et al. (2020, fig. S2).

Chapter 5. A Quantitative comparison of methods for analysing faecal spherulites

Preface

This chapter is written up in the style of a journal article which was submitted to the *Journal of Archaeological Science* on the 27th of June 2022. Although, it was not accepted for publication, I received very helpful reviews. This chapter is a modified version of the original article based on the reviewers' comments and feedback. I will reanalyse a small set of samples to include in the paper before resubmission, which I was unable to do within the time frame of completing my PhD. I am the sole author of the paper, however, the work could not have been carried out without the help of others, particularly Dr Marta Portillo who collected the modern dung samples, and Dr Wendy Matthews who provided comments and feedback on earlier drafts. Full acknowledgements are included at the start of the thesis. The paper is presented here in the same style of formatting as the rest of the thesis for consistency. Figure and table numbers have also been updated for consistency with the rest of the thesis.

5.1 Abstract

Dung is an important animal by-product, utilised from prehistoric times to the modern day, with diverse uses including manure, fuel and construction temper. The identification of dung in archaeological deposits is crucial to understanding human-animal relationships, wider land and resource management practices, and early agriculture. Many charred plant macro-fossils and phytoliths survive digestion and may be deposited by dung which has implications for interpretations of archaeological plant assemblages. Calcitic faecal spherulites, formed in the guts of animals during digestion are a key indicator of dung, but the comparability of different analytical methods has not been tested. This study quantifies the numbers of spherulites from identical samples, mounted on microscope slides using different methods to examine the effect of processing on the number of spherulites. The results indicate that samples of untreated raw sediment mounted on slides tend to have significantly higher numbers of spherulites per gram of sediment, compared with methods which involve extraction and stages such as sonication. The proportions of darkened spherulites, which form in burning temperatures of over 500°C, are relatively consistent between both methods. Faecal spherulites are the most time and cost-efficient method for identifying a potential faecal component in archaeological deposits, where preservation conditions are favourable. However, this paper calls for caution when comparing concentrations of dung within a site and between different sites and advocates a multi-proxy approach to interpret archaeological deposits with a faecal component.

Key Words: Faecal spherulite, dung, methodological development, microfossil, ethnoarchaeology

5.2 Introduction

The identification of dung in the archaeological record can be used to address a diverse set of archaeological questions (Miller, 1984; Shahack-Gross, 2011a; Spengler, 2018; Portillo and García-Suárez, 2021). It is now well-established that charred plant macro-fossils can survive digestion by ruminants (Valamoti, 2013; Wallace and Charles, 2013). Archaeobotanical studies of both micro and macro plant remains, therefore, cannot exclude the possibility of plants derived from animal dung in assemblages (Miller 1984; Miller and Smart 1984; Wallace et al., 2019). Dung provides important insights into changing human-animal relationships and co-interdependencies, particularly as penning deposits provide early indicators of animal management (García-Suárez et al., 2018; Portillo et al., 2020a). Animal dung has been a popular choice of fuel from prehistoric times (Miller, 1984; Miller and Smart, 1984; Matthews, 2005; Portillo et al., 2014; Smith et al., 2019; Spengler, 2018) to the present day (Anderson and Ertug-Yaras, 1998; Reddy, 1998; Elliott et al., 2015; Portillo et al., 2017a), and therefore, animal dung burnt as fuel represents a potential depositional pathway for macro and micro-botanical plant remains recovered in the archaeological record, as has been argued by Naomi Miller since the 80s (Miller, 1984; Miller and Smart, 1984). Dung is also a well-documented construction material used in the past and present, for example, within mortar and plaster surfaces (Gur-Arieh et al., 2019). It is essential to identify where dung is present in archaeological materials to ensure the most robust interpretations of plant remains which contribute to a better understanding of how people in the past interacted with the environment and managed local resources.

Dung is often difficult to detect in the archaeological record, appearing as amorphous organic material during excavation (Shillito et al., 2011b) and often disintegrates during flotation (Matthews, 2010), the most utilised and effective method for recovering charred plant macro-fossils. There are, however, a number of methods to identify dung in the archaeological record (Shahack-Gross, 2011b). One of the most rapid and cost-effective ways to identify the presence of ruminant dung is through the identification of faecal spherulites.

Faecal spherulites are spherical microscopic radially crystallised calcium carbonate surrounded by an organic coating, usually c. 5–20µm in diameter (Brochier, 1983; Brochier et al., 1992; Canti, 1997). They form in the guts of animals, most commonly ruminants, and are excreted in the animal faeces, and where preserved, provide an indicator of the presence of dung. Faecal spherulites are best preserved in alkaline soils with a pH of more than 7, and rarely preserved where the pH is below 6 (Canti, 1999). Bioturbation can severely inhibit the preservation of faecal spherulites which may not survive digestion through the guts of micro-organisms (Canti, 1999, p. 256). Faecal spherulites can survive combustion temperatures of more than 800°C in reducing conditions (Canti and Nicosia, 2018; Portillo et al., 2020b). Faecal spherulites have been shown to expand in temperatures of over 500°C and become “darkened” in the centre, losing their cross of extinction (Canti and Nicosia, 2018; Portillo et al., 2020b). Whilst a comprehensive understanding of the formation of darkened spherulites is still in its infancy, based on experimental work, the presence of “darkened” spherulites provides an indication of burning temperature, whereby, higher proportions of darkened spherulites and more complete “darkening” indicate higher temperature burning, possibly over a longer period.

Faecal spherulites are most easily identified using a polarising microscope in crossed polarised light (XPL) where they are usually between 5µm and 20µm in diameter and characterised by bright interference colours, low order white, to first order red and second order blue and a fixed extinction cross (Canti 1998, p. 437). Faecal spherulites are pseudo uniaxial negative, which can be tested using the λ plate, where low order white colour changes to blue and yellow in opposite quadrants.

Geo-ethnographic research is fundamental to understand and interpret archaeological deposits (Gur-Arieh et al., 2013; Elliott et al., 2015; Friesem, 2016; Portillo et al., 2017a; 2017b; 2021). Quantification methods of faecal spherulites often follow the procedures outlined by Canti (1999), whereby “raw” or “non-processed” sediment or dung is crushed and mounted onto a microscope slide (Tables 2.1, 2.2) (e.g. Albert et al., 2008, Portillo et al., 2014, Smith et al., 2019). Alternatively, in the protocol outlined by Gur-Arieh et al. (2013), spherulites are extracted is based on the Katz et al. (2010) method for the rapid extraction of phytoliths, which disaggregates material which could obstruct spherulite counting and is becoming an increasingly popular method to analyse faecal spherulites, as well as ash pseudomorphs (Tables 2.1, 2.2). While the Gur-Arieh et al. (2013) method does not centrifuge the material to separate heavy particles, some researchers (e.g. Portillo et al. 2021) have included the centrifuging step to a) remove material which may obstruct the counting of microfossils, and b) to increase the comparability with phytoliths extracted by the same methods. Key advantages and limitations to the Canti (1999) method, Gur-Arieh et al. (2013) method and the Gur-Arieh et al. (2013) method with the additional step of density separation by centrifuging are compared in Table 5.1. Spherulites can also be identified in micromorphological thin sections under a polarised microscope (Canti, 1998; Matthews, 2005; 2010; Canti and Brochier, 2017; Portillo et al., 2019), which provides the key advantage that spherulites are observed within their primary context. Micromorphology is a particularly powerful tool for identifying spherulites as it is often possible to distinguish between coprolites, penning deposits, refuse and dung fuel, for example (Matthews, 2010). Archaeological investigations, particularly in SW Asia are increasingly incorporating spherulite studies as an element of a multi-proxy approach, with the analysis often integrated with other microfossils studies such as phytoliths and starch (see Table 5.2). A number of methods have been employed to identify and quantify spherulites, particularly in the past two decades.

Table 5.1 Key advantages and limitations of two key methodological approaches for analysing spherulites based on Canti (1999) and Gur-Arieh et al. (2013)

	Advantages	**Limitations**
Canti (1999) **Mounting mediums** **Permanent (toxic)** Entellan New Merck Canada Basalm Histamount **Non-permanent (non-toxic)** Clove oil Methyl salicylate Liquid glycerol	• Method is rapid and requires minimal equipment • Unprocessed sediment mounted onto slide, so minimal risk of "losing" spherulites • Slides can be made using non-toxic, safe mounting mediums • Slides can be made permanent using mounting mediums • Material in liquid mounts can be rotated to help differentiate between faecal spherulites and microfossils with similar optical appearance • Effective and efficient means to compare with paired flotation samples and assess the likelihood macro-remains are dung derived (Smith 2019)	• Spherulites may be bound up in clays • Spherulites obscured by other materials and therefore difficult to accurately count • More difficult to ensure sediment is distributed evenly over whole slide • Require very high accuracy (5 decimal place) precision scales to accurately weigh material of less than 1mg (in the case that microfossil content is high) • Material in permanent mounts (e.g. with Entellan New, Merck) cannot be rotated once set
Gur-Arieh et al. (2013) based on Katz et al. (2010) **Mounting medium** SPT	• Material more homogenously distributed on slide • Clays dispersed during sonication • More homogenous mix of material on microscope slide • Suspected faecal spherulites can be rotated to improved identification • Possible to identify with ash pseudomorphs and phytoliths (Gur-Arieh et al. 2013)	• Sodium Polytungstate (SPT) dissolves spherulites quickly • More specialised, bulky, expensive equipment required
Portillo et al. (2021) method, based on Gur-Arieh et al. (2013) method **Mounting medium** SPT	• Phytoliths and spherulites are processed in the same way and may even be analysed on the same slide following one extraction • Heavy particles are removed through centrifuging • Suspected faecal spherulites can be rotated to improved identification • Efficient way to analyse and compared phytolith and faecal spherulite concentrations (Portillo et al., 2021)	• Sodium Polytungstate (SPT) dissolves spherulites quickly • More specialised, bulky, expensive equipment required • Centrifuging risks losing spherulites which theoretically have a specific density of 2.5-2.7g/cm^3 and may therefore "sink" rather than be suspended in the supernatant

In order to quantify concentrations of spherulites, methods also vary, and are often adapted to suit the specific research question and aims of the study. Some studies record the total number of spherulites counted (e.g. Elliott et al., 2015), while others relate the number of spherulites to the initial weight to achieve an estimated number of spherulites per gram of sediment or dung (e.g. Portillo et al., 2014). Canti (1999) estimated the number of spherulites relative to the weight of sediment or material from which they are derived. Table 5.2 summarises the key methods for preparation and quantification of spherulites which have been employed in both modern ethnographic and archaeological research. Where publications have not explicitly stated each step of the methodology employed for quantifying faecal spherulites, the processing steps are assumed to be the same as described in the methodology which they are following.

Table 5.2 Summary of the key stages of spherulite analysis in different studies. Key at foot of table. M=Method followed/based on (1 = Canti 1999, 2 = Gur-Arieh et al. 2013, 3 = own method); A=Ashing; S=Sieving; G=Crushing/grounding; DS=Density separation (Y=yes, N=no, ?=unknown)

Study	Method	Ashing	Sieving	Grinding	Density separation	Mounting agent	Other notes
Albert et al. (2008)	1	Y	N	N	N	Entellan New (Merck)	
Alonso-Eguíluz et al. (2017)	2	N	Y	N	?	SPT	
Amicone et al. (2021)	2	N	Y	Y	N	SPT	Briquette fired at 500°C
Brochier et al. (1992)	3	N	N	N	Y	Canada Basalm	Disaggregated with distilled water and hydrogen peroxide (30%)
Cabanes et al. (2009)	1	N	?	?	N	Entellan New (Merck)	
Canti (1999)	1	N	N	Y	N	Methyl salicylate	
Canti and Nicosia (2018)	1	Y	N	N	N	Cargille Meltmount	
Dalton and Ryan (2020)	1	N	N	Y	N		
Dunseth et al. (2018)	2	N	Y	Y	N	SPT	
Elliott et al. (2015)	1	Y	N	N	N		
Gur-Arieh et al. (2013)	2	Y	Y	Y	N	SPT	Burnt in experimental ovens
Gur-Arieh et al. (2014)	2	N	Y	Y	N	SPT	
Gur-Arieh et al. (2019)	2	Y	Y	N	N	SPT	
Kadowaki et al. (2015)	1	N	N	Y	N	Entellan New (Merck)	
Lancelotti and Madella (2012)	3	Y	Y	N	N	Entellan New (Merck)	Carbonates removed with 7% HCl and washed with deionised water.
Matthews (2005)	3	N	N	N	N	Clove oil	
Morandi (2018)	1	N	N	Y	N	Liquid glycerol	
Portillo and Albert (2016)	1	N	N	Y	N	Entellan New (Merck)	
Portillo et al. (2009)	1	N	N	Y	N	Entellan New (Merck)	
Portillo et al. (2014)	1	Y/N	N	N	N	Entellan New (Merck)	Modern dung ashed at 500°C

Portillo et al. (2017b)	1	Y/N	N	N	N	Entellan New (Merck)	Ashed in cooking installations
Portillo et al. (2019)	1	N	N	N	N	Entellan New (Merck)	
Portillo et al. (2020)	2	Y	N	N	Y	Entellan New (Merck)	
Portillo et al. (2021)	2	Y	N	N	Y		
Proctor et al. (2022)	1	N	Y	Y	N	Canada Basalm	
Shahack-Gross and Finkelstein (2008)	1	Y	N	N	N	Entellan New (Merck)	
Shahack-Gross et al. (2009)	1	Y	N	N	N		
Smith et al. (2019)	1	N	Y	Y	N	Canada Basalm	
Yeomans et al. (2021)	2	Y	Y	Y	N	SPT	

As demonstrated in Table 5.2, numerous variations exist to identify and quantify concentrations of faecal spherulites which have been employed in archaeological and ethno-archaeological studies, however, to date, no study exists which compares the quantitative results of each methodology. Although several studies rigorously assess the methodologies they developed or employed (e.g. Gur-Arieh et al., 2013; Smith et al., 2019). Without a comprehensive, methodological comparison between different methods for quantifying faecal spherulites, the capacity for comparison between different sites is limited. Furthermore, it is challenging to interpret the presence and concentrations of archaeological assemblages based on modern reference studies where different methods have been used. The key aim of this research, therefore, is to provide a comparative study of the estimated numbers of spherulites per gram of sediment recorded by two of the key methodologies on which most spherulite studies are based: analysis of spherulites in 1) untreated deposits (Canti, 1999), referred to as in this paper as “non-extracted” and 2) the method which requires more processing, referred to in this paper as “extracted” (Gur-Arieh et al., 2013; Portillo et al., 2021). A secondary aim of this study is to provide a pilot study to test whether phytoliths analysed and quantified by the same methodologies as applied to the faecal spherulites, described above, also produce consistent results. This is to inform on whether there is a basis for further methodological comparison of phytolith extraction and quantification procedures. The samples selected for this experimental comparative analysis are from modern cow and sheep dung and archaeological samples where high numbers of spherulites have been observed. The intention is to provide a foundation for assessing the comparability of each methodology which will enable more effective and accurate comparisons within and between archaeological sites and enable more robust interpretations to be drawn from modern reference studies. Furthermore, this study seeks to inform on the effectiveness of rapid assessments of sediment samples, particularly those which can be conducted in most field laboratories with minimal equipment during excavations.

5.2.1 Research area and case studies

The modern samples, three dung samples and a soil sample were collected from the modern village of Bestansur in Iraqi Kurdistan by Dr. Marta Portillo during the Central Zagros Archaeological Project (CZAP) Spring 2017 field season (Figure 5.1, Table 5.3). This study also analyses the spherulites present in two plaster floor fragments from the Neolithic site of Abu Hureyra in Syria (Figure 5.1), where high concentrations of faecal spherulites have been observed by the author (Chapter 8).

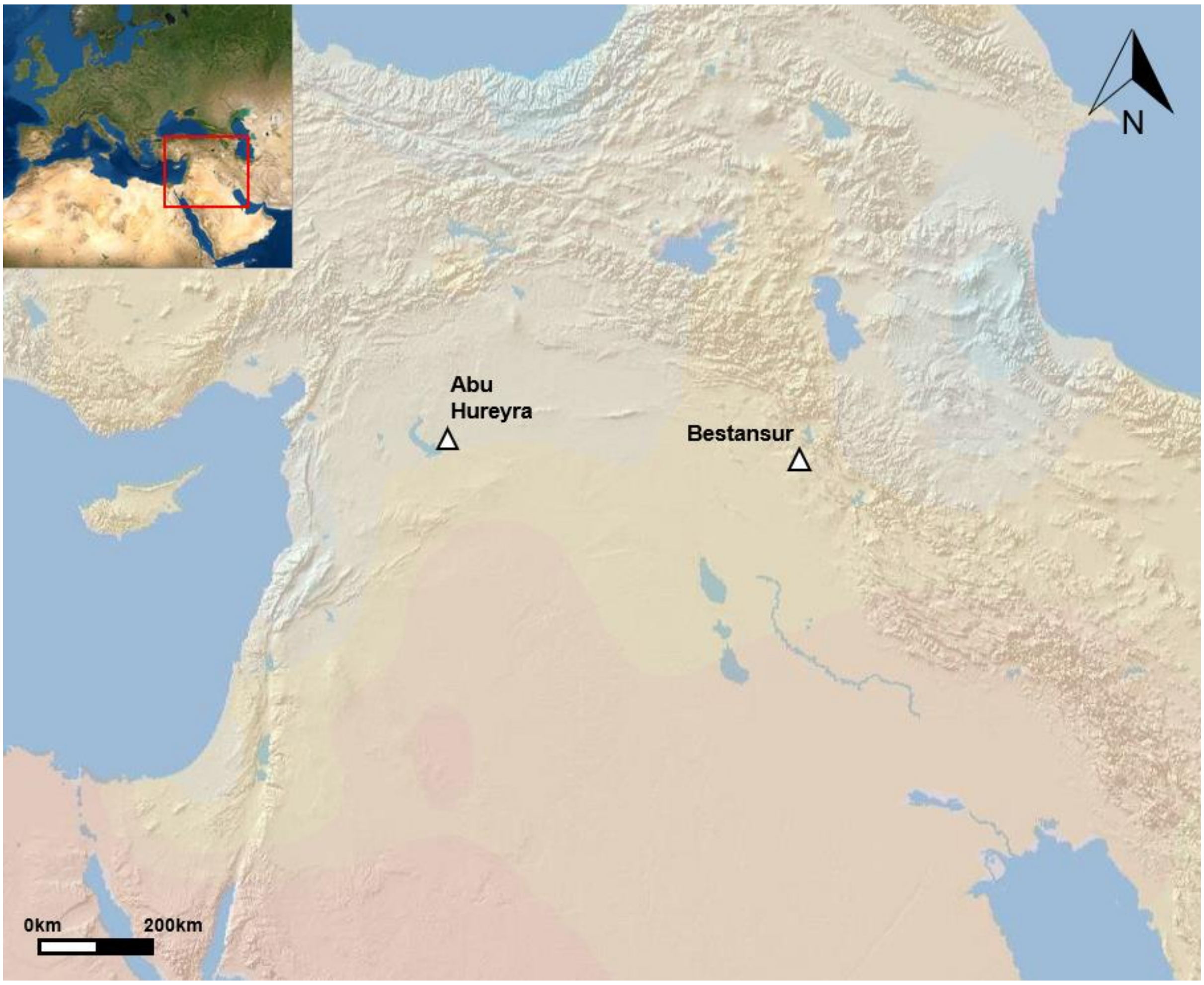


Figure 5.1 Map of research area showing the location of the modern village of Bestansur, Iraqi Kurdistan and Neolithic Abu Hureyra, Syria

5.3 Materials and Methods

Modern dung and associated penning deposits were selected for this study as they have a high concentration of faecal spherulites so that sufficient numbers of spherulites could be counted to produce statistically robust conclusions. Samples with known high concentrations of faecal spherulites were selected for this study based on an initial rapid assessment of the material, observed on a polarising microscope to check for presence and abundance of spherulites.

Table 5.3 Provenance and description for each of the samples analysed in this study, including organic content, calculated after ashing the modern material. LOI = Loss on Ignition; IW = Initial sample weight

	Sample number	Sample type	Sample provenance and season	LOI (g) and as % of IW
Ethnographic	BEST_MD17	Cow dung (adult)	Household 1 - C1 – adult cow - covered area (Spring 2017)	0.798 (79%)
	BEST_MD36	Ovicaprine dung	Household 2 - S4 (Spring 2017)	0.687 (68%)
	BEST_MD38	Sediment – penning deposit	Household 2 – S5 – sediment from ovicaprine pen (Spring 2017)	1.01 (52%)
	BEST_MD39	Sheep dung (adult)	Household 2 – S5 – fresh adult sheep dung (Spring 2017)	0.715 (56%)
Archaeological	AH_D48.65	Gypsum floor plaster fragment	Trench D, period 2A, external activity area (Moore et al., 2000, Appendix 1)	N/A
	AH_B34.20	Gypsum floor plaster fragment	Trench B, period 2C, external pit (Moore et al., 2000, Appendix 1)	N/A

Approximately two grams of the modern material was subsampled and weighed. Samples were ashed in reducing conditions in a furnace oven at 500°C for four hours to burn off organic material in which spherulites may be embedded that may impede counting and to ensure the dung was safer to handle, removing biohazards. As darkened spherulites also form from c. 500°C, ashing the modern dung samples enabled a quantitative comparison of the numbers of darkened spherulites identified between the two methodologies. The samples were weighed again after ashing to record the loss on ignition of organic material (Table 5.3). The archaeological Neolithic fired plaster fragments were subsampled by scraping a small quantity of material from the plaster with a metal spatula, then gently mechanically crushing the material (Chapter 4, section 4.2.2 for more detailed description). It was not necessary to ash the subsamples from the plasters as they contained little organic material which would obstruct the counts or in which spherulites may be embedded, and already contained darkened spherulites, presumably from the plaster manufacturing process.

While the focus of this study is on the quantification of faecal spherulites, phytoliths are often also analysed as part of an integrated multi-proxy methodology in studying dung to study plant components in dung or dung-derived deposits such as bedding in penning deposits, fuel sources, and animal diet. Furthermore, as one of the key methods being assessed here is based on a rapid phytolith extraction method, one of the key advantages for analysing spherulites using this method is that it enables direct comparison with phytoliths which are extracted following the same protocol. This study therefore analysed phytoliths from one modern cow dung sample (17) to assess if there was a basis for a further quantitative study. Following the same methodological approach as for the spherulites, the phytoliths were quantified both following the extraction method of Katz et al.

(2010a) and the ashed material was also mounted onto a microscope slide and mixed with clove oil without any further processing.

5.3.1 Non-extracted, clove oil mounted spherulite analysis

The prepared material was gently crushed on a clean piece of aluminium foil using a metal spatula. The material was then weighed onto a microscope slide, on a set of precision scales accurate to four decimal places, and mounted with 48µl of clove oil, which has a refractive index of 1.53. Initially ~1mg of modern dung/penning deposits were mounted onto a microscope slide, however, due to the very high concentration of spherulites, accurate counting was not possible. Therefore, for the modern dung and modern penning deposit, between 0.1-0.5mg of material was weighed onto the slide. It's worth noting that, precision scales often have an error margin of ~0.1mg, however, counting each sample in triplicate mitigated this issue to some degree. For the archaeological plaster samples, c. 0.001g of material was measured onto the slide, as a lower concentration of spherulites was expected than in the modern samples. The material was thoroughly mixed with the clove oil to ensure an even distribution across the area to be cover slipped. A cover slip measuring 22x22mm was placed onto the material and gently pushed down to remove air bubbles which can lead to uneven concentrations of spherulites.

5.3.2 Extracted, SPT mounted spherulite analysis

The "extracted method" is based on the protocol outlined by Katz et al. (2010a) for the rapid extraction of phytoliths, and adapted by Gur-Arieh et al. (2013), but with the additional step of centrifuging, as conducted by Portillo et al (2021). For the analysis of spherulites, approximately 20mg of modern material and 40mg of the archaeological samples were weighed into a 0.5ml conical plastic centrifuge tube. Each sample was treated with 500µl of sodium polytungstate (SPT) solution, calibrated to 2.4g/ml which deviates from the Portillo et al. (2021) method where dung was also centrifuged, which only treats samples with 450µl. The material was vortexed to ensure mixing, then sonicated for five minutes (following the Portillo et al., (2021) method, compared with Gur-Arieh et al., (2013) which sonicates for 10 minutes) to disperse clays and break up aggregates in which spherulites could be bound up. Samples were then centrifuged for five minutes at 5000rpm, to separate any minerals and heavy particles which may obscure counting. A 50µl aliquot of the supernatant, representing 10% of the sample was then mounted onto a microscope slide, and covered with a 24x24mm coverslip.

5.3.3 Faecal spherulites counting and quantification

A total of thirty-one slides were prepared and counted. For each method three slides were prepared for each sample following both methods (outlined above) with exceptions highlighted below. For the

SPT extraction method, each sample was processed three separate times to highlight any methodological errors or anomalous results. One SPT mounted slide (BEST_MD36.2) was not counted due to a slide breakage. One slide was prepared for archaeological floor plaster sample B34.20 for each of the methods, to preserve the fragment for further analysis as far as possible.

Spherulites were counted using a Leica DMEP polarising microscope at x200 magnification, with further examination at x400 where required. Micrographs were taken using a Leica DFC420 camera on a DMPL optical microscope.

A minimum of 200 spherulites were counted per slide for the ethnographic samples and a minimum of 1000 spherulites were counted in total between the three slides. For the archaeological samples, where fewer spherulites were present, it was not always possible to reach counts of 200 on one slide, but a minimum of 500 spherulites were counted between the three slides (except B34.20). Fields of view were selected randomly, but ensuring that some fields at the edge and some more central slides were counted to account for heterogenous distribution of the material on the slide. Darkened spherulites, which form when dung is heated, usually over 500°C (Canti and Nicosia, 2018), were recorded and presented as a percentage of the total number of faecal spherulites counted. The total number of spherulites recorded in a known number of fields was related to the weight of the initial sample and expressed as number of spherulites per gram of sediment, to identify concentrations and so that the results were standardised and comparable with one another. The estimated number of spherulites per gram of sediment was calculated using the following equations:

Equation 1a

$$\frac{no. faecal\ spherulites\ counted}{area\ counted} \times area\ of\ the\ slide\ = no. faecal\ spherulites\ on\ slide$$

Equation 1b (for "non-extracted" method)

$$\frac{no. spherulites\ per\ slide}{initial\ weight} = number\ of\ spherulites\ per\ gram\ of\ sediment$$

Equation 1c (for extracted method)

$$\frac{no. spherulites\ per\ slide}{initial\ weight} \div 10 = number\ of\ spherulites\ per\ gram\ of\ sediment$$

Spherulite abundance was compared with other ethno-archaeological data sets which follow a similar quantitative approach (Portillo et al., 2020b; 2021).

5.3.4 Comparative phytolith pilot

Phytoliths were also quantified for the modern cow dung sample (17-H1-C1) (Figure 5.3e, f), as phytoliths are often used as part of a multi-proxy approach to investigate plant material in animal dung and can be observed following the similar methodological procedures. Phytoliths were extracted on a method based on Katz et al., (2010), following the procedures outlined in section 4.2.1. Phytoliths were also counted on the same “non-processed” slide mounted in clove oil which faecal spherulites were counted on (section 5.3.1).

5.4 Results

5.4.1 Faecal spherulites – quantitative results

Faecal spherulites were present in all the material analysed in this study (Figure 5.2, Table 5.4) and were observed both in clusters (Figure 5.3a) and as individual particles (Figure 5.3a-d). For each method, three different slides were prepared and counted per sample to identify any anomalous results or potential issues with the method. There is some variability in the numbers of spherulites counted between the three slides (Appendix 3), however, the differences between the methods are clear between all counts. To compare the spherulite concentrations between the two methodologies, the mean of the three counts is used and numbers of faecal spherulites are rounded to the nearest million. All raw spherulite counts are available in Appendix 3.

5.4.1.1 Non-extracted – clove oil mounted quantitative results

In the non-extracted slides, between 46 million and 259 million spherulites per gram of sediment were observed in the modern dung and dung associated material (Figure 5.2, Table 5.4). Sample 38-H2-S5 (soil from pen) had the lowest number of spherulites out of the modern samples, in comparison to the samples directly from modern dung pellets (Table 5.3). The highest number of faecal spherulites, 259 million per gram, was identified in the sheep dung sample 39-H2-S5 (Table 5.3, Figure 5.2). In the archaeological plaster samples, significantly fewer spherulites were present (0.8 million and 1 million per gram of sediment) and the concentration of spherulites was relatively consistent across all three trials of each plaster fragment (Table 5.4).

Table 5.4 Quantitative results of faecal spherulites (millions) and proportions of darkened spherulites (%) identified in this study (spherulites counted is the mean of triplicate counting, individual counts available in Appendix 3)

	Sample	**BEST MD_17**	**BEST MD_36**	**BEST MD_38**	**BEST MD_39**	**AH D48.65**	**AH B34.20**
Non-extracted - clove oil mounted	Spherulites counted	5082	4729	3480	4903	800	201
	Spherulites/g of sediment (millions)	61	218	46	259	1	0.8
	Darkened n	8	148	348	182	126	36
	Darkened %	0.2	3.0	9.9	3.7	18.0	17.9
Extracted - SPT mounted	Spherulites counted	1930	4371	1953	2074	1687	110
	Spherulites/g of sediment (millions)	4	28	8	6	3	0.2
	Darkened n	7	161	228	117	203	17
	Darkened %	0.3	3.6	13.0	5.6	12.0	15.5

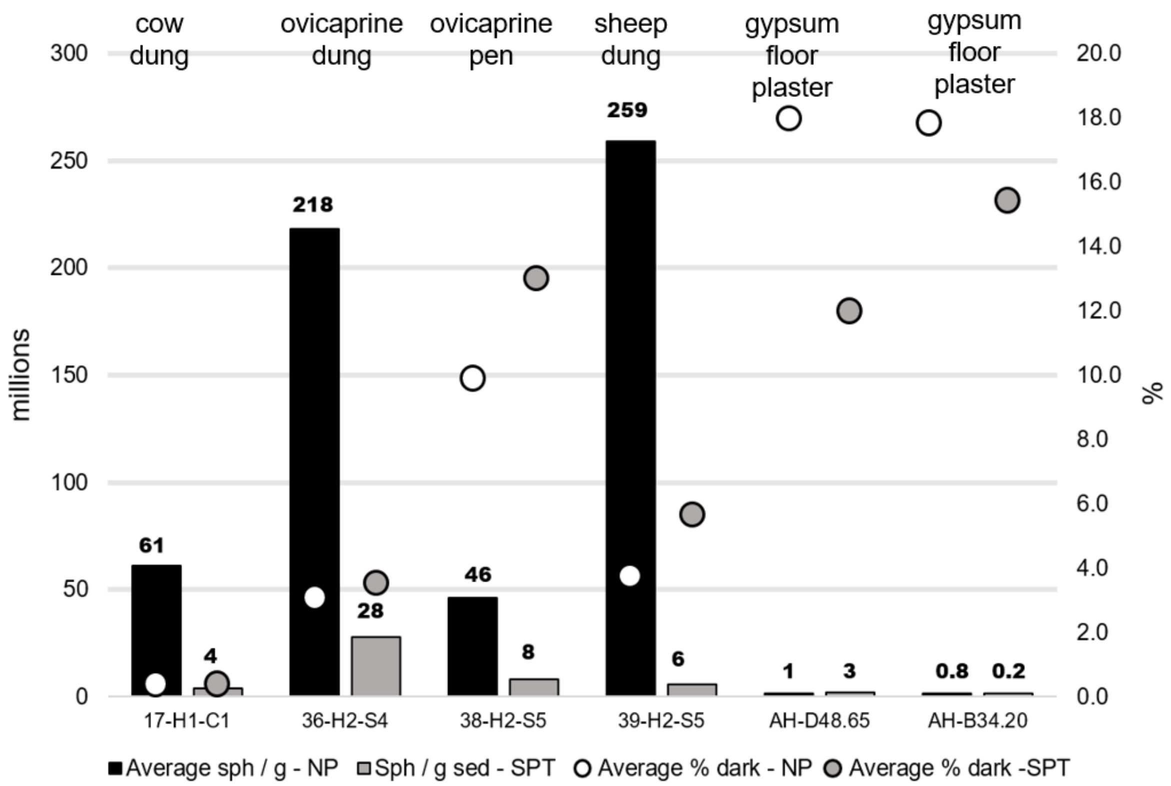


Figure 5.2 Mean number of faecal spherulites (in millions: primary y-axis) and proportions of darkened spherulites (as a %: secondary y-axis) for the non-extracted slides (black) and the extracted, SPT mounted slides (grey) for each sample

5.4.1.2 Extracted – SPT mounted quantitative results

Concentrations of spherulites were generally lower in the extracted SPT mounted slides compared with the non-extracted slides (Table 5.4, Figure 5.2). Concentrations of faecal spherulites range from an average of 4 million per gram to 28 million per gram of sediment in the modern dung and dung associated samples. As observed in the non-extracted slides, the concentrations of faecal spherulites were lower in the archaeological plaster samples (3 million and 0.2 million spherulites per gram) compared with the modern material. There was also increased variation between the two archaeological samples, AH-D48.65 and AH-B34.20, following the SPT method. Archaeological gypsum floor plaster fragment, D48.65, is unique, as the only sample where more faecal spherulites were counted in the slide of extracted spherulites mounted with SPT (n= 1687, 3 million spherulites per gram), compared with the non-extracted slide (n=800, 1 million per gram).

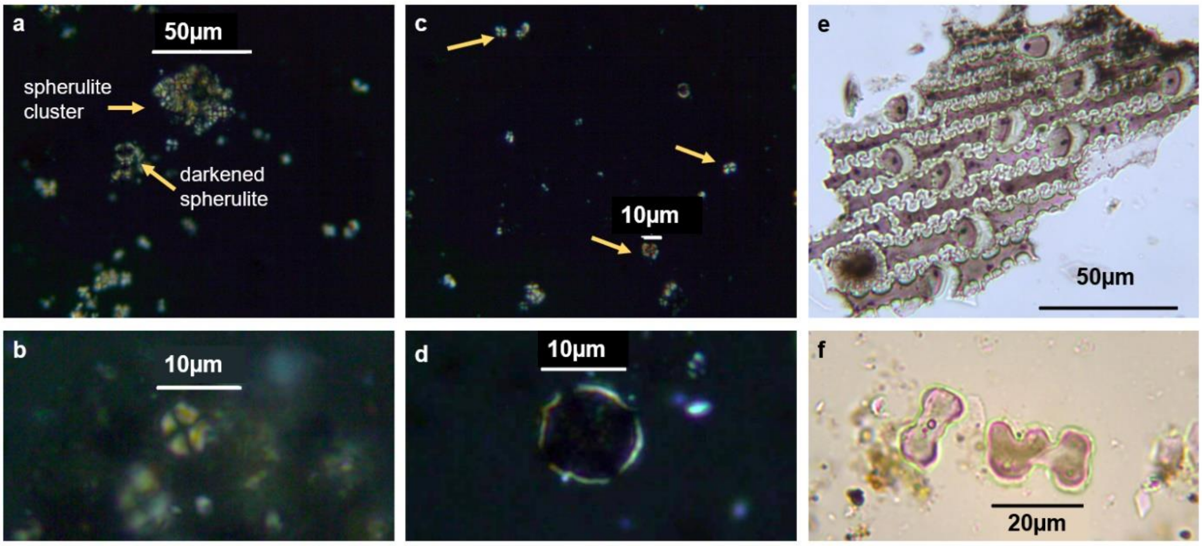


Figure 5.3 Photomicrographs showing a. a cluster of faecal spherulites and darkened spherulite in modern ovicaprine dung sample (BESTMD_36); b. Faecal spherulite from archaeological floor plaster sample (AH_B34.20); c. Individual spherulites in modern ovicaprine dung (BESTMD_36); d. Darkened faecal spherulite in archaeological plaster sample (B34.20); e. Multicell elongate dendritic with short cell rondel and papillate, c.f. cereal husk (BESTMD_17.3); f. Short cell bilobate (left) and polylobate (right) (AH_B34.20).

5.4.2 Darkened spherulites

Darkened spherulites (Figure 3a, c), formed in temperatures above c. 500°C (Canti and Nicosia, 2018) were identified in all material analysed within this study (Table 5.4, Figure 5.2). As illustrated in Figure 5.2, the proportions of faecal spherulites identified as “darkened” are relatively consistent between both methodologies for each sample. Proportions of darkened spherulites range from 0.2% to 9.9% in the modern material, non-extracted slides, and 0.2% to 13% in the extracted SPT slides. In the archaeological plaster samples the proportions of darkened spherulites are generally higher compared with the modern material. In the non-processed slides, 18% of the spherulites were identified as darkened in both samples and in the SPT slides c. 12% and 15% of spherulites were identified as darkened in samples AH-D48.65 and AH-B34.20 respectively. The relatively consistent proportions of darkened spherulites identified using both methods indicate that the processing methods compared in this study had little effect on the darkened spherulites.

5.4.3 Phytoliths

Phytoliths were also quantified for the modern cow dung sample (17-H1-C1) (Figure 3e, f), as phytoliths are often used as part of a multi-proxy approach to investigate plant material in animal dung and can be observed following the similar methodological procedures. Like the concentrations of spherulites, much higher numbers of phytoliths were observed in the non-processed material (32 million/gram of sediment), compared with the SPT method (13 million per gram of sediment). This observation demonstrates that further research into the effect of different phytolith extraction protocols is needed.

Interestingly, the proportions of multi-celled phytoliths (defined here as three or more phytoliths in anatomical connection) were almost the same in both methods (25% of total assemblage), suggesting the two methods tested in this study did not result in the breakdown of multi-cells. While previous studies have noted differences between numbers of multi-celled phytoliths which have been ashed compared to acid extraction methods to remove organic material (Jenkins 2009), in this study the material for both methods was ashed. A more detailed comparison of phytolith assemblages between different preparation methods with a larger set of samples is required to clarify discrepancies in the numbers of phytoliths per gram of sediment.

5.5 Interpretation and Discussion

As observed by Canti (1999), spherulites tend to be either scattered/distributed as individual particles (Figure 5.3b-d) or bound up in faecal aggregates (Figure 5.3a). In general, the non-extracted spherulites in slides mounted in clove oil, which had not undergone any processing, except where required, gentle crushing and ashing for the modern dung samples, were more frequently observed in large clusters (Figure 5.3c), particularly in the modern dung samples where faecal spherulites were particularly abundant. This sometimes presented a challenge to accurately count the spherulites and it is likely some spherulites were obscured by other material and therefore excluded from counts. However, since the counts of spherulites in the clove oil slides were consistently and considerably higher than in the SPT mounted slides for the ethnographic samples, it does not appear that this was a major issue. One solution could be to mount less material on the slides to prevent microfossil overloading, however, in some cases the initial weight was already very low (e.g. 0.0001g) and a more accurate precision scale would be required. It may therefore help to select dung samples with lower quantities of spherulites. Alternatively, the ashed material could be mixed with more clove oil and a larger cover slip could be used to spread the spherulites over a larger area.

Some clusters of spherulites were also observed in the samples of extracted spherulites in slides mounted with SPT, however, these tended to be less frequent and contain fewer spherulites compared with the slides mounted in clove oil. In the extracted slides mounted with SPT, individual particles were more commonly observed, which was likely due to the processing steps whereby the material is vortexed and sonicated, which would contribute to disaggregation of any clusters of spherulites and the material in which they were bound. Furthermore, the method of pipetting a 50µl aliquot onto the slide resulted in a more homogenous spread of spherulites compared with mixing the material into the mounting agent by hand using a spatula (Gur-Arieh et al., 2013).

This study did not record the rate of dissolution of the spherulites mounted in SPT, however, it appeared that individual particles dissolved more rapidly than clusters of spherulites or those embedded in other material, presumably, as this would offer some protection or buffering from the acidity of the SPT. Previous studies have indicated that the reaction between SPT and calcite does not start until after 1 hour (Skipp and Brownfield, 1993), and Gur-Arieh et al. (2013) notes that as the results of their study are replicable, it is methodologically sound to use SPT as a mounting medium. Further studies are required to measure the rate of dissolution of spherulites in SPT which take account of different variables, such as room temperature. More specifically to this study, further work is required to clarify how different processes in the lab, such as mixing with a vortex, sonication and centrifuging effect the rates of dissolution.

As calcitic spherulites have a specific density of 2.5-2.7g/cm^3, while the heavy liquid used in the "extraction" protocol, SPT, is made up to a specific density of 2.4cm^3, theoretically, some of the faecal spherulites may sink during the centrifuge step, rather than be suspended in the supernatant which is then mounted onto the slide for counting. This would account for the significant differences in concentrations of phytoliths on the SPT compared with the clove oil slides (Table 5.4, Figure 5.2). However, significantly, the results between both methods are far more comparable in the archaeological plaster samples (Table 5.4, Figure 5.2). A possible reason for this is that, the centrifuge step is appropriate for samples with a relatively higher mineral content (see Chapter 8, section 8.44 for elemental concentrations of plaster samples). On the other hand, the centrifuge step may be inappropriate for dung samples, which have a relatively higher organic content (Table 5.1).

5.5.1 Evidence for burning

Experimental research shows that darkened spherulites form where gaseous exchange is limited between 500°C and 700°C (Canti and Nicosia, 2018). Therefore, darkened spherulites are expected in the modern material analysed in this study for both methodologies as they were ashed at 500°C degrees for four hours prior to making up the slides. The proportions of darkened spherulites are fairly consistent with those identified by Portillo et al. (2020d), whereby concentrations of darkened spherulites of dung burnt at 500°C tended to be low (less than 10%), and similar to this study, there was a lower proportion of darkened spherulites identified from cow dung compared to sheep dung (Portillo et al., 2020b, p. 5, table 1). It is possible that there may have been fluctuations in temperature within the muffle furnace in which the samples were ashed, which could have caused slight variations in length of exposure to specific temperatures resulting in the variation in darkened spherulite formation evident in Figure 5.2 between different sample material.

5.5.2 Comparison with other studies

The quantitative results from this study are compared here to similar studies that follow the same methods of extraction using SPT, and crucially centrifugion to separate the heavy particles, in order to assess the comparability of the results (Portillo et al., 2020b; 2021) (Figure 5.4). The results from this study are compared with a study of modern ruminant dung from Menorca in the Balearic Islands (Portillo et al., 2021) and a study which also uses material from the modern village of Bestansur, Iraqi Kurdistan (Portillo et al., 2020b). Material where no faecal spherulites were identified, presumably because of an external taphonomic factor, and dung from young animals where spherulite formation and ubiquity is still relatively understudied is excluded to reduce the number of variables which may affect spherulite production and abundance. The mean number of spherulites per gram of sediment identified in cow dung from Portillo et al.'s (2021) study is 9.5 million, which is higher than the 4 million identified in this study. In this study, the fresh modern sheep dung produced 6 million spherulites per gram of sediment, while the average number of spherulites counted from modern sheep dung collected in Menorca was 10.7 million per gram of sediment (Portillo et al., 2021). However, for both sheep and goat, the results from this study, do fall within the range identified in the modern study carried out in Menorca, albeit, most comparable with the minimum numbers of spherulites identified.

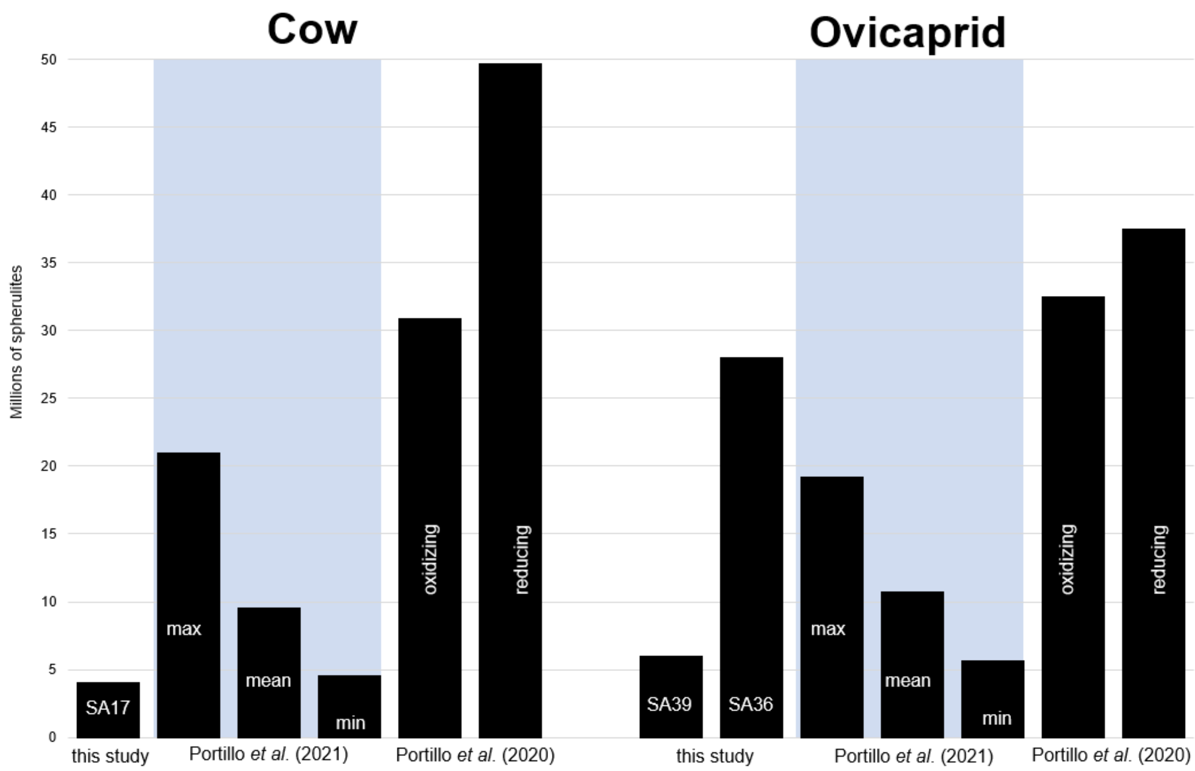


Figure 5.4 Comparison of faecal spherulite abundance in this study compared with Portillo et al. (2020) and Portillo et al. (2021) where an identical protocol was followed

As highlighted in this study and in comparison with a study of modern dung samples from ruminants in Menorca, Balearic Islands (Portillo et al., 2021), there can be significant variation between the numbers of spherulites produced, even when the species, environment, feeding regimes and sample preparation methods are the same (Portillo et al., 2021, p. 7202, table 4). Some of the variation between different studies could be attributed to the use of different lab equipment. As identified in phytolith counting and identification, different researchers, sometimes produce different counts, highlighting the need for strict and standardised identification criteria (Katz et al., 2010). In this study, all counts were undertaken by the author, therefore the parameters used for identifying and quantifying spherulites were consistent between the two methodologies.

Overall, this study highlights the significant variation between the number of faecal spherulites even when sampled from modern material from the same location.

5.6 Conclusion and future directions

Since the early 90s, the identification of faecal spherulites from archaeological sites has successfully identified animal dung. However, the methods for quantification remain unstandardised and diverse. Studies often fail to explicitly or comprehensively outline the methodologies which have been employed to identify or quantify faecal spherulites which hampers the reproducibility of experimental or modern reference studies and inhibits the usefulness of comparisons between different sites where analysis has been conducted by different methodologies. This study has demonstrated that the quantification of faecal spherulites using different, widely applied methodologies, can result in significantly different quantities of faecal spherulites being recorded.

Proportions of darkened spherulites which reflect burning temperature and conditions are relatively consistent between the two methods of quantification tested in this study. The importance of identifying faecal spherulites cannot be understated and should be more widely incorporated into standard practice both during excavation, where facilities are available and in post-excavation analysis. However, further studies are required to quantify numbers of spherulites and compare them within and between sites in a meaningful way. The results from this study, indicate that the rapid identification of spherulites in raw sediment (e.g. Canti, 1999) is sufficient to provide a general overview of deposits with a faecal component, although for this purpose, both methods are effective.

Further modern reference and experimental studies are needed to make lengthy quantification more meaningful. Experimental research of faecal spherulite dissolution rate would be helpful, especially if conducted in conjunction with other variables such as temperature (room/solution), pH of material and mounting medium.

Within a site, quantification of relative numbers of spherulites in different areas is a useful tool to provide information about uses of space, such as penning deposits, and use of resources such as dung fuel and dung as a building material, which informs on a range of themes including human resource management, sustainability and changing human-animal relationships.

The identification of faecal spherulites is particularly informative when integrated with other methodological approaches such as phytoliths, micromorphology and paired with flotation samples to recover charred macrofossils. Significantly, the presence of dung spherulites, particularly when paired with flotation samples (e.g. Smith et al. 2019), provides essential information about potential presence of dung which could be a significant pathway for charred plant remains (Miller 1984, Miller and Smart 1984). Only through a better understanding of the depositional pathways of archaeobotanical plant remains can we advance our understanding of significant changes in human economy and lifeways, such as the development of agriculture in SW Asia.

Chapter 6. Phytolith and faecal spherulite prospection and taphonomy

The aim of this chapter is to explore simple, rapid, mostly non-destructive methods to explore phytolith and spherulite preservation, taphonomy and abundance in archaeological samples. This is important because phytoliths and spherulites which are often found in archaeological sediments, have the potential to provide novel information not gathered by other proxies (Chapter 2 for full discussion and references). During excavation, feedback can be provided to excavators and other specialists to inform on excavation and sampling strategies and contribute to interdisciplinary discussions (Hodder, 2000). Phytolith and spherulite studies have been particularly fruitful in Epipalaeolithic and Neolithic sites in SW Asia, where preservation conditions (discussed below) tend to be quite favourable. However, unlike macro-artefacts and plant macro-fossils, faecal spherulites cannot be seen with the naked eye, and phytoliths are rarely seen with the naked eye, or during excavation, which is a challenge for selecting which material to sample, and particularly which material to export. Rapid screening techniques also have huge potential benefits for archived environmental archaeological sediment samples, such as the Abu Hureyra archive of occupation residues at the UoR (Appendix 1). Researchers may have limited time and resources to assess collections housed in museums and institution, and therefore rapid screening methods can help to select the most appropriate samples for further analysis. The phytolith and spherulite quantitative data are presented more fully in Chapter 7 and available in full in Appendix 4.

6.1 Soil pH

Phytoliths survive in soils and sediments with pH ranging from 2 to 8.2 (Weiner, 2010, p. 175) and calcareous faecal spherulites dissolve in pH conditions of less than 6.5 (Canti, 1999). The soil pH in all sediments tested (n=17) was between 6 and 8, which is favourable for the preservation of silica phytoliths and calcitic dung spherulites (Figure 6.1). Given the soil pH conditions identified in this study, current soil pH is not likely a factor which has influenced any variations in the concentrations of phytoliths or spherulites in this study. However, soil properties evolve and change over time and therefore, it is possible that the alkalinity of the soils has fluctuated since deposition (Goldberg and Macphail, 2006, p. 65). Sample E338.146 has both a relatively high pH (7.5) as well an exceptionally large proportion of phytoliths designated as “weathered morphotypes” (33%) (Figure 6.1, 6.2a), and in this instance, the pH could have been a factor in the dissolution, pitting and etching of the phytoliths. A range of factors apart from pH also effect phytolith preservation and taphonomy (Albert et al., 2006; Cabanes et al., 2011; Cabanes and Shahack-Gross, 2015; Osterrieth et al., 2009), discussed in Chapter 2, Chapter 7 and Chapter 11. Pearson’s R correlation coefficient was carried out to test if there was any statistically significant relationship between pH and the concentrations of phytoliths and spherulites, and proportions of weathered morphotypes and multi-cells. In Pearson's correlation coefficient a score of 1 represents a perfect correlation, 0.8 or more is considered "good", 0.5-0.8 is "moderate" and 0.3 is "weak". A "p" value of less than 0.05 indicates that the result is statistically significant.

There was no correlation between the estimated numbers of phytoliths per gram of sediment and the soil pH ($r = -0.185$, $p>0.05$, $p = 0.478$) (Figure 6.1, 6.2a) or pH and spherulites/g of sediment ($r = 0.27$, $p>0.05$, $p = 0.917$) (Figure 6.1, 6.2b). This study also did not find any relationship between pH and the proportion of weathered morphotypes ($r=0.167$, $p>0.05$, $p = 0.522$) (Figure 6.1, Figure 6.2c), or the proportion on the assemblage made up of multi-cell phytoliths and pH ($r=0.184$, $p > 0.05$, $p = 0.479$) (Figure 6.1, Figure 6.2d).

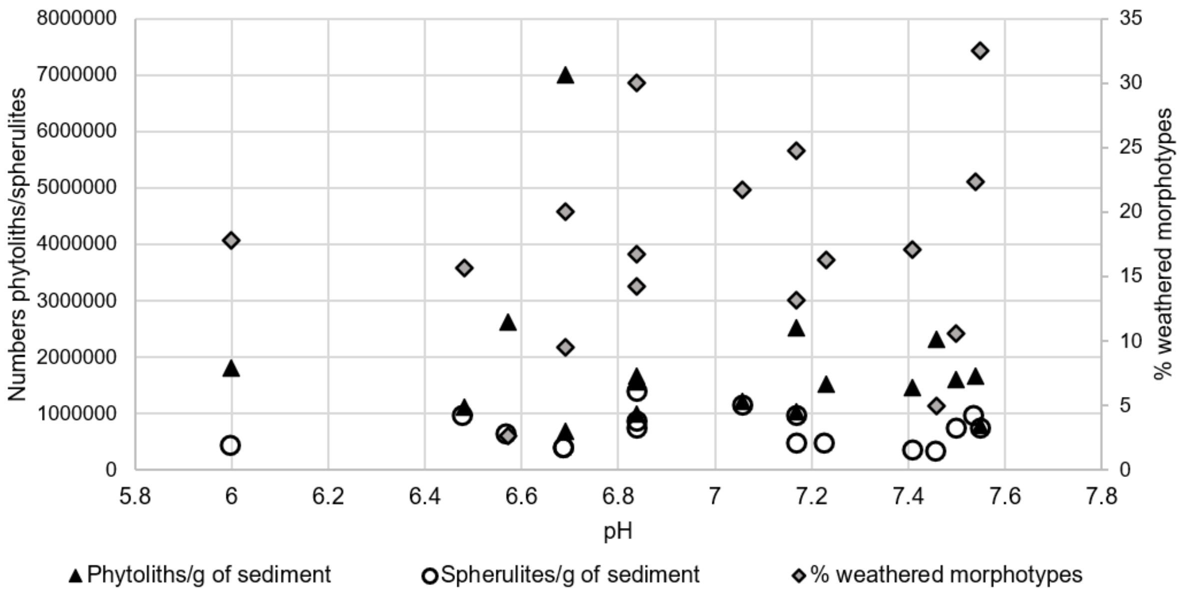


Figure 6.1. Scatter plot illustrating the relationship between the concentrations of phytoliths, faecal spherulites and weathered morphotypes with soil pH

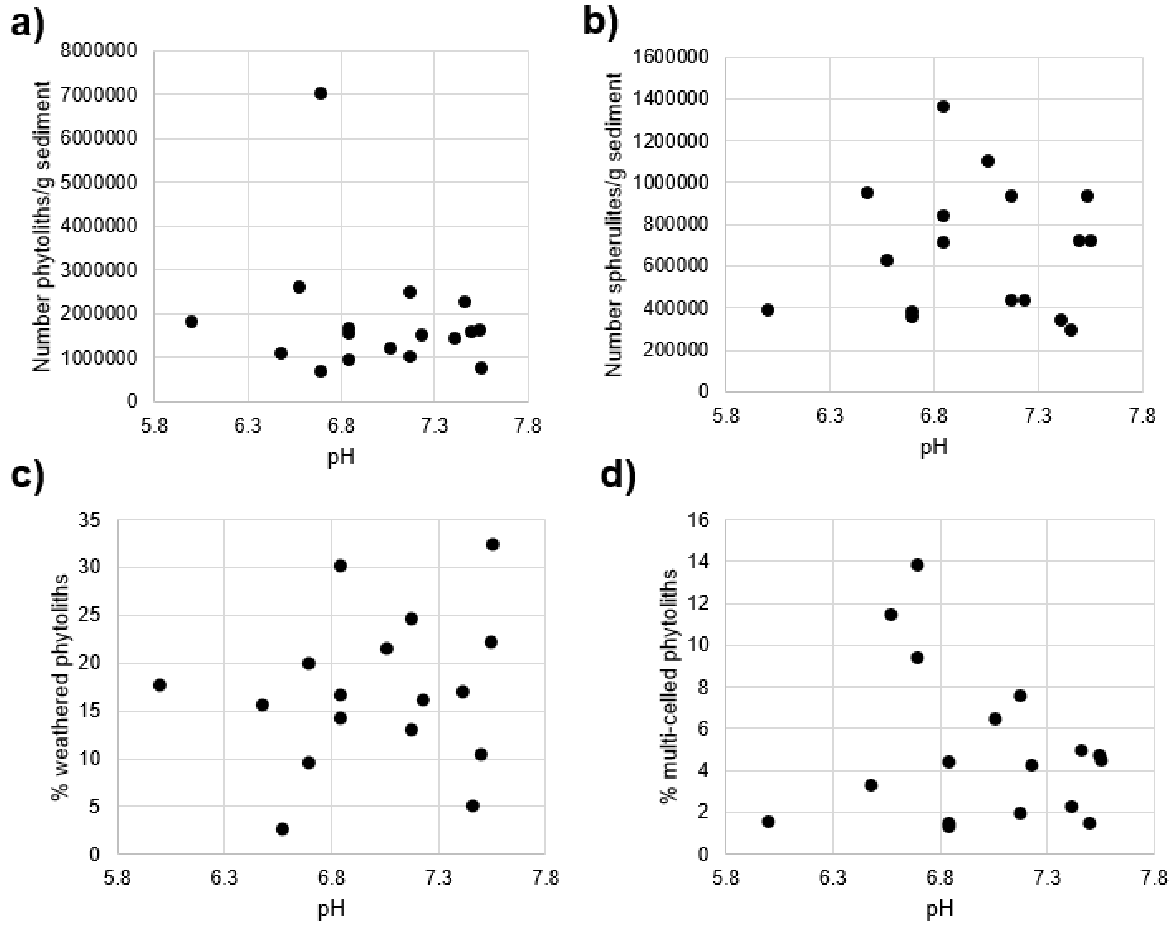


Figure 6.2 Individual scatter plots showing the relationship between pH a) the estimated numbers of phytoliths per gram of sediment, b) the estimated number of spherulites per gram of sediment, c) the proportion of weathered morphotypes per gram of sediment and d) the proportion of multi-celled phytoliths per gram of sediment.

6.2 Impact of phytolith degradation on assemblages

The proportion of degraded phytoliths, here referred to as "weathered morphotypes", within a phytolith assemblage, that is, phytoliths with surface pitting and etching to the point that they are unidentifiable, which may have occurred pre-deposition or post-deposition, has often been used to assess preservation of an assemblage (Cabanes et al., 2011). However, "weathered" characteristics have also been attributed to incomplete silicification of plant cells (Cabanes et al., 2011). Scatter plots were created, and Pearson's r carried out to test whether there was any relationship between the overall concentration of phytoliths and the proportion of weathered morphotypes present in each assemblage (Figure 6.3a). A negative correlation whereby samples with lower proportions of phytoliths tend to have higher proportions of weathered morphotypes, could suggest that the overall phytolith concentration may have been affected by dissolution or the incomplete silicification of plants. This would then be important to consider when comparing phytolith concentrations between different deposits in Chapters 6 and 10.

Weathering may also affect the overall phytolith morphotype composition of the phytoliths assemblages, as some morphotypes are more fragile and readily dissolved and therefore under-represented (Cabanes and Shahack-Gross, 2015). Pearson's r indicated that there was a slightly weak negative correlation between the number of phytoliths per gram of sediment and the number of weathered morphotypes ($r=-0.525$, $p < 0.05$) in the same set of seventeen samples which were analysed for pH. Therefore, samples with high concentrations of phytoliths tended to have lower proportions of weathered morphotypes. The relationship between phytolith concentrations and weathered morphotypes was then tested on a larger set of samples (n=91) to provide a more reliable result. In the larger sample set, no relationship was detected between the concentrations of phytoliths and the proportions of weathered morphotypes, $r=0.109$, $p>0.05$ ($p=0.305$). The analysis of all of the samples analysed in this study (n=91) suggested that weathering has not had a significant impact on the overall concentrations of phytoliths.

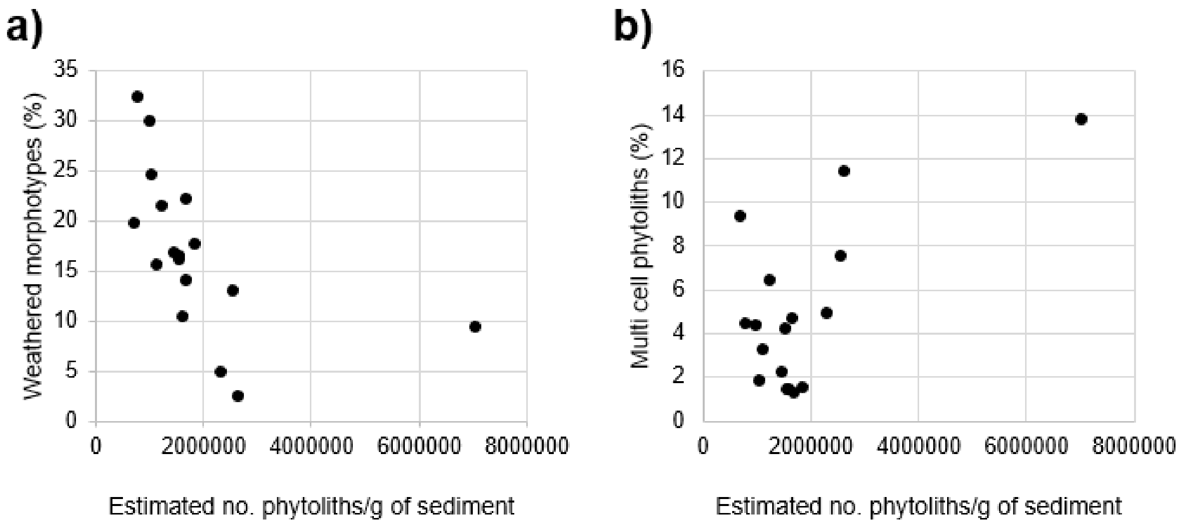


Figure 6.3 Scatter plots to visually display the relationship between the estimated number of phytoliths per gram of sediment with a) the proportion of weathered morphotypes (weak negative correlation) and b) the proportion of multi-cell phytoliths (very slightly positive correlation)

The relationship was also tested between the concentration of faecal spherulites and proportions of weathered phytolith morphotypes observed in the corresponding phytolith sample. This was to assess whether some of the same factors which caused the degradation of phytoliths could have also affected the overall concentration and preservation of faecal spherulites. Pearson's r, tested the relationship between faecal spherulites and weathered morphotype phytoliths, and showed no correlation ($r = 0.181$, $p > 0.05$). As there are a variety of different factors affecting the preservation of faecal spherulites and causing the weathering of phytoliths (Canti, 1999; Albert et al., 2006; Cabanes et al., 2011; Cabanes and Shahack-Gross, 2015; Portillo et al., 2020), it is not unexpected that there is no correlation between phytolith weathering and the concentration of faecal spherulites.

6.3 Multi-celled phytoliths as an indicator of sample preservation

The numbers of multi-celled phytoliths are often used as an indicator of phytolith abundance and preservation. The numbers of multi-cells within a sample, as well as the size of multi-cells can be affected by a number of factors. The processing methods which are used to extract phytoliths from sediments produce different quantities of multi-cells. Dry ashing to remove organic matter produces more multi-cells compared to acid extraction (Jenkins, 2009; Shillito, 2011). It has been proposed that the number and size of multi-cell emmer wheat phytoliths is related to water availability during growth (Rosen and Weiner, 1994). The frequency and size of multi-cell phytoliths at Çatalhöyük has been applied to make inferences about economic strategies, and in combination with charred macrofossil evidence, suggest the fission-fusion of the population to obtain resources (Roberts and Rosen, 2009). Based on experimental studies, the frequency and size of multi-cells also decreases following processes such as grinding (Portillo et al., 2017b).

The seventeen samples which were tested for pH were also compared to assess if there was any relationship between the concentration of phytoliths and the number of multi-cells within a sample. Pearson's r showed a moderately positive correlation ($r = 0.646$, $p < 0.05$, $p = 0.005$) between the estimated number of phytoliths per gram of sediment and the proportion of multi-cells present, whereby the samples with a higher concentration of phytoliths also had a higher proportion of weathered morphotypes (Figure 6.3b). The same variables were tested again on a larger sample set ($n=91$), which showed a much weaker, although still statistically significant relationship ($r = 0.266$, $p < 0.05$, $p = 0.011$). The results demonstrate that multi-cell phytoliths are likely to be more frequent in better preserved samples which have higher concentrations. However, as demonstrated by previous research (Jenkins, 2009; Portillo et al., 2017b; Rosen and Weiner, 1994; Shillito, 2011), a number of other factors also likely effect the proportions and sizes of multi-cell phytoliths present within a sample.

Since multi-cell phytoliths are frequently identified in both modern and archaeological animal dung (Elliott et al., 2020; Matthews, 2005; Portillo et al., 2021), the relationship between faecal spherulites and the proportion of multi-cell phytoliths in paired samples was also tested to assess whether the proportion of multi-cells may provide an initial indicator of the presence of dung. No correlation was detected between the estimated number of faecal spherulites and the proportion of multi-cells ($r = -197$, $p > 0.05$, $p = 0.449$). The lack of relationship between faecal spherulites and multi-cell phytoliths is likely attributable to several factors. Firstly, the samples analysed in this study represent occupation residues, and where spherulites have suggested that dung is a component, it is not clear the amount of dung which makes up the deposits, which could have a very low input of dung. While faecal spherulites provide an accurate indicator of the presence of dung, the concentration of spherulites is determined by a number of factors (e.g. species, environment, animal diet, preservation conditions), and does not provide an indicator of the amount of dung present or the proportion of a sample which may be composed of dung.

As ruminant dung often has high concentrations of phytoliths (Elliott et al., 2020; Portillo et al., 2021; Shahack-Gross, 2011), Pearson's r was also employed to test the relationship between the estimated number of phytoliths and spherulites per gram of sediment. The aim of this analysis was to ascertain whether the concentration of phytoliths was correlated with the presence of dung. There was no significant relationship between the numbers of phytoliths and spherulites per gram of sediment ($r = 0.107$, $p > 0.05$, $p = 0.567$). This is likely because the concentration of phytoliths can vary according to numerous factors relating to the quantity of plant material, the type of vegetation and preservation conditions (Albert et al., 2003; Tsartsidou et al., 2007).

6.4 pXRF of occupation residue sediments

Thirty-one sediment samples, representing Epipaleolithic and Neolithic occupation residues (Chapter 4, Table 4.1) from Abu Hureyra were analysed by pXRF to determine their elemental composition, all of which were also analysed for phytoliths and spherulites. The primary aim of the pXRF analysis was to assess whether any single elements correlated with the concentrations of phytoliths and spherulites, expressed as the estimated number of phytoliths and spherulites per gram of sediment. Associations between elements, detected by pXRF, and phytolith/spherulite concentration could therefore be used as a rapid screening method to select samples for phytolith analysis or infer the presence of ruminant dung, which could then be clarified through the presence of faecal spherulites and/or most costly and time-consuming techniques such as thin section micromorphology, or GC-MS to detect faecal biomarkers.

The pXRF analysis identified the concentrations of thirty-nine elements, of which sixteen were below the limit of detection (LOD) in all samples and a total of twenty-three elements had readings above the limit of detection for some of the sediment samples (Appendix 5). Pearson's correlation was carried out in IBM SPSS statistics to explore the possible statistically significant relationship between single chemical elements and the estimated number of phytoliths and spherulites per gram of sediment within a sample.

Elevated phosphate levels are often indicative of high levels of organic matter in archaeological sediments, and thereby also associated with animal dung (Shahack-Gross, 2011, p. 206, table 1).

There was no identifiable correlation between the number of spherulites per gram of sediment and amount of a phosphorous (r = -0.235, p > 0.05, p = 0.202), which is an element commonly associated with dung or Calcite (r = 0.054, p > 0.05, p = 0.772), which is the primary element that faecal spherulites are composed of (Canti, 1997). Other materials present in the sediments including bone and ash could also be high in P and Ca (Shahack-Gross, 2011), which may explain the absence of a correlation between P, Ca, and spherulites. Phytoliths are predominately composed of silica, however, there was also no correlation between the number of phytoliths per gram of sediment and the amount of silica (r = 0.135, p > 0.05, p = 0.469). This is possible because silica also occurs naturally in sediments (Piperno, 2006, p. 6).

6.4.1 Data exploration

Eleven of the elements identified by pXRF (Zr, Sr, Fe, Ti, Ca, K, Al, P, Si, Cl, S) were selected for further data exploration, through Principal Component Analysis (PCA). Elements were selected where all, or almost all of the samples analysed (n=31) had values which were not below the limit of detection (LOD) and did not have large error readings of >10% (at two sigma precision). The aim was to test whether concentrations of some elements combined could be used to screen sediments for dung and might also provide an indicator of space type (internal/external) or material (soil/ashy). The PCA analysis also provided the opportunity to identify if elemental composition grouped according to the period of occupation (1, 2A, 2B, Chapter 3), or according to the trench, possible reflecting either different activities taking place, or varying soil conditions across the mound.

PCA analysis was initially conducted on thirteen items, which included eleven elements and the concentrations of phytoliths and spherulites per gram of sediment. Phytoliths, spherulites, P, K and S were excluded from the analysis following an initial assessment because of the low correlations with other variables (Appendix 6). The final PCA analysis was conducted on 8 elements (Zr, Sr, Fe, Ti, Si, Cl, Al, Ca) with orthogonal rotation (Varimax). KMO verified the sampling adequacy (= 0.754) and all KMO values for individual elements were >0.5. Bartlett's test of sphericity (χ^2 (28) = 143.642, p < 0.01) indicated that correlations between elements were sufficiently large to conduct PCA analysis. Two eigenvalues were >1, and in combination explained 71% of the data. After rotation, the elements that clustered on the same components suggested that PC-1 accounted 51.8% of the variance, and represented variation between Al, Zr, Si, Cl, Ti and Fe. PC-2 explained 19.2% of the variance, and represented Zr, Ti, Ca, Sr and Fe. A scatter plot was generated to further explore associations and groupings between different samples (Figure 6.4).

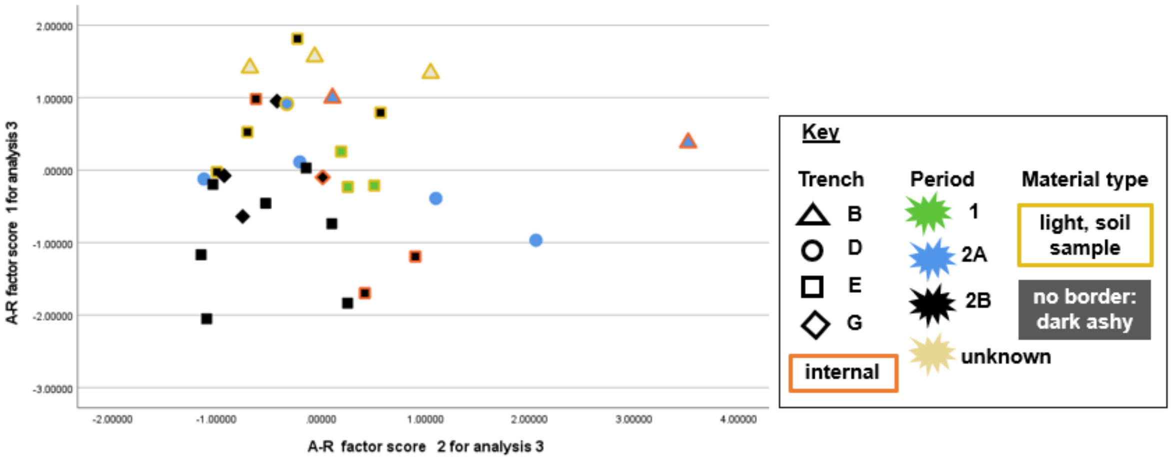


Figure 6.4 Scatter plot showing principal components 1 and 2.

The PCA scatter plot (Figure 6.4) revealed no obvious groupings, however, some deductions can be made about the elemental compositions according to spatial category, period and material type. Light coloured soil samples (as opposed to more ashy, burned material), tended to group together (gold bordered). The Trench B soil samples (triangles) grouped relatively closely together, as did the soil samples from Trench E, period 1 (green square, gold borders) and Trench E, period 2B (black square, gold border). However, these grouping were also interspersed with some of the ashy, charcoal rich samples.

Whether the material was sampled from an internal (orange border, n=6) or external area, seemingly had no relationship to the elemental composition which was clearly more influence by material type. Samples grouped very loosely by trench, although, this is likely more related to the material type as well, rather than being determined by the area from which the sample came from. Samples also appear to be grouped by their assigned time period, but similarly to the trench, it is also likely more strongly determined by the material types. The PCA analysis did not reveal any groupings, or associations between different elements which could be used to determine or clarify the archaeological deposits from which they were derived. This is likely because, as bulk samples, most of which were preferentially sampled to provide material for radiocarbon dating, and are therefore of a similar compositions to one another.

6.5 Summary of results

This chapter has explored some factors which may affect the concentrations of phytoliths and spherulites identified in bulk sediments analysed in this study. Significantly, although most samples contained some “weathered” phytoliths, which are unidentifiable due to surface pitting and etching, this does not appear to be related to the overall concentration of phytoliths which are present. However, a very weak correlation was identified in a small sub-set of samples whereby higher proportions of weathered phytoliths were associated with lower phytolith concentrations. This indicates that phytolith concentrations may be slightly affected due to weathering, although not to the extent that it makes a significant difference when comparing phytolith concentrations from different deposits. However, weathering may cause the over or under-representation of some morphotypes, which is discussed more fully with the phytolith morphotype analysis interpretation in Chapter 7.

Although the frequency and size of multi-cells can be affected by a number of factors, they are often used as an indicator of good phytolith preservation. This research identified a weak correlation between the number of multi-cells and the concentrations of phytoliths, indicating that in this set of samples, higher proportions of multi-cells tend to be associated with higher concentrations of phytoliths. Although previous studies have shown phosphorous is higher in dung deposits compared with natural sediments (Elliott et al., 2015, p. 295), in this study, no correlations were identified between, P and Ca and the concentration of faecal spherulites. The evidence from this study therefore suggests that pXRF is not an effective method for detecting the presence of faecal spherulites and therefore dung in mixed bulk samples, where a range of other materials could contribute to high phosphorous readings. Similarly, in the bulk samples analysed, in this study, there was no correlation between Si and phytolith concentration, probably because it is a major component in natural sediment, indicating that elevated levels of Si are not indicative of high concentrations of phytoliths. The PCA analysis revealed no significant groupings between elemental compositions (Figure 6.4).

The most significant factor to affect the elemental composition of the bulk sediments analysed, was likely determined by the material type (i.e. unburnt soil sample or ashy sediment with charcoal inclusions). The results from this chapter were applied to the phytolith and spherulite analysis reported in Chapter 7 to provide a more nuanced and clearer understanding of what different concentrations of phytoliths and spherulites represent, and the interpretations which can be drawn from each sample.

Chapter 7. New perspectives on plant-use at Neolithic Abu Hureyra, Syria: an integrated phytolith and spherulite study

Preface

Chapter 7 presents the key results from the phytolith and spherulite analysis which has been written up in the style of a journal article and was submitted to *Vegetation History and Archaeobotany* on the 11th of May. Reviewers comments and feedback were received in January 2023 and have been incorporated into this chapter. The final manuscript is under preparation to resubmit to VHA as of the submission of the thesis. I am the sole author of this paper, however, it would not have been possible to write without help and guidance from others, detailed in the acknowledgements at the start of this thesis. As the paper is not yet published, it is presented here in the same style of formatting as the rest of the thesis for consistency, including updated figure and table numbers. Some very minor amendments have been made since the submission of this paper, and references to other sections of the thesis are included to orient the reader.

7.1 Abstract

Archaeobotanical remains contribute crucial evidence for shifts in human economy from foraging to farming, understanding early village life and the strategies employed by people in the past to cope with changing environmental conditions. However, differential preservation of plant proxies often leads to the over or under representation of some plant types. This research analyses phytoliths and faecal spherulites to provide new perspectives on human economy at the Neolithic site of Abu Hureyra, N. Syria (~11,100-6000BC) and plant taphonomy by comparing results with those from previous extensively analysed charred plant macro-fossils. This site is of especial importance as one of the earliest and largest pre-pottery Neolithic B farming settlements in the world, however, it was flooded following the construction of the Tabqa dam in the 1970s. This research therefore presents a case study for some of the methods that can be applied to archival material to continue research in areas of high archaeological significance that are no longer accessible. The results from these new phytolith analyses show that a diverse range of vegetation types were exploited throughout the lifespan of the site, whilst the presence of faecal spherulites highlights dung as a potential fuel source and depositional pathway for plant remains.

Key words: Neolithic, plant-use, phytoliths, faecal spherulites, dung

7.2 Introduction

Current research increasingly demonstrates the diversity of the Neolithisation process, pathways and variations between different regions in SW Asia (Fuller et al., 2012; Arranz-Otaegui et al., 2016). There is increasing evidence of highly localised crop practices, community and household scale strategies/adaptations (Bogaard et al., 2017) and the reflection of unique cultural identities of communities through the selection of plant and animal resources (Kabukcu et al., 2021). The Middle Euphrates Valley holds key evidence on pre-domestication cultivation and the development of increasingly sedentary farming communities (Willcox et al., 2008; Fuller et al., 2011; Willcox, 2012). The archaeological site, Tell Abu Hureyra (Figure 7.1) is significant for its longevity, with evidence for occupation from ~11,100 cal. BC as an Epipalaeolithic hunter-gatherer settlement spanning ~1000 years (Hillman, 2000), and a later pre-pottery Neolithic B (PPNB) occupation over several millennia, ~8600-6000BC (Moore et al., 2000, pp. 527–529). Abu Hureyra, therefore, provides an important case study for advancing our understanding of the development of early agriculture, sedentism, sustainable environmental management practices, resilience and adaptation to changes in the environment (Roberts et al., 2018).

Figure 7.1 Map showing location of Abu Hureyra

Carbonised plant remains provide key evidence for domestication and changes in human economy as the Neolithic developed in SW Asia. However, these remains are preserved under a restricted set of conditions and may represent less than 20% of an assemblage compared with desiccated plant remains (van der Veen, 2007, p. 977) and sometimes less when compared with micromorphological studies of the diverse plant materials preserved in archaeological deposits (Matthews, 2010; Matthews *et al.*, 2020). This research aims to add to the growing body of research on phytoliths as representations of a range of plant materials for comparison to charred remains (Ramsey et al., 2017).

Phytoliths are highly durable, as they are inorganic and therefore more resistant to destructive processes than other plant materials (Piperno, 2006, p. 5). They are present on many sites globally, particularly in SW Asia, in soils with pH between ~2 and 8. Phytoliths are microscopic bodies of silica, absorbed in a soluble state by plants through groundwater (Piperno, 2006). After the decomposition of organic matter, silica is deposited into soil or sediment, often replicating the cells in which it was deposited in the plants. Following the decomposition of organic matter, the phytoliths are deposited into the soil, providing an indicator of the type of vegetation from which it was derived. Some phytolith morphologies are diagnostic of the parts of the plant in which they formed, for example, the stems and leaves or inflorescences of grasses, and the types and proportions of these can inform on cereal processing activities (Harvey and Fuller, 2005; Portillo et al., 2017b) and fuel choices (Gur-Arieh et al., 2013; Portillo et al., 2014; Portillo et al., 2017a). Unlike pollen, phytoliths are not usually highly airborne and tend to provide a localised signal of the plants present in a specific deposit and have been used to determine variations in uses of space at archaeological sites (Tsartsidou et al., 2008; 2009; Portillo et al., 2012). Phytoliths are usually identified to the order or family, rather than genus or species level, often providing a lower taxonomic resolution compared with charred macro-fossils. Integration of both proxies, however, provides the potential for a more complete overview of past-plant use. Table 7.1 highlights some of the key preservation differences between phytoliths and charred macro-fossils as past plant proxies. This study integrates new phytolith data with the previously published charred macro-fossil record from Abu Hureyra (de Moulins, 2000; Hillman, 2000) to provide new perspectives on plant-use and resource management practices during the Neolithic and on plant taphonomy in archaeology more widely.

Table 7.1 Preservation condition and representation of plant material in the archaeological record for charred plant-macro fossil compared with phytoliths

Charred macro-fossils	**Phytoliths**
• Represent plants burnt at low temperatures <400-500°C (Boardman and Jones, 1990), above which carbon is generally oxidised and not present • May over-represent seeds and wood which are more robust than stems, leaves and roots • Can over-represent agricultural bi-products, deliberately burned as waste, weed seeds and fuel (Hillman, 1981) • Usually under-represent cereal chaff which burns quickly (Boardman and Jones, 1990) (an important by-product to identify processing strategies); roots and tubers which are often roasted by direct heat (Colledge, 1991); and oily seeds which may explode	• Withstand burning exceeding 400-850°C and are preserved in non-burnt contexts • Preserve in a broad spectrum of soil types (pH 2-8) (Cabanes et al., 2011; Cabanes and Shahack-Gross, 2015) • Include some morphotypes that are diagnostic of parts of plants in which they are formed • May over represent agricultural by-products and cereal chaff as these often have high silica content • Under-represent dicot plants as monocots produce up to 20 times more phytoliths (Albert et al., 2006, Tsartsidou et al., 2007) • Taxonomic resolution is often lower than for charred macro-fossils

Knowledge of the depositional and taphonomic processes of plant remains is essential for understanding the wider significance of an assemblage (van der Veen, 2007; Matthews, 2010). A major challenge in archaeobotany is to disentangle the potential origins of plant material which may contribute to a single context. Animal dung, for example, has been used as fuel since at least the Neolithic in SW Asia (Miller and Smart, 1984; Matthews, 2005; Portillo et al., 2014; Smith et al., 2019; 2022; Spengler, 2018) to the present day (Reddy 1998; Portillo et al., 2017a; Miller 1984) and may be a source of seeds and phytoliths as these are preserved in modern and archaeological human and animal coprolites (Shillito et al., 2011; Wallace and Charles 2013; Valamoti 2013; Portillo et al., 2020a ; 2020b; Elliott et al., 2020, Portillo and García-Suárez 2021). A new study of faecal spherulites from flotation residues points to the presence of dung at Abu Hureyra during the Epipalaeolithic, and the use of dung fuel from Period 1B, as suggested by Naomi Miller, who argued that herbivore dung could have contributed to the charred assemblage at Epipalaeolithic Abu Hureyra (Miller, 1996). Therefore, an increase in small-seeded grasses and legumes, could represen animal diet, rather than a diversification of the human diet in response to environment changes (Hillman et al., 1997) or weed seeds signalling early cultivation (Hillman et al., 2001). This possibility for misinterpretation demonstrates the importance of resolving the origins of plant material in addressing important issues about developments in human strategies and resilience to cope with change.

It is important to understand the role of dung as agriculture developed in the Neolithic period at Abu Hureyra when charred domesticated cereals and pulses become more prevalent (de Moulins 2000, Hillman, 2000), to resolve whether the continued presence of small-seeded grasses and legumes indicate continued reliance on a broad-spectrum biodiverse diet of wild, gathered food (de Moulins, 2000), or whether some of these remains were deposited by animal dung burnt as fuel. Furthermore, the presence of dung provides key insights into changing human-animal relationships during the Neolithic, and early stages of animal management, as changes in animal bone morphology indicative of domestication can take up to 1000 years to manifest and may be influenced by environmental and anthropogenic factors (Zeder, 2008; Matthews, 2010, p. 107; Fuller et al., 2011). Also of major significance and interest is the use of dung as an important secondary product for fuel, manuring or construction (e.g. Bull et al., 2002; Zapata et al., 2003; Matthews, 2010; Portillo et al., 2014; Portillo et al., 2017a; Gur-Arieh et al., 2019). The use of dung as fuel also provides information on the nature and sustainability of fuel selection and it may also be the preferred fuel choice for specific activities for its long, regular burning properties (Zapata et al., 2003). However, dung is challenging to identify during excavation as it often appears as amorphous organic material (Shillito et al., 2011) and requires specialised and targeted analytical techniques to detect in archaeological deposits and materials (for overview see Shahack-Gross 2011, p. 206, table 1). Dung disintegrates during flotation and during the phytolith extraction process, resulting in the mixing of plant remains derived from dung with those from other depositional activities (Matthews 2010). One method to assess whether dung is present in archaeological sediments is the identification of faecal spherulites (García-Suárez et al., 2018; Portillo et al., 2017a; Matthews 2005; Smith et al., 2019), microscopic, calcitic particles which form in animals' guts, particularly ruminants (Brochier et al., 1992; Canti, 1997, 1998, 1999). This study integrates the study of phytoliths with spherulites to resolve whether dung represents a potential depositional pathway for plant-remains at Abu Hureyra. This study aims to a) provide new evidence for plant-use and resource management at PPNB Abu Hureyra through the analysis of phytoliths and b) identify the extent to which dung may have been used as a resource or was present on site and therefore contributed to the plant fossil assemblage, which will inform more broadly on changes in human-animal relations as farming developed. The implications and new information provided by micro-fossil analysis for specific spaces within the site will be published separately.

7.2.1 Study area: Abu Hureyra

Tell Abu Hureyra is located in northern Syria in the Middle Euphrates valley, 35.866 °N and 38.400 °E, ~ 130km east of the modern city of Aleppo (Figure 7.1). The Neolithic occupation of Abu Hureyra spans from ~8600 to 6000 cal. BC (Figure 7.2a) consistent with dates for the mid to late PPNB (Asouti and Fuller, 2012, p. 150, table 1). The site was built in an area which is predominately calcareous with a chalk substrate, on a well-drained terrace above the floodplain on the South bank of the river Euphrates, which would have provided a dependable water supply (Moore et al., 2000, p. 28). Abu Hureyra is situated in a key heartland of the development of agriculture, which hosted potentially favourable environmental conditions in the rain-fed agricultural zone and on the banks of the Euphrates. Many cultural developments associated with the Neolithic first occur in the Middle Euphrates and surrounding regions (Akkermans and Schwartz, 2003) and through cultural exchange and trade networks, Neolithic Abu Hureyra was linked with contemporary societies in Anatolia, the Southern Levant and Eastern Fertile Crescent (Moore et al., 2000, p. 166 fig. 7.1). Situated on the border of several ecozones, the inhabitants of Abu Hureyra would have had advantageous access to a broad resource base which included riverine forest, woodland steppe, stands of wild cereals (wheats, ryes and feather grasses) and park woodland (Moore et al., 2000, pp. 43–91).

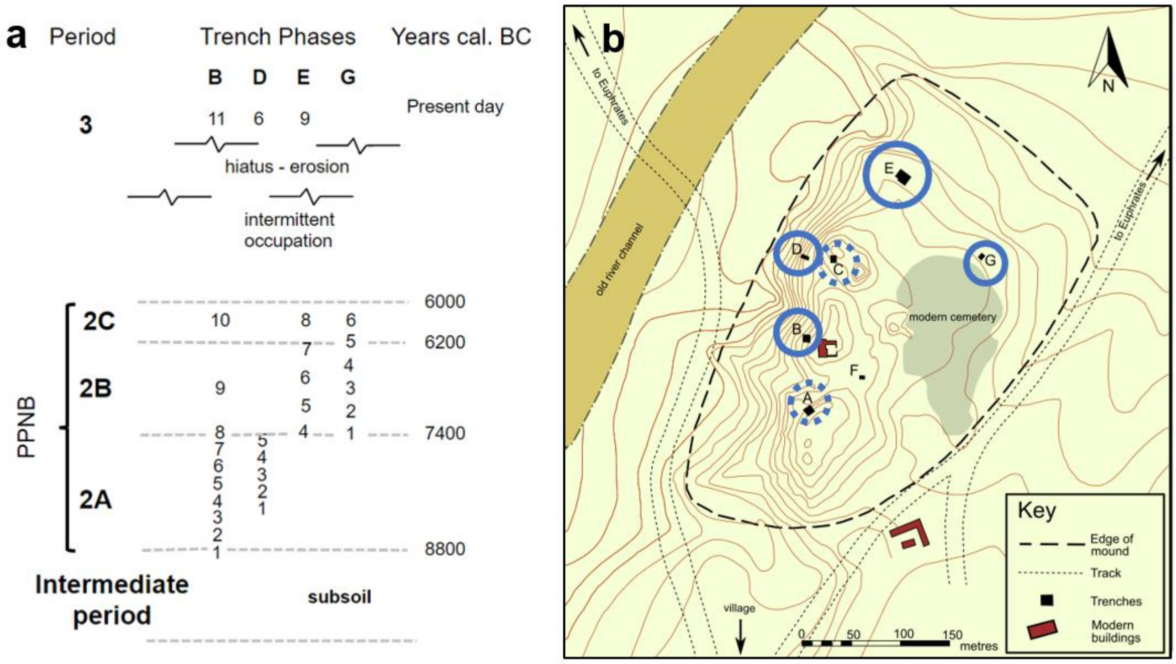


Figure 7.2 a) Schematic diagram showing the years cal. BC for key periods and trench specific phases at Abu Hureyra, adapted from Moore et al. *(2000,* p.257, fig. 8.75) *and b) site map showing locations of trenches and immediate environs of Abu Hureyra adapted from Moore* et al. *(2000 p.34, fig. 2.14).*

The site is now flooded and inaccessible following the construction of the Tabqa dam. Professor Andrew Moore and colleagues excavated seven trenches (Figure 7.2b) during two seasons in 1972 and 1973, revealing densely packed, rectilinear mudbrick buildings across the substantial 11ha+ mound (Moore et al., 1975). However, it is not possible to assess whether all areas of the mound were occupied contemporaneously or represent a series of small settlements over a long period of time. Due to the unique lifespan and significance of the site, and its imminent destruction, an extensive archive of material was recovered (Moore et al., 2000, pp. 547–548). Over one hundred environmental archaeological bulk samples from the site including soil, occupation residues and charcoal are housed at the University of Reading, providing a unique archive for new environmental analyses.

7.3 Materials and methods

Ninety-three samples were selected for phytolith and spherulite analysis in this study to span the lifetime of the site, where possible. Forty-five samples, recovered from Trenches B, D, E and G (and one mudbrick control sample from Trench A) are discussed in detail. The remaining material was sampled from Trenches A and C, for which no detailed contextual information has yet been published. Professor Andrew Moore has provided preliminary phasing for these samples which are assigned to periods 2A and 2B (Appendix 1), and therefore the phytolith and spherulite analysis from this material was used to explore broader trends in plant and resource management between periods 2A and B. This data will be published in full at a later date, as a key focus of this study is to understand the shift in economy between Neolithic period 2A and 2B (Figure 7.2a), when the settlement expanded and plant/animal remains suggest a shift away from the exploitation of wild resources to an increased reliance on domesticates. The material analysed in this study is from bulk samples of occupation residues which were collected during two seasons of excavation in 1972 and 1973 by Professor Andrew Moore and colleagues, and represent different periods, phases and spaces (e.g. internal, external) within the site (Table 7.2) (Moore et al., 1975). Full context descriptions and sample descriptions are available in Appendix 1. Further contextual information including matrices, section drawings and plans are available in Moore et al., (2000, pp. 105–131, 189–259).

Table 7.2 Summary of results from phytolith and spherulite quantification. Spatial context: EX = external, IN = internal, P = pit, F = feature. Deposit type/material: F = fill, OR = occupation residues, A = Ashy, CH = charcoal, H = hearth/fire spot, M = mudbrick

Sample, period, phase	Spatial context	Deposit type / material	Phytoliths / 1g of sediment	Multi cells %	Weathered %	Melted %	Spherulites / 1g of sediment	Darkened spherulites (%)
E55.31: 1A-1	EX, F, P	F, OR, A	2,600,000	11	3		3,200	0
E435.15: 1A-1	EX, F, P	F, OR, A	1,600,000	1	10		0	N/A
E402.14: 1C-3	F	OR, A	1,800,000	2	17		3,200	0
B203.99: 2A-5	IN	OR, A, CH	870,000	8	13		48,000	6
B163.71: 2A-7	IN	OR, A, CH	1,600,000	5	22		14,000	0
B163.74: 2A-7	IN	OR, A, CH	670,000	6	1	0	13,000	0
D14.28a: 2A-4	EX, F, P	F, OR	550,000	11	5	3	0	N/A
D14.28b: 2A-4	EX, F, P	F, OR, CH	590,000	4	5	5	24,000	0
D23.23: 2A-4	EX	OR, A, CH	660,000	1	3	2	0	N/A
D30.?: 2A-4	EX	OR, A	470,000	3	7	2	20,000	17
D36.36: 2A-4	EX	OR, A, CH	560,000	6	2	1	3,700	0
D54.71: 2A-4	EX	OR, A, CH	850,000	5	2	0	6,800	50
D55.69: 2A-4	EX	OR, A	2,000,000	3	21		25,000	0
D57.75: 2A-4	EX	OR, A, CH	770,000	3	3	0	20,000	0
D58.79: 2A-4	EX	OR, A, CH	1,100,000	2	2	0	0	N/A
D59.84: 2A-4	EX	OR, A	1,200,000	7	22		7,000	50
D62.88: 2A-4	EX	OR, A	1,700,000	1	14		3,300	0
D68.100: 2A-4	EX	OR, A	1,500,000	4	16		3,300	0
D66.95: 2A-4	EX	H, OR, A	1,000,000	2	25		6,100	0
A207.64: 2B		M	680,000	9	20		0	N/A
E36.22: 2B-5	EX	OR, A	2,100,000	20	2	2	100,000	7
E36.23: 2B-5	EX	OR, A	1,000,000	12	6	4	44,000	0
E39.33: 2B-5	EX, F	H, OR, A	7,000,000	14	10		64,000	37
E325.113: 2B-5	EX	OR, A, CH	2,300,000	5	5		6,500	0
E329.123: 2B-5	EX	OR, A, CH	1,200,000	17	4	3	3,400	67
E337.7: 2B-5	EX	OR, A	370,000	21	7	4	0	N/A
E338.146: 2B-5	IN	OR, A, CH	770,000	5	33		18,000	40
E339.245: 2B-5	EX	OR, A, CH	930,000	2	27		18,000	0
E344.143: 2B-5	IN	OR, A, CH	1,500,000	2	17		36,000	36
E358.30: 2B-5	EX	OR, A	1,400,000	2	17		13,000	0
E361.10: 2B-5	EX	OR, A	1,100,000	3	16		130,000	7
E362.11: 2B-5	EX	OR, A	2,600,000	7	26		160,000	18
E21.7: 2B-6	IN	OR, A	1,400,000	5	14		7,500	50
E30.17: 2B-6	EX	OR, A	420,000	6	2	1	160,000	18
E210.62: 2B-6	EX	OR, A	700,000	1	28		27,000	0
E231.71: 2B-6	IN	OR, A	930,000	9	4	4	20,000	40
E265.76: 2B-6	EX, F	F, OR, A, CH	970,000	4	30		3,300	0
E268.79: 2B-6	EX	OR, A	1,600,000	10	6	5	0	N/A
E18.3: 2B-7	EX	OR, A	1,000,000	16	5	3	8,800	0
E19.4: 2B-7	EX	OR, A	520,000	6	5	2	58,000	4
G67.35: 2B-1	EX, F	H, OR, A, CH	370,000	6	4	5	0	N/A
G57.32: 2B-2	IN	OR, A, CH	330,000	4	6	1	20,000	0
G62.33: 2B-2	IN	OR, A, CH	350,000	5	6	1	37,000	45
G18.9: 2B-3	EX	OR, A, CH	460,000	13	3	1	0	N/A
G24.15: 2B-3	EX	OR, A, CH	310,000	5	1	3	24,000	33

7.3.1 Phytoliths

Phytoliths were extracted following the rapid extraction method of Katz et al., (2010). Sediments were sieved to remove fractions greater than 0.5mm and combusted at 500°C for ~90 minutes in a muffle furnace to remove organic material. An aliquot of ~40mg was weighed into a 0.5ml conical plastic centrifuge tube. 50µl of 6NHCl was added to dissolve carbonates, followed by 450µl of Sodium Polytungstate (SPT) ($Na_6(H_2W_{12}WO_{40})H_2O$) with a density of 2.4g/ml to concentrate the phytoliths. The solution was sonicated for 5 minutes then centrifuged for 5 minutes at 5000 RPM. Microscope slides were mounted with 50µl of the supernatant, which represents 10% of the total number of phytoliths in the initially weighed sample and enabled quantitative comparisons between samples. A minimum of 200 phytoliths with diagnostic morphologies were counted per sample in a known number of fields (between 10 and 50) based on the counting method outlined by Katz et al., 2010. Numbers of phytoliths were related to the initial weight of material to provide an estimated number of phytoliths per gram of sediment. Phytoliths were counted using a Leica DMEP optical microscope at x200 magnification and x400 for further morphological analysis. Digital images were recorded using a Leica DFC420 camera and DMPL optical microscope.

Phytolith morphologies were identified using standard published literature (Twiss et al., 1969; Brown, 1984; Piperno, 1988; Mulholland and Rapp, 1992; Rosen, 1992), the PhytCore online (Albert et al., 2016) and the University of Reading phytolith reference collection. Nomenclature used within this study follows the most recent International Code for Phytolith Nomenclature, ICPN 2.0, (Neumann et al., 2019a; 2019b) where possible, particularly for geometric morphologies. Modern reference studies (Portillo et al., 2014; Albert et al., 2008; Tsartsidou et al., 2007) were referred to for the interpretation of phytolith morphologies. Phytoliths which could not be identified because of surface pitting and etching caused by dissolution, were recorded as 'weathered morphotypes' which are expressed as a % of the total phytolith assemblage for each sample. Three or more conjoined cells are counted as multi-cells and the individual cell morphologies noted to identify the plant type or part it originated from.

7.3.2 Spherulites

The methodology for identifying and quantifying spherulites is based on Canti (1999). Approximately 1mg of dried sediment was weighed on to a 25x75mm microscope slide, mixed with ~48µl of clove oil which was distributed evenly over an area of ~22x22mm and cover slipped. The number of spherulites were counted in a known number of fields then related to the initial sediment weight and expressed as number of spherulites per gram of sediment.

Spherulites were identified by size, the presence of a fixed cross of extinction and colour; low order white becomes blue/yellow in opposite quadrants when using the λ plate (Canti, 1998) and compared with spherulites derived from modern cow and sheep/goat samples. Spherulites were counted on an optical microscope DMEP at x200 magnification in crossed polarised light (XPL) with further examination at x400 as required. The number of spherulites present in five transects (at x200) were counted. Spherulite concentrations were compared with ethnoarchaeological datasets which follow similar quantitative approaches (Gur-Arieh et al., 2013; Portillo et al., 2014; Portillo et al., 2017a; Tsartsidou et al., 2008). A Mann-Whitney U test was conducted to assess if there were statistically significant differences between the numbers of spherulites present overall between periods 2A and 2B.

7.3.3 Soil pH

Soil pH was measured in a subset of samples, selected to represent different areas of the site, time periods and deposit types (n=16) to check the soil preservation conditions for phytoliths and spherulites. Ca. 10g of sediment was sieved at 2mm, transferred to a 50ml centrifuge tube, mixed with 25 ml distilled water and shaken.

7.4 Results

7.4.1. Phytolith preservation

The soil pH in all sediments tested was between 6 and 8, which is favourable for the preservation of silica phytoliths and calcitic dung spherulites. Phytoliths were identified in all material analysed in this study (Figure 7.3, Appendix 4A), though the estimated number of phytoliths per gram of sediment varies considerably between samples. The lowest concentration of phytoliths (G24.15, 310,000/g of sediment) was identified in ashy occupation deposits recovered from a courtyard in Trench G. The highest concentration of phytoliths (E39.33, 7,000,000/g of sediment) was ashy material sampled from the base of an external hearth in Trench E (Moore et al., 2000, p. 233 fig. 8.51).

Phytoliths no longer identifiable due to surface pitting and etching, classified as “weathered morphotypes”, were present in all samples (1% to 33% of the phytolith assemblages). The highest proportion of weathered morphotypes (33%) was observed in E338.146, ashy, occupation residues from room 1 of the Trench E, phase 5 house (Moore et al. 2000, p. 233, fig. 8.51). Samples B163.74 and G24.15 have the lowest proportions of weathered morphotypes (~1%), as well as low (B163.74) and very low (G24.15) concentrations of phytoliths (Table 7.2, Figure 7.3).

Multi-cell phytoliths, three or more cells in anatomical connection, were present in all material (15 to 21% of the phytoliths assemblages), which, where soil conditions are favourable for multicell phytolith production, is indicative of the relatively good state of preservation of the phytoliths (e.g. Rosen and Weiner, 1994; Jenkins et al., 2016). The highest proportion of multi-cell phytoliths (21.1%) was identified in sample E337.7, sampled from a period 2B external activity area (Table 7.2, Figure 7.3). However, the multi-cells in E337.7 largely consist of degraded multi-cell elongate entire phytoliths, and the overall concentration of phytoliths is low (370,000 per gram of sediment), therefore, in this case, the high proportions of multi-cells is neither indicative of good preservation nor a high concentration of phytoliths. Samples with low proportions of multicells tended to be recovered from external spaces (e.g. D23.23, D.62.88 and E210.62, although much higher proportions of multicells were observed in samples from similar spaces and deposit types.

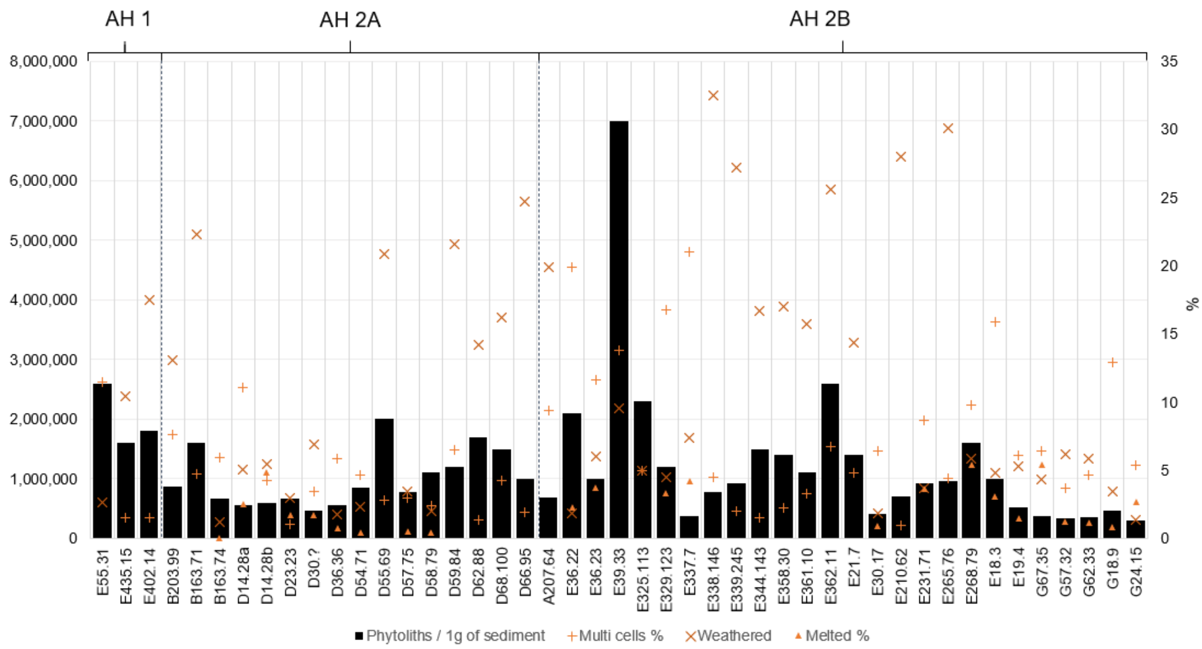


Figure 7.3 Quantitative phytolith results showing the number of phytoliths per gram of sediment (primary y axis) and percent of multi-celled, weathered morphotypes and melted phytoliths per gram of sediment (secondary y axis)

7.4.2 Phytolith morphotypes and vegetative associations

Thirty-nine single cell phytolith morphotypes were identified in this study, including some which are associated with specific vegetation types, as well as redundant phytoliths produced by many different taxa (Vrydaghs et al., 2016). All material analysed contained phytoliths derived from both monocot and dicot plants, though were generally dominated by phytoliths associated with monocot plants.

7.4.2.1 Dicot phytoliths

Phytoliths most likely derived from dicots were represented by a minimum of 4% of the assemblage in sample E55.31; occupation soil from the pit in complex 2 of the Epipalaeolithic phase of the site (Moore et al. 2000, p. 114). The highest concentration of dicot derived phytoliths were identified in sample D54.71, an ashy sample with abundant charcoal fragments, recovered from an external, open area of Trench D, identified as a hub for domestic activities by frequent patches of burning, flints and bones (Moore et al., 2000, p. 218). Phytoliths derived from dicot leaves, identified by epidermal appendage hair and hair bases, platelets and polygonal morphotypes in this study, (Figure 7.5a, b), and from dicot wood and bark, identified by ellipsoid, block and irregular morphotypes in this study, were identified in all samples (Figure 7.4b). The highest proportions of wood/bark phytoliths (>20% of the total assemblage) were observed in material extracted from bulk samples which contained high (>80%) charcoal fragments.

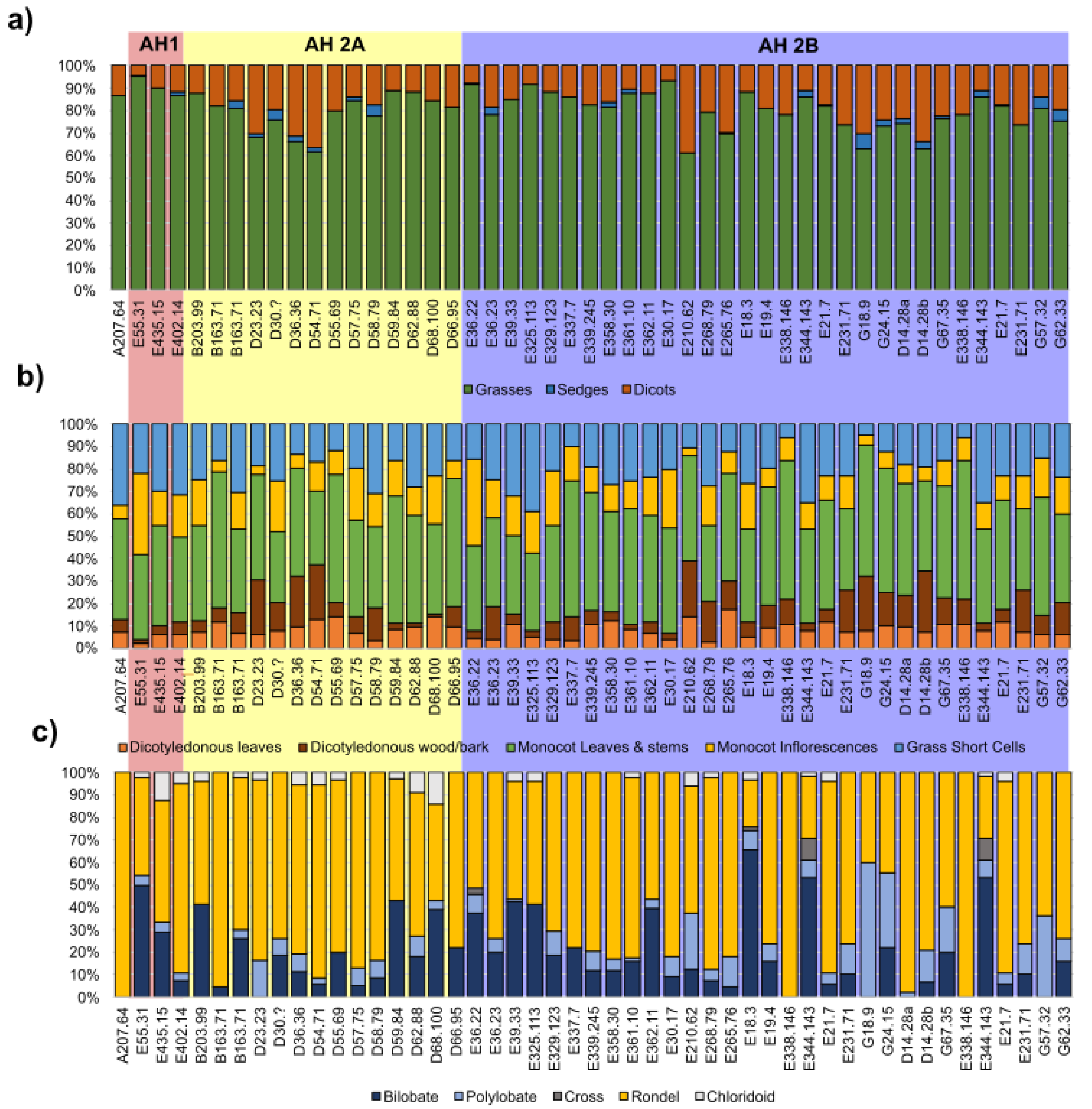


Figure 7.4 Relative abundance of a) key vegetation types, b) parts of plants and c) grass short cells. Samples are arranged by trenches and in chronological order, earliest to latest, left to right according to the phasing published in (Moore et al.*, 2000, p. 257 fig. 8.75).*

7.4.2.2 Monocotyledonous plants

Phytoliths derived from monocots comprised >50% of all phytolith assemblages (Figure 7.4a). The material where the lowest proportion of dicot phytoliths was identified (E55.31) correspondingly had the highest number of monocot phytoliths, with no visible charcoal fragments in the bulk sample. The lowest proportion of monocot derived phytoliths, 44% of the total assemblage, was identified in sample E210.62, which was also characterised by low proportions of multicells and high proportions of weathered phytoliths (Table 7.2). This material was sampled from external occupation residues (period 2B, phase 6) which had abundant evidence for domestic activities such as cooking and waste disposal. The deposit overlay an ash filled channel and surrounding area between buildings (Moore et al. 2000, p. 225, fig. 8.42, p. 235, fig. 8.54 for plan and section diagram).

Some phytolith morphologies identified were indicative of the part of the plant in which they formed. Phytoliths produced in the stems/leaves (e.g. bulliform from monocot leaf, Figure 7.5c) and inflorescences (e.g. dendritics, Figure 7.5d, e) were present in all samples analysed in this study, generally dominated by those produced in the leaves/stems. The exception is E36.22, which has a leaf/stem to inflorescence ratio of about 1:1, and therefore very little difference between the plant parts represented.

Short cells are produced in all parts of monocotyledonous plants and some morphologies are indicative of the type of vegetation they are produced in. Most samples were dominated by short cell rondels (Figure 7.5d, e), most frequently produced by C3 Pooideae grasses (Twiss et al., 1969, p. 111), which would have been prevalent in wild, natural stands surrounding in close proximity to the site of Abu Hureyra (Moore et al. 2000, pp. 78–80 fig. 3.18). Rondels are the only short cell identified in occupation residues from room 1 of the phase 5 building in Trench E (E338.146).

In sample E338.146, occupation residues from room 1 of the period 2B, phase 5 building in Trench E (Moore et al., 2000, p. 233, fig. 8.51), rondels are the only short cell identified (100%, n=4). The lowest proportion of rondels compared with other short cells is identified in sample E18.3, occupation residues from an external area used for domestic activities between buildings, Trench E, period 2B, phase 7 which contained the highest number of bilobate shorts cells, the second most common short cell morphology identified in this study (Figure 7.4c). Bilobates are most commonly produced by Panicoid grasses (Twiss et al., 1969), although also occur in lower numbers in other Poaceae sub families, including Pooideae, especially in the Stipeae tribe (Fredlund and Tieszen, 1994; Strömberg, 2004; Piperno, 2006). Some of the bilobate morphologies identified in this study resembled those found in Pooideae grasses, that is, they are more trapezoidal in cross section compared with Panicoid bilobates which tend to be more flat and symmetrical (Fredlund and Tieszen, 1994). Furthermore, both bilobates and rondels were present in some of the multi-cells identified, suggesting these were derived from Pooid grasses (Figure 7.5e). Although some of the bilobates were also likely derived from Panicoid grasses such as small seeded edible wild grasses of the millet family or reeds, identified in the charred macrofossil record (de Moulins, 2000) and both of which would have grown on the Euphrates Valley bottom.

7.4.2.3 Wetland plant resources

Phytoliths derived from sedges (Cyperaceae) were quantified as a sub-category of monocot phytoliths, as they produce some distinctive morphologies and contribute important information about local wetland resources and environment. Sedge type phytoliths were identified in just under half of all samples (47%). Where present, sedges tend to make up a relatively low proportion of the phytolith assemblage (1-7%). Sedges are present consistently in both periods 2A and 2B, in material from Trenches A, B, C, D, E and G, in ashy occupation residues from both internal and external areas. The highest proportion of sedges were identified in G18.9 ashy occupation residues, recovered from an external courtyard between houses in Trench G, which similarly to the spaces between buildings in Trench E, had abundant evidence for domestic activities taking place in these spaces (Moore *et al*. p.247, fig. 8.65).

Reeds are also often readily identified by distinctive phytoliths morphotypes (Sangster and Parry, 1969; Liu et al., 2013). Bulliform multi-cells (c.f. “stacked” bulliforms), which often form in reed leaves, were present in 18% of all samples analysed. Few elongate multicells were identified that could be securely attributed to reed stems (Ryan, 2011). Single cell “bulliform flabellate” morphotypes were observed in all samples (Appendix 4B) and closely resembled those derived from the subfamily Arundinoideae (Chen et al., 2020, p. 18, fig. 12), which includes *Phragmites* sp. (reeds).

Diatoms and sponge spicules are often associated with wetter conditions when observed in phytolith assemblages and were observed in thirteen out of forty-five and out of forty-five sample respectively (Appendix 4A).

7.4.2.4 Multi-cells

Multicellular phytoliths were identified in all material analysed and usually contained between 3 and 7 individual phytoliths, though often the preservation was not sufficient to accurately count the number of individual phytoliths. The most prevalent multicells were elongate entire (psilate) morphologies, most commonly formed in the stems and leaves of grasses which were identified in 91% of samples analysed. 64% of the samples contain elongate dendritic phytoliths which are usually derived from the inflorescences of grasses, and are commonly associated with cereals. Based on ‘wave patterns’ (Rosen, 1992) of multicellular elongate dendritic phytoliths, several samples contained multicellular phytoliths which resemble those found in wheat and barley, which is consistent with the charred macrofossil record (de Moulins, 2000). Multicellular phytoliths with sufficient attributes to assign to a more specific genus such as wheat and barley were low in number and did not provide a useful avenue for exploring the prevalence of different cereals over time. Furthermore, it is not possible to conclusively assign these multicells to a species or differentiate between wild and domesticate types, based on wave pattern alone. Some studies have shown that wild grasses can be differentiated from cereals based on the multicellular composition, where wild grasses tend to have a more narrow and erratic wave of the cell wall with frequent small sized papillae and high numbers of pits on the periphery (Ball et al., 1996; 1999; Tsartsidou et al., 2007, p. 1270). Few of the multicell phytoliths observed in this study had sufficient numbers and preservation of cells to differentiate between wild grasses and cereals (Figure 7.5 d, e).

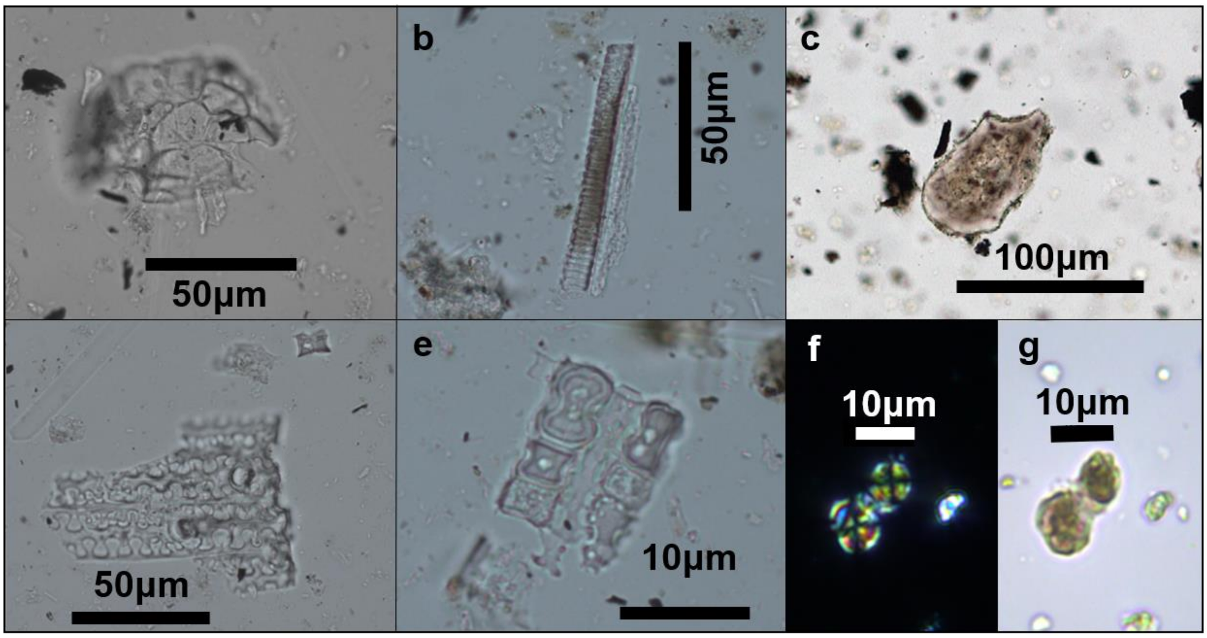


Figure 7.5 Photomicrographs of some key phytolith and faecal spherulites identified in this study: a) dicot hair base (dicot leaves), b) tracheary element (dicot leaves),), c) bulliform (grass leaves, c.f. phragmites *sp.), d) multicell elongate dendritic with shorts cell rondels (grass inflorescence), e) multicell elongate dendritic with short cell rondel and bilobates, f) faecal spherulites in xpl and g) ppl*

7.4.3 Spherulite concentration and temporal patterns

Faecal spherulites were identified in 80% of all samples analysed, although the concentrations varied considerably between samples (Table 7.2, Figure 7.6). Darkened spherulites, which usually form in burning conditions between 500°C and 700°C (Canti and Nicosia 2018, Portillo et al., 2020b) were identified in just under half (49%) of the samples where spherulites were identified. All samples with particularly high concentrations of spherulites (>50,000 per gram of sediment) were associated with external areas of Trench E in material associated with phases 5, 6 and 7 of period 2B. The samples in which no spherulites were identified spanned all three phases of the site and came from external occupation residues in Trenches D, E and G. Spherulites were identified in low numbers in all occupation residues from internal spaces.

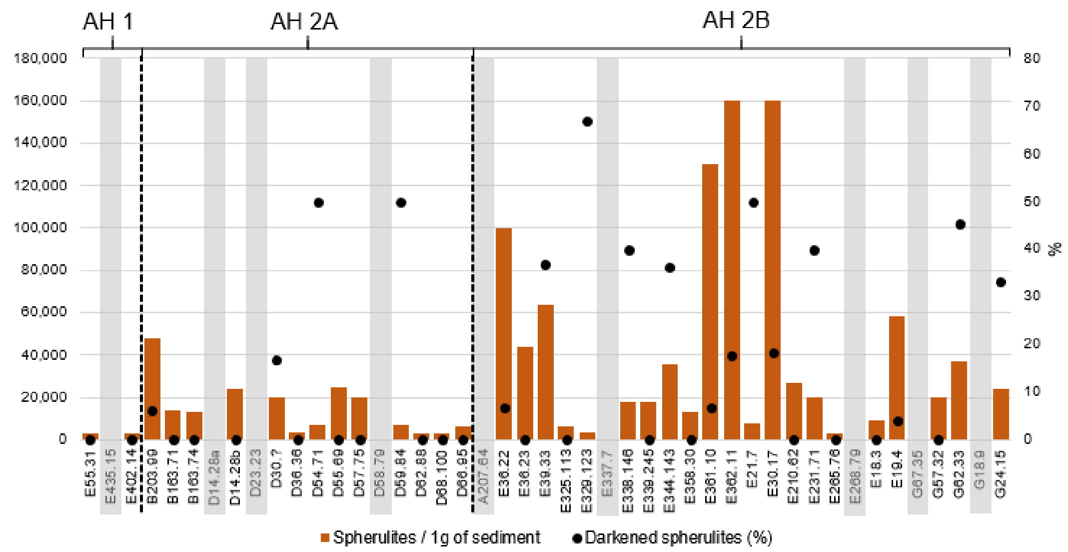


Figure 7.6 Estimated number of faecal spherulites per 1 gram of sediment (bar, primary y-axis)), including the number of dark spherulites observed (%, circle, secondary y-axis)

7.4.3.1 Abu Hureyra 1: Epi-palaeolithic

Spherulites were present in material from two out of three samples recovered from the Epipaleolithic phase of the site (E55.31 and E402.14) (Figure 7.6). However, the paucity of extant samples (n=3) limits interpretations about the presence or ubiquity of dung, and its role as a potential depositional pathway during the Epipalaeolithic occupation. The very low numbers of spherulites identified within them (Table 7.2), and the possibility of contamination from overlying Neolithic deposits cannot be discounted.

7.4.3.2 Abu Hureyra 2: Neolithic periods 2A and 2B

The highest concentrations of faecal spherulites were identified in material associated with period 2B of the site. A Mann-Whitney U test showed no statistically significant difference between the concentrations of spherulites in periods 2A compared with 2B, when the p value is set to 0.05 ($U = 1048$, $p<0.05$ ($p=0.057$). Faecal spherulites were identified in 75% (n=33) of the samples analysed from period 2A, compared with 80% (n=51) for period 2B. The mean concentration of spherulites from period 2A (14,000 spherulites per gram of sediment), was lower than in period 2B (50,000).

7.4.3.3 Spatial variation in spherulite concentrations

Overall, the mean number of spherulites in internal areas (21,125 spherulites/g sediment, n=9) was consistent with external areas (29,350 spherulites/g sediment, n=32). The highest average number of spherulites (43,875/g of sediment) was identified in Trench E, where spherulites were present in 90% of samples analysed. Trenches D and G, both had relatively lower average numbers of spherulites (9169/g of sediment, n=13 and 16,200 per gram of sediment, n=5, respectively). In Trench D, spherulites were identified in 77% of samples analysed, and 60% in Trench G.

7.4.4 Other bio-microfossils and mineral inclusions

Other microfossils identified in this study include diatoms which were observed in thirteen out of forty five samples (29%) and sponge spicules in five out of forty-five samples (Table 7.2, Appendix 4A).

7.5 Discussion

7.5.1 Integrated charred macro-fossil and phytolith assemblages

Charred macrofossils have been analysed and published for five of the same contexts as phytoliths have been analysed from in this study; E402, D59, G57 and B163, and are compared in Figure 7.7. In addition, the charred macrofossil assemblage from B202 is compared with the phytolith assemblage from B203, as these contexts are closely related stratigraphically and are similar in terms of context and deposit type. The integration of both phytolith and charred macrofossil records from the same assemblages enables this study to investigate how plants are represented in these two different proxies at Abu Hureyra. When comparing patterns in overall assemblages for charred plant remains and phytoliths, presence of phytoliths which represent different vegetation types (Figure 7.4, Table 7.3) is consistent with the charred macro-fossil record from the site, showing utilisation of a wide variety of resources including plants from the moist bottom of the Euphrates valley, wood and herbaceous shrubs and dryland grasses from the surrounding steppe (Hillman, 2000, 341-348; de Moulins 2000, 399-416). The variety of plant types also attests to Abu Hureyra's location on the boundary of two eco-zones; the riverine environment and surrounding undulating steppe. Whilst pooid, panicoid and chloridoid grasses may exist in the same environment, the significant dominance of rondels, is consistent with the site's valley location where the local environment was likely to have been more temperate compared with surrounding semi-arid areas (Moore et al., 2000, p. 28).

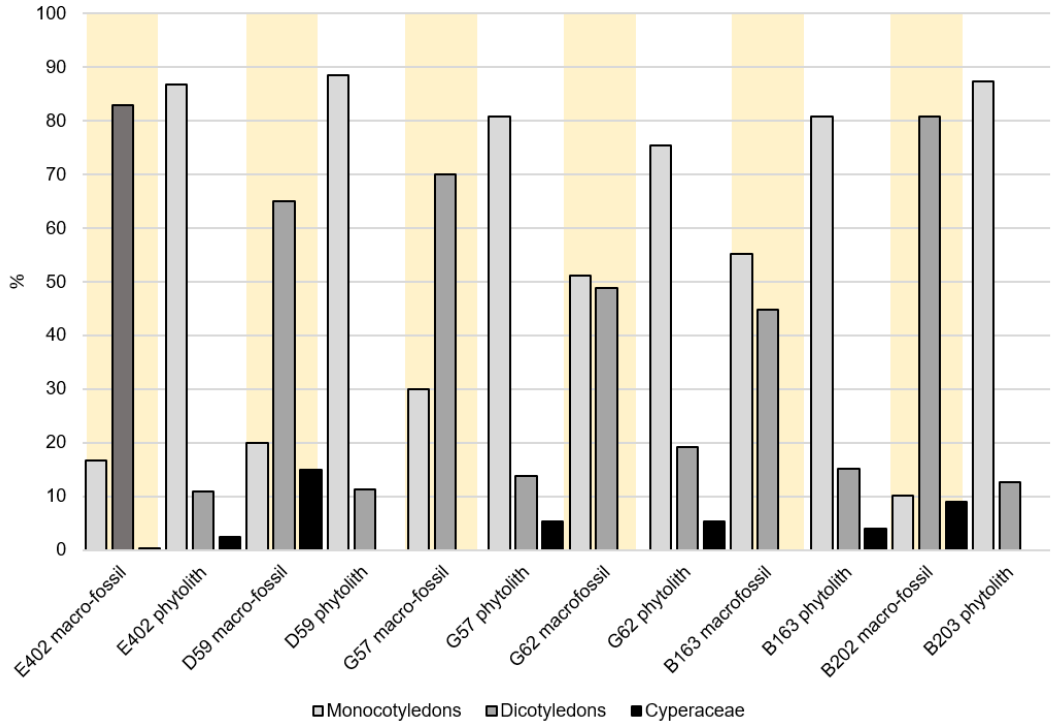


Figure 7.7 The proportions of monocots, dicots, and sedge type plant material represented by charred macrofossil compared with phytolith assemblages, data obtained from de Moulins and Hillman (2000, 2000c)

Table 7.3 Phytolith morphotypes derived from dicots, monocotyledons and sedges as a sub-category of monocots, with the key charred macrofossil associated with each vegetation type at Abu Hureyra. Ecozones defined by Hillman (Moore et al.*, 2000)*

Phytolith morphotypes	Key ecozones of charred macro-fossil remains identified at Abu Hureyra
Dicots – trees and herbaceous shrubs	
EA hair with base – dicot type Platelet MC jigsaw MC polyhedral Ellipsoid Parallelepiped blocky Irregular Tracheids Spheroid psilate	Trees – wild nuts of park woodland and terebinth woodland steppe Native to park woodland and least arid areas of woodland steppe – possibly cultivated Weeds of dryland cultivation, native to steppe and woodland Small-seeded legumes of the tribe Trifoleae (e.g. cloves and medicks) Weeds of irrigated fields and N-enriched ruderal habitats, native to wadis, wadi-banks, and valley bottoms Euphrates valley bottoms environments and weeds of valley bottom cultivation
Monocots – cereals (domestics/wild), wild grasses	
GSSCP rondel – C3 grasses, Pooideae family, temperate climate GSSCP saddle – chloridoids, warm, dry environments, likely grew amongst wild grasses on the semi-arid steppe MC LC dendritic – cereal husks	Domestic cereals Park wood-woodland and least arid areas of woodland steppe (& weeds of dryland cultivation) Moist steppe and open areas of park woodland (& weeds of crops) Steppe, woodland steppe, and park woodland (sometimes weeds of crops) Small-grained grasses mainly of steppe and woodland steppe, and also major weeds of dryland cultivation Weeds of irrigated fields and N-enriched ruderal habitats, native to wadis, wadi-banks, and valley bottoms
Monocotyledonous – reeds and sedges	
Hat/cone MC sedge cone MC elongate dendritic/dentate & stomata Bulliform flabellate GSSCP bilobate (panicoid type) GSSCP polylobate	Euphrates valley-bottom habitats, and weeds of valley bottom cultivation

Key: EA = Epidermal appendage; MC = Multi-cell; GSSCP = Grass silica short cell phytolith

The charred macro-fossil assemblages are dominated by dicot plants while monocots dominate the phytolith assemblages (Figure 7.7). One of the factors contributing to this contrast is that monocotyledon plants produce up to 20 times more phytoliths than dicots which are therefore underrepresented in the phytolith record (Albert et al., 2006; Tsartsidou et al., 2007). Another significant factor is that archaeological charred plant remains are dominated by fuel and burnt storage (Hillman, 1981) while phytoliths represent a broader set of plant uses (Table 7.1). In addition, dicot wood and bark, which may be used for fuel, can be contaminated by up to 40% or 50% by airborne particles which land on the bark (Albert et al., 2003; Tasrtsidou et al., 2007). While both the charred macrofossil and phytolith assemblages were sampled from the same contexts, the methods of recovery and sampling strategy were different, and therefore represent different plant uses and depositional pathways. The flot fractions at Abu Hureyra were recovered in a 1mm mesh and heavy residues in a 3mm mesh (Moore et al. 1975, p. 55), though modern standards generally use a 0.250mm mesh to ensure the recovery of all small-seeded grasses, legumes and chaff (Asouti et al., 2018, p. 25). Furthermore, phytolith representation can vary significantly within a single context (Zurro et al., 2009), which is a consideration in this study, as phytoliths were extracted from bulk samples, and are therefore unlikely to be representative of the whole context. The parts of the plants represented are also different in the charred macrofossil assemblage compared with the phytolith assemblage, which may represent different uses of different plant types. For example, although Pooid cereals tend to produce high numbers of phytoliths, experimental studies have shown phytoliths to be absent or very low in number in the cereal grains (Tsartsidou et al., 2007, p. 1268, fig. 2e). Therefore, the cereal grains themselves, identifed in the charred macrofossil assemblage at Abu Hureyra (de Moulins, 2000, p. 400) are not necessarily synomonous with high numbers of phytoliths.Sedges, as a sub-category of monocots, where present, make up low proportions of the overall plant assemblages in both the charred macrofossil and phytolith records. Where sedges are present in the macro botanical record, they are not identified in the phytolith record and vice versa in this study, with the exception of E402, where sedges are present in both. As sedges represent a potentially important resource for a variety of uses including building, bedding, basketry and fuel (Ramsey et al., 2018), this contrast highlights a key value of adopting a multi proxy approach. One reason for the identification of sedges in phytolith but not charred macrofossil assemblages is that phytoliths do not require burning for preservation. Sedge type phytoliths, identified in G57, G62 and B163, but absent from the macrofossil assemblage were all sampled from internal occupation residues. The sedges could therefore represent construction material or matting which would not have been burned and therefore seeds and other macro plant remains would not have been preserved. Conversely, in D59 and B202/203, sedges are identified in the charred macrofossil record but absent in the phytolith assemblage which could be due to less favourable preservation in these deposits as sedge phytoliths are fragile and particularly susceptible to chemical dissolution (Cabanes and Shahack-Gross, 2015, p. 10). This study found no statistically significant relationship between the proportions of weathered morphotypes and sedges (r^2=0.0882 for samples where sedge phytoliths were present). This is likely because of the wide variety of factors causing phytoliths to “weather” as well as the different factors which will lead to sedges being present or not within the archaeological plant assemblage in the first place. However, samples with the highest proportions of sedge phytoliths identified in this study (>4%) have relatively lower proportions of weathered morphotypes (<7%), suggesting that the proportions of sedges represented in the phytolith assemblage may be affected by partial dissolution, and are more prevalent in better preserved samples.

A number of the plants identified in the charred macrofossil assemblage at Abu Hureyra were potentially consumed by animals (herbivores) and therefore entered the archaeological record through animal dung, likely burnt as fuel (Miller, 1996). Faecal spherulites were identified in all samples where the charred macro-fossil results are available for the same contexts, though spherulite concentration was relatively low in all these samples (Table 7.4). The presence of faecal spherulites from bulk sediment samples does present the possibility that some of the plant remains from those contexts were deposited by animals. The presence/absence of key plant remains identified at Abu Hureyra, which could have been consumed by herbivores in contexts where dung spherulites have been identified are recorded in Table 7.4.

Table 7.4 Presence and absence of some plants consumed by herbivores in samples where the charred macro-fossil results were available to compare with the spherulite quantification conducted in this study (Arranz-Otaegui et al., 2018, p. 276, table 5, and references therein; Dunseth et al., 2019, pp. 177–178, table 5)

Sample number	**Faecal spherulites**	**Heliotropes** ***Heliotropium***	**Feather-grass** ***Stipa* sp.**	**Dock** ***rumex***	**Goose foot** ***Chenopodium***	**Melde** ***Atriplex***	**Domestic/wild barley** ***H. sativum/ spontaneum***	**Wild einkorn** ***T. monococcum* ssp. *boeticum***
E402	3200	0	X	0	0	X	X	X
D59	7000	0	0	X	0	0	0	0
G57	20,000	X	0	0	0	X	0	0
G62	37,000	X	0	0	0	0	X	X
B163	14,000	X	0	0	0	X	0	X
B202	48,000	X	0	X	0	0	X	0

The ingestion of ripe *Stipa* sp. grains at Abu Hureyra by herbivores has been controversial, as Hillman et al. (1997) argued that ripe Stipa grains would be harmful to the digestive tracts of herbivores and therefore avoided, which was contested by Miller (1996, 1997), who argues that, as it is also dangerous to humans, Stipa would be more likely to occur in herbivore dung than human refuse. The identification of faecal spherulites in flotation residues from the Epipalaeolithic period of occupation at Abu Hureyra in a recent investigation by Smith et al., (2022) provides further evidence that some of the charred plant assemblage could have been deposited by animal dung, either burnt as fuel, or otherwise deposited on site then burnt. *Stipa capensis* has been identified in studies of modern dung pellets, demonstrating that they are ingested by animals, including sheep and goat (Dunseth et al., 2019, p. 182). The contexts compared in Table 7.4 all contain at least one type of plant remain which could be derived from animal dung, either accidentally burnt or deliberately burnt as fuel.

7.5.2 Environment, plant-use and resource exploitation

Monocot type phytoliths dominate all assemblages analysed, although there is some variation in the ratios of monocot to dicot phytoliths between samples. Many of the phytoliths identified can be attributed to specific environ types and compared with charred plant macro-fossils identified at Abu Hureyra from similar plant types and environments (Hillman, 2000) (Table 7.3). The ashy nature of most of the samples analysed in this study (Table 7.2) accounts, in part, for the relative consistency between samples from different spaces and time periods, identified in this study, as previous studies have identified few differences in phytolith assemblages between hearths in Hayonim cave (Albert et al., 2003). The variety of morphologies present in each sample attest the heterogenous nature of the sediments analysed, which, collected as bulk samples likely included plant input from a number of depositional events. Similarly to at Abu Hureyra, ashy phytolith assemblages identified at Sheikh-e Abad and Jani in the Central Zagros also exhibit highly variable compositions of phytolith morphotypes within each samples, though all ashy samples are fairly similar to one another (Shillito and Elliott, 2013, p. 19, fig. 16.9). Samples with higher proportions of dicot phytoliths, especially those derived from the wood/bark, tended to be from ashier samples with higher proportions of charcoal (Table 7.2). This material likely reflected wood burnt as fuel, consistent with the abundant wood charcoal from Abu Hureyra, which made up ~90% of all identified charred remains (Hillman et al., 1997). Where present it is common for charcoal to make up high proportions of charred assemblages compared with other charred plant remains (e.g. seeds/chaff). The consistent presence of dicot phytoliths (Figure 7.4a, Table 7.3), despite being lower phytolith producers (Albert et al., 2003, p. 470; Tsartsidou et al. 2007), suggests both woody vegetation types were important and exploited throughout the occupation of Abu Hureyra, alongside grasses and wetland resources. This is supported by the charred macro-fossil record and charcoal records (de Moulins, 2000, pp. 399–416; Hillman, 2000, pp. 341–348; Roitel and Willcox, 2000, p. 545). During the Early Holocene from c. 9700 cal. BC (11,650 cal. BP), woodland gradually expanded as a result of increased precipitation and rapid warming (Roberts et al., 2018, p. 49). By period 2A, ~8600 cal. BC (10550 cal. BP), regional vegetation reconstructions and charred plants in occupation deposits suggest woodland resources were abundant (de Moulins, 2000). Dicots make up an average of 11.8% of the phytolith assemblage during this period, a very slight increase from the proportions of dicots represented in the AH1 samples (Figure 7.4a). This could be a reflection of the increasingly wetter conditions in the region, demonstrated by the decrease in $\delta^{18}O$ isotopes from Lake Zeribar (Stevens et al., 2001) and Lake Van (Wick et al., 2003; Kwiecien et al., 2014). However, more likely, this reflects the compositions of the bulk samples which contained more fragments of charcoal. A climate anomaly resulting in cooler, drier conditions across much of the Middle East occurred at ~9200 cal. BP (7250 cal. BC) (Fleitmann et al., 2008), although its impact was varied (Flohr et al., 2016). There are no significant changes in the types of vegetation identified in the phytolith record in this study between Periods 2A and 2B which represent occupation prior to and following the cooler, drier conditions which occurred at about 7250 cal. BC. However, it could have driven the intensification of already practiced cereal and animal agriculture, resulting in the more widespread agricultural practices in Abu Hureyra 2B compared with period 2A.

Within the monocotyledons, rondel short cells (Figure 7.5e), produced by the Pooideae grass sub-family and associated with temperate climates (Table 7.3; Twiss, 1992, pp. 115–16) are the most ubiquitous amongst the samples analysed (Figure 7.4c). This is consistent with the charred macro-botanical record at Abu Hureyra where Pooideae grasses, including cereals; wheat, barley and rye, have been identified (de Moulins, 2000, pp. 399–416; Hillman, 2000, pp. 341–348). Saddles, which are present but rare within this study (Figure 7.4c), and are indicative of Chloridoids, which usually prefer warm, dry climates and likely grew amongst wild cereals on the semi-arid steppe. Bilobate short cell phytoliths (Figure 7.5e) are commonly associated with Panicoideae grass sub-family (Twiss et al., 1969), however, also form in Pooid grasses. Several multi-celled phytoliths contained both bilobates and rondels (Figure 7.5e) suggesting that some of the bilobates identified in this study originated in Pooideae grasses, growing on the surrounding steppe. Some multi-cells which contained bilobates, also resembled Panicoid types (e.g. Ryan 2011). In many cases it was not possible to distinguish whether the bilobates identified in this study were derived from Pooid or Panicoid grasses, and likely represented the input of both types of vegetation.

Distinctive “sedge-type” phytolith morphotypes are present in 47% (21 out of 45) of the samples analysed, though when present make up a small proportion of the assemblage. The poor silicification of sedge cone cells means they are rarely preserved in archaeological sediments (Albert et al., 2006) which may account for their overall low representation in this study with some of the non-diagnostic morphotypes, such as ‘cylindroids’ and weathered morphotypes likely deposited by sedges, although multicell forms from sedge stems would still be expected to be present. Diatoms and sponge spicules are indicative of wetter conditions and are present in 29% and 11% of 45 samples respectively, albeit in low numbers and do not correspond to those samples where sedge types phytoliths or multi-cells likely derived from reeds were identified. The low numbers of phytoliths which can be securely identified as reed-like is unexpected given the importance of reeds at Abu

Hureyra, demonstrated by abundant impressions in mudbrick, macro-charcoal and modern ethnographic evidence from close to the site and the proximity of the site to the moist floodplain of the Euphrate's Valley (Moore et al., 2000, pp. 406, 485, 499; Roitel and Willcox, 2000). However, the low numbers of reeds compared with sedges are consistent with phytolith assemblages from Late Neolithic Tepe Marani, Iraqi Kurdistan (Marsh et al., 2018, pp. 961–962, fig. 6d). Reed bulliform phytoliths, have been shown to be resistant to partial dissolution, attributed to their geometric surface to bulk ratio of less than one, compared with other more fragile morphotypes (Cabanes and Shahack-Gross, 2015, p. 7). Bulliforms are ubiquitous in every sample, and it is likely that many of these are derived from reeds.

Certain bilobate morphologies are also common in this study (Figure 7.4b, Figure 7.5e) and are often produced by the Panicoideae grass sub-family, generally most prolific in warm and wet climates (Twiss et al., 1969, p. 112) and may represent wetland plants used for basketry in storage facilities (e.g. Rosen, 2005, p. 208, Ryan, 2011). However, based on the morphology and that some bilobates were identified in the same multicells as SC rondels, some of the bilobates are likely also formed in Pooid grasses.

7.5.3 Dung as a resource at Abu Hureyra

The identification of faecal spherulites across the majority (80%) of samples within this study indicates that ruminant dung was both present, and in places, ubiquitous on the site. This study therefore highlights the need to consider animal dung as a potential depositional pathway for both macro-fossil and micro-fossil plant remains from the site. Faecal spherulites require specific soil conditions for preservation, and therefore, while the presence of faecal spherulites provides a good proxy for ruminant dung, an absence of faecal spherulites is not synonymous with the absence of dung, as the spherulites may have dissolved or been degraded overtime or were not present in the highly localised sample taken from a context, especially compared with the large quantities of soil processed in flotation. Spherulite presence can be highly localised and concentrations of spherulites may vary significantly within one archaeological context or even sample. The increase in spherulite abundance between periods 2A and 2B is consistent with the documented increase in domesticated animals during the Neolithic (Legge and Rowley-Conwy, 2000) which may have provided more accessible and reliable supplies of animal dung as a resource as well as increased opportunities for managed animals to defecate on site and waste to accumulate.

There is no evidence for domesticated or herded animals during the Epi-palaeolithic at AH1, which Hillman et al. (1997) argue would have made dung difficult to obtain. Isotopic studies from Çatalhöyük, where dung was burnt as fuel (Matthews, 2005), demonstrate that in the later Neolithic animals were herded some distance from the site (Pearson et al*.,* 2015). Therefore, collecting dung from managed livestock may provide few additional benefits, compared with collecting dung from passing migrating herds on the steppe. Wild gazelle were hunted seasonally from the start of the occupation at Abu Hureyra, it is suggested using the mass hunting strategy of desert kites (Legge and Rowley-Conwy, 2000, pp. 449–450). This may have allowed concentrations of dung to accumulate, which could be collected, dried and stored as dung cakes for use throughout the year.

The identification of dung at Abu Hureyra is crucial for interpreting plant-use and resource management at the site, including evidence for early cultivation. AMS dated, morphologically domesticated grains provide some evidence for cultivation during the Younger Dryas (Hillman, 2000, 376-389), however, they occur in low numbers and may also occur in small proportions in wild assemblages (Nesbitt, 2002). During the occupation of AH1, increased proportions of small-seeded legumes, small-grained grasses and dryland gromwells are interpreted as weeds of arid land cultivation (Hillman et al. 2001, p. 388, figure 3). Similar patterns identified at other Natufian sites such as Eynan, Hilazon Tachtit and el-Wad, however, are interpreted as a broadening of the diet as a coping strategy to maintain stability during the Younger Dryas in the absence of any evidence for cultivation (Portillo et al., 2010; Rosen, 2010), also supported by a more recent reassessment of the charred plant assemblage from AH1 (Colledge and Conolly, 2010). Despite the limiting sample size (n=3), the identification of spherulites in two out of three samples from AH1 suggest, at the least a background faecal component in sediments from which macro and micro plant fossils have been analysed, adding support to Miller's (1996) proposal that at least some of the charred seeds represent dung burnt as fuel, although the possibility that the spherulites represent contamination from higher levels cannot be excluded. The presence of dung, equally, need not exclude the suggestion that people broadened their dietary diversity to cope with deterioration of the environment and the retreat of key food staples (Flannery, 1969; Colledge and Conolly, 2010). Equally, the presence of dung-derived seeds does not exclude the possibility that small-scale cultivation occurred during the Epipalaeolithic. At Çatalhöyük, for example, *Stipa* sp. is strongly correlated with cereal crops (Fairbairn et al., 2002; Weide et al., 2018). A larger Epipalaeolithic sample set, ideally corresponding with the contexts from which archaeobotanical remains were retrieved, could further substantiate Miller's (1996) hypothesis. A study demonstrating the survival of dung spherulites in flotation residues provides a potential new avenue through which to explore dung as a potential depositional pathway, where there are no extant sediment samples (Smith et al., 2019). Even the conclusive identification of dung represents one of many potential depositional pathways for plant remains, which could also represent waste, other fuel sources, construction debris or cooking spillages.

7.6 Conclusion and further research

This study has demonstrated the relatively good preservation of micro-fossils, particularly silica phytoliths and calcitic dung spherulites at Abu Hureyra in archived samples excavated in the early 1970s. Other microfossils including starch grains, diatoms and sponge spicules are also present. Phytolith analysis reveals a variety of different vegetation types are present and used consistently throughout the occupation of Abu Hureyra. As attested in the charred macrofossil assemblage, the phytoliths also indicate that the inhabitants of Abu Hureyra made use of the rich resource base, including park woodland, steppe grasslands and the valley bottom, which was a likely a factor which contributed to the longevity of the site. Significantly, the identification of dung spherulites in over half of all samples analysed indicates a consistent background faecal component in sediments. It should therefore be considered that at least some of the charred plant remains, as well as phytoliths analysed within this study, could be derived from animal dung burnt as fuel, or dung otherwise deposited on site, particularly during the Neolithic. This study highlights the value of archival material, particularly environmental and soil archives which hold the potential to address new and old research questions though constantly evolving methodologies and techniques. The material still exists should future techniques be developed to address the research questions posed here, or new research questions as the field develops.

To identify more conclusively the presence and source of dung, GC/MS analysis has been conducted (Chapter 9). Investigations have also been carried out to identify the use of plant material and dung in building materials (Chapter 8), to better inform on the wider uses of dung as a resource and the implications for changing human-animal relationships at this pivotal time in human history which laid the foundations for the infrastructure of the modern world.

Chapter 8. Insights into resource management and technological development through microbotanical and geoarchaeological characterisation of floor plasters from Neolithic Abu Hureyra, Syria, 8600-6000 cal. BC

Preface

Chapter 8 presents the results of phytolith, spherulite and geochemical analysis conducted on gypsum plaster floor fragments, as well as some other plaster types and materials (Table 4.3 and 4.4). The data is written up as an article which was submitted to *Quaternary International* on the 5th of July 2022, and following reviewer feedback was resubmitted on the 15th November 2022 and accepted for publication in February 2023. I am the sole author of this paper, however, the work could not have been completed without the support of others, detailed in the Acknowledgements section of this thesis. As the paper has not yet been published, the formatting style is consistent with the rest of the thesis, including figure and table numbers. Minor amendments have been made to the original submission to refer to other sections of the thesis.

8.1 Abstract

Plaster is a key technological innovation, manufactured and widely used during the Neolithic across SW Asia. One of the key sites which has contributed significant evidence to our understanding of the development and construction of early farming settlements is pre-pottery Neolithic B site (PPNB), Tell Abu Hureyra, 8600-6000 cal. BC, located in the Middle Euphrates Valley, Syria. Key features of the site's built environment include rectilinear mudbrick structures, painted plaster floors and substantial storage vessels. To investigate these technological developments and chronological and contextual variation in human resource selection, this study integrates analysis of phytoliths, faecal spherulites and geochemical characterisation by pXRF, of multiphase floor plasters from Abu Hureyra. The ubiquity of faecal spherulites present in the plaster matrix indicate animal dung was an important resource for floor plaster construction at Abu Hureyra. A high proportion of the plant remains identified within the plaster matrix through phytolith analysis are likely also dung derived and indicate a mixed foddering regime of managed animals. Consistencies in the plaster make-up reveal traditions and sustainable practices which likely spanned several millennia. Differences in plaster technology when compared to other sites within the same resource catchment area suggest localised, site-specific preferences and cultures may have influenced technological choices.

Key words: Neolithic, plaster, phytoliths, dung spherulites, pXRF

8.2 Introduction

Agricultural societies first developed in SW Asia, c. 10,000 years ago, marking a shift in human-animal-environment and laying the foundations for the world and food systems of today (Zeder, 2011; Bar-Yosef, 2017). The Pre pottery Neolithic B (PPNB hereafter), ~8700-7000 cal. BC (Asouti and Fuller, 2012, p. 150, table 1) is characterised by sedentary agricultural communities and the widespread domestication of plants and animals, which occurred in multiple locations across SW Asia (Fuller et al., 2011; 2012), often in different ways, reflecting the diversity of resource availability and human selection (Kabukcu et al., 2021). During the PPNB, alongside the rise of sedentary agricultural communities, plaster manufacture and use becomes more widespread across SW Asia, with a variety of plaster types (i.e. mud, gypsum, lime) used for a multitude of cultural and economic functions (Kingery et al., 1988; Clarke, 2012 and references therein).

Lime and gypsum plaster are frequently associated with pre-Pottery Neolithic sites across SW Asia (Gourdin and Kingery, 1975), both of which are time consuming to manufacture, resource intensive, and on a large scale would have required some degree of specialisation amongst inhabitants (Nilham et al., 2006), although the manufacture of gypsum plaster is substantially easier and less fuel intensive (Kingery et al., 1988). Plaster production practices and techniques are very diverse across SW Asia in the Neolithic, even within a relatively small geographical zone (Goren and Goldberg, 1991). Therefore, it is important to understand the plaster manufacturing process to understand how people selected and managed local resources. However, the identification of the processes to manufacture gypsum and lime plaster can be challenging as the chemical composition of gypsum rock and plaster are the same, and the calcium carbonate formed in the finished lime plaster has an identical chemical composition to limestone which may have been added as a temper (Gourdin and Kingery, 1975, p. 134). Specialist techniques required such as infrared spectroscopy (IR), micro spectroscopy (μ-IR), and scanning electron microscope-energy dispersive X-ray analysis (ESEM-EDX) and micromorphological thin section analysis are highly effective at characterising plasters (Godleman et al., 2016), however, are often expensive and time consuming. This study therefore explores the utilisation of proxies which are relatively rapid and cost-effective to inform on plaster manufacture practices.

Tell Abu Hureyra, located in modern day N. Syria (Figure 8.1a-c), is significant for its scale and longevity, occupied initially as an Epipalaeolithic hunter-gatherer settlement (11,200-9800 cal. BC), and later as a large (estimated 11ha) PPNB village which comprised densely packed rectilinear mudbrick buildings (8600-6000 cal. BC). Large quantities of plaster were used in the creation and maintenance of the built environment with plastered floors, walls, roofing and substantial "vaiselle blanche" white ware storage vessels. X-ray diffraction, energy dispersive x-ray analysis, sulfuric acids tests and scanning electron microscopy (SEM) indicate the Abu Hureyra plaster was made from gypsum (Kingery et al., 1988; le Mière, 2000; Moore et al., 2000).

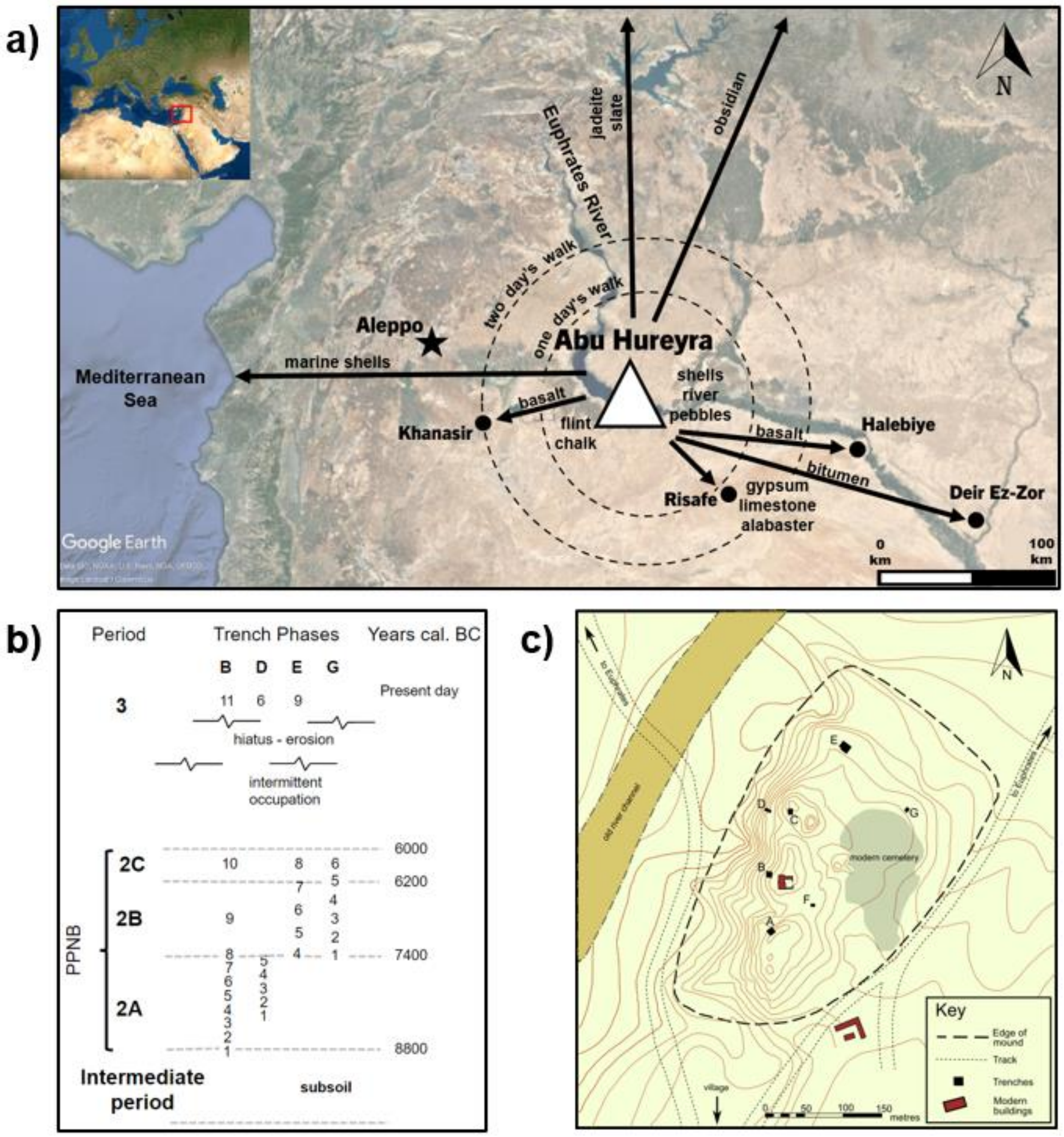


Figure 8.1 Map showing a) study area location and sources of raw materials. Adapted from: Moore et al. 2000, p. 166, Fig. 7.1, and b) the chronology and phasing of Neolithic Abu Hureyra. Adapted from: Moore et al. 2000, p. 257, fig. 8.75.

This study builds on previous analysis which have identified the use of gypsum plaster for construction (Kingery et al., 1988; le Mière, 2000) to provide new information about floor plaster construction practices at Neolithic Abu Hureyra. Raw material acquisition strategies, human resource management and technological choices more widely are identified by applying an integrated micro-botanical and geochemical investigation of the plaster materials. To identify plant material present in the plaster matrix and what vegetation type it was derived from, phytoliths, microscopic silica bodies derived from plants were extracted from the plaster matrix and identified (Piperno, 2006). Faecal spherulites, calcitic particles excreted in the dung of ruminants (Canti, 1997; 1999), are also analysed, to identify if dung is used in the plaster manufacture process to investigate whether phytoliths identified represent plants intentionally selected as temper or incorporated into the plaster matrix by animal dung, which can be further clarified using micromorphology. The elemental composition of plasters was identified by portable X-Ray Fluorescence (pXRF) to explore how plaster composition may vary in different areas of the site and in different time periods. The relatively rapid and cost-effective methodologies applied in this study provide a basis for employing more labour and time intensive techniques such as micromorphology which could provide further clarification to plaster composition. Microfossil and geochemical data are integrated to inform on wider human-plant-animal-environment interactions and cultural innovations during the development and expansion of the Neolithic in SW Asia to better understand how people sustainably managed resource use.

8.2.1 Plaster manufacturing: cultural and technological innovations

The earliest current evidence for lime plaster manufacture is from a lime burning area, at Hayonim Cave, dating to ~12,000 years cal. BC (Kingery et al., 1988; Chu et al., 2008) where calcite has been identified that is more disordered than natural geogenic limestone, but not to the extent that would be expected for a fully calcined lime plaster (Friesem et al., 2019). The utilisation of plaster has been identified from ~10,000 years BC, initially as an adhesive (Kingery et al., 1988, p. 226), but significantly also in burials (Friesem et al., 2019; Grosman et al., 2020), providing new insights on cultural and technological innovations which precede the emergence of the Neolithic and sedentary agricultural settlements. Gypsum plaster has been frequently identified in PPNB (~8700-7000 cal. BC) sites along the Middle Euphrates and in wider Northern Syria, and is used in floor plaster, white ware vessels and burials (Van Zeist and Waterbolk-Van-Rooijen, 1985; le Mière, 2000; Moore et al., 2000; Akkermans et al., 2006; Portillo et al., 2014).

The manufacture process of lime plaster compared with gypsum plaster is more resource and labour intensive, although, the time and energy required to form lime and the environmental impact of its manufacture have been debated (Goren and Goring-Morris, 2008). However, both types require a significant investment of time, raw materials and expertise (Nilham et al., 2006). To manufacture gypsum plaster, gypsum rock ($CaSO_4{\bigcirc}2H_2O$) is heated to between 150-400°C. Between 100°C and 190°C, three quarters of the water is driven off to form the hemihydrate ($CaSO_4{\bigcirc}\frac{1}{2}H_2O$) (plaster of Paris), which when mixed with water reacts to form the dihydrate ($CaSO_4{\bigcirc}2H_2O$) (Equation 1) (Gourdin and Kingery, 1975, p. 135).

Equation 1

$$CaSO_4 \cdot 2H_2O\ (s) \rightarrow CaSO_4 \cdot \tfrac{1}{2}H_2O\ (s) + 3/2\ H_2O\ (g)$$

Plaster of Paris is relatively easy to make and use, however, it has limited use as an architectural material because the mixture sets quickly, unless additional additives are added to retard the crystal development (Gourdin and Kingery, 1975, p. 135). The properties of gypsum plaster make it unsuitable for exterior use unless the climate is very dry as it is soft, relatively soluble and absorbs water (Nilham et al., 2006). A harder plaster can be formed if the gypsum rock is calcined at a higher temperature, which slows setting time considerably, although the addition of accelerators can speed up setting time, if required (Gourdin and Kingery, 1975, p. 135).

Ruminant excrement (e.g. sheep, goat and cow dung) is one of the most widespread products used for organic stabilisation globally (Vissac et al., 2017, p. 16) and would have been widely available during the PPNB and at Abu Hureyra from domesticated animals (cattle and sheep/goat), whose numbers increased between the initial period of occupation 2A and subsequent period 2B (Figure 8.1b). Gazelle dung collected from migrating herds could have also been collected for fuel, as argued by Miller (1996), particularly during the Epipalaeolithic and Period 2A, where faunal records indicate gazelle were the main source of protein (Legge and Rowley-Conwy, 2000). Although the use of dung fuel was deemed unlikely by the excavators (Hillman et al., 1997), new evidence suggests dung fuel was used in both the Epipalaeolithic and Neolithic phases of occupation at the site (Smith et al., 2022; Chapter 7; Chapter 9). This study assesses whether animal dung was intentionally incorporated into gypsum floor plasters at PPNB Abu Hureyra through the identification of faecal spherulites.

The consumption of animal meat as a source of protein has been an important component of human diet long before animal domestication, although the use of secondary products (e.g. for clothing, dairy, manure, construction) also significantly shaped changing human-animal relationships as agriculture developed. Previous ethnoarchaeological research has identified the use of livestock dung in floors, plasters, roofing and mudbrick through micromorphology and dung spherulite analyses (Shahack-Gross et al., 2003; Zapata et al., 2003; Tsartsidou et al., 2008; Portillo et al., 2012; 2014; 2017a; Berna, 2017; Gur-Arieh et al., 2019). A major challenge in archaeological research is to interpret the extent to which people in the past, used secondary products such as dung as a response to the problem of waste management or intentionally selected it as a fuel and construction material of choice for its favourable properties, both of which were likely factors, which influenced domestication pathways. A paramount aim of this study is to understand the use of secondary animal products more widely and to clarify the relationship dynamics between people and animals as agricultural communities developed, particularly as part of the concept of niche construction (Laland and O'Brien, 2010), whereby human selection of resources may impact animal domestication trajectories (Stiner and Kuhn, 2016).

8.2.2 Regional setting and case study

Prof. Andrew Moore and colleagues excavated Abu Hureyra as part of a rescue mission in 1972/73, preceding the construction of the Tabqa dam, which flooded the site. Tell Abu Hureyra was initially occupied as an Epipalaeolithic hunter-gatherer settlement, and later as one of the earliest global examples of an agricultural village, featuring a suite of early domesticates. Located in the Middle Euphrates Valley, modern day northern Syria, 35.866 °N and 38.400 °E, on what would have been the south bank of the river Euphrates, so the inhabitants had access to a reliable and constant water supply (Moore et al., 2000, p. 28). Abu Hureyra was located on a well-drained terrace above the Euphrates' flood plain, in a predominately calcareous environment with a chalk substrate (Moore et al., 2000, p. 28). The subsoil is calcareous sandstone, with high clay content, chalk and pebbles inclusions (Moore et al., 2000, p. 113). The location, on the border of several ecozones, would have provided advantageous access to a wide resource base including riverine forest, park woodland, woodland steppe and stands of wild cereals. Mud, wood and reeds would have been readily available from the Euphrates' floodplain and sources of gypsum were available several kilometres from the site (Figure 8.1a). As the riparian species available from the valley floor were quick growing, they would have provided a renewable source of timber (Moore et al., 2000, p. 267).

The Neolithic occupation of the site has been split into three phases, Periods 2A-C, spanning ~8600-6000 cal. BC (Figure 8.1b). The transition between PPNB periods 2A and 2B, ~7400 cal. BC was marked by significant settlement growth from ~8ha to 11ha and an increased reliance on domesticated sheep/goat, alongside a decline in gazelle exploitation (Moore et al., 2000, p. 257). The transition to period 2C, ~6200 cal. BC, marks a significant shift in the composition of the settlement, whereby spaces between buildings were increased, often with large pits of burnt debris between them and a further increase in the exploitation of cattle and pig (Moore et al., 2000, p. 258).

In recognition of the importance of the site and its imminent destruction, a large archive of samples from the site was recovered and distributed to a number of museums and institutions across three continents to present opportunities for further research (Moore et al., 2000, pp. 247–248). The University of Reading holds an archive of plaster samples from Abu Hureyra for scientific analyses, provided by Prof Andrew Moore, which includes fragments of floor plaster, white ware "vaiselle blanche" vessels, wall plasters, and fragments of plaster with reed impressions (Moore et al., 2000, p. 263).

During the PPNB at Abu Hureyra, most of the excavated building floors were made of a hard plaster (Figure 8.2), usually around 5cm thick, although there were also clay and trodden earth surface floors (Moore et al., 2000, p. 263). Most of the identified floors within buildings were renewed two to three times, but some up to 10 times, demonstrating a significant investment in the constructions. Floor surfaces clearly were not created as purely functional, while some were left plain, others were decorated in black, red and yellow with pigments of soot and red/yellow ochre (Moore et al., 2000, p. 263). Internal plaster floors may have been covered in reed matting and bedding straw, although there was little direct evidence of this identified during the excavation (Moore et al., 2000, pp. 264–265). Reed, straw and cereal chaff impressions have been identified in mudbricks and plaster fragments (de Moulins, 2000). Most internal spaces had hearths which were generally ash filled, without food remains, which led the excavators to suggest that the internal hearths were used for heating rather than cooking (Moore et al., 2000, p. 265). In contrast, external spaces between buildings were filled with refuse, including abundant charcoal, charred plant remains and animal bones, suggesting that most domestic activities, such as cooking and food preparation, took place in these spaces (Moore et al., 2000, p. 268).

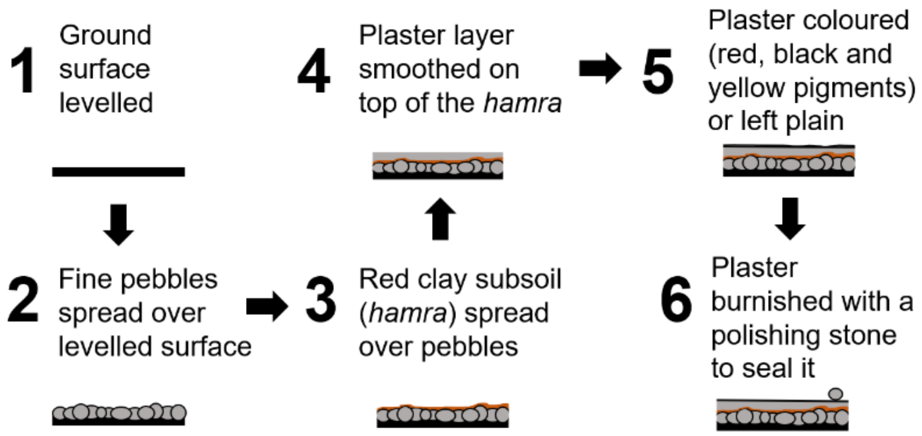


Figure 8.2 Typical sequence of floor construction at Abu Hureyra, after the descriptions in Moore et al. 2000, p.263

Previous analysis by X-ray diffraction, energy dispersive x-ray analysis, sulfuric acids tests and scanning electron microscopy (SEM) suggested the Abu Hureyra floor and vessel plasters were made of gypsum plaster (le Mière, 2000, pp. 532–533). There was a significant investment in the built environment, demonstrated by the range of techniques and materials utilised across the site. Mud was mixed with straw, pressed into moulds and likely sun baked to form the mudbricks from which the buildings were constructed (Moore et al., 2000, p. 267). Wood would be required for both building (e.g. poplar poles) and to heat the gypsum rock for long periods at 200-400°, as well as for domestic cooking and heating (Moore et al., 2000, p. 267). New analyses are being conducted to identify whether other fuels, such as dung, may have also been used at Abu Hureyra. Abu Hureyra therefore provides a good case study to assess how people were interacting with, selecting and managing resources as the PPNB settlement grew and developed.

8.3 Materials and Methods

Twelve samples, comprising a sample from the surface and base of six floor plaster fragments were selected for phytolith, spherulite and pXRF analysis in this study (Table 8.1, Figure 8.3, Appendix 8A, Fig. 1), to represent different phases (Figure 8.1b) and areas of the site (Trenches, B, D and E, Figure 8.1c). Five different material types, including three non-floor plasters and amorphous black carbonised material and material labelled as "bitumen" by the original excavators, were also analysed for spherulites for comparative purposes, n=9, (Appendix 8B; Appendix 9A). Twenty-seven plaster samples were selected for PCA analysis to further explore the elemental compositions, identified by pXRF (Appendix 9B).

Table 8.1 Contextual information and sample descriptions of the selected floor plaster fragments which were analysed for phytoliths, spherulites and pXRF. Contextual information from Abu Hureyra online database (https://www.rit.edu/academicaffairs/abuhureyra/). In the sample ID, the first letter represents the Trench, the first number is the level/context and the second number is the sample number

Sample ID	Period	Context description	Plaster description	Analysis
D100.14 Figure 8.3a/b	2A, phase 3	Clay and plaster floors, internal	Painted floor plaster Yellowish brown plaster Faded red and black surface	Phytolith Faecal spherulites pXRF
D117.34 Figure 8.3c/d	2A, phase 3	Mudwash with occupation soil, some floors, external	Coloured floor plaster Yellowish brown plaster Faded red and black surface	Phytolith Faecal spherulites pXRF
D48.65 Figure 8.3e/f	2A, phase 4	Mudwash with ash, occupation debris, external	Coloured plaster Yellowish brown plaster Faded red and black surface	Phytolith Faecal spherulites pXRF
E20.27 Figure 8.3g/h	2B, phase 7	Mudbrick wash and collapse, external	Plaster Brownish yellow with very eroded black and red surface	Phytolith Faecal spherulites pXRF
B32.16 Figure 8.3i/j	2C, phase 10	Pit filling, grey, ashy, decayed mudbrick, external	Plaster Yellowish grey plaster, smooth black surface	Phytolith Faecal spherulites pXRF
B34.20 Figure 8.3k/l	2C, phase 10	Mudbrick wall, collapse, ashy occupation soil, external	Plaster Reddish grey plaster Smooth red surface, speckled with black	Phytolith Faecal spherulites pXRF
B126.44	2A, phase 8	Neolithic burial, mudbrick and plaster collapse in room 3	Gypsum plaster with basket impressions and red paint	Faecal spherulites pXRF
B164.78	2A, phase 7	Loose fill of room 1, occupation debris and mudbrick fragments	White plaster with reed impressions (roof?)	Faecal spherulites pXRF
E204.149	2B	Clay fill and plaster	Bitumen crumbs	Faecal spherulites pXRF
E331.129	2B	Clay fill and mudbrick collapse	Carbonised material dark amorphous organic material	Faecal spherulites pXRF
E465.320	1	Occupation soil in postholes, smallest pits and burrows	Polished plaster (floor?)	Faecal spherulites pXRF
E71.4	2C	Brown silty wash and occupation debris	Black wall plaster	Faecal spherulites pXRF

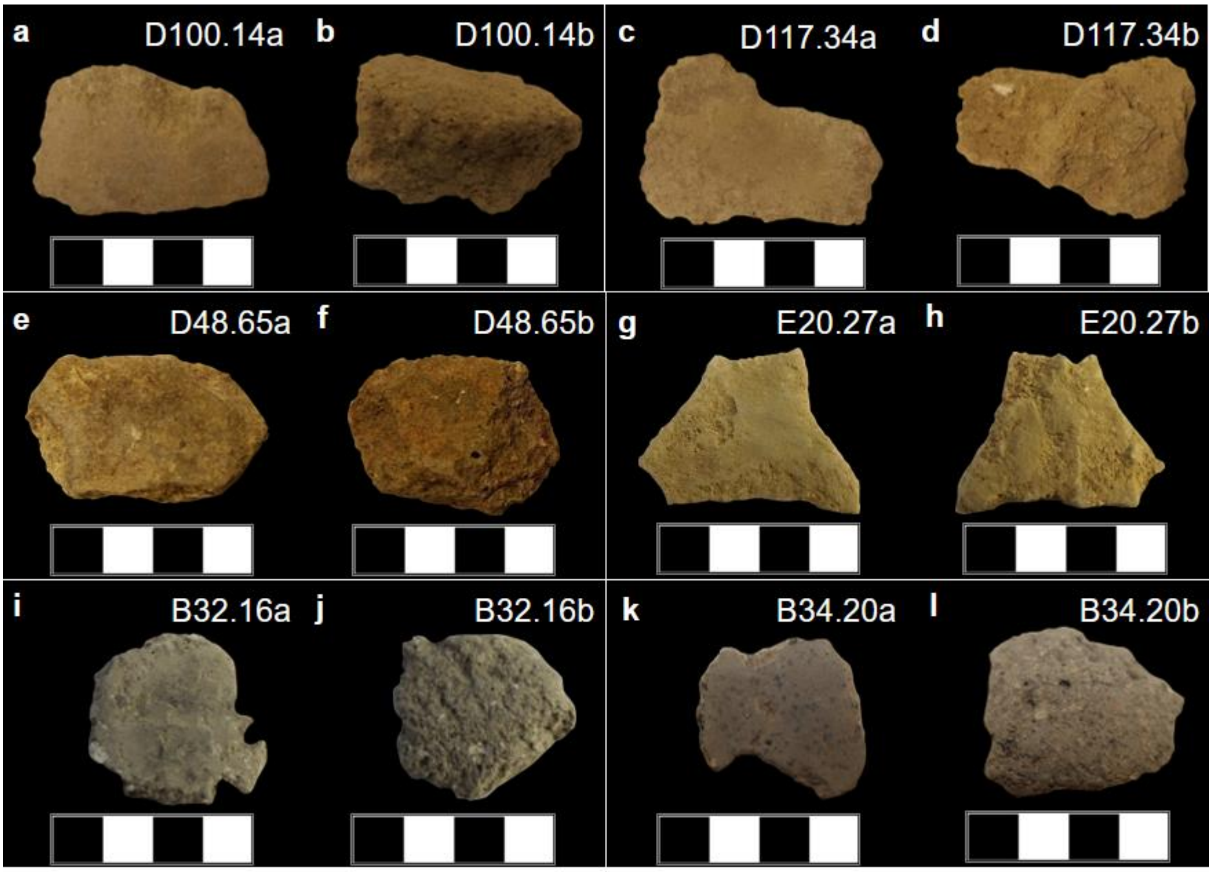


Figure 8.3 Pictures of plaster fragments analysed for phytoliths and spherulites in this study. a. D100.14 surface, b. 100.14 base, c. D117.34 surface, d. D117.34 base, e. D48.65 surface, f. 48.65 base, g. E20.27 surface, h. E20.27 base, i. B32.16 surface, j. B 32.16 base, k. B34.20 surface, l. B34.20 base. "a" at the end of the sample ID = surface of plaster fragment and "b" at the end of the sample ID = base of plaster fragment

8.3.1 Phytolith analysis

Phytoliths are microscopic bodies of silica (SiO_2nH_2O), formed when silica is absorbed in a soluble state (H_4SiO_4) by plants through groundwater, then deposited in cell interiors and the in-fillings of cell walls and solidifies (Piperno, 2006, p. 5). Phytoliths are minerogenic, therefore more durable than other plant remains commonly used in archaeology and are preserved in a broader range of conditions compared with other past plant proxies commonly used in archaeology. Phytolith morphologies can be characteristic of the type of vegetation in which they were formed, enabling differentiation, for example, between monocotyledons (e.g. grasses, reeds and sedges) and dicotys (woody and herbaceous). Phytoliths derived from monocots are often identifiable to a higher taxonomic resolution than dicot phytoliths. Some phytoliths are also diagnostic of the part of the plant in which they were formed, in monocots distinguishing between stems, leaves and inflorescences, and in dicots between wood/bark and dicot leaves. In this study, phytolith analysis is conducted to provide new information about the types of plants selected for use in the construction of plaster floors, and thereby offer new perspectives on the way humans were interacting with and managing local environmental resources as sedentary agricultural settlements grew, which could have increased the demand on the environment.

Phytolith extraction followed a protocol based on the method outlined by Katz et al. (2010a). Material was scraped from plaster fragments using a sterilised metal spatula. A sample of material was taken from the plaster surface (“a”) and a sample taken from the plaster matrix (“b”). When necessary, the scraped material was crushed with a pestle and mortar, however, most of the material was sufficiently fine not to require additional crushing and sieving. An initial check of the material showed very low proportions of charred and organic material which may obstruct phytolith counting, therefore the material was not removed prior to phytolith extraction, i.e. samples were not ashed. Therefore, any indications of burning which were identified (e.g. melted phytoliths or darkened spherulites) could be attributed to the plaster making process. Approximately 40mg of plaster was measured into a 0.5ml conical plastic centrifuge tube and treated with 50µl 6NHCl to dissolve carbonates. 450µl of Sodium Polytungstate (SPT) ($Na_6(H_2W_{12}WO_{40})H_2O$) was added and centrifuged at 5000RPM for 5 minutes to concentrate the phytoliths. 50µl of the supernatant was mounted on a microscope slide, representing 10% of the initially weighed material and related to the initial weight to provide an estimated number of phytoliths per gram of sediment. For each sample, a minimum of 200 phytoliths were counted on a Leica DMEP polarising microscope at x200 magnification and further morphological analysis at x400. Micrographs were taken using a Leica DFC 420 camera and DMPL optical microscope.

Phytolith morphologies were identified using standard published literature (Twiss et al., 1969; Brown, 1984; Mulholland and Rapp, 1992; Rosen, 1992; Piperno, 2006), the PhytCore online database (Albert et al., 2016) and the University of Reading phytolith reference collection. Where appropriate, nomenclature used within this study follows the most recent International Code for Phytolith Nomenclature, ICPN 2.0, (Neumann et al., 2019a), particularly for geometric morphologies. Modern reference studies (Portillo et al. 2014; Albert et al. 2008; Tsartsidou et al. 2007) were referred to for the interpretation of phytolith morphologies. “Weathered morphotypes” refer to phytoliths which could not be identified due to surface pitting and etching and are expressed as a percentage of the total phytolith assemblage. Phytoliths were classified as “Melted” when they were not identifiable due to heat exposure. Morphological changes in phytoliths are not uniform across different morphotypes when exposed to heat, and variation within a heat source (e.g. hearth) also influences visible signs of melting. Therefore, both “melted” and phytoliths with no signs of heat damage often occur within a single assemblage and from the same source of vegetation, and melting or degree of melting, cannot differentiate between different depositional pathways or sources of phytoliths. “Multicells” were recorded when three of more phytoliths were joined in anatomical connection. Each multicell was counted as one phytolith, and the individual cell morphologies noted to enable further identification of its origin.

8.3.2 Spherulites

Dung spherulites are composed of radially crystallized monohydrocalcite, and form in the guts of animals, most commonly ruminants, and are excreted, providing a good indicator of animal dung in the archaeological record (Canti, 1997; 1998; 1999).

Spherulites are identified and quantified on a method based on Canti (1999a). Approximately 1mg of the material scraped from the plaster for the phytolith analysis was mounted onto a slide and thoroughly mixed with clove oil (~50µl) to ensure a homogenous, even spread of material over an area of 22mm x 22mm, which was cover slipped. The number of spherulites were counted in a known number of fields, representing five transects of the slide, and related to the initial sediment weight to be expressed as the number of faecal spherulites per gram of plaster and enable comparisons between samples.

Spherulites were identified by size (5µm-20µm), the presence of a fixed cross of extinction and colour; low order white becomes blue/yellow in opposite quadrants when using the λ plate (Canti, 1998) and compared with spherulites derived from modern cow and sheep/goat samples. Spherulites were counted on an optical microscope DMEP at x200 magnification in crossed polarised light (XPL) with further examination at x400 as required. The number of spherulites present in five transects of the slide (at x200) were counted. Spherulite concentrations were compared with ethnoarchaeological datasets which follow similar quantitative approaches (Tsartsidou et al., 2008; Gur-Arieh et al., 2013; Portillo et al., 2014; 2017a) and modern ashed dung samples from cow, sheep, goat and penning deposits.

8.3.3 pXRF

pXRF was conducted to assess the elemental composition of plaster fragments and explore changes in plaster making sources and technology in different time periods and areas across the site. Twenty-seven floor plaster fragment readings were selected for analysis in this study (Appendix 9B). Different parts of each plaster fragment were analysed as part of a pilot study to assess whether the elemental composition on the surface might provide information about the use of space, compared with the plaster matrix which would provide information about construction practices. An initial batch of samples (n= 11) were analysed on the plaster surface, the base and on a cross-section of the plaster matrix. Each area (surface, base, cross-section) was analysed three times to identify any erroneous readings. The readings in each area of the plaster were consistent with one another, therefore, readings were only taken from the surface and from the base/matrix in the remaining samples (n=14). Each area continued to be analysed three times, and where there were no anomalous readings, the mean was used for further data analysis. Non-floor plasters were also analysed for comparative purposes, including a fragment of white plaster with reed impressions and wall plaster (Appendix 9B). A standard composed of Camberley sands, was analysed at the start of each run of samples and repeated approximately every forty readings, to ensure consistency.

pXRF was conducted using a Thermo Fisher Niton Goldd+ XL3t pXRF Analyser. Material was analysed in the Cu/Zn Mining mode for four minutes per sample (30 seconds for main, 90 seconds for low, 30 seconds for high and 90 seconds for light elements).

8.3.4 Statistical methods

The pXRF analysis identified the concentrations of thirty-nine elements. Twenty-seven samples were selected for principal component analysis (PCA), which included a larger set of floor plaster samples, where the readings from the plaster base were used, and non-floor plasters for comparative purposes (SI 4). The data was checked for large error readings (>10%), but all analysed material had low error readings (<1% at two sigma precision). Elements with a high proportion of the results below the limit of detection (< LOD) were excluded from the analysis. The elemental concentration of P in sample E20.6 was below the limit of detection, therefore, this value was replaced with the corresponding lower limit of detection, which is provided by the analyser as the error reading value. Strontium (Sr) was excluded from the analysis because of the low correlations with other variables. PCA was conducted in IBM SPSS.

8.4 Results

8.4.1 Quantitative phytolith and faecal spherulite results

Phytoliths and faecal spherulites were observed in all floor plaster fragments analysed in this study (Table 8.2, Figure 8.4, Appendix 9C). Phytolith concentrations range from 0.6 million phytoliths per gram of sediment (plaster matrix of E20.27) to 1.7 million phytoliths per gram of sediment (plaster matrix of D117.34). In half of the samples (D100.14, D117.34, B34.20) there is a higher concentration of phytoliths in the plaster matrix compared to the sample taken from the plaster surface (Figure 8.4). The variability between the concentrations of phytoliths in the plaster surface and base of each fragment is less than 30%, except for sample D117.34 where a 53% higher concentration of phytoliths was observed in the plaster matrix compared with the plaster surface sample.

Table 8.2 Quantitative phytolith and spherulite data

Sample ID	Phytoliths/g of sediment (millions)	Phytoliths counted (n)	" Weathered" phytoliths (%)	Multi-cell phytoliths (%)	Melted phytoliths (%)	Spherulites/g of sediment (millions)	Spherulites counted (n)	Darkened spherulites (%)
D100.14a	0.9	244	1.2	3.3	3.3	0.2	58	10.3
D100.14b	1.2	237	3.0	3.4	3.8	0.3	82	17.1
D117.34a	0.9	248	2.8	3.6	3.2	0.1	41	19.5
D117.34b	1.7	253	3.6	4.3	5.1	0.4	93	24.7
D48.65a	1.2	280	2.5	3.9	3.6	0.8	243	10.3
D48.65b	1	289	4.5	5.9	3.5	1	261	13.0
E20.27a	0.8	230	1.7	12.6	1.7	0.4	96	15.6
E20.27b	0.6	225	7.1	6.2	3.6	0.2	68	17.6
B32.16a	0.9	262	0.0	2.3	0.0	1.3	319	0.6
B32.16b	0.9	214	0.9	4.2	0.0	0.6	171	2.9
B34.20a	1	280	6.8	6.1	6.4	0.9	214	14.0
B34.20b	1.4	201	3.5	8.5	4.0	1	232	15.5

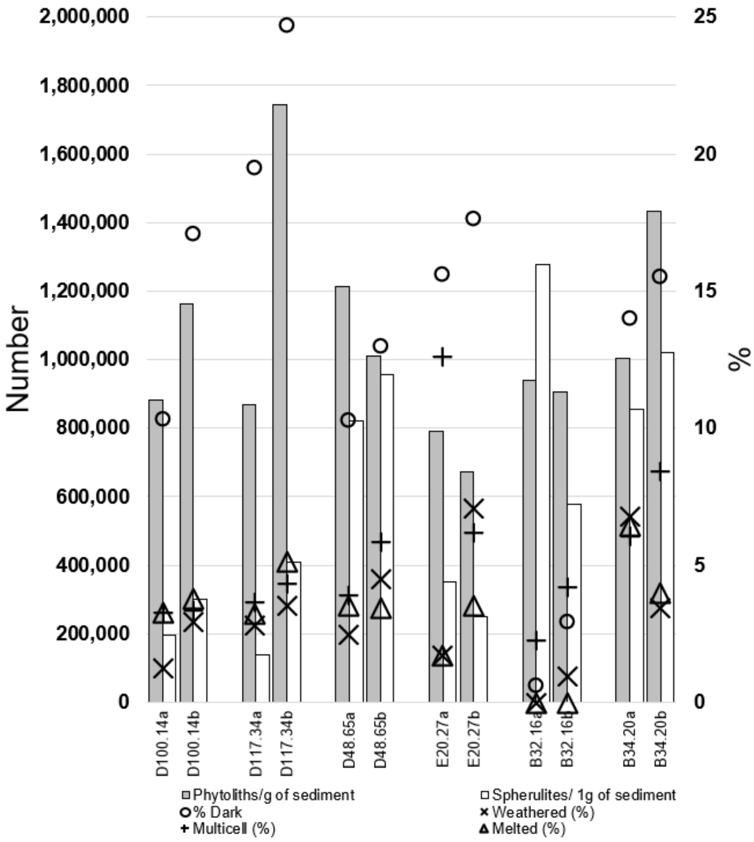


Figure 8.4 Estimated numbers of phytoliths and spherulites per gram of material analysed (primary y-axis) including percentages of darkened spherulites, weathered phytoliths, multi-cellular phytoliths, and melted phytoliths (secondary y-axis) in floor plaster samples.

The proportions of "weathered" morphotypes were relatively low in all material ranging from absent (surface of B32.16) to 6.8% of the total assemblage (B34.20a) which indicates the relatively good phytolith preservation conditions (Table 8.2). B34.20 was the only sample which had a higher proportion of weathered morphotypes in the surface sample compared with the plaster matrix (Table 8.2, Figure 8.4). Multi-cellular phytoliths, defined as three or more phytoliths in anatomical connection, are present in all material analysed in this study and make up between 2.3% and 12.6% of the total phytolith assemblage (Table 8.2). Multi-cellular phytoliths usually contained between three and twelve individual phytoliths, however, quite often the material was too degraded to accurately record the exact number and types of cells within the structures. All samples, except for B32.16 (surface and plaster matrix) contained melted phytoliths, which made up between 1.7% and 6.4% of the phytolith assemblage (Table 8.2, Figure 8.4). The numbers of melted phytoliths are relatively consistent between the samples taken from the surface and the plaster matrix in samples D100.14 and D48,65. There is a higher proportion of melted phytoliths in the plaster matrix sample from D117.34 and E20.27, and in sample B34.20 there is a higher proportion of melted phytolith in the surface compared with the base, which is also the sample with the highest proportion of melted phytoliths overall.

8.4.2 Phytolith morphotype analysis

8.4.2.1 Vegetation indicators

Phytoliths derived from monocots dominated all of the material analysed in this study, (Figure 8.5a, Figure 8.6a-d) and made up a minimum of 70% of the total assemblage (surface of D117.34) and a maximum of 96% of the total assemblage (surface of B32.16). The proportions of different vegetation types represented by phytoliths (grasses, sedges, dicot leaves, dicot wood/bark) were consistent between the sample taken from the surface and the sample taken form the plaster matrix for each sample (Figure 8.5a). Sample D100.14 had a slightly higher variation between the proportions of monocots and dicots in the surface compared to the plaster matrix (16% and 14% respectively), in contrast to the rest of the samples where there was less than 7% variation between the surface and the plaster matrix.

Phytoliths which are diagnostic of sedges, from moister environments, were identified in all samples except for the plaster matrix of D100.14b. Sedges made up a maximum of 6.4% of the total phytolith assemblage in the surface sample of D48.65, where a similar proportion of sedge phytoliths were identified in the plaster matrix (5.5%). In the other two plaster fragments from Trench D (D100.14, D117.34), the proportions of sedges were higher in the surface samples, 5.3% and 5.6% respectively, compared with the plaster matrix where sedge phytoliths were absent (D100.14b) and present only in very low proportions (0.4% of the total assemblage, n=1, D117.34). The proportion of sedges is also relatively consistent between the surface and plaster matrix in E20.27 (1.7 and 1.3% respectively). In contrast to the samples from Trench D, both samples from Trench B have higher proportions of sedge type phytoliths in the plaster matrix sample (3.3% and 5.0%) compared with the plaster surface (1.2% and 1.1.% respectively).

Reed type bulliforms were identified in all samples and made up between 8 and 18.2% of the total phytolith assemblage (Appendix 9C). The surface to bulk ratio of bulliforms is less than one and they tend to be relatively large and well silicified, meaning they are more resistant to dissolution than other morphotypes (Cabanes and Shahack-Gross, 2015, p. 7). Sedges on the other hand are more fragile and susceptible to dissolution and often under-represented in the archaeological record (Cabanes and Shahack-Gross, 2015). Reed type bulliforms were combined with sedge type morphotypes to provide an indicator of the input of wetland type vegetation which made up between 9.2 and 19.6% of the total phytolith assemblage (Appendix 10D).

The proportions of reeds and sedges in the phytolith assemblages are likely a conservative estimate of the input of wetland type vegetation, as many of the morphotypes associated with reeds and sedges are also produced by most other monocot type plants such as grasses and are therefore not included in the wetland count. Higher proportions of wetland type (sedges, reed bulliforms, panicoid bilobates) phytoliths were identified in the surface of all three Trench D samples compared with the plaster matrix. In the Trench E and B fragments, higher proportions of wetland type phytoliths were identified in the plaster matrix compared with the plaster matrix.

The most common short cell phytoliths in all samples were rondels (3.8-20.7% of the total assemblage) derived from C3 Pooideae grasses which include cereals and other wild grasses. Short cell polylobates and bilobates which are most commonly derived from Panicoid C4 grasses (Twiss et al., 1969) were also present in many of the samples but in lesser numbers than the rondels (Appendix 4). Although, similarly to the bilobates identified in the sediment samples (Chapter 7), some bilobates were trapezoidal in long section and more closely resembled those derived from Pooid grasses such as *Stipa* sp. (Fredlund and Tieszen, 1994). Short cell “saddle” phytoliths which usually form in C4 chloridoid grasses are present in low numbers in the surface sample from E20.27a, and the plaster matrix from samples B32.16 and B34.20 (Figure 8.6d).

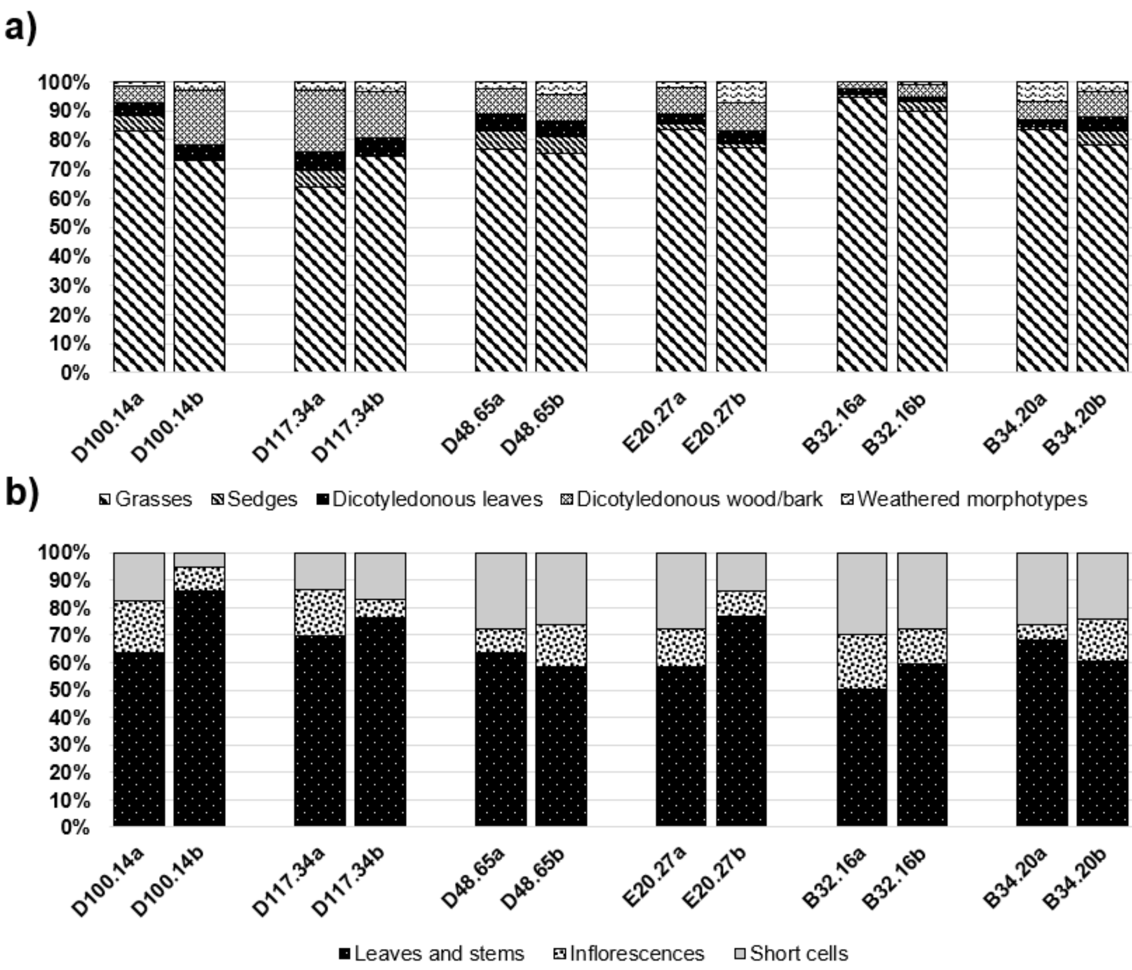


Figure 8.5 Proportions (%) of a) types of vegetation represented by phytoliths and b) the parts of the plant monocot phytoliths are derived from

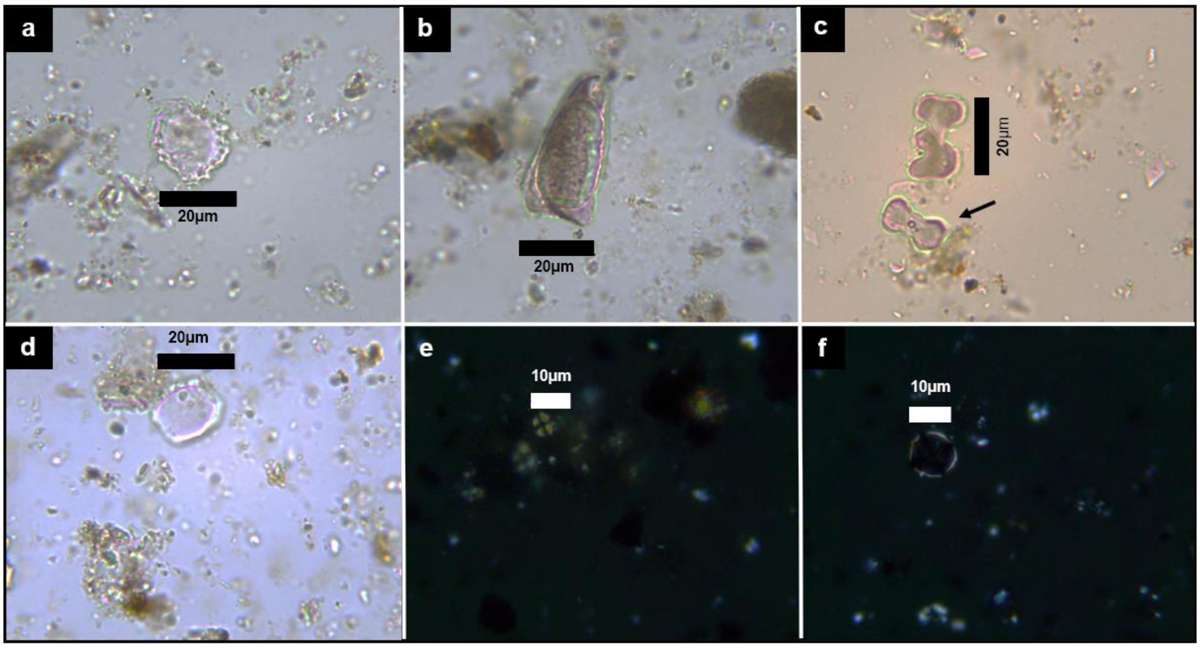


Figure 8.6 Photomicrographs of phytoliths and spherulites observed in this study showing, a) epidermal appendage papillae from a grass inflorescence (D100.14), b) prickle from monocot leaf (B34.20b) c) short cell polylobate (left of scale bar) and short cell bilobate (arrow), possibly from C4 panicoid sub family (B34.20), f) short cell saddle, from C4 chloridoid grass family (B32.16) e) faecal spherulite in XPL (B34.20) and f) darkened faecal spherulite (B34.20)

8.4.2.2 Plant parts

Phytoliths derived from both dicot wood/bark and dicot leaves were identified in all samples (Figure 8.5a). In both the base and surface samples of all plaster fragments, a higher proportion of dicot wood/bark phytoliths was identified, compared with phytoliths derived from dicot leaves (2.3% to 21.1% and 1.6 to 6.5% respectively, Figure 8.5a). Dicot wood/bark was most frequently represented by parallelepiped blocky phytoliths morphotypes which made up between 1.1 and 16.1% of the total phytolith assemblage (Appendix 10C). Platelets were the most frequent phytolith morphotype derived from dicot leaves and were present in half of the samples analysed. No phytoliths which can be assigned exclusively to dicot leaves were identified in either of the samples from B32.16, or in the surface sample from B34.20. All material analysed contained phytoliths from monocot inflorescences which made up 4.6 to 18.7% (e.g. papillae, Figure 8.6a) but a higher proportions of phytoliths from monocot stems/leaves, which made up 44.5 to 62.8% of the total assemblage, (Figure 8.5b, Figure 8.6b). There is no clear association between the part of the plant and whether the sample is taken from the surface or the plaster matrix.

8.4.3 Faecal spherulites

Faecal spherulites (Figure 8.6e) were abundant in all the plaster material analysed in this study, with estimated concentrations ranging from 0.1 to 1.3 million faecal spherulites per gram of sediment. In four out of six of the samples, spherulite concentration was higher in the plaster matrix, compared with the plaster surface, except for E20.27 and B32.16, where the surface sample had a high concentration of spherulites. Both the highest and lowest concentrations of faecal spherulites were identified in samples taken from the plaster surfaces. The highest concentration of faecal spherulites (1.3 million per gram of sediment) was identified in the samples from the surface of B32.16. The lowest concentration of phytoliths (0.1 million spherulites per gram of sediment) was identified in the surface sample of D117.34. There was very little difference between the average number of spherulites in the plaster matrix compared with the plaster surface overall (606,923 million/g of plaster in the surface compared with 585,962 in the plaster matrix). There was a higher concentration of spherulites in the plaster fragments recovered from the later phase of the site, period 2C, (average 933,000/g of sediment) represented by the Trench B fragments (Figure 8.4) compared with the earlier phase, period 2A, (average 471,000) represented by the Trench D fragments (Figure 8.4).

Darkened spherulites (Figure 8.6f) were observed in all material analysed in both surface and plaster matrix samples, though the proportions varied considerably between samples, the surface of B23.16 had the lowest proportion of darkened spherulites (0.6%, Table 8.2, Figure 8.4). In contrast, almost a quarter of all spherulites observed in the plaster matrix of D117.34b were darkened. There was not a large difference in the proportions of darkened spherulites in the surface of each sample compared with the plaster matrix, however, in all samples, a higher proportion of darkened spherulites was observed in the plaster matrix compared with the plaster surface. Faecal spherulites, including darkened spherulites were also identified in sample E465.320, recovered from the earliest phase of occupation which was analysed for comparison, however, is excluded as it was recovered from a mixed context which included animal burrows, and could be intrusive from a later Neolithic layer, and therefore was not selected for phytolith and faecal spherulite analysis. Spherulites were also identified in other materials including a fragment of white plaster with reed impressions, black wall plaster and a fragment of gypsum plaster with reed impression and red paint (Appendix 8A, a-c; Appendix 9B). No spherulites were identified in the material labelled “bitumen” and only four faecal spherulites were identified in the material labelled “carbonised material” (Appendix 8B (d, e); Appendix 9A and 9B).

8.4.4 pXRF

All the floor plasters were relatively uniform in terms of their elemental compositions (Figure 8.7a). All fragments were characterised by high concentrations of Calcite (Ca) and Silica (Si) as well as significant peaks of Sulphur (S) and Iron (Fe) (Figure 8.7a). In the material that was analysed both by pXRF and for phytoliths and spherulites, the Trench D fragments, on average, have the highest concentrations of Ca (152,810 ppm), while the lowest average Ca content is in the Trench B samples (115,413 ppm). The Trench D material also has the highest concentration of S (38910 ppm), while trench E has the lowest average concentration of S (22452 ppm). E20.27 stands out for its relatively lower peak of Magnesium (Mg), and also had the lowest concentrations of Potassium (K), Aluminium (Al), Phosphorous (P), Si, and Chlorine (Cl) (Figure 8.7a), which also has the lowest concentrations of phytoliths and spherulites (Table 8.2, Figure 8.4). In contrast B34.20 has the highest concentrations of Mg, K, Al, P and Si (Figure 8.7a) as well as the highest number of spherulites and a high concentration of phytoliths (Table 8.2, Figure 8.4). The Trench D samples are from the same period and appear to be relatively similar to each other in their elemental composition.

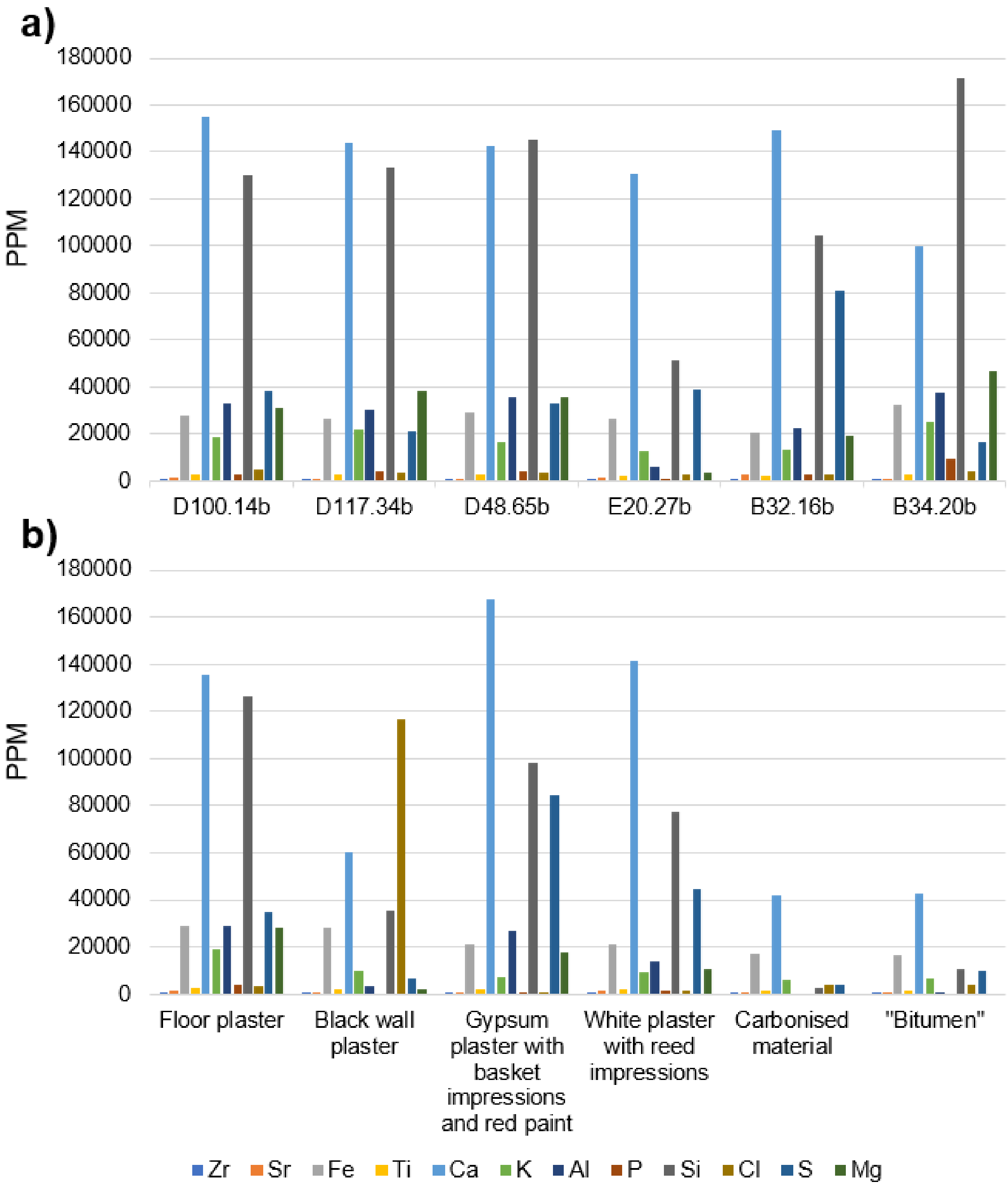


Figure 8.7 Elemental composition of a) floor plaster matrix of five samples analysed in this study and b) comparison between average floor plaster composition and other material types

8.4.5 Integrated phytolith, spherulite and pXRF data

Associations between the concentrations of different elements and quantitative phytolith results were explored in the twelve sample (surface and base of six plaster fragments) which were analysed for phytoliths, spherulites and pXRF (Table 8.1) to better understand plaster composition. Plotting the data in scatter graphs indicated a positive correlation between the concentration of Si and the number of phytoliths per gram of sediment, whereby higher proportions of silica are associated with higher concentrations of phytoliths. The correlation is weak (R^2 = 0.2404) because of several outliers, however, when D117.34b, B32,16a and B34.20a are removed there is a moderately strong correlation between the concentration of silica and the number of phytoliths per gram of sediment ($R^{2=}$0.7409) in the remaining nine samples. The concentration of P appears to be positively correlated to the number of spherulites per gram of sediment, however, the regression value is low (R^2 = 0.3749) because of two outliers, D117.38a and B32.16a. When these values are removed, there was a strongly positive correlation between the concentration of P and the estimated number of spherulites per gram of sediment (R^2= 0.7028) in the remaining ten samples.

8.4.6 Comparison with other material

The other material types analysed in addition to floor plasters were also characterised by high peaks of Ca (Figure 8.7b). In contrast to the floor plasters, the black wall plaster had a high peak of Cl, and a relatively lower peak of S compared with most of the floor plaster samples. Both the fragment of gypsum plaster with reed impressions and the white plaster with reed impressions had very similar compositions to the floor plasters. The material categorised as "carbonised material" and the material categorised as "bitumen", appeared morphologically very similar at the macro-scale. The pXRF analysis also shows that these samples are also similar in terms of their elemental composition, both characterised by high peaks in Ca and relatively high peaks of Fe.

8.4.7 Data exploration

Principal Component Analysis (PCA) was conducted on eleven items with orthogonal rotation (Varimax). KMO verified the sampling adequacy (= 0.702) and all KMO values for individual items were > 0.5. Bartlett's test of sphericity (χ^2 (55) = 344.503, $p < 0.01$) indicated that correlations between items were sufficiently large for PCA. Two eigenvalues had values > 1, and in combination explained 77.1% of the data. After rotation the items that clustered on the same components suggested that PC-1 represented variation in Fe, Ca, Zr, Ti, Zn and K, and PC-2 represented variation in Mg, Si, Al and P. A scatter plot was generated to further explore associations and groupings between different samples (Figure 8.8).

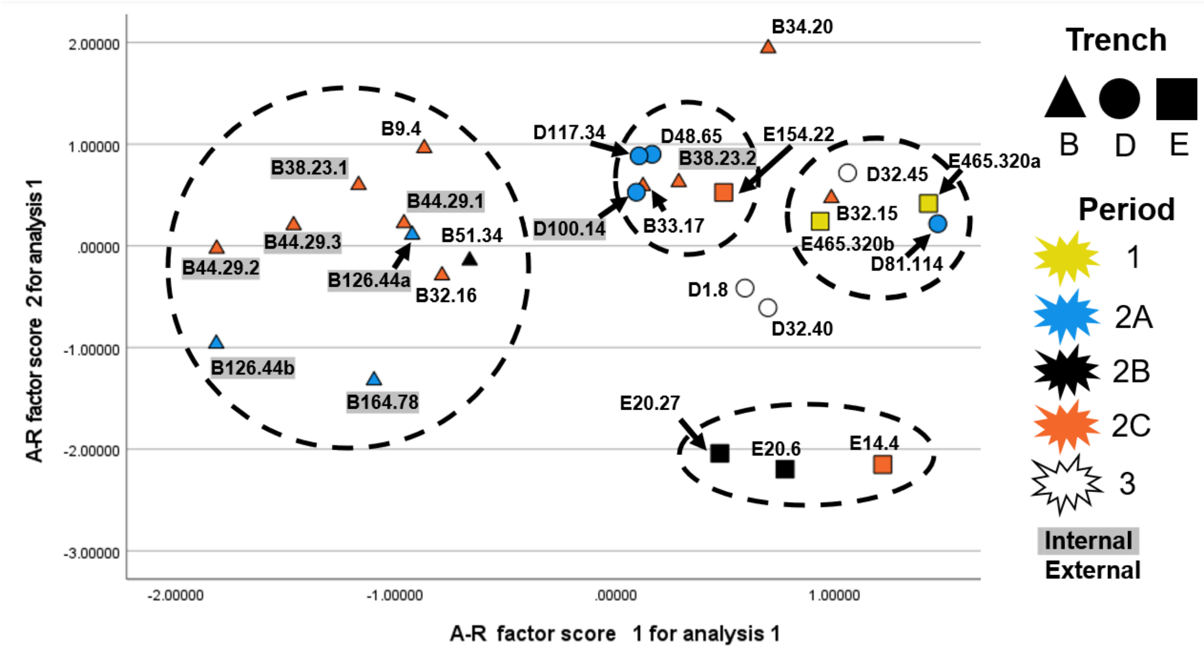


Figure 8.8 Scatter plot to visually display PCA-1 (x-axis) and PCA-2 (y-axis) for the geochemical data. Figure generated in IBM SPSS

The scatter plot revealed groupings between different sample categories (Figure 8.8). Many of the plaster fragments clustered in groups according to the Trench (B, D, E) and thereby area they were recovered from, and to a lesser extent, the period (Figure 8.8). Most of the plaster fragments from all periods of Trench B, represented by triangles, grouped together. B164.78 is a large fragment of white plaster with reed impressions, more likely representing former roofing material than floor, and lay slightly outside of the main cluster of samples from Trench B, alongside sample B126.44, a fragment of gypsum plaster with reed impressions and red pigment, where the reading was taken from the base of the sample (Figure 8.8). In contrast, the reading taken on the red pigment groups very closely with some of the floor plaster samples (e.g. B44.29.1). Sample B44.29 contained multiple fragments of plaster, three of which were selected for analysis to check how the elemental composition differed between material sampled together and help to ascertain if differences between samples reflected different morphologies, time periods or spaces, or represented natural variability. All three fragments from B44.29 grouped relatively closely. In contrast, fragments of plaster which were sampled separately from the same context, (B32.15, B32.16) and (B38.23.1, B38.23.2) did not group very closely together (Figure 8.8).

The plaster fragments representing different contexts from Trench D, period 2A, all cluster tightly together (blue circle, Figure 8.8) except D81.114. Two of the plaster fragments which were recovered from the later post-Neolithic period 3 (white circles), also grouped relatively closely, with a third clustered more closely with other plaster fragments (Figure 8.8).

Polished plaster sample E465.320 was recovered from one of the earliest layers of occupation at Abu Hureyra, period 1, phase 1, which is part of the Epipalaeolithic hunter-gatherer settlement (yellow square in Figure 8.8; Appendix 9C). The pXRF readings from both the base and the surface of E465.320 were included in the PCA analysis and scatter plot to investigate whether different parts of the fragments grouped together. The surface and base readings clustered relatively close together (Figure 8.8), also with two samples from Trench D (period 2A and period 3) and one sample from Trench B (period 2C). The black wall plaster sample E14.4 (period 2C) lay outside the main cluster of floor plaster samples, though grouped with the two floor plaster samples also from Trench E (E20.6 and E20.27), which were recovered from the same context.

8.5 Interpretation and Discussion

8.5.1 Plaster composition and manufacture

The high concentrations of Ca are expected given the local geology and close proximity of the site to sources of limestone and gypsum (Figure 8.1a). The sulphur peaks in all of the sample are consistent with previous analyses of plaster fragments from Abu Hureyra which show them to be made from gypsum (Calcium sulfate, $CaSO_4$) (Kingery et al., 1988; le Mière, 2000). Gypsum crystals can also be seen in some fragments under a stereo microscope. Elevated concentrations of Zr have been associated with roofing material in modern ethnographic reference studies (Jenkins et al., 2017, p. 423), however, the suspected roofing material analysed in this study ("white plaster with reed impressions", Figure 8.7b) did not have elevated concentrations of Zr, which likely reflects differences in the local geology. Plaster floor samples, characterised by high Ca, and also with significant peaks of Si, Fe, and Al, are consistent with floors analysed at the modern, abandoned village of Al M'tan, Jordan, and would have been abundant in the local geology. The high peaks of S identified the Abu Hureyra floor plaster fragments are not present in Al M'tan (Jenkins et al., 2017, p. 423), likely because a different raw material, rather than gypsum has been used.

The relatively high concentration of phytoliths in all floor plaster fragments analysed is indicative of the significant and intentional input of plant material, either incorporated as vegetation for temper or through the inclusion of highly organic dung remains (Table 8.2). Although a low number of phytoliths could have been incorporated into the plaster matrix from the source of gypsum, through contamination from local sediments, this is unlikely to have contributed to the overall phytolith assemblage in a significant way. The total phytolith assemblage of the floor plaster material was composed of between 9.2% and 19.6% wetland plant material, identified by the presence of reed bulliforms, sedge phytoliths and panicoid grass short cells. Some of the wetland plant phytoliths could have been incorporated into the plaster matrix through mud collected from the valley bottom, but similarly, would have made up a smaller proportion of the assemblage if contributed only through contamination. Therefore, some of the wetland plant material could have been incorporated through animal dung, as managed animals may have been herded in the rich valley bottom.

Phytoliths survive digestion by animals and are frequently observed in high concentrations in dung (Gur-Arieh et al., 2013; Portillo et al., 2020a; 2020b). As dung spherulites are so prolific in all material analysed it is likely that much of the plant material was incorporated into the matrix through animal dung, as has been a common practice from prehistoric times to the modern day (Miller and Smart, 1984; Reddy, 1998; Miller et al., 2009; Portillo et al., 2014; 2017a). Cow dung is an additive frequently used in earthen architecture in the Kerala region of India (Paul and Changali, 2020). The ubiquity of faecal spherulites suggests animal dung was an important component of the plaster matrix. Dung may have been used as an additive for the gypsum plaster which may have retarded crystal development to reduce the plaster setting time (Kingery et al., 1988). The strength of plaster would be enhanced with the addition of cow dung (Vissac et al., 2017) as the fibre content in cow dung has been shown to make fired plasters stronger (more tensile) improve water resistance and reduce cracking (Henry and Therrien, 2018).

The presence of darkened spherulites in all plaster floor fragments (0.6 - 24.7% of the spherulites observed, Table 8.2) and melted phytoliths (up to 6.4% of the total phytolith assemblage) suggests that the dung was exposed to heat. Most of the plaster fragments had more than 10% darkened spherulites which indicates burning temperatures of between 400° and 600°C (Canti and Nicosia, 2018), although burning temperatures could have been higher in anaerobic conditions (Portillo et al., 2020b). Phytoliths also start to undergo morphological changes from c. 600°C (Brochier, 2002), suggesting that both the phytoliths and spherulites underwent the same heating process. In sample B32.20, the relatively lower proportions of darkened spherulites (0.6% and 2.9% in the base and surface sample respectively) and absence of melted phytoliths suggest a lower burning temperature, although it is clustered with other plaster fragments in terms of elemental composition (Figure 8.8). Dung may have been exposed to heat prior to being added to the plaster of Paris mixture, possibly introduced as ashes, or incorporated untreated and burnt afterwards as part of the plaster matrix. Some of the phytoliths and faecal spherulites could represent contamination from dung fuel used to manufacture the plaster, however the very high concentrations of faecal spherulites exceed concentrations analysed directly from archaeological and experimental dung fuel deposits (Portillo et al., 2020; 2021; Chapter 7). Amorphous charred flecks are also visible in the plaster matrix (Appendix 8A), further supporting the inclusion of burnt material. This study has taken the first step to demonstrate that dung was an important component of the gypsum floor plasters and is likely the pathway through which many of the phytoliths present were incorporated into the plaster matrix. Further research is needed, however, to clarify the pathway of dung into the plaster matrix, which could be resolved through micromorphological analysis of thin sections of plaster fragments.

8.5.2 Intra site similarities and differences in plaster technology

8.5.2.1 Changes in plaster technology over time

Two of the samples included in the PCA analysis came from readings of a floor plaster fragment (surface and base, E465.320) which was sampled from the earliest Epipalaeolithic phase of the site. This sample contained abundant dung spherulites, however, as the context included pit fills and burrows, the fragment may have been intrusive from later (Neolithic) levels. The concentration of spherulites was much higher in the samples recovered from the later phase of the site, period 2B (~7400-6200 cal. BC), compared with those from the earlier phase of the PPNB site, period 2A (~8600-7400 cal. BC) which could be indicative of the increased utilisation of animal dung over time. Some of the plaster samples were not recovered in situ but in secondary deposits, and therefore in some cases would not have been manufactured in the time period assigned to the deposit they were recovered from. As PPNB Abu Hureyra developed there is a marked increase in domesticated animals (sheep/goat/cow), and reduced exploitation of wild animals (gazelle) between period 2A and 2B (Figure 8.1b) (Legge and Rowley-Conwy, 2000), and dung would have therefore become more plentiful as the settlement expanded. However, as the fragments representing different time periods are also from different trenches, the different quantities of spherulites could also represent different uses of space, and/or different construction techniques in different parts of the site. Spherulites may otherwise have contaminated the samples through use of the space (e.g. dung trodden in from external areas), though given the plasters are hard and fired, this would not account for the ubiquity of spherulites in the plaster matrix. Plaster manufacture may not have been uniform across the site and could have differed between households or over generations as traditional technologies changed over time. Spherulite production is not always uniform, and while it is well documented that spherulite production varies significantly between different species (Canti, 1999; Portillo et al., 2021), there can also be substantial differences in spherulite concentrations from the dung of the same species raised in the same environment (Portillo et al., 2021). Preservation of spherulites is also known to vary according to burning temperature and pH for example (Portillo et al., 2020b). Conditions for faecal spherulite preservation may also vary in different areas of the site, for example, dissolved in dung/pens with high uric acid content, which could account for some of the variation in the concentrations of spherulites between different materials. Further analysis of more plaster fragments will provide additional evidence about whether there is an increase in the use of dung in construction material over time, possibly related to increased accessibility to dung as more animals were domesticated. However, more targeted research into the taphonomy of dung spherulites in plasters and the relationship between the quantity of dung and number of faecal spherulites is necessary to refine interpretation, analysing controlled experimental and ethnographic material and the additional use of more powerful analytical techniques such as SEM microscopy and micromorphology.

8.5.2.2 Use of space

Phytoliths and spherulites were analysed from both the base and surface of the plasters to assess if it was possible to infer information about the use of different spaces from the microfossils on the surface compared with in the plaster matrix. It has been suggested that activities are probably the main drivers of phytolith signatures on the surfaces, rather than the floor make-up (Jenkins et al., 2017, p. 427). However, as the plasters in this study are relatively solid, and not analysed in association with residues from the surface found *in* situ, the elemental and microfossil signature most likely represents the plaster composition. Furthermore, overall, the surface and plaster matrix were quite similar, even though, macroscopically, the plasters appear quite heterogeneous (Figure 8.3, Appendix 8A), which may account for any variability between concentrations of phytoliths samples from the plaster matrix, compared with those sampled from the surface. Ethnographic research which forms an important baseline from which to interpret archaeological data sets has found phytolith assemblages to be similar across floors surfaces, including those sampled from the edges of hearths (Vos et al., 2018, p. 684). Other studies, however, have identified variations in microfossil assemblages which reflect different uses of space (Tsartsidou et al., 2009; Portillo et al., 2014). Plaster surfaces in this study are relatively similar in terms of phytolith composition, although proportions of monocots to dicots do vary and the floor surfaces also have a high variety of different phytolith morphotypes within each sample, as observed in floor samples from the modern abandoned village, Al Ma'tan, Jordan (Jenkins et al., 2017, p. 426). Further analysis of more plaster floor fragments will help to clarify if differences between phytolith and spherulite concentrations and assemblages are representative of different activity areas or spaces and additional techniques such as micromorphology will also clarify microscopic variations and formation processes.

pXRF readings were taken from various points on the plaster, but were also quite similar. The PCA analysis revealed some clear groupings of plaster fragments from Trench B, Trench D and to a lesser extent, Trench E, with some overlap (Figure 8.8). Different samples recovered from the same contexts also grouped together. There was no clear pattern to distinguish fragments recovered from internal compared with external contexts, however, at the time of use, it is likely all the plasters were constructed internally, and therefore those recovered from external contexts probably represent secondary deposition. As discussed above, the similarities between samples from the same trench compared to other trenches could suggest different uses of space, or different plaster manufacture technologies employed by different households in different areas on site. Geoarchaeological investigations into mudbricks at Çatalhöyük suggested the manufacturing process, rather than the raw material selection, has a greater influence over the compositional variation of mudbricks (Love 2012). Differences between trenches could also represent temporal changes, as the chronology of the site is not a high enough resolution to be certain if some areas were occupied contemporaneously or represent the shifting of the settlement over time, perhaps as some areas filled up with refuse. It is also possible that the differences in elemental composition which create groupings based on the PCA analysis reflect localised burial conditions and post depositional changes.

8.5.3 Plasters at Abu Hureyra in a regional context

Livestock dung has been identified as a component of the built environment in ethnographic studies of different regions around the world, in floors, walls and roofing material (Shahack-Gross et al., 2003; Zapata et al., 2003; Tsartsidou et al., 2008; Portillo et al., 2012; 2014; 2017a; Berna, 2017; Gur-Arieh et al., 2019). Faecal spherulites have also been identified in some of the floor plasters from PPNB (late 8th to early 7th millennium cal. BC) Tell Seker al-Aheimar, located in the Upper Khabur region, Syria, which were also made from gypsum plaster (Portillo et al., 2014, p. 108). However, the numbers of spherulites identified in different floor samples are consistently lower (0 to 0.061 million per gram of sediment) than the numbers identified in this study (Portillo et al., 2014, p. 111, table 3). The numbers of spherulites identified in the plaster fragments analysed in this study are significantly lower than have been identified in archaeological compacted dung in conditions favourable to spherulite preservation which have been observed up to 76.1 million in a study following the same quantitative approach (Portillo et al., 2019). Gypsum floor plasters were common at the contemporary Neolithic site of Bouqras (Van Zeist and Waterbolk-Van-Rooijen, 1985), however, it is currently not known whether dung could have similarly been incorporated into the built environment and highlights the need for assessment of dung presence on a wider scale to clarify shared practices or site-specific choices between settlements in the same region. Gypsum or lime were also used to coat early white ware vessels at Sabi Abyad (Akkermans et al., 2006), however, the focus of research on secondary animal products has been on the identification of dairy and meat through lipid analysis of pottery, with no published considerations of dung as another potentially important secondary animal product.

Dung was an integral part of the built environment at Aşıklı Höyük and it has been suggested that the build-up of dung between buildings encouraged the use of dung in construction, whereby raw waste was recycled to be used as a binder (Stiner and Kuhn, 2016). At Abu Hureyra, the zooarchaeological record shows an increased reliance on domestic animals between periods 2A and 2B, and therefore the use of dung as a construction material could also have been in part a response to manage waste build up. At the same time, as the settlement size of Abu Hureyra increased, an increased need for dung could have driven increased management of animals bred and kept on or near the site to provide a renewable and easily accessible source of food and secondary products, including dung as a raw material which was already in use. The latter provides an example of niche construction whereby animal management and domestication at Abu Hureyra could have been accelerated by human requirement for secondary animal products. A major challenge in the archaeological record is to ascertain whether dung was recycled into the built environment as a response to the increasing build-up of waste, or whether it was selected as a material of choice for its many favourable properties such as water resistance (Henry and Therrien, 2018), as both are likely to have been factors.

8.5.4 Interpreting plant-use from phytoliths in dung and plaster

High concentrations of spherulites in association with high proportions of grass derived phytoliths have been used in previous studies to imply a grass rich, managed diet (Yeomans et al., 2021). On the basis that a high proportion of the phytoliths present in this study are dung derived, this assemblage also suggests a grass rich diet, as monocot derived phytoliths dominate all plaster fragment phytolith assemblages (Figure 8.5a). However, monocots produce up to 80% more phytoliths compared with dicots (Albert et al., 2003), and therefore dicots are often under-represented in the phytolith record. The consistent presence of phytoliths from monocot inflorescences could suggest foddering practices based on agricultural by products and waste. Inflorescence phytoliths (elongate dendritic, papillae) are present in lower proportions than phytoliths from the leaves/stems, however, elongate dendritic morphotypes can be susceptible to dissolution, and are therefore sometimes under-represented in archaeological phytolith assemblages (Cabanes et al., 2011). The consistent presence of dicot wood/bark may represent material integrated into the plaster matrix through the heating process and is consistent with the charred flecks visible macroscopically. The dicot leaves present in low proportions could therefore represent contamination from the wood used as fuel to heat the gypsum, as wood/bark phytoliths can be contaminated by up to as much as 49% with monocots and dicot leaves (Tsartsidou et al., 2007, p. 1270). However, it is also possible that some of the dicot leaves entered the plaster matrix through the animal dung, reflecting the browsing habits of herded animals, or foddering during winter (Halstead et al., 1998).

Increasingly, new archaeological investigations in SW Asia highlight that culinary choices are not entirely dictated by the local environment and resource base, but culture and human selection likely also played a significant role (Kabukcu et al., 2021) and a move away from viewing food as an entirely economic entity. Plaster technology is also intrinsically entangled with culture and function, used in a variety of different ways as it became widespread in SW Asia as the Neolithic developed (Clarke, 2012). At Çatalhöyük, for example, variations in mudbrick composition have been argued to reflect differences in recipes between households and thereby demonstrating human agency and selection at a house level (Love, 2012). This study contributes to the extensive body of data on the diversity of Neolithic plaster practices in SW Asia, and further demonstrates that the choices made by communities were not tied exclusively to their local environmental resource base, as practices differ between sites in a relatively small geographic region. On the other hand, shared practices across vast geographical zones attest the interconnectedness of Neolithic communities across SW Asia. Like the use of plants during the Neolithic, plaster manufacture practices likely reflected community preferences and traditions at a site-specific level.

8.6 Conclusion and future directions

This study has demonstrated the application of integrated microfossil and geochemical analyses to provide new information about floor plaster construction at PPNB Abu Hureyra. The high concentrations of faecal spherulites identified in all plaster fragments suggest ruminant dung was a significant component of PPNB gypsum floor plasters. Dung was therefore likely a major pathway for the abundant plant material within the plaster matrix, identified in this study through phytolith analysis. Phytoliths identified in the plaster, if dung derived, could represent the diet of managed animals, which provides a novel proxy through which to better understand the development of human-animal relationships as utilisation of domesticates increased in a shift away from wild animals during the PPNB. Dung may have been easier to collect when animals were penned on or close to the site and the diversity of vegetation types identified in the phytolith analysis indicate a mixed foddering regime.

The elemental composition of all the gypsum floor plaster fragments analysed in this study were relatively consistent. However, further data exploration of the geochemical data though PCA analysis suggested closer relationships between some samples recovered from the same trenches and to a lesser extent the same periods of occupation. All techniques were applied to both the plaster matrix (at the base) and the surface of each fragment to assess whether construction material could be differentiated from possible activity residues. The elemental composition, concentration of spherulites and phytolith assemblages tended to be consistent with one another on the base and surface, and therefore it was concluded that the material primarily related to the plaster make-up, which could reflect variations in recipes between households, as suggested for mudbrick composition at Çatalhöyük (Love, 2012).

This study provides the foundations for further analytical work to better understand the development of plaster manufacture practices through techniques which are relatively time and resource efficient. Phytolith and spherulite analysis on different plaster types and materials, such as white ware vessels, plasters with reed impressions and wall plasters will provide further information about the range of resources utilised for construction, while the analysis of a larger set of floor plaster fragments will enable a more reliable interpretation of changes and sustainable practices between spaces and over time. The black amorphous material characterised as "carbonised material" and bitumen are similar at the macro scale and elementally. Further work, including SEM microscopy will help to clarify the nature of these materials.

Plaster manufacture becomes widespread across SW Asia during the Neolithic, however, practices and manufacturing techniques are diverse, even within a fairly constrained geographical region. The apparent preference for gypsum plaster tempered with dung at Abu Hureyra was most likely not solely about resource availability, as both gypsum and carbonates were locally available, or exclusively an economic decision, but reflects human selection, choices and possibly site-specific traditions, passed on through generations.

Chapter 9. The detection of faecal biomarkers by GC-MS

This chapter firstly presents and evaluates the results of the GC-MS analyses conducted on sediments and plasters to detect faecal biomarkers. The results are then integrated with other analyses conducted as part of this research and discussed with reference to other studies which are similar in terms of themes and methodological approaches. The GC-MS analysis had two key aims. Firstly, to address Aim 2, to explore possible depositional pathways of plant remains by identifying if dung is present, particularly in deposits where faecal spherulites were not identified. Dung could be present in deposits where faecal spherulites were not identified either because of preservation conditions (e.g. too acidic, see section 2.3.3), or because dung had been deposited by a human or animal which produces low or no spherulites (Canti, 1999). The second aim of the GC-MS analysis was to compare and evaluate the two methods applied in this research for identifying dung; the identification of faecal spherulites (Chapters 5, 6 and 7) and the identification of faecal biomarkers by GC-MS analysis (this chapter) to develop the most effective methodological approaches for identifying depositional pathways in archival environmental, archaeological and archaeobotanical material.

9.1 Results of the GC-MS analysis

Virtually all of the forty-eight samples analysed contained small quantities of cholesterol, a compound often associated with carnivores (Shillito et al., 2020). However, cholesterol rarely survives in archaeological samples, and when present is often with squalene, which is a steroidal precursor associated with human skin lipids (Whelton et al., 2021, pp. 2, 3). A “blank” sample was prepared and analysed with each set of samples (six batches total, section 4.2.6), and these too contained similar peaks of cholesterol. For this reason, the cholesterol present is deemed most likely to be related to contamination during sample preparation or handling of the material prior to analysis, and therefore, only samples which also contain other faecal associated compounds were further analysed and included in this chapter.

Nineteen out of the forty-eight samples analysed contained at least one compound associated with faecal material (excluding cholesterol) (Figure 9.1). The relative percentage of each faecal containing compound is provided in Table 9.1. Three ratios were calculated to identify if there was a faecal component in the material (ratio 1, > 0.7 = presence of faecal material; Grimalt et al., 1990), account for diagenetic changes which transform coprostanol to epi-coprostanol (ratio 2; Bull et al., 2002) and identify if the faeces most likely originated from an omnivore or a ruminant (ratio 3; >1 = omnivore, < 1 = ruminant) (Chapter 4, Table 4.6, Figure 4.3).

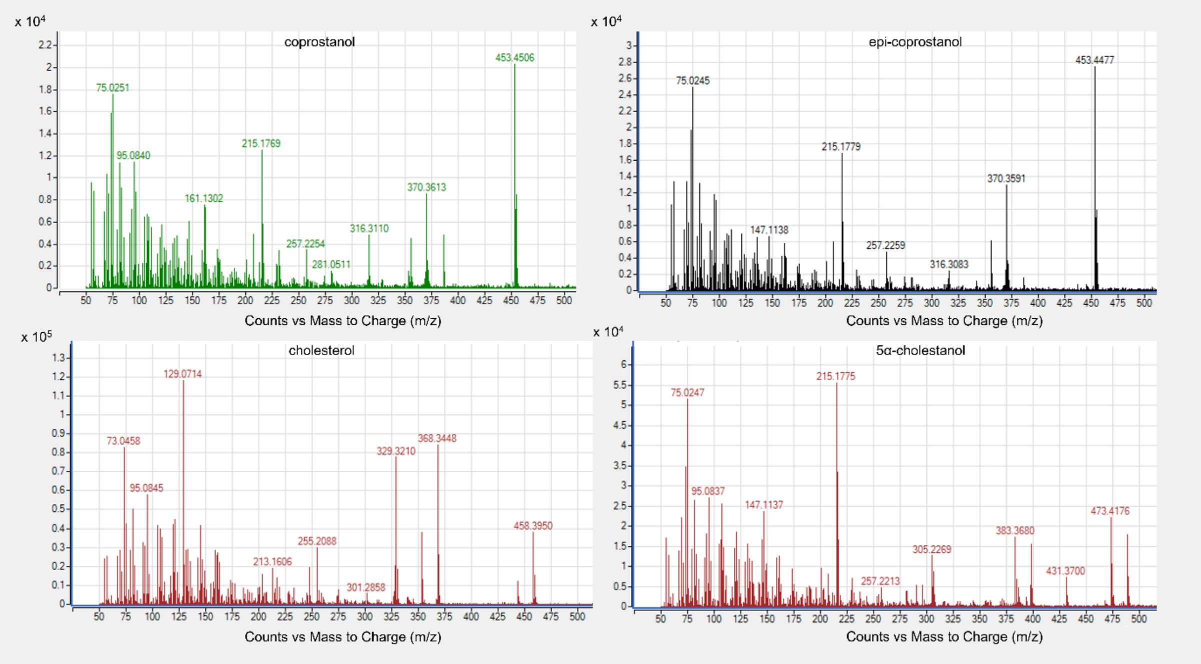


Figure 9.1 Mass spectra of the most common faecal-associated sterol compounds identified in this study

Six of the nineteen samples had a value of more than 0.7 for ratio 1, which confirmed the presence of faecal material (Grimalt et al., 1990; Bull et al., 2002). One sample, E361.10 was indeterminate as ratios 1 and 2 were on the border, with values of 0.67 and 0.73 respectively (Table 8.1). It was not possible to calculate the ratios of the remaining samples because they did not contain some of the compounds required for the ratios, or only contained trace amounts of the compounds. The compounds required to calculate ratio 3 to determine whether faecal matter was omnivore or ruminant derived, were only present in sample E39.33 (Figure 8.2). The value of 0.51 (less than 1) for ratio 3 in sample E39.33 is indicative of a ruminant origin.

Table 9.1 Relative abundance (%) of compounds associated with faecal material. T = trace amount of compound was present. Ratios indicative of a faecal origin of material are highlighted in green.

Sample ID	Period	Phase	Material type	Space	No. spherulites / gram material	Coprostanol	Epicoprostanol	Cholesterol	5α-cholestanol	5β-Campestanol	Campesterol	5β-Stigmastanol	Epi-5β-Stigmastanol	5α-Campestanol	Sitosterol	5α-Stigmastanol	Ratio 1	Ratio 2	Ratio 3
E55.31	1	1	Sediment	F - P - O	3200	0	T	T	0	0	0	0	0	0	0	0			
E402.14	1	3	Sediment	F - PH - O	3200	0	T	0	0	0	0	0	0	0	0	0			
D57.75	2A	4	Sediment	E - O	20,000	T	T	T	T	0	0	0	0	0	0	0			
D14.28	2A	4	Sediment	F - P - O	0	52.1	10.6	37.3	0	0	0	0	0	0	0	0	1.00	1.00	
E338.146	2B	5	Sediment	I - O	18,000	0	T	100	0	0	0	0	0	0	0	0			
E361.10	2B	5	Sediment	E - O	130,000	15.6	5.6	71	7.8	0	0	0	0	0	0	0	0.67	0.73	
E344.143	2B	5	Sediment	I - O	36,000	0	0	100	T	0	0	0	0	0	0	0			
E39.33	2B	5	Sediment	E - F - H	64,000	6.9	0.9	22	0.8	2.8	6.8	15.2	0	8.7	29.5	6.7	0.95	0.95	0.51
E329.123	2B	5	Sediment	E - O	3400	0	0	100	0	0	0	0	0	0	0	0			
E231.71	2B	6	Sediment	I - O	20,000	5.3	T	92.9	1.8	0	0	0	0	0	0	0	0.75	0.76	
E30.17	2B	6	Sediment	E - O	160,000	7.2	T	92.8	0	0	0	0	0	0	0	0	1.00	1.00	
E268.79	2B	6	Sediment	E - O	0	0	T	100	T	0	0	0	0	0	0	0			
E19.4	2B	7	Sediment	E - O	58,000	T	T	100	T	0	0	0	0	0	0	0	1.00	1.00	
E18.3	2B	7	Sediment	E - O	41,000	43.8	7.6	48.6	0	0	0	0	0	0	0	0	1.00	1.00	
D116.34	2A	3	Plaster	E - S	409,000	0	0	76.0	24.0	0	0	0	0	0	0	0	0.00	0.00	
D48.65	2A	4	Plaster	E	957,000	5.4	3.5	87.2	3.9	0	0	0	0	0	0	0	0.58	0.69	
D44.29	2A	4	Plaster	E - S	Present	0	T	100	0	0	0	0	0	0	0	0			
B34.20	2C	10	Plaster	E - F - P	1,002,000	0	0	93	7	0	0	0	0	0	0	0	0.00	0.00	
B38.23	2C	10	Plaster	I	Present	9.8	T	77.8	12.4	0	0	0	0	0	0	0	0.44	0.44	

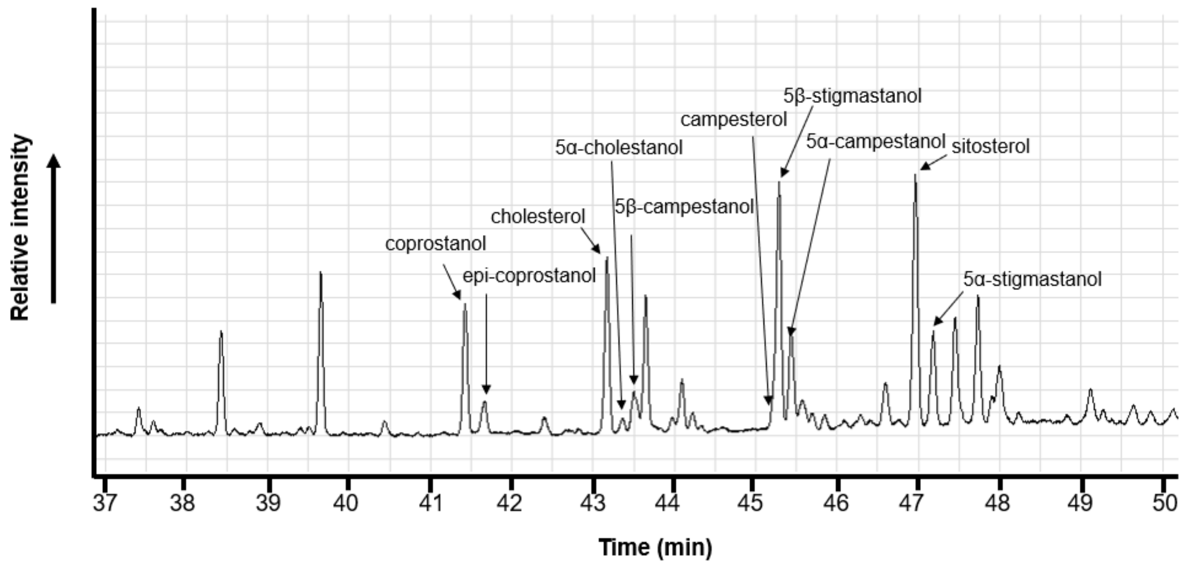


Figure 9.2 Annotated partial GC-MS profile of sterols in hearth base (E39.33)

Based on the presence of coprostanol and epi-coprostanol, four samples were selected for further bile acid analysis; D14.28, E361.10, E18.3 (sediments) and D48.65 (plaster). Seventeen out of nineteen of the samples which had at least one faecal-associated compound identified by GC-MS, also contained faecal spherulites, except for D14.28 and E268.79, in which no faecal spherulites were identified (Table 8.1). In E268.79, occupation residues from an external activity area in Trench

E, only trace amounts of epi-coprostanol and 5α-cholestanol were identified, as well as cholesterol, which, as discussed above is present in most of the samples and not necessarily indicative of a faecal component. In sample D14.28, there are more significant quantities of coprostanol, epi-coprostanol and cholesterol, which, combined with the absence of spherulites, could be indicative of a human faecal origin, and therefore the bile acids were also analysed from this sample.

Four out of six of the samples which had ratio 1 values of more than 0.7, indicating a faecal component, are from the period 2B phase of Trench E, and a further one sample, E231.71 came from a building in Trench E, period 6. The sample which has a borderline value for ratio 1 and 2, E361.10, is also from an external activity area between buildings in Trench E, and also had the highest number of spherulites recorded in any of the sediments analysed in this study (130,000 spherulites per gram of sediment, Table 8.1, Chapter 7). Therefore, although not demonstrated conclusively by the GC-MS analysis in isolation, combined with the faecal spherulite results, this sample likely has a faecal component. Lithocholic acid (LCA) and deoxycholic acid (DCA) were present in sample E361.10, which combined with the presence of coprostanol and epi-coprostanol are indicative of a human faecal origin. Bile acids indicative of human, omnivore or ruminant faeces were not identified in samples D14.68, D48.65 and E18.3 which were also selected for bile acid analysis. In the sample from the hearth base, E39.33, DCA was also identified which is consistent with the sterol ratios which are indicative of a ruminant origin (Prost et al., 2017; Shillito et al., 2020).

9.2 GC-MS analysis: interpretation and discussion of the results

9.2.1 Sediment and hearth samples

Half of the occupation residues which contained at least one faecal compound (n = 8) are from external areas of Trench E, period 2B, four of which have a ratio 1 value which confirms a faecal component. This area was identified as possibly having relatively higher quantities of dung based on the presence of faecal spherulites and other contextual information such as a trodden route between buildings, interpreted as having been formed through routinely herding animals through this part of the site (Moore et al., 2000, p. 235, fig. 8.54) (Chapter 7). The low concentrations and inconclusive identification of dung or dung associated elements from these samples could be because of the conditions required to preserve sterols. High temperature burning can cause the degradation of organic compounds including faecal sterols, even in moderately low temperatures from 400°C (Reber et al., 2019; Matthews et al., 2020, p. 277). Gypsum rock is usually heated to temperatures between 150°C and 400°C to form plaster, though calcination at higher temperatures creates a harder plaster (Gourdin and Kingery, 1975, p. 135), which may have been preferable for floor plaster construction. Analysis of the faecal spherulites included in the plaster matrix identified relatively high proportions of “darkened” spherulites, which form in burning conditions usually exceeding 500°C (Canti and Nicosia, 2018; Portillo et al., 2020b), and comprised between 0.6 and 24.7% of the spherulites identified (Chapter 8, Table 8.2). As discussed in Chapter 8, the proportions of darkened spherulites are consistent with burning temperatures of between 400°C and 600°C. Therefore, whether the animal dung was burned prior to inclusion in the plaster matrix, or within the plaster, the temperatures indicated by the proportions of darkened spherulites and also presence of melted phytoliths are likely to have destroyed the faecal sterols.

Sample E39.33 was recovered from the base of a hearth and contained a relatively high number of spherulites (64,000 per gram of sediment) compared with other archaeological sediments analysed in this study, and also faecal sterols which are indicative of ruminant dung (Table 8.1, Figure 8.2). The presence of faecal biomarkers in sample E39.33 is significant as it demonstrates ruminant dung used as a fuel. Interestingly, a high proportion of the spherulites present in E39.33 were darkened (37%, Chapter 6, Table 6.2). Spherulites start to darken under burning conditions exceeding 500° (Canti and Nicosia, 2018). Based on experimental dung burning and quantification of faecal spherulites, the high proportions of 37% darkened spherulites is mostly likely to occur in burning conditions between 550° and 700° C (Portillo et al., 2020b, p. 10, table 3). Herbivore dung has also been identified in fire spots and burnt samples from the Neolithic site of Bestansur in Iraqi Kurdistan (Elliott et al., 2020, p. 391). Although faecal sterols are degraded by high burning temperatures, the survival of sterols in the hearth base form Abu Hureyra and multiple samples from Bestansur, demonstrate the potential of this technique to provide new information about fuel selection and wider resource management practices.

Hearth base sample E39.22 also had the highest number of phytoliths per gram of sediment of all of the archaeological material analysed in this study (7 million per gram of sediment), which is consistent with other contemporary archaeological dung deposits (Portillo et al., 2019c, p. 101106, table 3). Phytolith concentrations tend to be relatively high in archaeological dung deposits, particularly in herbivores, compared with omnivores, due to the high plant content of their diet (Elliott et al., 2020, p. 391). Within E39.33, there was a higher proportion of dicot leaves compared with dicot wood/bark which made up a relatively low proportion of the overall phytolith assemblage. Of the grass short cells identified in the phytolith assemblage in E39.33, there was a relatively higher proportion of bilobates compared with other sediment samples analysed for phytoliths. Some of the bilobates were trapezoidal in cross section, which is consistent with morphologies derived from Pooideae grasses, further testified by the presence of bilobates and rondels identified with elongate dendritic phytoliths in anatomical connection (Figure 7.5e). A relatively high proportion of the phytolith assemblage in E39.33 were multicelled (defined in this study as 3 or more phytoliths in anatomical connection). Reeds often have distinctive multicellular structures, through which they can be identified in the archaeological record (Ryan, 2011; Ramsey et al., 2016). However, despite the excellent preservation of phytoliths and abundance of multicelled structures, none recorded in E39.33 were diagnostic of reeds.

Several studies have inferred that the phytoliths identified represent foddering practices and animal diet in deposits where high numbers of spherulites have been identified (Elliott et al., 2020; Matthews et al., 2020; Yeomans et al., 2021). Based on the positive identification of ruminant dung through faecal spherulite analysis and GC-MS, it is possible that most of the phytoliths present in E39.33 are derived from animal dung (sheep, goat or cow). The dominance of monocotyledonous plants which include phytoliths derived from the stems, leaves and inflorescences could be indicative of animals being herded on the steppe grasslands. Alternatively, the monocot phytoliths may represent processing waste fed to the animals as fodder, which could have included the stems and leaves of cereals and husks from smaller weed crops accidentally collected with the harvest. The phytoliths which are derived from dicot leaves (e.g. dicot hair bases, tracheary elements, Figures 6.5a and 6.5b), could represent the browsing habits of animals herded in the environment surrounding Abu Hureyra, where herbaceous dicot shrubs and trees would have grown on the grassy steppe, or foddering practices (Halstead et al., 1998).

The presence of faecal spherulites in over half of the sediment samples analysed in this study (Table 7.2, Figure 7.6.) demonstrate a faecal component in many of the occupation residues, particularly those from external areas. Therefore, dung could be a potential depositional pathway for some of the plant materials identified in the assemblage. The survival of sterols in hearth and fire spot samples in previous studies (Elliott et al., 2020) and this study, demonstrate the survival of sterols under some burning conditions. The low quantities or complete absence of faecal associated compounds in most of the ashy and charred sediments, combined with relatively low concentrations of faecal spherulites, are not necessarily a preservation issue, but more likely because of low input of dung in the mixed ashy occupation residues. It is therefore appropriate to consider dung as a potential depositional pathway for many of the occupation residues analysed, however, for most of the samples, there is little evidence for the significant input of faecal material, and the samples can certainly not be interpreted as dung deposits.

9.2.2 Gypsum floor plasters

In all floor plaster samples analysed, high concentrations of faecal spherulites were identified and it is clear from the analysis reported in Chapter 7, that ruminant dung was a significant component of floor plasters. Five of the floor plaster fragments analysed in this study contained faecal associated compounds, however, none in significant quantities or ratios to suggest the presence of any faecal material in the plasters (Table 8.1). One possible reason for this could be that the sterols did not survive the potential firing temperatures of 400°C+ which may be been used to manufacture the gypsum plasters (Gourdin and Kingery, 1975). This indicates that the material in the ashy, charred occupation sediments, where the sterol analysis indicates a dung component in some samples, was charred in a different way compared with the plasters. Faecal spherulites start to darken when burned, usually at temperatures of more than 550°C (Canti and Nicosia, 2018).

Based on experimental burning of modern dung samples, and as discussed in Chapters 5 and 7, sections 7.5.3 and 5.5.1), the proportions of darkened spherulites are indicative of burning temperatures between 550°C and 700°C, at which point, faecal sterols may be destroyed. It is interesting, however that in the ashy sediment sample from the hearth base (E39.33), there is an even higher proportion of darkened spherulites than identified in any of the plasters, yet faecal sterols are also preserved in this samples. This could be because during the plaster manufacture, all of the material was heated to a high temperature. In contrast, the ashy material at the base of the hearth could represent accumulation over multiple fire events, during which different the input of different dung could have been subject to different burning temperatures. The formation, taphonomy and preservation of darkened spherulites is still relatively unknown and can vary significantly depending on the species and diet of the animal. There is also variation in proportions of darkened spherulites between similar samples derived from the same species. Therefore, while the presence and proportion of darkened spherulites provides a relatively good indicator of burning range, it is not sufficient in isolation to identify more specific burning conditions.

9.2.3 Sampling strategies and selection of material for GC-MS analysis

Fourteen out of twenty-four sediment occupation residue samples and five out of twenty-two gypsum floor plaster fragments contained faecal associated sterols, though only seven out of forty-eight of the samples analysed were shown to have a probable faecal component. The proportion of positively identified faecal biomarkers is generally much higher at other Neolithic sites in SW Asia, including at Çatalhöyük, Anatolia, Bestansur, Jani, Sheikh e Abad and Shimshara which have followed a similar procedure (Bull et al., 2005; Shillito et al., 2011a; 2013a; 2013c; Elliott et al., 2020; Portillo and Matthews, 2020). This can be explained by the highly-targeted selection of material to be analysed by GC-MS, whereby suspected coprolitic material had already been identified either during excavation or subsequently through micromorphological analysis. For example, at Bestansur, four out of eight spot samples were shown to be of a human faecal origin (Matthews et al., 2020, p. 275). This study has shown that in the absence of other evidence or techniques to suggest the presence of dung, spherulite analysis provides a rapid and cost-effective screening method, as only two of the samples where faecal associated sterols or bile acids were identified contained no spherulites. As discussed above, this could be because the dung was produced by a human or pig which do not tend to produce large quantities of spherulites, if any at all (Canti, 1997; 1999). Therefore, a limitation of using faecal spherulites as a screening method for possible dung derived sediments is that it will likely only account for ruminant dung.

9.3 Summary and conclusion of GC-MS analysis and future directions

Overall, the detection of faecal biomarkers by GC-MS was low in both sediments and plaster floor samples in this study. However, the positive identification of faecal-associated sterols and bile acids in nineteen out of forty-eight of the samples analysed demonstrates the potential of GC-MS analysis on this material. This study has provided a unique opportunity to identify the potential presence of dung on a large sample set, which includes material both with, and without, a suspected faecal component. The probable presence of faecal material in two samples where no spherulites were identified, highlights the value of multi-proxy approaches to identify dung. However, GC-MS is a relatively expensive and time-consuming method which requires highly specialist equipment and laboratory conditions. The relatively low returns of faecal biomarkers identified in this study have demonstrated that it is not appropriate to analyse large numbers of samples without a more targeted sampling approach and rationale. Faecal spherulites have been shown to provide an excellent indicator of ruminant dung, however, further research is required to develop techniques to screen for faeces from low or non-spherulite producing animals.

Chapter 10 Integrated contextual interpretation and discussion

This chapter integrates and synthesises the results reported on in Chapter 5 to 9 to provide new insights and perspectives into specific areas, spaces and contexts at Abu Hureyra to inform on how people were managing selected plant and animal resources (Aim 1). The aim of integrating the data generated in this research with contextual information is to provide higher resolution insights into uses of different plant types (Aims 1, 3), to explore their potential depositional pathways into the archaeological record in specific areas (Aim 2) and demonstrate the value of environmental archaeological archives through the methodological approaches applied in this study (Aim 5). This informs on activities, lifeways and technological choices made by people to compare with other contemporary sites and assess the extent to which changes in economy were regional, local or site specific to provide new evidence about the different pathways of agricultural development in SW Asia (Cauvin, 2000; Asouti, 2006; Fuller et al., 2010; 2011; 2012; Ibáñez et al., 2018). The chapter is structured in four sections to examine each of the key trenches, periods and phases analysed in this research from broadly in chronological order from earliest to latest; Trench D, period 2A, phases 3 and 4, Trench G, period 2B, phases 1-3, Trench E, period 2B, phases 1-7 and Trench B, period 2A, phases 5 and 7, Trench B period 2C. The spatial and chronological relationships between the phases in each trench are provided in more detail in Chapter 3 (Table 3.1: dates and main attributes of each trench and phase included in this research; Figure 3.5: schematic diagram showing relationship between trench phases) and Chapter 4 (Figure 4.1; contour plans of Abu Hureyra mound and trench locations, Figure 4.2; plans of key areas discussed in this research).

10.1 Abu Hureyra 2A (8600-7400 cal. BC): Trench D, phases 3 and 4

Trench D was excavated down the western slope of the Abu Hureyra mound (Figure 4.1), initially to ascertain the extent of the settlement although much of the upper layer had eroded away (Moore et al., 2000, pp. 209, 218). Trench D exposed part of a building in phase 3, with two burials underneath the initial flooring layer, which had then been filled up with mudbrick debris, on top of which a series of clay and plaster floors had been laid (Moore et al., 2000, p. 213). The external area outside of the phase 3 house had been used intensively and included a substantial accumulation of debris, including flint and bone, layer of ash and patches of burning. Trench D, phase 4 consists predominately of a large open area, between two buildings which extended beyond the limit of the trench (Figure 4.3, Figure 10.1). The deposits in the large areas were ashy and burnt, with high quantities of flint and bone, suggesting the intensive use of this area for domestic activities such as cooking (Moore et al., 2000, p. 218).

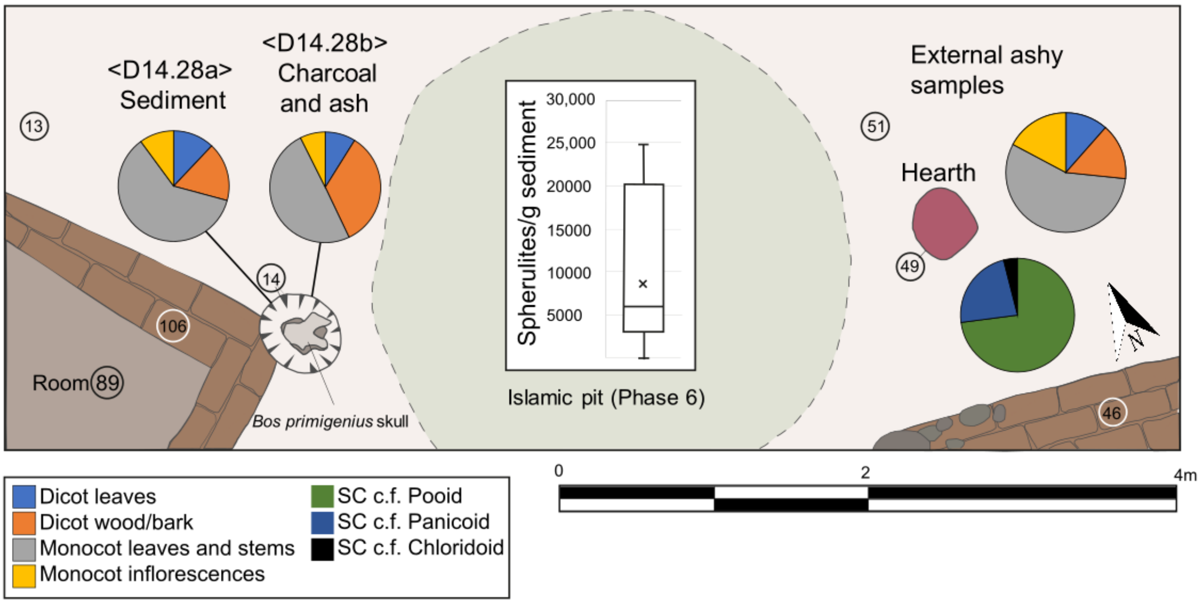


Figure 10.1 Annotated plan of Trench D, phase 4, showing the proportion of different phytolith types and box plot showing estimated number of spherulites per gram sediment for the external ashy samples. Adapted from Moore et al. 2000, p. 216, fig. 8.33

In total, thirteen occupation residue sediment samples were analysed for phytoliths and spherulites (Chapter 7, Appendix 4) from Trench D, phase 4, eleven of which were also analysed by GC-MS to detect dung biomarkers (Chapter 9). Eleven occupation residues sediment samples, representing successive layers of occupation were selected from an external open area (section 4.1.1, Figure 10.1) to examine the nature and range of domestic activities, represented by the numerous hearths, ash patches, and abundant flint and bone identified during excavation (Chapter 6) (Moore et al., 2000, p. 218). Phytoliths and spherulites were also analysed from six samples of gypsum floor plaster, from the surface and plaster matrix of three fragments, from phases 3 and 4, one of which was identified *in situ* and the other two from secondary deposits to study construction practices (Chapter 8, Figure 8.1).

The following section (10.1.1) discusses and evaluates the evidence for dung in Trench D, to inform on the use of secondary animal products in Period 2A (8600-7300 cal. BC) and the most likely scenarios through which it was deposited in the archaeological record. This is important to be able to assess if there was a change in the use of dung in later in Period 2B (7300-6200 cal. BC), represented in this study by Trenches G and E (sections 10.3 and 10.4 of this chapter).

10.1.1 Evidence for dung in Trench D

Faecal spherulites are present in nine out of eleven of the occupation residue samples analysed from the open external activity area in consistently low numbers, with estimated concentrations of 3300-25,000 spherulites per gram of sediment. Seven of the occupation residue sediment samples (one from the pit fill B14.28 and six from the external activity area) were also analysed by GC-MS to identify if faecal biomarkers were present. One of the external, ashy, occupation residue samples, D57.75 contained trace amounts of faecal associated sterols (Chapter 9, Table 9.1), though not in sufficient quantities to indicate a faecal component (Grimalt et al., 1990; Bull et al., 2002).

In D14.28a, occupation residues in a pit containing a *Bos* skull, coprostanol, epi-coprostanol and cholesterol were all identified in quantities which suggested a faecal component (Chapter 9, Table 9.1). All of the occupation residues recovered from the external activity area in Trench D contained some charcoal and ashy material, primarily having been collected for radiocarbon dating, as well as some of the surrounding sediment, in contrast to D14.28a which was lighter brown and not visibly burnt (Appendix 1). It is possible therefore that the absence of faecal sterols, despite the presence of faecal spherulites in material from the external area, relates primarily to the preservation conditions required for each proxy, as organic compounds are usually destroyed in temperatures exceeding 400°C (Reber et al., 2019). The presence of spherulites could therefore indicate dung being used as part of a mixed dung-wood fuel regime, the presence of animals, or the presence of animal waste being dumped in this area.

Faecal spherulites were abundant in all of the Trench D plaster fragments analysed, and phytoliths were also preserved in relatively good concentrations in the plaster fragments (Chapter 8). Given the high quantities of spherulites present in the plaster fragments, it is also possible that the Trench D phase 4 external area was used for plaster preparation, perhaps collecting, burning or mixing the dung with the fired gypsum. Although, considering the relatively low numbers of spherulites identified in the ashy external deposits (Figures 7.6, 10.1), this could have also occurred elsewhere.

In terms of the types of phytoliths present there are no consistent patterns between those where spherulites are present compared with those where they are absent (Figures 7.3, 7.4, 7.5). In addition, the low concentrations of faecal spherulites suggest that dung is probably a relatively minor component of the external area, and that while some of the phytoliths may be dung derived, they are also likely deposited by other domestic activities, such as fuel and cooking.

10.1.2 Plant-use and activities in Trench D

All of the Trench D, phase 4 samples were dominated by monocot phytoliths from both the leaves and stems, but also had significant quantities of dicot wood/bark and dicot leaves (9-29% of the total phytolith assemblage, Figure 7.4a, Figure 10.1). The preservation of elongate dendritic multi-cells was not sufficient to be able to identify any of them to a higher taxonomic resolution beyond, grass husk. However, the short cell phytoliths provide some information about the different types of vegetation present (Figure 7.4c). Rondels, usually formed in pooid grasses, bilobates/polylobates which were probably derived from panicoid grasses (Twiss et al., 1969), although some of which may also have been deposited in Pooid grasses (section 2.2.1), were identified in all of the samples, and chloridoid saddles in five of the samples, suggesting the input of different types of grasses which may have represented different activities that took place in the external space of material which was discarded there (Figure 10.1).

Sedge type phytoliths were present in eight of the samples (Figure 7.4a) and all of the Trench D material contained bulliform phytoliths c.f reed (Appendix 4C). The variety of vegetation types and plant parts present may suggest that the open area between buildings was used for a variety of different activities, and the plants likely reflect fuel residues and processing waste. One of the samples analysed for phytoliths, D68, had one of the largest concentrations of gazelle bones, with sufficient variety in the types of bones present to suggest that the entire carcass was processed on site (Legge and Rowley-Conwy, 2000, p. 453). As faecal spherulite concentrations are very low in all of the samples from the phase 4 open space, it is possible that some of the spherulites could have been mixed into the sediment following the decomposition of the parts of the animal which were not used. The variety of plant types present is also a testament to the rich and varied resource base, including steppe grasses, valley bottom plants and park woodland (Figure 3.3).

The charred macrofossil assemblages from Period 2A (Trenches B and D) were composed predominately of small-seeded legumes, mostly Trifolieae such as clovers and medicks, interpreted as arable weeds, along with ~10% cereal grains and low quantities of chaff (de Moulins 2000, pp. 404-406, fig. 12.29). Although categorised as a weed seed of cultivation in Moore et al. (2000), some Trifolieae species are edible, and also used as fuel and medicine (Mithen et al., 2007, p. 445, table 22.1). Trifolieae may also be consumed by animals such as sheep or goat (Dalton and Ryan, 2020b, p. 191) and is grown to the present day as fodder (Charles, 1998, p. 113). Legumes are generally very low phytolith producers (Tsartsidou et al., 2007, p. 1269, fig. 2C) so the presence of many potential arable weeds are unlikely to be represented in the phytolith record, demonstrating the importance of integrated approaches to identify a wider spectrum of plant resources and uses.

10.1.3 Pit containing *Bos primigenius* skull

Samples D14.28a and D14.28b were recovered from an external pit, cut into the corner of a decaying house wall, which contained occupation residues, brick fragments and a *Bos primigenius* skull, placed intentionally upright and covered over, Figure 4.2, Figure 10.1, Figure 10.2, (Moore et al., 2000, p. 218). Phytolith and spherulite analyses were conducted on two samples from the pit fill, D14.28a, which is greyish brown sediment and D14.28b which is darker and ashier with more frequent charcoal inclusions. Both samples were analysed for phytoliths and spherulites. Interestingly, spherulites were identified in a low concentration in the “charcoal” sample, but were absent from the soil sample from D14.28 (Figure 7.6, Appendix 4). This may suggest that the low numbers of spherulites present in Trench D represent dung burnt as part of a mixed wood-dung fuel regime, rather than being a background component of the sediment due to the presence of animals on this part of the site.

Figure 10.2 Photograph of Bos Primigenius *skull placed upright and covered over in a pit (scale 20cm). Adapted from Moore et al. 2000, p. 218, fig. 8.34*

D14.28b (ashy with charcoal inclusions) has a higher proportion of dicot types phytoliths (32%) compared with the "soil" sample where dicots represented 22% of the phytolith assemblage (Figure 10.1). Consistent with this observation, there were higher proportions of phytoliths derived from the wood/bark of trees in the charcoal sample compared with the soil sample (25% compared with 13% of the total assemblage). The proportion of dicot material (22%) in the unburnt samples is relatively high given dicots produce ~80% fewer phytoliths that monocots (Albert et al., 2003; Tsartsidou et al., 2007), and perhaps represents the use of woody plants for other activities as well as burning (e.g. crafts, tools). Interestingly, the proportion of sedge type phytoliths is the same in both sample types (3%). The phytoliths and spherulite concentrations and phytolith morphotypes are similar to those from the surrounding deposits, which is consistent with the excavator's observation that the *Bos primigenius* skull was intentionally covered, likely with material from the surrounding area (Moore et al. 2000, p. 218).

10.2 Trench B: Abu Hureyra 2A, phases 5-8 (8600-7300 cal. BC) and Period 2C, phase 10 (6200-6000 cal. BC)

Trench B was excavated along the north-south axis of the site (Figure 4.1) and represents a particularly significant area of the excavation because it documents sequential occupation spanning periods 2A, 2B and 2C, through 11 phases of building (Figure 3.5, Figure 10.3). The change from reliance on wild gazelle to managed caprine occurred during Trench B, phase 8 (7465-7175 cal. BC at 95.4% probability, Jacobsson, 2017), Figure 10.3, and is therefore particularly significant as it demonstrates that the change occurs over time rather than reflecting use of different spaces (Legge and Rowley-Conwy, 2000, p. 434). During the latest Neolithic phase of Abu Hureyra, period 2C (~6200-6000 cal. BC) a 30cm-1m deep deposit of ashy occupation debris accumulated over the previous constructions. No standing buildings were identified, leading Moore et al. to conclude that buildings were spaced much further apart compared with the previous densely packed buildings identified in periods 2A and 2B (2000, p. 209). Although the layer was highly disturbed, three small shallow pits (1.45m diameter and 1m deep) were identified which had been dug into the surface, where ashy occupation debris, animal bones, charcoal and flint had accumulated (Moore et al., 2000, p. 208, fig. 8.21).

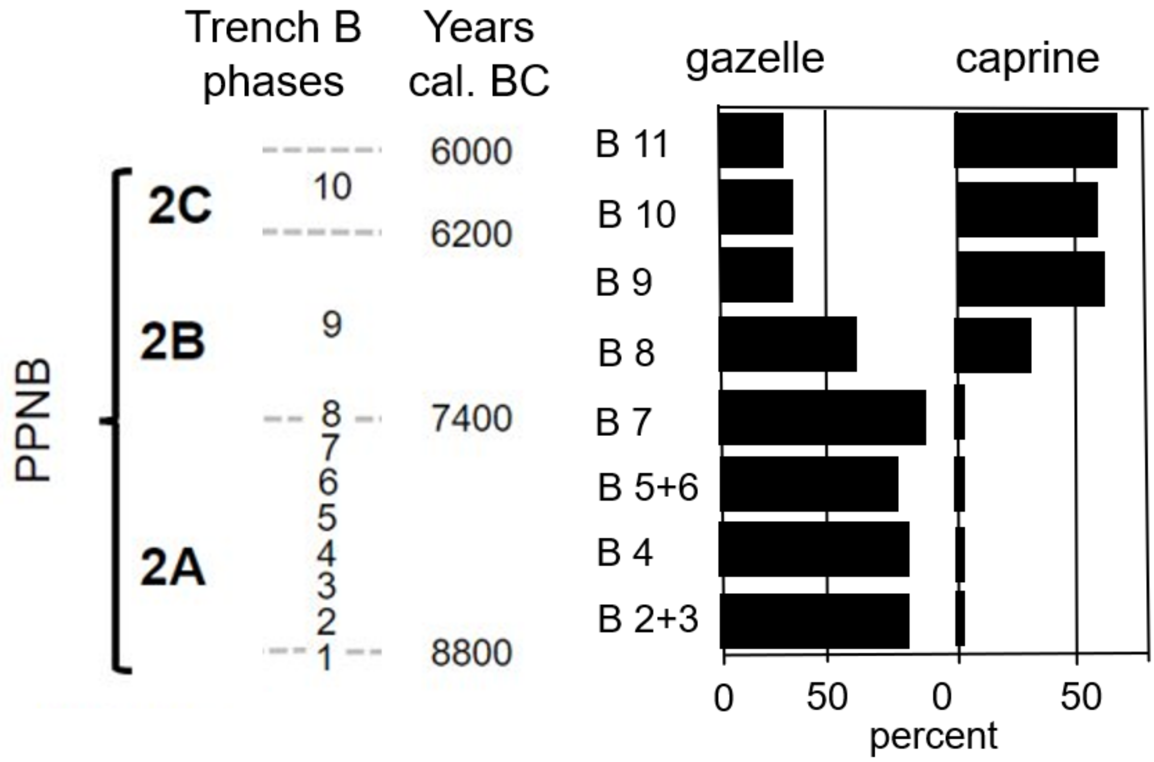


Figure 10.3 Schematic diagram showing Trench B Phases in relation to the zooarchaeological evidence for a change in reliance from gazelle to caprine between Periods 2A and 2B. Adapted from Moore et al. (2000, p 257, *fig. 8.75 and p.432, fig. 13.8)*

Three internal occupation residue sediment samples from two contexts were analysed for phytoliths and spherulites; B203.99, phase 5 and B163.71, and B163.74, phase 7. The Trench B samples are particularly valuable in this study, as charred macro fossils from the same (B163) or very similar levels (B202) have also been analysed and published, providing a context specific opportunity to compare the phytolith and charred macro fossil assemblages from the same (or very closely related) contexts, which provides the opportunity to explore the different plant-types represented by each proxy (Chapter 7, Figure 7.7, section 10.2.1 and 10.2.2).

A fragment of gypsum plaster with red paint and basket impressions (phase 8) and a fragment of white plaster with reed impressions (phase 7) were also analysed for faecal spherulites, elemental composition by pXRF and for the detection of faecal biomarkers by GC-MS (Chapter 8, Chapter 9, Appendix 9, Appendix 10). From period 2C, phase 10, two fragments of floor plaster were analysed for phytoliths and spherulites, but as no buildings were identified in this phase likely represented secondary deposition, perhaps from a Period 2C building outside the extent of the Trench (Chapter 8).

10.2.1 Trench B, phase 5

The only sample analysed from Trench B, phase 5 came from (203) which was a layer of red clay with multiple traces of internal floor surfaces applied over the top of the phase 4 structure (Figure 10.4a, b). A series of black plaster floors were identified in the room during excavation, built on top of layer of red clay and pebbles (Figure 8.2) of which one (197), had a red designed painted, interpreted as a sun, Figure 10.4a (Moore et al., 2000, p. 194). Two samples were also floated and analysed for charred macro-fossils from Trench B, phase 5; (202) is a black plaster floor, overlying the layer of red clay (203) analysed for phytoliths in this study, and (198) is a hearth at the southern end of the room shown in Figure 10.4a (but does not appear in the plan).

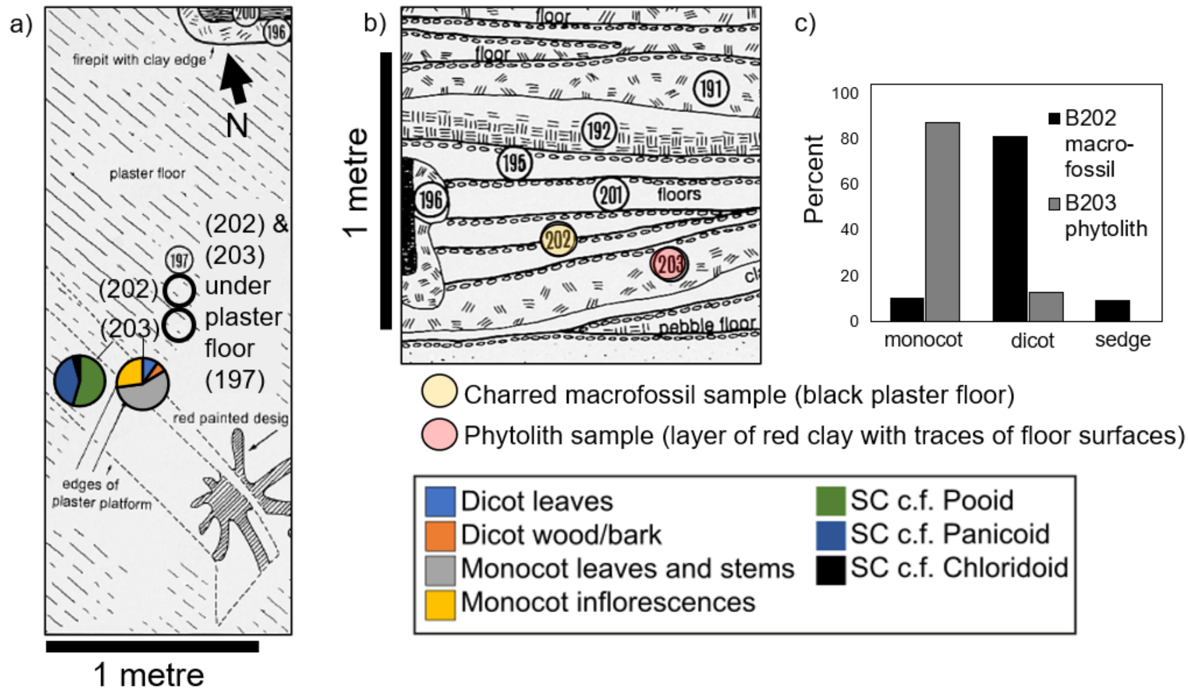


Figure 10.4 a) Plan of excavated Trench B, phase 5 room (adapted from Moore et al. 2000, p. 198, fig. 8.9), and section drawing (adapted from Moore et al. 2000, p. 191, fig. 8.2) showing locations of sample analysed for charred macrofossils (202) and for phytoliths in this study (203) and c) comparison bar chart showing proportions of monocots, dicots and sedges represented in the charred macro-fossil record from B202 and the phytolith record from B203 analysed in this study (B203.99).

The charred plant macrofossil assemblage analysed form Trench B, phase 5 is relatively consistent with the rest of Period 2A, that is, characterised by ~10% domestic cereals and dominated by small-seeded legumes such as clovers and medicks, interpreted as arable weeds, as well as small-seeded grasses (de Moulins, 2000, p. 400-403). The phytolith assemblage in B203.99 is dominated by monocots, mostly derived from the leaves/stems (Figure 7.4, Figure 10.4a). The multi-cell phytoliths also tended to be derived from the stems/leaves of grasses (e.g. elongate psilate), but a low number of elongate dendritics with short cells rondels from grass husks were also identified (Appendix 4B). The most frequently observed grass short cells were rondels, commonly associated with Pooid C3 grasses. There was also a relatively high proportion of bilobates probably formed in Panicoid grasses, based on their relatively short, wide shafts and large straight or semi-rounded lobes (Piperno, 2006, p. 32) in comparison with some other samples where bilobates had a more trapezoidal shape and therefore more closely resembled bilobates derived from Pooids (Fredlund and Tieszen, 1994)(Figure 7.4c for comparison of short cells proportions with other samples, Figure 10.4a for proportion of c.f. Pooid, c.f. Panicoid and c.f. Chloridoid in B203.99). Considering the context is internal and within the phase are multiple layers of floor and occupation residues, the relatively high proportions of bilobates c.f. Panicoid likely represent the remains of matting and/or basketry, as observed at Çatalhöyük (Rosen 2005; Ryan 2011).

It is particularly interesting that no sedges were identified in the phytolith assemblage, since they are present in the macrofossil sample analysed from above. As discussed in Chapter 7, sedges only occur in very low quantities in all samples analysed in this study, possibly due to their relatively fragility compared with other phytolith morphotypes (Cabanes and Shahack Gross, 2015). It could, however, also represent contextual variation, as the phytolith sample (203) represents a layer of red clay, while the charred macrofossil sample is a floor layer which sedges may have been laid on top of for bedding or as a surface covering, as has been shown to be common practice from the Epipalaeolithic (and probably before) across different regions of the world to the to the present day (Sievers, 2013; Ramsey et al., 2017; Robertson and Roy, 2021).

The proportions of monocots, dicots and sedges in the macro-fossil sample (B203) and phytolith sample (B202) are strikingly different, as discussed in section 7.5.1 (Figure 7.7, Figure 10.4c). A key reason for this is that the charred small-seeded legumes which dominate the macro-fossil assemblage are mostly low phytolith producers (Tsartsidou et al., 2007), as are most dicot plants (Albert et al., 2003). On the other hand, most of the phytolith assemblage in B203.99 is made up of parts of the plant, such as monocot leaves/stems and dicot leaves which would not survive charring (Figure 10.4a, Table 2.3).

10.2.2 Plant-use in Trench B, phase 7

Four rooms were identified in the phase 7 buildings, of which room 1 was the most extensively excavated (Figure 4.2). In room 1, an oval niche (65cm high, 55cm wide and 21cm deep), coated with whitewash, was constructed into a wall, also whitewashed, and probably used for storage (Moore et al., 2000, p. 201, fig. 8.14). A large, broken, whiteware vessel, sat on top of a mudbrick and plastered reed box, on the floor of room 1, likely a permanent feature of room 1 box (Moore et al., 2000, pp. 201, 202, fig. 8.12, fig. 8.15).

Two samples were analysed from context 163 which is grey ashy occupation soil and mudbrick collapse which covered the internal walls of the phase 7 building (Moore et al., 2000, p. 202). Both samples appeared very similar macroscopically and were brown (B163.71) and greyish brown (B163.74) both with inclusions of charcoal and some bone, though the charcoal fragments were slightly larger and more frequent in sample B163.71. Despite being sampled from the same deposit and appearing similar in composition macroscopically, both are quite different in terms of the concentration of phytoliths (1,600,000 and 670,000 respectively, Table 7.2) and composition of the phytolith assemblage (Figure 7.4a-c, Appendix 4B, 4C). Faecal spherulites were identified in both samples in similar, very low quantities (estimated 13,000-14,000 per gram of sediment) indicative of a possible faecal component, but not likely a major depositional pathway. Although both samples were dominated by rondels, likely derived from Pooid grasses, B163.74 had a higher proportion of bilobates and polylobates which could be derived from panicoid grasses, often indicative of wetter conditions (Twiss et al., 1969). One elongate multi-cell with short cells (rondels) and stomata, c.f reed leaf was identified (Ryan, 2011), as well as a multi-cell elongate with frequent bilobate short cells c.f. reed leaf (Ramsey et al., 2016, p. 13, fig. 5). Other wetland indicators include sedge phytoliths, diatoms and sponge spicules, which were identified in low numbers in B163.74, but absent in B163.71 (Figure 7.4a, Appendix 4A, Appendix 7B). Most of the multi-cell phytoliths identified in both samples (5% and 6% of the total phytolith assemblage) were elongate dendritics, often in anatomical connection with short cell rondels, likely derived from Pooid grass husks (Twiss et al., 1969; Twiss, 1992), however, there were not sufficient characteristics, for example "wave patterns" or countable pits in the papillae (Rosen, 1992) to further provide a higher taxonomic resolution (e.g. differentiate between wheat, barley or rye).

Most of the charred plant macro remains in Period 2A were characterised by cereal grains (~10%) and high proportions of arable weeds, mostly consisting of small-seeded legumes and small-seeded grasses, discussed in section 10.1.2 (de Moulins 2000, p. 410). In Phase 7 of Trench B, at the end of Period 2A, however, there was an increase in the proportion of domestic cereals, including barley, domestic einkorn, and rachis fragments of free threshing wheat and even higher arable weed-seeds, particularly small-seeded legumes and grasses such as gypsophilia, purslane, *Portulaca olearacia* (de Moulins, 2000, Figure 12.28, p. 400).

There was a particularly high recovery of plant remains from phase 7, and quantities of charcoal indicate a fire had occurred in this space, which was possibly used for storage of partially cleaned grains with an admixture of weed seeds, based on the charred assemblage and storage vessels identified (de Moulins 2000, p. 410). Therefore, the apparent changes in plant represented in the charred macro-fossil record could represent context related variation rather than a shift in plant-use preceding the switch to gazelle.

The relatively high proportions of domestic cereals, as well as small-seeded grasses in the charred macro-fossil record is consistent with the grass husk multi-cells identified in the phytolith assemblages. On the other hand, the wetland phytolith indicators could represent plant material used to make storage vessels and baskets (e.g. Rosen 2005, Ryan 2011), where seeds would not have necessarily been incorporated, and therefore, even if burnt would be less likely to be represented in the charred macrofossil assemblage.

The differences between B163.71 and B163.74 highlight the variation which can exist within a single context when analysing microfossils such as phytoliths. There is an increase in the proportions of domestic cereals, small-seeded legumes and small-seeded grasses, interpreted as weed seeds (92%) in the macrofossil assemblage analysed from Trench B, phase 7 (163) and (164) (de Moulins, 2000, p. 410). The very high quantities of wood charcoal indicate a fire could have occurred, however, no melted phytoliths or darkened spherulites were identified in this study, which, together with the high proportions of charred material, indicates that the fire unlikely exceeded temperatures of c. 500°C. De Moulins (2000) suggests partially cleaned grain was possibly stored in the phase 7 building with an admixture of weeds seeds, which is consistent with the storage vessels and plaster niche in room 1 (Figure 4.2). The apparent increase in domestic cereals towards the end of period 2A, could also be due to could be context related variation (de Moulins 2000, p. 410).

Two samples from a fragment of white plaster with reed impressions (B164.78) which was also from Trench B, phase 7, recovered from the fill of room 1, Figure 4.2 (for section drawing see Moore et al. 2000, p. 191, fig. 8.2) were also analysed to check if spherulites were present to inform on the use of dung in different plaster types. A very low number of faecal like spherulites (n=5) were observed in a subsample of the white plaster taken from within a reed impression (B164.78a) and none were observed in the second subsample taken from the opposite side of the white plaster to the reed impression (Appendix 10a). One fragment of gypsum plaster with basket impressions and red paint, B126.44, (Appendix 9B) from Trench B, phase 8 (Appendix 1, Appendix 10.2) was also analysed for faecal spherulites to assess whether dung was used in its manufacture like the floor and wall plasters. Two subsamples were analysed, one from an area of red paint with basket impressions (B126.44a) and the other from a bit of the plaster which had no colouring or basket impression (B126.44b). As observed on the white plaster with reed impressions (B164.78), a very low number of faecal like spherulites (n=5) were identified in subsample from the red area with basket impressions (B126.44a) but not from the other part of the plaster (B126.44b). Both the white plaster with reed impressions (B164.78) and the gypsum plaster with basket impressions (126.44) were analysed by GC-MS to identify faecal biomarkers, which were not detected in either sample (Appendix 10).

The very low presence of dung spherulites in the reed/basket impressions but absent from the plaster, could represent contamination, but it could also be indicative of dung, or a dung derived product used as an adhesive between the reed and the plaster. Dung is a well-established material used in construction since prehistoric times, to the present day (Reddy, 1998; Boivin, 2000; Matthews, 2005; Karkanas, 2006; Portillo et al., 2014; Berna, 2017; Gur-Arieh et al., 2019), and as demonstrated by the ubiquity of faecal spherulites in floors plasters identified in this study (Chapter 8, Figure 8.4), was well used at Abu Hureyra, and therefore could have also been used as an adhesive to fix reeds to plaster. Further research is required to clarify if this is the case, possibly by identifying if faecal spherulites are present in more of the reed impressions present in Abu Hureyra plaster samples, where they are absent from the plaster matrix.

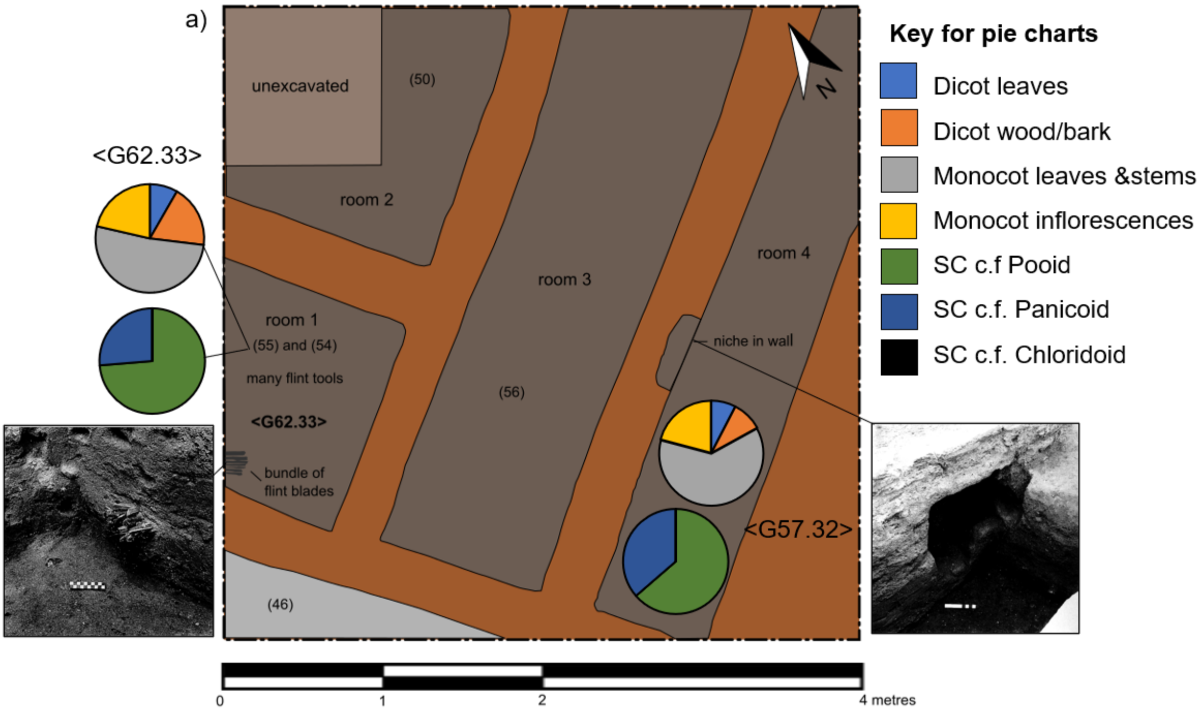
In part, the possible spatial variation between phytolith assemblages, which tend to provide a relatively localised signal of plant-use, over a large context could account for the differences between the charred macro-fossil assemblages and phytolith assemblages from the same contexts which is demonstrated and discussed in Chapter 7 (Figure 7.7). However, the taphonomic differences and preservation conditions required to preserve charred macro-fossils, compared with phytoliths (Chapter 2) would have a significant influence over the types of vegetation which are preserved in the archaeological record.

Faecal spherulites were present in low to moderate quantities in all three internal occupation residue samples from Trench B (13,000 – 48,000 spherulites per gram of sediment). Faecal spherulites were also identified in a fragment of plaster with red paint and basket impressions (17,000 spherulites/g of plaster) and in a fragment of white plaster with reed impressions, which was probably roofing material (34,000 spherulites/g of plaster). Given the high numbers of spherulites present in the plaster fragments analysed from Trench B, as well as other areas of the site, it is possible that the spherulites identified in the internal occupation residues represent contamination from the construction materials.

10.3 Abu Hureyra 2B (7400-6000 cal. BC): Trench G, phases 1-3

Trench G was excavated to determine the sequence of occupation in the northeast of the mound, the upper parts of which were heavily eroded (Moore et al., 2000, p. 241). Seven occupation residue sediment samples from Trench G were analysed for phytoliths and spherulites, representing phases 1-3 from external (n=4) and internal (n=3) occupation residues (Figure 10.5a, b).

In Phase 1, this area was an open space, with significant deposits of dark occupation soil, patches of burning, with pits and hearths dug into the surface (Moore et al., 2000, p. 242). During phase 2, a mudbrick building was constructed which extended beyond the edge of the trench to the northwest and northeast, of which several rooms were excavated (Figure 4.5, Figure 10.5a). Room 4 (phase 2) is an exceptionally narrow room, ~ 70cm x 100cm, with a series of trodden surfaces, renewed as debris built up and possibly used for storage. A large number of flint tools were recovered from room 1, including 230 fresh flint blades and lightly retouched blade knives bundled together, but with little flint waste in the deposit (Moore et al., 2000, pp. 244, 248).



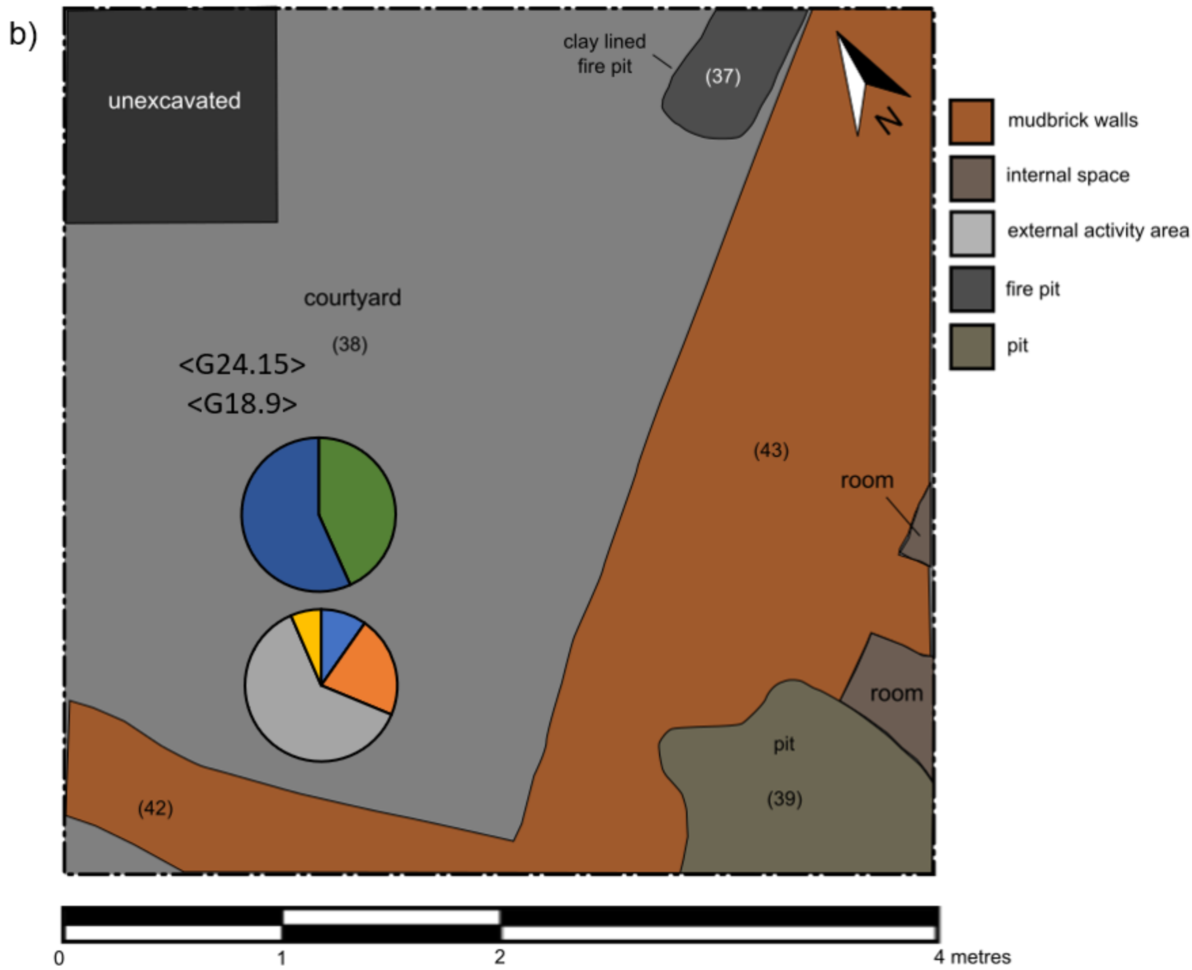


Figure 10.5 Annotated plan of Trench G, showing a) phase 2 with proportions of phytolith types in room 1 and room 4. Plan adapted from: Moore et al. 2000, p. 247, fig. 8.65. Photograph on left (taken from the east, scale 10cm) of bundle of flint blades from Moore et al. (2000, p. 249, fig. 8.69). Photograph on right (scale 20 cm) of niche in wall 60 of room 4 from Moore et al. (2000, p.248, fig. 8.67) and b) phase 3 with average proportion of phytolith types from ashy occupation residues from the external courtyard. Plan adapted from Moore et al. (2000, p. 247, fig. 8.65)

10.3.1 Trench G, phase 1

No spherulites were identified in the only sample to be analysed from Trench G, phase 1, which was brown sediment with frequent charcoal inclusions. Given the absence of spherulites and sediment colour (brown rather than grey and ashy), there is no evidence in this analysis that dung was burnt as fuel in this hearth, although, as discussed in section 2.3, many factors affect the preservation of spherulites. The representation of plants and absence of spherulites in the hearth is further discussed in section 10.5. The low concentration of phytoliths (370,000 per gram of sediment) compared with other material analysed in this study, from other trenches (Table 7.2, Figure 7.3) could be because most of the material put into the hearth was wood, which produces considerably fewer phytoliths compared with monocots, discussed in Chapter 2 (Albert et al., 2003; Tsartsidou et al., 2007). Phytoliths from monocots could have been incorporated into the archaeological record through contamination adhering to the barks of burnt wood (Albert et al., 2003). Cereals and domestic grains were identified in the charred macrofossil record for Trench G, phase 1, but in relatively low quantities compared with the later phases 2 and 3 (de Moulins, 2000, p. 411, fig. 12.31). Therefore, some of the monocots could be from spilled foodstuffs, especially as the majority of short cells identified were rondels (Figure 7.4c), indicative of Pooid grasses, including cereals (Twiss et al., 1969).

10.3.2 Trench G, phase 2 building and phase 3 external courtyard

Spherulites were identified in "moderate" concentrations in three samples, including from the occupation residues of room 4 (G57.34) and room 1 (G62.33), phase 2, Figure 4.5, as well as from material excavated from the occupation soil in the courtyard outside the Trench G building (phase 3, Figure 4.6, Figure 10.5a, b). Both rooms have relatively low concentrations of phytoliths, which is similar to external samples from Trench G. Both external deposits and rooms 1 and 4 are dominated by monocot derived phytoliths, with higher proportions of leaf/stem phytoliths compared with those derived from grass inflorescences (Figure 10.5a). However, the occupation deposits from both rooms do exhibit higher concentrations of phytoliths from grass inflorescences compared with those from external areas (Figure 10.5a, b). The higher inflorescences in internal occupation residues could be derived from plant material stored in these spaces, as hypothesised by Moore et al. (2000, p. 244) given the small sizes of the room, and the plaster niche in the wall of room 4 (Figure 4.5, Figure 10.5a).

10.4 Abu Hureyra 2B (7400-6000 cal. BC), Trench E, phases 5-7

Trench E was the only trench with evidence for the Epipalaeolithic occupation of the site (phases 1-3), however this study focuses on the Neolithic phase of occupation in Trench E as the main aim is to understand the development of agriculture during the Neolithic.

During the earliest phase of Neolithic occupation identified in Trench E, phase 4, three rectangular mudbrick houses were identified (Moore et al., 2000, p. 231, fig. 8.49). The phase 4 buildings set the plan for the construction of buildings throughout the period 2B in Trench E (Moore et al., 2000, p. 225), Figures 10.6, 10.7. Spaces in between buildings were generally narrow and would have reduced the scope for changing the building plots. The phase 6 buildings were built on the same alignment as the phase 5 buildings (Figure 4.1c), however, the house in the centre of the trench in phase 6, had one less room than its phase 5 predecessor, which enlarged the external space between the buildings (Moore et al., 2000, p. 233). During phase 6, the exterior house walls were made much thicker (80cm long, 30-40 cm wide), although interior walls remained thin (Moore et al., 2000, p. 235). A channel, 30cm wide and 10cm deep, filled with ash and charred cultivated chickpea seeds cut between the phase 6 buildings, which excavators hypothesised had been formed by erosion from trampling when animals were herded through the site over a long period of time (Moore et al., 2000, p. 236). Similar to the external areas in Trenches B, D and G, the external areas between houses were filled with lenses of ash and other debris, and were likely the hub of domestic activities, such as cooking (Moore et al., 2000, p. 237).

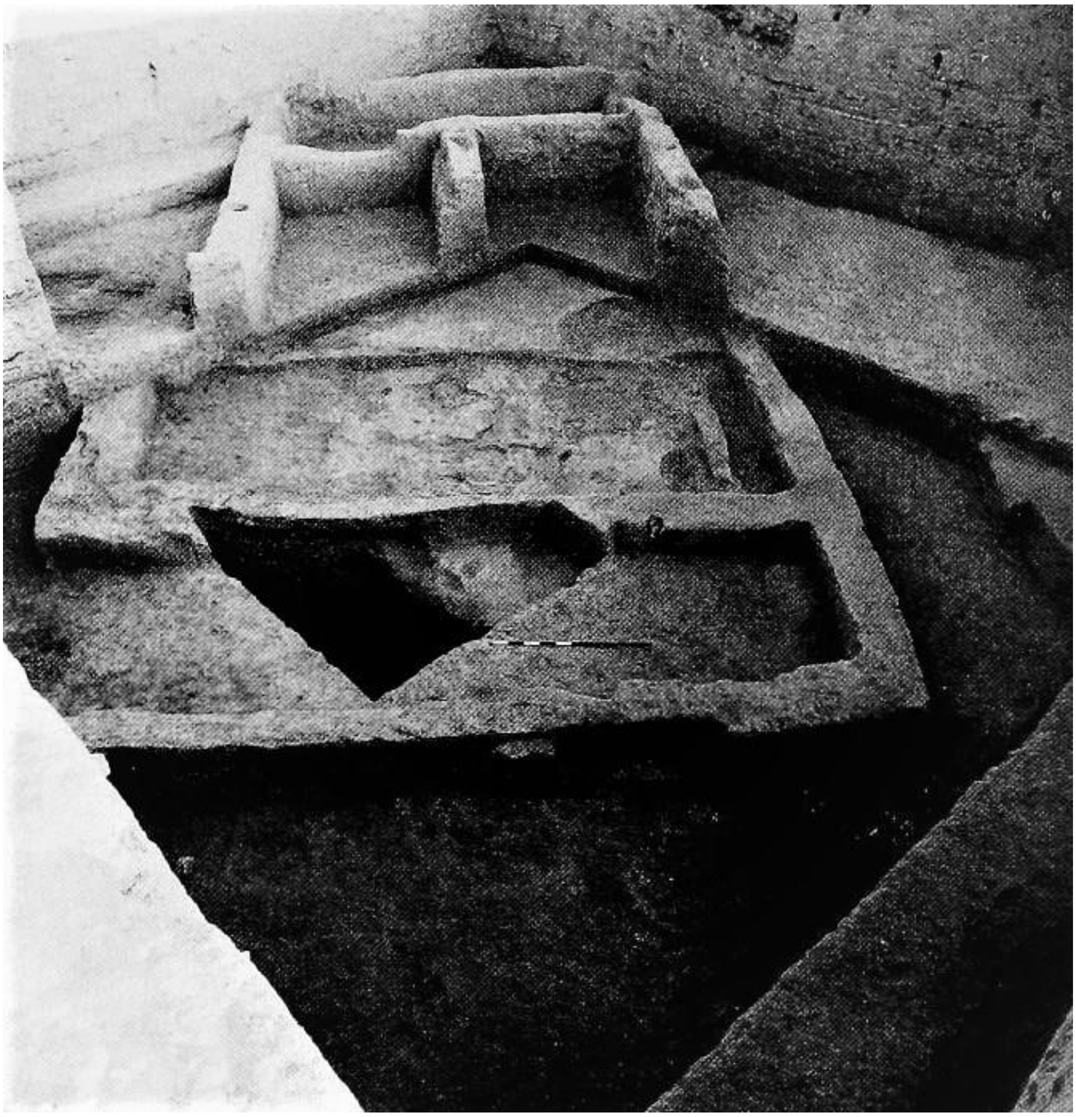


Figure 10.6 Photograph of the Trench E, phase 5 building superimposed on the phase 4 building (scale 1 metre) from Moore et al. (2000, p.232, fig. 8.50)

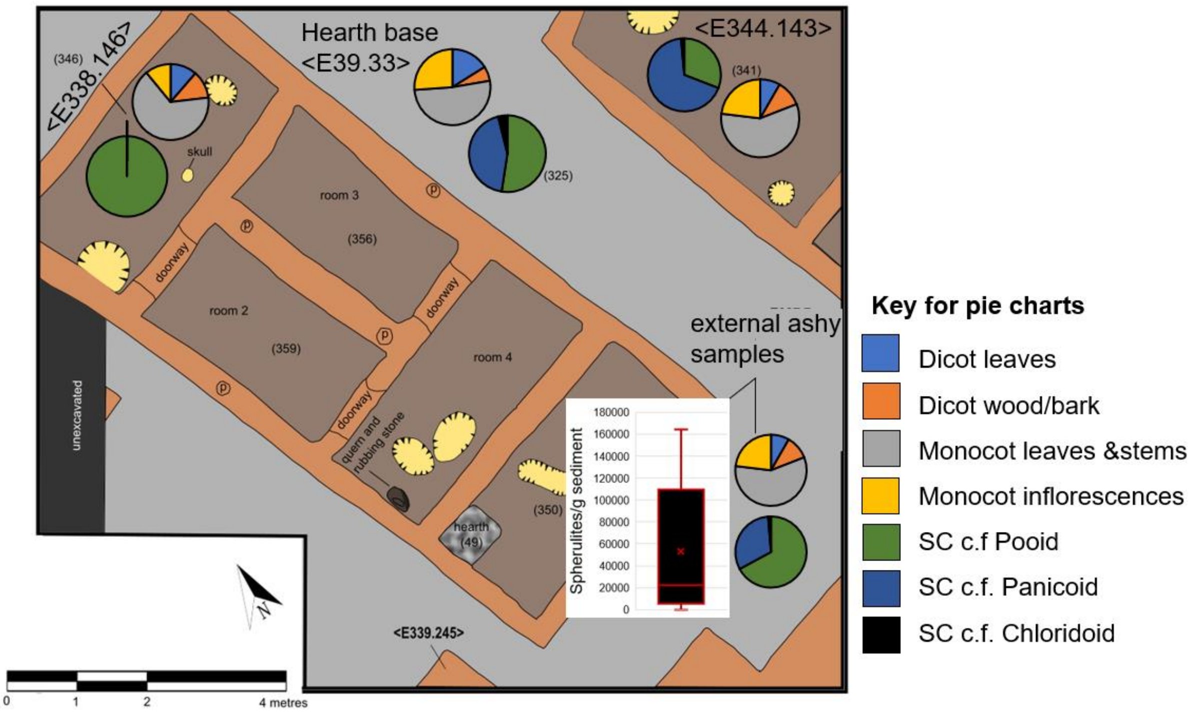


Figure 10.7 Annotated plan of Trench E, phase 5, showing the proportion of different phytolith types in different spaces and box plot showing the estimated number of faecal spherulites per gram of sediment for external ashy samples, adapted from Moore et al. 2000, p. 233, fig. 8.51

Twenty-eight occupation residue samples were analysed for phytoliths and spherulites from the Neolithic 2B period of Trench E occupation, most of which were recovered from external activity areas (n=16), including one sample from a hearth base (E39.33) and one pit fill which consisted of ash and occupation debris (E265.76). Four samples were also analysed from internal occupation residue deposits, which included the fill of a clay lined pit (E21.7). Two samples from a plaster fragment recovered from Trench E, phase 7, were also analysed for phytoliths and spherulites. A further three fragments of polished floor plaster were analysed by pXRF.

10.4.1 Dung and plant-use in Trench E, period 2B

The highest concentration of spherulites, E362.11 (160,000 per gram of sediment), came from a space between two buildings in phase 5, in a context described by the original excavators as “dark occupation debris” (Moore et al., 2000, p. 233, fig. 8.51), Figure 10.7. The overlying deposit, sample E361.11, also contained a high concentration of faecal spherulites (130,000/g of sediment) and is described as “Dark occupation soil, mudbrick wash and trodden surfaces”. A similar number of spherulites was also identified in sample E30.17, from a later deposit (phase 6) of the same external space between buildings where excavations revealed an ash filled channel running through this area, hypothesised by the excavators to have been formed by animals herded through the site (Moore et al., 2000, p. 236). Sample E19.4, which also has a relatively high number of spherulites (58,000 per gram of sediment), is again from a later deposit (phase 7) from the same external area. Other deposits where high numbers of spherulites were identified in this study include two samples from external areas between buildings in Trench E, phase 5, E36.22 (100,000/g of sediment and 64,000 per gram of sediment respectively), indicating the likely continuation of this space as an established passageway through which managed animals may have been led. E39.33, is sampled from the base of an external hearth (Figure 10.7), suggesting the possible use of animal dung as fuel, particularly as this sample also had a relatively high proportion of phytoliths derived from dicot leaves, often associated with animal dung as herded animals often browse on the leaves of dicot shrubs and trees. Significantly, five of the samples from the Trench E external activity areas contain low numbers of multicell dendritic phytoliths, with short cell bilobates, c.f panicoid grasses, which have been associated with basketry at PPNB Çatalhöyük (Rosen, 2005; Ryan, 2011). These types of phytoliths are usually observed in internal spaces (Portillo et al., 2014), presumably where baskets are used for storage, so their presence in the external area at Abu Hureyra is interesting, and could represent a space where baskets were being made.

10.5 Comparison of hearths

In contrast to the relatively high number of phytoliths and faecal spherulites identified in the Trench E, phase 5, hearth base (E39.33), the concentration of phytoliths was very low and no faecal spherulites were identified in the fill of a fire pit from trench G, phase 1 (G67.35), Figure 10.7a, b. This sample did, however, show a moderately high number of dicot derived phytoliths (22%), just over half of which likely originated in dicot wood/bark, as opposed to leaves (Figure 10.7c). The absence of spherulites combined with the phytolith evidence would seem to indicate that primarily wood fuel was used in this hearth. The comparison of E39.33 with G67.35 could indicate different functions of fire spots in different places, or a possible shift over time to utilise different fuel types.

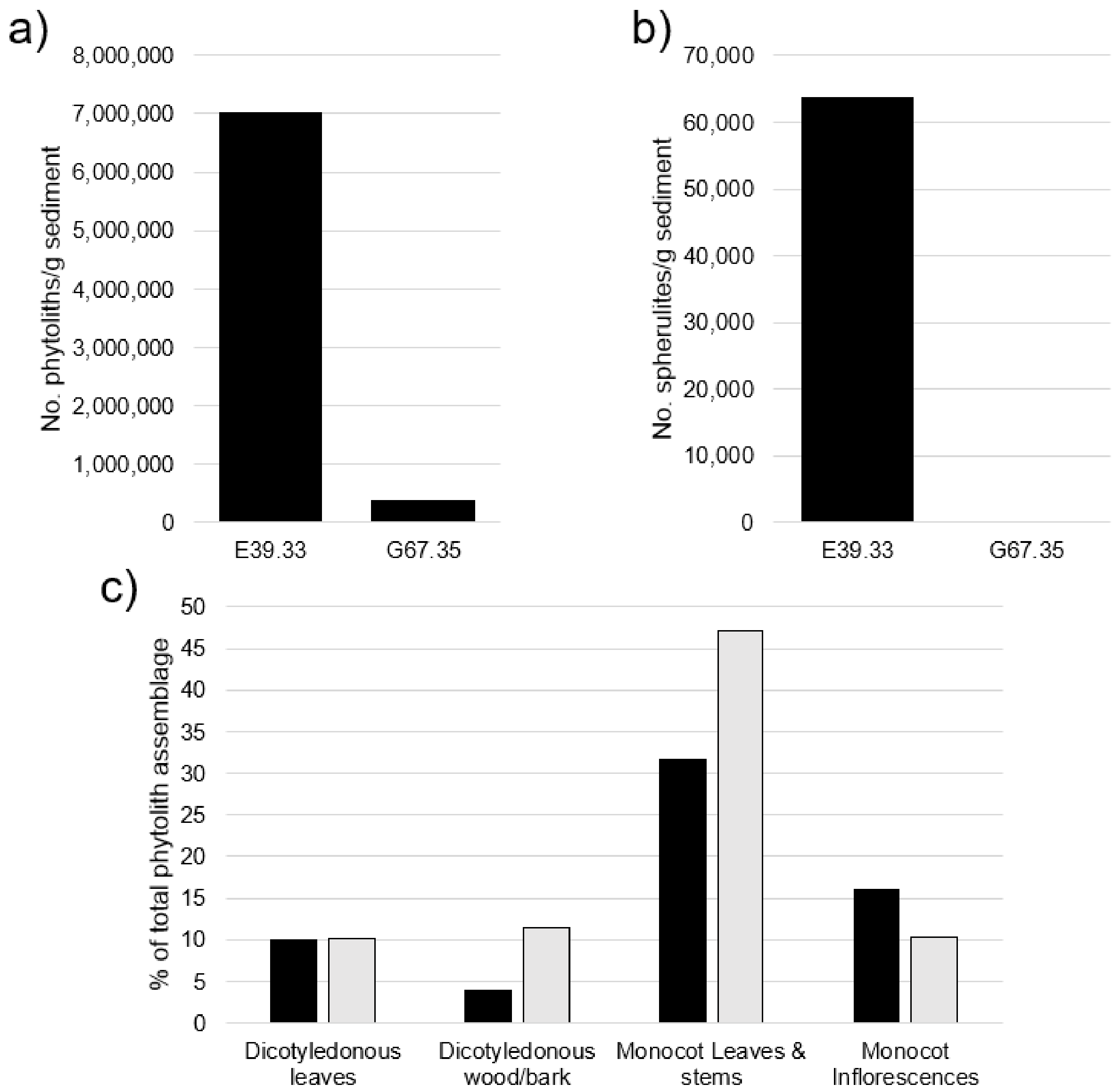


Figure 10.8 Graphs comparing the hearth from Trench G, phase 1, with the hearth from Trench E, phase 5 showing a) number of phytoliths per gram of sediment, b) number of spherulites per gram of sediment and c) different vegetation types and plants represented by the phytoliths.

10.6 Summary of Integrated contextual interpretation

In summary, this section has demonstrated the value of a multi proxy approach to clarify the use of plants and resources in different spaces at Abu Hureyra. Faecal spherulites, which indicate the presence of animal dung were present in all areas and time periods of the site analysed in occupation residues (Chapter 6) and gypsum floor plaster fragments (Chapter 7). The significantly higher proportions of faecal spherulites identified in plaster fragments compared with occupation residues suggests that the phytoliths identified in the plater fragments could be derived from the animal dung incorporated into the plaster matrix and therefore represent animal diet, rather than the intentional inclusion of plant material as temper. Chapter 11 discusses continuity and change in the presence of plant material from comparable contexts and deposit types, primarily focussing on external material from Trench D (Period 2A) and Trench E (Period 2B).

Chapter 11. Integrated Discussion

This chapter first summarises the key findings of this research in relation to the three key questions addressed in this study, with reference to the specific aims and objectives outlined in section 1.2, on the themes of environment, resource management, early agricultural and sedentary developments and variations, and use of archives, summarised below (Table 11.1). Each section (11.1 – 11.3) also discusses the key outcomes of this research with reference to other case studies. Section 11.4 then discusses how the main findings of this research contribute to theories and frameworks on human responses to climate change, human-socio-economic changes and the development of agriculture in SW Asia during the PPNB.

Table 11.1 Summary of key research questions, specific aims and objectives addressed in this research (section 1.2)

Research Question	Aims	Objectives
1. In what ways do plant-use and resource management change during the development of agriculture and sedentism in the PPNB? **Section 11.1**	**Aim 1:** Assess what plants were used and how plant-use changes over time and for different purposes **Aim 3:** Identify construction practices to inform on technological developments and resource-use	**1i:** Analyse phytoliths from occupation residues **1ii:** Integrate phytolith analysis with charred macro-fossils **3i:** Identify the elemental composition of plaster fragments by pXRF **3ii:** Analyse phytolith from plasters to identify plant-use for construction **3iii**: Identify faecal biomarkers by GC-MS
2. What are the potential depositional pathways for different plant proxies analysed and how can we better understand the complex taphonomy of archaeobotanical assemblages? **Section 11.2**	**Aim 2:** Identify whether animal dung was a component of mixed occupation residues, fuel and construction materials, and could have contributed to the charred macro-fossil assemblage **Aim 4:** Compare and evaluate methodologies for detecting dung in the archaeological record	**2i:** Identify if faecal spherulites are present in occupation residues and construction materials (plaster) **2ii:** Identify faecal biomarkers by GC-MS **4i:** Conduct experimental analyses to compare methods of quantifying faecal spherulites **4ii:** Compare the positive identification of dung spherulites with presence of faecal biomarkers **4iii:** identify if concentrations of elements are related to the detection of dung
3. How can we use environmental archaeological archives to their full potential to inform on current questions in archaeological research? **Section 11.3**	**Aim 5:** Demonstrate the ways in which archaeological environmental archives can be used for addressing current questions in archaeology	**5i:** Conduct analyses (Aims 1-4) on environmental archaeological archived material from Abu Hureyra **5ii:** Integrate results of this study with previous analysis at AH at other sites in the region

11.1 Environment, plant-use and resource management during the development of agriculture and sedentism in the PPNB

This section discusses how this research has provided new information to address research question 1; In what ways do plant-use and resource management change during the development of agriculture and sedentism in the PPNB?

11.1.1 Plant-use and environment

The phytoliths assemblages analysed in this research have contributed to a more comprehensive understanding about how people were using plant material at Abu Hureyra during the Neolithic (Chapter 6, Aim 1i), as has been demonstrated at numerous other case studies across SW Asia (e.g. Asouti et al., 2020; Elliott et al., 2020; García-Suárez et al., 2018; Jenkins and Rosen, 2007; Matthews et al., 2020; Portillo et al., 2009, 2010, 2013, 2014, 2019, 2020; Power et al., 2014; Ramsey et al., 2016, 2017, 2018; Ramsey and Rosen, 2016; Rosen, 2005; Shillito and Elliott, 2013; Shillito and Matthews, 2013). While some of the characteristics identified at Abu Hureyra are consistent with evidence from other contemporary PPNB settlements in the region, there are also some interesting differences discussed below.

The phytolith assemblages tended to be dominated by monocotyledons, but also with a significant input of vegetative material from dicot leaves and wood/bark in all samples analysed (Figure 7.4). A key aim of this research was to assess whether there were any changes in plant-use identified in the phytolith record between periods 2A (~8600-7300 cal. BC) and 2B (~7300-6200 cal. BC) at which time there was a major switch in the animal economy from a reliance on wild gazelle to managed caprines (Aim 1i). Phytolith concentrations and morphotype assemblages varied between samples (Table 7.2, Figure 7.4), although differences were not related to time period, specifically when comparing periods 2A and 2B. Differences in phytolith assemblages are more likely related to spatial category, deposit type and material in this study which may be why there is little change in the phytolith record over the same time period there is a significant shift in animal management practices. Different phytolith ecozone indicators were compared for samples which represent similar context-types, in this case ashy occupation residues from external activity areas assigned to Period 2A in Trench D and Period 2B in Trenches G and E (Figure 11.1a). Figure 11.1 compares the phytolith representation of different ecozone indicators/vegetation types (wetlands, grassy, woody) between samples from Period 2A and Period 2B. The types of vegetation in the phytolith record were remarkably similar between the ashy external deposits in Trench D (Period 2A) and Trench E (Period

2B) which could suggest continuity and thereby sustainability in the use of plant resources between periods 2A and 2B.

Interestingly, the external material from Trench G (n=2), which was broadly contemporary with Trench E (Period 2B) (Figure 11.1a), contains higher proportions of woody material and less grassy material compared with Trenches D and E. In Trench G, there were slightly higher proportions of grass short cells, cf. panicoid (bilobates and polylobates), compared with rondels c.f. Pooid (rondels and bilobates with trapezoidal cross sections) (Figures 10.1, 10.5b, 10.7). The differences in phytolith assemblages could indicate different activities were taking place in the Trench G courtyard (Figure 10.5b), compared with the external spaces between houses in Trench E, Figure 10.7) and in the open space between buildings in Trench D (Figure 10.1). A much higher density of bone, flint and other occupation debris were observed in the Trench D and E external areas compared with Trench G (Moore et al. 2000), perhaps suggesting a wider variety or higher intensity of domestic activities took place in the Trench D and E external areas, which in part could account for some of the differences between the plant remains (Figure 11.1b).

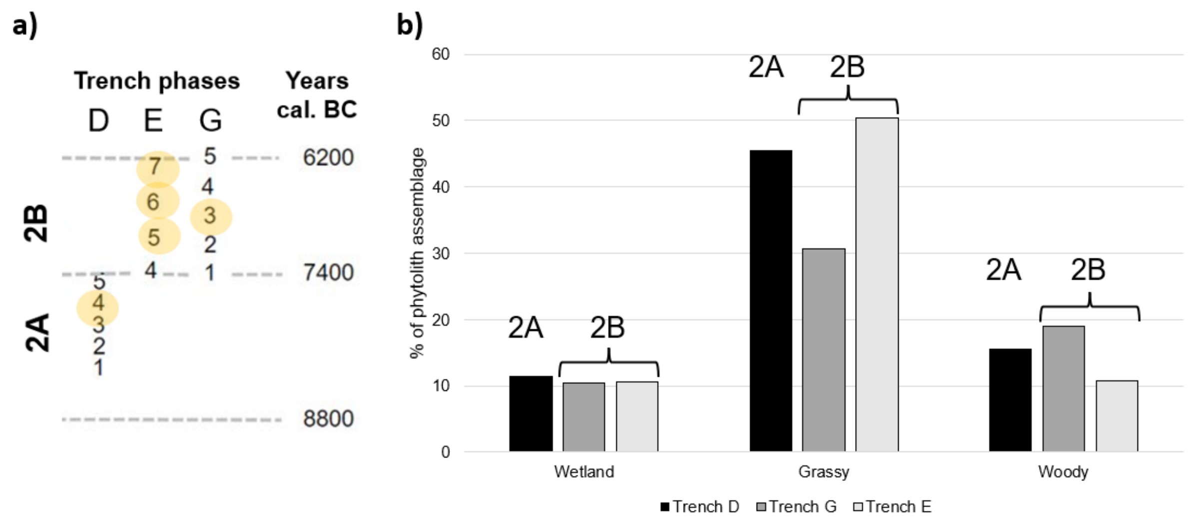


Figure 11.1 a) Schematic showing associations between Trench phases with external samples and b) proportions of different ecozone indicators identified by phytoliths from ashy material from the external area of Trench D (Period 2A), G (Period 2B) and E (Period 2B)

The overall consistency in the phytolith assemblage spanning a period of c. 2500 years, withstanding some variations between different space and deposit types (Chapter 7, Figure 11.1) is significant for understanding whether and how people sustainably managed environmental resources. The variety of plant resources in the phytolith record of different ecozone indicators, such as wood, grasses and wetlands, (Chapter 7, Figure 7.4, Appendix 4B, Appendix 4C, Figure 11.1) as in the charred macro-fossil record (de Moulins 2000) indicates the availability of a wide range of resources. The continued use of a range of resources is probably attributable to the rich environs of Abu Hureyra on the ecotone between the Euphrates Valley to the north and extensive steppe to the south. The location would have provided a combination of advantageous resources not provided at any other location in the Middle Euphrates (Moore et al. 2000, p. 475), discussed in Chapter 3, section 3.2, and summarised here. Although many Neolithic sites were situated on or close to the banks of the Euphrates River and other major water sources (section 3.2, 3.3), Abu Hureyra's proximity to Wadi Shetnet es-Salmas gave inhabitants an additional advantage. Water from the wadi would have been closer and easier to obtain than from the main Euphrates, and provided a corridor for xeric woodland, such as Terebinth to grow, providing a refugia during even brief periods of aridity which may have occurred year to year (Moore et al. 2000, p. 395). The "natural highway" provided by the Euphrates River would have provided access to forested hills to the north and minerals not available in the immediate environment (Figure 8.1a). While the rich resource base would have enabled inhabitants to experiment and innovate (Zeder, 2012), as argued at Çatalhöyük (Bogaard et al., 2017), it may also have buffered against the need to adapt, whether to changes in environment or increased population growth.

The high prevalence of Pooid grass derived phytoliths identified in this study (e.g. short cell rondels), in all samples, reflects a key element of the environment surrounding Abu Hureyra, where stands of wild cereals and other wild grasses would have grown on the steppe to the south, whether as a wild resource, managed, cultivated or domesticated. Small variations observed in the proportions of phytolith short cells, which reflect different types of vegetation, such as panicoid bilobates and chloridoid saddles (Twiss et al., 1969; Twiss, 1992) in this study (Chapter 7, Figure 7.4c, Appendix 4B, 4C), most likely reflect different deposit types (e.g. ashy, non ashy) and activities (e.g. basketry, fuel) across the site (Rosen, 2005; Ryan, 2011; Colledge and Conolly, 2014; Portillo and Matthews, 2020), discussed in Chapters 7 and 10.

The low numbers of husk phytoliths in this study that could be identified to one of the major cereals was surprising. Domestic cereals, including rye, einkorn, emmer, bread wheat two-row and six-row barley, grew in importance as PPNB Abu Hureyra developed, based on an increase in arable weed seeds in the charred plant assemblage, although domestic cereal grains make up a relatively small proportion of the charred macrofossil assemblage (de Moulins, 2000). The density of buildings in the settlement led Moore et al. (2000, p. 495) to suggest processing, e.g. threshing, took place outside of the settlement, which would also account for the low proportions of charred chaff in the assemblages. Furthermore, the domestic seeds are more likely to have been carefully conserved for use while weed seeds and small grains would have been discarded in the fires and therefore ended up in the charred assemblage (Hillman 1981). Since many of the samples analysed for phytoliths are from ashy deposits in external areas, probably used for cooking, the same reasons could explain the low number of cereal phytoliths which could be identified as wheat, barley or rye, based on wave patterns or number of papillae pits in this study (Rosen 1992).

Given the rich wetland resources available to the inhabitants of Abu Hureyra, the relatively low proportions of phytoliths from reeds and sedges identified in this study was unexpected (Chapter 7, section 7.4.2.3, Figure 7.4a, Figure 10.2). It is well established that reeds were an important, and well-utilised resource at Abu Hureyra, based on reed charcoal, Cyperaceae seeds in the charred macro-fossil record, abundant reed impressions in mudbrick and plaster, and parallels with the modern village of Abu Hureyra (de Moulins, 2000; Moore et al., 2000; Roitel and Willcox, 2000). The taphonomy of sedge phytoliths, which are often fragile and easily dissolved is discussed in Chapter 7 and section 11.1.2 below, may be one probable explanation for the low numbers of sedges identified in this study. More unexpected is the low number of reed stems and leaves in the phytolith record form Abu Hureyra, as these are prolific phytolith produces (Sangster and Parry, 1969; Ramsey and Rosen, 2016). Moreover, reed phytoliths are commonly identified in Late Pleistocene and Early Holocene sites from across SW Asia, situated in different environment types (Rosen, 2005; Jenkins and Rosen, 2007; Ryan, 2011; Matthews et al., 2013; Shillito and Elliott, 2013; Ramsey and Rosen, 2016; Ramsey et al., 2016; 2017; 2018; Elliott et al., 2020).

The relatively low proportions and occurrence of diagnostic reed phytoliths identified in this study at Abu Hureyra is likely due to several factors. Firstly, most of the samples analysed in this study were ashy and included frequent fragments of charcoal, and probably, to a large extent reflect materials selected for fuel (Chapter 4, section 4.1.2, Appendix 1). With fast growing and renewable sources of timber growing on the valley floor (e.g. poplar), and the xeric woodland (e.g. Terebinth) available along Wadi Shetnet es-Salmas (Moore et al., 2000, p. 267), as well as river access to forested hills to the north, wood resources could have been sustainable under careful management. Furthermore, in the Southern Levant, it has been suggested that people not only sustained, but also expanded woodland resources (Asouti et al., 2015).

Furthermore, as discussed in Chapter 8, and below in section 11.1.3, if wood were to become scarce, there was a readily available, continuous supply of dung available from the domesticated caprines and cattle, as also evidenced in this research (Chapters 7, 8 and 9). This study has demonstrated the likely use of dung fuel through the identification of faecal spherulites (Chapter 7, section 7.4.3), and faecal biomarkers, in the base of a hearth (Chapter 9, Figure 9.2, section 9.2.1). Therefore, the people of Abu Hureyra would have had little need to burn reeds and sedges which served a range of other purposes at the site.

11.1.2 Integration of macro and micro plant perspectives

As phytoliths and charred plant macrofossils are preserved under different conditions and therefore represent different plant types and uses (sections 2.1, 2.2), and are often analysed and reported separately, a key aim of this research was to compare the charred plant macrofossil assemblage (de Moulins 2000) with the phytoliths recorded in this study (sections 7.4, 7.5.1, Aim 1ii).

The representation and proportions of monocots to dicots was almost completely inversed when the charred macrofossil and phytolith assemblages were compared from material sampled from the same context (Chapter 7, Figure 7.7). As discussed in Chapter 10 (10.2), this can be explained in part by phytolith production in different plant types (e.g. Albert et al. 2003; Tsartsidou et al., 2007), while woody material and seeds are most commonly charred. As shown in Figure 10.4, the types of plant parts which make up most of the phytolith assemblage, are also unlikely to survive charring (Boardman and Jones, 1990; van der Veen, 2007; Matthews, 2010), and therefore, the combination of both proxies provides a broader perspective of plant-use.

Sedges provided a particularly interesting comparison of plants represented in the charred macrofossil record when compared with the phytolith record, as out of the six contexts where both charred plant macro fossils and phytoliths were analysed, sedges were only represented in both assemblages in one sample, E402, from the Epipalaeolithic phase of the site.

The samples where sedge phytoliths were identified but sedge seeds were not present in the charred assemblages were all from internal occupation residue deposits. The sedges could therefore represent basketry, matting or construction material (Rosen, 2005; Ryan, 2011) which under normal circumstances would not be burnt and preserved in the charred macrofossil record. This demonstrates the value of analysing multiple plant proxies which are preserved under different conditions to identify plants used for diverse purposes (Table 2.1, Aim 1i, Aim 1ii).

A similar approach to that in this research was used to compare the phytoliths and charred macrofossil plant remains at the Late Pleistocene site Ohalo II, where they found slightly lower discrepancies between both records, demonstrating that the differences are not only because of the low production of phytoliths in dicots (Ramsey et al., 2017, p. 710, fig. 3). The Ramsey et al., (2017) study grouped phytoliths by ecozone types; wetland, woodland, steppe/parkland grasses, whereas this study chose to group the plant remains more generally as dicots, monocots and sedges as a subsample of monocots. The reason this study assigned the plant remains in this way was, in part because of the high prevalence of small-seeded legumes in the charred assemblages which are dicots, but do not necessarily represent “woodland” vegetation, as small-seeded legumes, such as those in the Trifolieae family, were likely growing in stands of wild or managed cereals and grasses (de Moulins 2000). However, as legumes tend to be low phytolith producers, and few studies exists on specific phytolith morphologies unique to these taxa, it is not possible to split the "dicot" type phytoliths identified in this study in this way (Bozarth, 1992; An and Xie, 2022). Furthermore, as discussed above, the inhabitants of Abu Hureyra, likely made use of renewable timber sources growing in the valley bottom and along the nearby wadi, and therefore, the dicots do not necessarily represent a woodland ecozone, and are better defined as "woody" type vegetation. At Ohalo II, on the other hand, the occurrence of plant remains such as small-seeded legumes is lower, so that dicot type phytoliths can be more securely attributed to "woodland-type" taxa (Weiss et al., 2004). The relative proportion of phytoliths which were identified as wetland-type (e.g. MC phragmites culms and leaves) was higher at Ohalo II than at Abu Hureyra where few multi-cell phytoliths could be securely attributed to wetland type plants. A key reason for these differences is that the economy of Ohalo II is based on hunting and gathering, compared to at Abu Hureyra, where farming is developing and arable weeds growing amongst crops made a significant contribution to the charred macro-fossil assemblage. The comparison between Ohalo II and Abu Hureyra, however, is valuable and demonstrates the importance of adapted comparisons between different plant proxies to suit the types of plants represented in each assemblage.

11.1.3 The use of dung as a resource

Naomi Miller's seminal work in the 80s highlighted the importance of considering dung fuel as a depositional pathway for charred plant remains (Miller, 1984; Miller and Smart, 1984). Naomi Miller significantly argued in 1996 that some of the charred plant remains from Epipalaeolithic Abu Hureyra could be derived from animal dung burnt as fuel (Miller, 1996) rather than representing a broadening of the diet in response to the deterioration of climate and thereby plant resources following the onset of the Younger Dryas (Moore and Hillman, 1992), or weeds of cultivation (Hillman et al., 1997; Hillman et al., 2001) (Chapter 2). Dung was widely used and present on sites during the Neolithic in SW Asia (e.g. Spengler 2018 and references therein). The development of agriculture has been traditionally associated with an increased reliance on domestic cereals and legumes (Moore et al., 2000; Weiss et al., 2004; Savard et al., 2006), although a more recent synthesis of archaeobotanical data sets from SW Asia argues there is no evidence for a narrowing of the diet in Neolithic agricultural societies (Wallace et al., 2021). Therefore, this research aimed to identify if dung was a potential depositional pathway for plant macro and micro-remains in occupation residues and construction materials at Abu Hureyra (Aim 2i, 3iii). The identification of dung informs on a) depositional pathways of plant remains and b) the use of secondary animal products and resource management, for example the selection of different fuel types.

Faecal spherulites were identified in low concentrations in two out of three of the samples from the Epipalaeolithic phase of the site, which could suggest the use of dung preceding the intensification of animal management. However, radiocarbon dating of charred plant remains showed that around a third of dates obtained (eleven out of thirty-six) were intrusive from the Neolithic layers, and it is therefore also very possible that the dung spherulites could also be intrusive. The ambiguity and small sample size available for this study for the Epipalaeolithic period of occupation at the site is why the focus here is on periods 2A and 2B, as more samples are available and from well-defined contexts. However, faecal spherulites have also been identified in a recent study of flotation residues from the Epipalaeolithic period of occupation at Abu Hureyra (Smith et al., 2022), adding further evidence to Miller's (1996) suggestion that dung may have been burnt as a fuel.

Faecal spherulites were identified in c. 80% of the occupation residues sediments analysed and in all of the floor plaster fragments (Chapters 7, 8). In the occupation residues, the concentrations of spherulites and number of samples which contained spherulites were higher in the later Period 2B (~7300-6200 cal. BC) compared with Period 2A (~8600-7300 cal. BC) (Figure 7.6). The increase in faecal spherulites coincides with the increase in managed caprines and cattle, identified from zooarchaeological studies, ~7300 cal. BC (Legge and Rowley-Conwy, 2000). The presence of faecal spherulites which are most likely deposited by ruminant dung (Canti, 1997) in occupation residues, could represent the build-up of animal waste. Particularly as the highest concentrations were observed in external areas between buildings, the dung may have been collected and dumped in a refuse area, as occurred, for example at Çatalhöyük (Shillito and Matthews, 2013; Shillito et al., 2013b).

Some dung may have been deposited in external areas by animals being led through the site, as suggested by Moore et al. (2000) based on the channel between buildings identified during excavation in Trench E (Figure 4.4, Figure 10.7). No spaces which were likely animal penning areas were identified during the excavation, and it was hypothesised that animals were kept on the outskirts of the site (Moore et al. 2000). However, penning deposits may not be distinguishable during excavation without techniques such as micromorphology. Indications of trampling, for example, through compaction or horizontal inclusions in association with faecal material indicate penning areas (Matthews et al., 1996; Milek, 2012). Animal penning areas have been identified within other densely packed early farming settlements such as Çatalhöyük, Anatolia and Sheikh e Abad, Iranian Zagros (Portillo et al., 2019; Shillito et al., 2008), therefore it is also possible that penning areas existed at Abu Hureyra, although, micromorphological analysis of dung accumulations at Pınarbaşı and Boncuklu found no evidence for penning (Portillo et al., 2020a). As all of the occupation residues analysed include burnt material, it is also possible that the dung had been burnt as part of a mixed fuelling regime, as attested by the identification of faecal spherulites and faecal biomarkers in the ashy material from a hearth base, E39.33, (Figure 7.6, Figure 9.2). New research analysing dung spherulites from archived flotation residue material has suggested the use of dung fuel since Period 1B in the Epipalaeolithic phase of occupation at Abu Hureyra as well as in material from the Neolithic period of occupation, which supports the findings of this study.

The presence of darkened spherulites, which usually form in reducing burning conditions exceeding 500°C (Canti and Nicosia, 2018; Portillo et al., 2020b), in just under half (33 out of 71, 46%) of the occupation residues from both internal and external contexts across all trenches, which contained spherulites, also suggest that the dung had been burnt at some stage. Moore et al. suggest that where waste had built up in the narrow spaces between buildings the smells would have likely been significant (Moore et al. 2000, pg. 232). Waste material, including dung, may have been burnt (Moore et al. 2000, p. 240) as a practical solution to reduce noisome odors and biohazards. At Çatalhöyük, for example, *in situ*, burning associated with cess layers has been identified through micromorphology (Shillito and Matthews, 2013, p. 41).

The ubiquity of dung spherulites identified in gypsum floor plaster fragments suggest that dung was a well utilised resource in construction throughout the Neolithic occupation of Abu Hureyra, meeting Aim 3iii to identify whether dung was used in construction. PPNB Tell Seker al-Aheimar, was broadly contemporary with Abu Hureyra (late 8th to early 7th millennium cal. BC) and is one of the largest settlements of its period (300m x 180m and 11m high) located in the Jazireh plain, Upper Khabur region, north-eastern Syria (Nishiaki et al., 2013). Similar quantities of spherulites were identified from gypsum plaster floors, and other floor surfaces at Tell Seker al-Aheimar as at Abu Hureyra. The concentrations of spherulites in all the plaster fragments were significantly higher than in the sediments, suggesting a significant faecal input to the plasters, rather than a lower faecal component in the occupation residues, identified by much lower quantities of spherulites. The average number of spherulites per gram of plaster increases slightly between the period 2A fragments from Trench D and the period 2C, fragments from Trench B. As in the occupation residue sediment samples, the increase in spherulite concentrations between earlier and later phase possibly indicates the increased presence of dung and more intensive use as a material as the numbers of managed and domesticated animals increased ~7300 cal. BC (Legge and Rowley-Conwy, 2000). The increase in managed sheep and goat, as well as domestic cattle would have both provided increased access to dung, as also potentially generated more waste which needed to be managed. At Aşıklı Höyük, where there is similar, relatively rapid transition to managed caprines, Stiner and Kuhn (2016, p. 182) highlight the connection between increased waste build up and recycling, for example, dung used as a binder in construction.

As well as the significant reversal from a reliance on wild gazelle meat to managed caprines, at Abu Hureyra, there is a slight increase in cattle between periods 2A and 2B (Legge and Rowley-Conwy, 2000). Cows are highly resource expensive, heavy feeders and require large quantities of water, which is a significant challenge facing the world today (Poore and Nemecek, 2018). Moore et al. (2000, p. 497) attribute the increase with requirements for meat, milk and hides, but also hypothesise that they could have been used as draft animals to intensify agricultural production as the settlement grew. Multivariate statistical analysis of bovine bone dimensions and identification of pathologies has provided some evidence for the use of working cattle at other PPNB sites including Tell Halula based on bovine phalanxes with thick shafts, Tell Aswad based on multivariate analysis of bone dimensions are indicative of castration and Cafer Höyük, based on pathologies (Helmer et al., 2018, p. 85). The identification of dung as a major component of construction materials in this study (Chapter 8) could have been sourced from cow manure. It is possible that as the requirements for increased amounts of dung for expansion and regular repair of the settlement grew, it became more economically viable to keep an increased number of cattle, which may have provided a more efficient means of acquiring dung for plaster manufacturing compared to sheep and goats. Sheep and goats may have been taken into the fields to graze on the stubble of crops following their harvest, and therefore manuring the soil in the process (Moore et al. 2000, p. 497).

11.1.4 Construction practices and resource management

The high peaks of sulphur in the plaster fragments analysed in this study by pXRF (Figure 8.7, Appendix 10) are consistent with x-ray diffraction of a vessel fragment from Trench G and analysis of a fragment of floor plaster from Trench B which identified that both plaster types were made form gypsum (Kingery et al., 1988; le Mière, 2000, pp. 532, 533). The PCA analysis of the elemental composition of plaster fragments showed some groupings which relate to the period that the plaster is sampled from, however, sample clusters seem to be more determined by the trench from which the plaster fragments were sampled from than the time period (Figure 8.8).

Previous studies have demonstrated that artefacts may be contaminated by elemental composition of their burial environment (Wilke et al., 2016; Williams et al., 2020). Ti, Mn, Fe and Zr, which are all naturally occurring in sediments and present in all sediment and plasters analysed by pXRF in this study, may contaminate artefacts, though studies demonstrating this focus on their effect on bone (Carvalho et al., 2004; Piga et al., 2011). Therefore, it is possible in this study, that very localised burial conditions in each of the trenches, influenced the elemental composition of plaster fragments and affected the distribution of different samples in the PCA analysis.

Perhaps there were also subtle variations between the manufacturing of construction material between different households who may have experimented with different techniques which could have manifested over several generations. At Çatalhöyük, for example, variations in plant-use are observed at a household level (Bogaard et al., 2017), and variations in mudbrick composition have also been attributed to differing recipes which reflect human agency and selection at a household level (Love 2012).It is possible that at Abu Hureyra, as a site of a similar period and scale, there too, were opportunities for households to experiment and express individual preferences through construction practices. At Tell Halula, a study based on burial practices and goods, has suggested household autonomy increased during the PPNB, while inhabitants continued to observe a shared organisational framework (Kuijt et al., 2011). At Abu Hureyra, the construction of multiple phases of houses conforming to the same alignment as their predecessor and frequent burials within successive housing layers (Moore et al., 2000, pp. 189–273) possibly suggests ancestral ties to specific areas or households, and therefore, the very slight variations between some plasters could represent household preferences or traditions passed down and adapted over generations (Kuijt 2018). On the other hand, some differences could be accounted for by random choice, or availability of a specific material at the time of construction. For example, at Al'Matan, inhabitants suggested there was no particular reason for the selection of different clay sources (E. Jenkins, personal communication). This research could therefore fit with evidence from other sites which suggest a degree of household autonomy, and thereby flexibility, within a common overarching set of lifeways.

The very slight variation and groupings in the elemental composition between plaster samples from different trenches (Figure 8.8) could also represent changing practices over time. As discussed above, small changes in plaster manufacture practices could reflect the adaption or morphing of traditions passed down over generations or reflect a management strategy to cope with an increasing build-up of animal dung (e.g. Stiner and Kuhn, 2016).

Minor adaptations in plaster manufacturing could also reflect changes in environmental conditions. Adjusting the quantities of different materials and firing temperatures could have altered the properties of the plaster, perhaps to cope with changes in precipitation and temperature. For example, the calcination of gypsum at a higher temperature forms a harder plaster and the increased use of dung as a constituent of plaster may also have made it stronger and more water resistant which would have been beneficial during periods of increased precipitation (Gourdin and Kingery, 1975, p. 135; Vissac et al., 2017). Observation of the plaster fragments under a stereo microscope and in the clove oil spherulite slides under a polarising microscope indicated at least some of the dung was ash. As well as increased dung accumulation, residues (e.g. ash), from increased need for energy sources in a growing population with greater technological complexity could also have been recycled into the plasters as a practical solution to manage waste. Although, ash is hydrophobic and therefore could have been deliberately selected to increase the water-repellent properties of the gypsum plaster (Matthews 2010, p. 109), which is less waterproof than lime plaster (Kingery et al., 1988, p. 221). For example, micromorphological observations at Saar revealed ashy layers in between gypsum plasters (Matthews, 2010, p. 109) and ash has also been identified in mortars at Çatalhöyük (Matthews et al., 1996).

Overall, however, despite the minor variations in elemental and phytolith composition between plaster fragments, the technology shows remarkable continuity, particularly considering the longevity of the Neolithic phase of the site which could have been inhabited for more than 2500 years (Moore et al., 2000, p. 257, fig. 8.75, Figure 3.5). The main components required to construct the plasters; gypsum and animal dung, must have been acquired and managed sustainably, to support the substantial settlement (estimated 11ha). Moore et al. consider Abu Hureyra could have been as large as 16 ha, which possibly supported populations of up to 6000 people during the peak of period 2B (Moore et al. 2000, pp. 272, 274, fig. 9.6). The estimation of a 16ha site supporting 6000 inhabitants represents the maximum boundaries, and caution is required when estimating population size from site size (Bernardini and Schachner, 2018; Birch-Chapman and Jenkins, 2019). However, even based on much more conservative estimations, the size and population of Abu Hureyra would have been unprecedented at the time and required huge quantities of resources to maintain. The location of the site close to the Euphrates would have been favourable as fuel requirements increased, as inhabitants had access to quick growing riparian wood on the valley floor which would have provided a renewable source of timber (Moore et al., 2000, p. 267), probably contributing to the longevity and growth of Abu Hureyra. Section 11.3 further discusses the environs of Abu Hureyra and the ways in which the economy of other large, long-lived sites contemporary with Abu Hureyra, relates to their local environment and wider interaction networks.

11.2 Depositional pathways and taphonomy of plant remains in the archaeological record

This section draws together the evidence produced in this study to address research question 3: What are the potential depositional pathways for different plant proxies analysed and how can we better understand the complex taphonomy of archaeobotanical assemblages? The focus in this study was to identify whether dung was a potential depositional pathway for charred macrofossils and phytoliths. Identifying whether dung was a potential depositional pathway is significant as it affects the interpretation of plant assemblages (e.g. food, fodder or food) and provides information about human-animal-plant-environment relationships (Hillman, 1973; Miller, 1996; Smith et al., 2019; Spengler, 2018).

This study has shown that dung is a component of the ashy occupation residues analysed through the presence of faecal spherulites (Aim 2i) (section 11.1.3, 11.1.4). Therefore, it cannot be excluded that dung was a depositional pathway for at least some of the plant remains identified in this study through phytoliths and also charred macro fossils. All of the material which also had macro fossils published from the same context contained spherulites indicating that at least some of the identified charred plant macrofossils in these contexts could be dung derived (Figure 7.7). The most likely plants that could be dung-derived are explored in Chapter 7 and include heliotropes, feather-grass, dock, goose foot, melde, and wild grasses (Table 7.4).

Faecal spherulites were present in all of the floor plaster samples analysed in significantly higher concentrations than in the occupation residues. Based on the ubiquity of faecal spherulites both on the surface and in the plaster matrix, this study has concluded that there was a significant input of ruminant dung in the manufacturing of gypsum floor plasters. Ruminant dung is highly organic and contains abundant plant remains, and therefore, it is reasonable to assume that many of the phytoliths identified in the plaster matrix originated from dung. High concentrations of faecal spherulites in archaeological sediments have been used to indicator animal diet and fodder (Yeomans et al., 2021). In the sediment occupation residues, even the presence of spherulites likely determines one of many depositional pathways for the plant remains and therefore interpreting animal diet based on the phytolith evidence would be problematic. The high concentrations of faecal spherulites present in the plaster, however, presented an opportunity to identify animal diet and foddering regimes to inform more widely on animal management as the Neolithic developed and ongoing change in human-animal relationships (discussed in Chapter 8).

Micromorphology provides an opportunity to analyse archaeological materials within their primary context, and clarify a wider range of depositional pathways and associations between different plants and artefacts (Matthews, 2005; 2010; Matthews et al., 2013; Banerjea et al., 2015; Elliott et al., 2020). An initial aim of this research was to identify depositional pathways of plant remains from ovens at the contemporary Neolithic settlement of Bestansur, Iraqi Kurdistan, to provide comparative data on different ways in which phytoliths enter the archaeological record. Time restraints caused by the ongoing covid-19 pandemic and subsequent laboratory closures and postponement of fieldwork meant that this element of the PhD research could not be completed (see Covid-19 impact statement). However, this represents an exciting and imperative avenue for future research, to better understand archived environmental archaeological samples through new analysis and comparative studies of material more recently excavated material.

11.3 Integrated methodological approaches and environmental archaeological archives to address current questions in archaeological research

This section provides a brief evaluation and synthesis on ways in which we can use environmental archaeological archives to their full potential to inform on current questions in archaeological research (research question 3).

In order to fully address research question three, a key element of this research was on methodological evaluation. As the identification of dung was an important aspect of this research, an experiment was conducted to compare the quantitative results of two different methods for quantifying faecal spherulites (Chapter 6). The study demonstrated that the results of quantitative faecal spherulite analysis can vary significantly depending on the protocols adopted to quantify spherulites. This is important, as currently, a range of methodological variations are applied to identify and quantify spherulites in archaeological studies (Table 6.2), and inconsistencies between methods lower the interpretative value for spherulites. Therefore, caution is required when making interpretations of archaeological deposits based on spherulite concentration, particularly where different methods have been employed to analyse and quantify the faecal spherulites.

Darkened spherulites form in burning temperatures usually exceeding c. 500°C (Canti and Nicosia, 2018), though can vary considerably between different species and under different conditions (Portillo et al., 2020b). The study of darkened spherulites is still in its infancy, and further experimental work is required to clarify the formation and preservation conditions for them (Canti and Nicosia, 2018; Portillo et al., 2020b; 2021). This study has contributed to the growing body of research into darkened spherulites, as it is essential to understand the effect of the analytical method in order to conduct further effective research into formation and preservation conditions of darkened spherulites. Although the total concentrations of spherulites varied considerably between the two methods assessed, the proportions of spherulites which were "darkened" were relatively consistent, both between samples and across both methods (Figure 5.2). This has demonstrated that the identification and proportion of darkened spherulites present within an assemblage can provide a good indicator of burning temperature, regardless of the method used to analyse the spherulites. This is important to consider when analysing archival remains and selecting which techniques will most effectively address the research question. For example, organic compounds, including faecal sterols rarely survive in temperatures exceeding 400°C (Reber et al., 2019).

Gypsum plaster can be formed from gypsum rock in temperatures of between 150° and 400°C (Kingery et al., 1988), which indicated some faecal sterols, detected by GC-MS, may have survived in the plasters, particularly given the high sensitivity of the equipment and method applied in this research (section 4.2.6). However, no faecal component was detected in any of the plaster fragments analysed, although some compounds associated with dung were identified in low quantities and trace amounts in five of the floor plaster fragments (Table 9.1). In this instance, the high proportions of darkened spherulites (>10%) reported in ten out of twelve of the samples analysed, could be used as an indicator and useful new screening method for the low potential of GC-MS analysis. Faecal spherulite analysis is a particularly effective screening methos as it is relatively rapid and low cost requires little expensive lab equipment, except for a polarising microscope with a cross-polarising filter, compared with the time, resources and expertise it takes to prepare material for GC-MS analysis.

pXRF analysis was conducted on bulk occupation residue sediments to assess whether the elemental composition, or specific elements were related to the concentration of phytoliths and spherulites and could therefore be applied as a non-destructive screening method to assess the potential of material for further analyses. Elevated levels of phosphorous have been associated with faeces and plant material, as well as more generally with areas of human activity (Holliday and Gartner, 2007). Therefore this study assessed whether there was any relationship between concentrations of P, faecal spherulites and phytoliths. Faecal spherulites are calcitic (Brochier et al., 1992; Canti, 1997; Canti and Brochier, 2017) and phytoliths are predominately made up of silica (Piperno, 2006), so this research also assessed whether there was any relationship between Ca and faecal spherulite concentration and Si and phytolith concentration. No relationship, however, was detected between concentrations of P, Ca and Si with concentrations of phytoliths and spherulites (Chapter 5).

As elevated P signals are produced by a wide range of materials frequently found in the archaeological record (e.g. bone, plants, dung) and the occupation residue sediment samples are heterogenous this is likely why no relationship was detected between P and faecal spherulites or phytolith concentration in this study (Entwistle et al., 2000; Oonk et al., 2009; Canti and Huisman, 2015; Williams et al., 2020). Furthermore, Ca and Si are abundant in the local environment given the highly calcareous geology of the region (Moore et al., 2000, p. 47).

Specific spaces and activities have also been differentiated between on the basis of their elemental composition in both modern ethnographic studies and in archaeological investigations (Middleton, 2004; Rondelli et al., 2014; Jenkins et al., 2017; Vos et al., 2018). In this research, PCA analysis was conducted to explore potential patterns in elemental composition between different sample types (trench, spatial category, time period, material type). Some groupings were apparent, and samples clustered very loosely by trench, however the deposit material types (dark ashy, or light soil) seemed to be the most significant factor determining the groupings (Figure 6.4).

The identification of dung as a major component of the gypsum floor plasters at Abu Hureyra provided the basis through which to assess how elemental composition, analysed by pXRF, related to faecal spherulite and phytolith concentrations. In contrast to the bulk sediment samples, slightly positive relationships were identified between Si and the concentration of phytoliths and P and the concentration of spherulites in the floor plaster samples (Chapter 8). This could be because of a higher concentration of dung, and lower mixing of other materials within the plaster samples.

The comparison of methodologies for quantitatively analysing faecal spherulites (Chapter 5) aided in the selection of the methodology used throughout this research to identify faecal spherulites. The method used, involved mounting c. 1mg of material (e.g. sediment/plaster) onto a microscope slide, mixing the material with clove oil and cover slipping, which was found to be the fastest, safest and most cost-effective way to quantify the spherulites. The quantification of spherulites using the same method for all archaeological material analysed in this study enabled a comparison of concentrations between different spaces at Abu Hureyra (e.g. trenches, internal/external) and different time periods (with a focus on periods 2A and 2B), with additional samples from the Epipalaeolithic occupation residues (low to no spherulites) and plaster fragments from period 2C (very high spherulite concentrations).

More reference and experimental studies are also needed to gain a better understanding of the formation and taphonomy of darkened spherulites (Canti and Nicosia, 2018; Portillo et al., 2020b). Through the review and comparison conducted in Chapter 6, it is also clear that there is huge variation in spherulite production and concentration which may depend on the animal, environment, diet, soil conditions and processing methods employed to quantify faecal spherulites (Canti, 1997; 1999; Portillo et al., 2020b; 2021). Further research is needed which replicates previous studies and further assesses the effects of different combinations of variables known to influence faecal spherulite production and taphonomy in modern and ethnographic studies to provide a baseline and reference for interpretating archaeological dung deposits.

This study has benefitted from the thorough, detailed publications on Abu Hureyra, particularly the *Village on the Euphrates* monograph (Moore et al., 2000), which includes detailed section drawings, plans, matrices and descriptions of each phase of occupation. Professor Andrew Moore has also been very helpful and provided additional information about the samples and excavation which have contributed to the interpretation of material analysed in this study. The detailed stratigraphic and excavation records from Trenches A and C, which are Neolithic deposits spanning period 2A and 2B, are yet to be published, limiting the interpretations of materials from those trenches in this study, which is why this research has focused on the material from Trenches B, D, E and G. However, it is hoped that in the future collaborations will be possible to combine the analyses undertaken in this research with the ongoing publication of the Abu Hureyra excavations. This study has demonstrated the capacity of environmental and archaeological material which was excavated ~50 years ago to contribute new data to ongoing debates in archaeological research.

11.4 Aligning new research with the development of agricultural societies

This final section of the discussion reviews and synthesises the new data generated in this study with wider frameworks (Chapter 1, section 1.3) to better understand the development and intensification of farming practices during the Neolithic in SW Asia. This section firstly evaluates how people may have responded (or not responded) to minor changes in climate during the Neolithic occupation of the site (section 11.4.1). Then goes on to draw comparisons between Abu Hureyra and other contemporary large PPNB farming settlements, to disentangle the choices of people at a site-specific level, compared with shared practices across the local and wider region.

11.4.1 Climate and socio-economic changes at Abu Hureyra

Human socio-economic developments have been associated with climate change from the past to the present day (Richerson et al., 2001; Weninger et al., 2006; 2014; Rosen, 2007; Bar-Yosef, 2011; Kelley et al., 2015, Jones et al., 2019), but the extent to which this is the case remains debated (Bar-Yosef, 2011; 2017; Roberts et al., 2011; 2018; Jones et al., 2019). The samples analysed in this study span key periods of change from the earliest occupation of the site (Period 1, phase 1, ~11,200 cal. BC) to some of the latest, prior to the site's abandonment (Period 2C, ~6000 cal. BC). This section first very briefly discusses climate and plant-use during the Epipalaeolithic period of occupation (11.4.1), although the focus of this section is on changes in climate, environment and plant-use as the Neolithic developed at Abu Hureyra (8600-6000 cal. BC).

11.4.1.1 Climate and socio-economic changes at Epipalaeolithic Abu Hureyra (11,200-9800 cal. BC)

As discussed in Chapter 3 and summarised in Table 3.2, during the Epipalaeolithic phase of occupation at Abu Hureyra, c. 11,200-9750 cal. BC (Period 1 A-C), based on palaeoecological records from Lakes Ghab, Huleh, Zeribar and Mirabad, the climate was likely initially warm and wet, with abundant resources and stands of wild cereals growing close by during the earliest identified occupation of the site (sections 3.2.3 and 3.2.4, Alley et al., 1993; Moore et al., 2000; Weaver et al., 2003, Colledge and Conolly 2010). The onset of colder, drier conditions during the Younger Dryas, ~10,800-9700 cal. BC, likely changed the distribution and availability of local vegetation (see Figure 3.3), as identified in regional palaeoecological records (Roberts et al., 2018 and references therein; Jones et al., 2019).

The increased aridity and temperature decline during the Epipalaeolithic occupation of the site have been argued to have had a significant impact on the subsistence strategies used by the inhabitants of Abu Hureyra, whereby an increase in small-seeded legumes and grasses has been interpreted as evidence for pre-domestication cultivation and/or a broadening of the diet to manage depletion in resources, detailed in section 2.3.1.1 (Hillman et al., 2001; Colledge and Conolly, 2010). The increase in small-seeded grasses in the charred archaeobotanical assemblages at Natufian sites including Eynan, Hilazon Tachtit and el-Wad in the Southern Levant, have been interpreted as increased dietary breadth to maintain stability during the Younger Dryas (Portillo et al., 2010; Rosen, 2010). Phytoliths from Natufian sites in the Levant suggest a decrease in the use of woody plants in later Natufian periods, compared with the early Natufian (Rosen, 2010, p. 119). In contrast, the one available occupation residue sample from the later Epipalaeolithic period (E402.14) at Abu Hureyra has higher proportions of dicot derived phytoliths compared with the earlier samples (E55.31, E435.15) (Chapter 7, Figure 7.4). However, such a small sample size (n=3 for the Epipalaeolithic) has no interpretative value for identifying trends over time, as the phytolith assemblages likely represent the specific context, material and deposit type from which they were recovered (section 2.2). Furthermore, some dicot type phytoliths could be derived from legumes which grew amongst stands of wild cereals (discussed in section 11.1.1), rather than representing wood from trees, which would be consistent with the charred macrofossil assemblage (Hillman et al., 2001). However, at this time, the taxonomic resolution of the phytoliths identified is not sufficient to differentiate between these plant types. Therefore, the focus of the discussion here is on the reoccupation of the site (from ~8600 cal. BC) during the relatively stable Holocene (~9900 cal. BC onwards), section 11.4.1.2.

11.4.1.2 Climate and socio-economic changes at Neolithic at Abu Hureyra (8600-6000 cal. BC)

At ~8200 cal. BP or 6250 cal. BC, a global climate event occurred (discussed in section 3.2.3,Table 3.2) which resulted in a period of increased aridity in SW Asia (Bar-Matthews et al., 1999; 2003), although the effects on climate would have varied in different parts of the world. Often referred to as the 8.2ka event, this period of climate change has received much attention as it is argued to have caused widespread abandonment, collapse, reorganisation and migration of Neolithic societies in

SW Asia (Weninger et al., 2006; 2014). A global climate anomaly has also been identified earlier at ~9200 cal. BP (~7250 cal. BC), referred to as the 9.2ka event, similar to the 8.2ka event but less severe, which likely resulted in cooler and more arid conditions in SW Asia (Fleitmann et al., 2008). A more recent synthesis of radiocarbon dates with climate records and archaeological records has demonstrated that there is no evidence for wide scale collapse, abandonment or migration in response to the 9.2ka or 8.2ka events, although people may have responded through local, small-scale adaptions to manage changes in the climate conditions and resource availability (Flohr et al., 2016). This study, therefore, offers new insights into how people may have responded to the 9.2ka event at a site-specific level at Abu Hureyra.

The PPNB period of occupation at Abu Hureyra is divided into periods 2A (8600-8200 cal. BC) and 2B (8200-7000 cal. BC), which is characterised by a change from the dominance of wild gazelle in period 2A to managed caprines in Period 2B, alongside an increase in proportion of cattle (Chapter 3, Table 3.5). Based on charred macro-remains, changes in the use of plants between periods 2A and 2B are less pronounced than in the faunal record (de Moulins, 2000). There appears to have been a peak in the proportions of domestic cereals around the transition between periods 2A and 2B, though as this shift is only represented in two samples, from Trench B, phase 7, it could reflect different uses of space (de Moulins 2000, p. 410). The size of the settlement clearly increased between periods 2A and 2B, even give debates regarding the estimation of site size and population estimates (e.g. Kuijt, 2000; Bernardini and Schachner, 2018; Birch-Chapman and Jenkins, 2019). The transition between periods 2A and 2B took place between 7515 and 7225 cal. BC (9465 and 9175 cal. BP) (Jacobsson, 2017), according to a reassessment and model of quality checked radiocarbon dates (Table 3.1, Figure 3.7), and may, therefore, have coincided with the 9.2ka event.

There are multiple ways in which the 9.2ka event could have affected the inhabitants of Abu Hureyra which are discussed here with reference to the available evidence. It is possible that the aridification and temperature decrease associated with the 9.2ka event could have shifted gazelle migration patterns away from the site. Although given the extensive areas of grasslands close by (Moore et al., 2000, p. 80), it seems unlikely that they would have completely changed their migration course significantly, though perhaps changes in climate could have shifted the time of year the gazelles passed by Abu Hureyra. The charred macrofossil record (de Moulins, 2000) and phytoliths analysed in this study show that a wide variety of plant resources (e.g. steppe grasses, wetland valley bottom plants and woody plants) continued to be used during and following the 9.2ka, and there is no suggestion of a significant change in plant-use between periods 2A and 2B (section 11.1.1). The suggested population increase, therefore, could have been due to people migrating to Abu Hureyra from more marginal areas, as the rich resource base on the border of several ecozones at Abu

Hureyra (Chapter 3, section 3.2.4) would have provided a buffer against any impact of the 9.2ka event. In addition, large rivers such as the Euphrates can take between 1 and 10 years to respond to drought (Jones et al., 2019), and therefore the flood plains and the vegetation/cultivation they hosted may have been resilient to shorter term periods of aridity. The continuous use of plant-resources indicated localised management strategies for the sustainable use of plants over the ~2500 year occupation of the site. For example, there is no evidence in the phytolith record for an intensification or decrease in the use of wetland resources (sedges, bulliforms c.f. reed exploited consistently throughout the occupation) as the site grew, or during possible periods of environmental change, such as aridification during the 9.2ka event (Chapter 3, section 3.2.4).

Legge and Rowley-Conwy (2000) argue that an increase in hunting pressure as the settlement grew could have encouraged the intensification of caprine management evidenced in the faunal record, which could have been caused by the migration of people from more marginal areas. People could also have migrated with their herds which would also account for the dramatic increase in managed caprines between period 2A and 2B. In this study, an increase in the number of faecal spherulites identified in occupation deposits from period 2B compared with 2A is consistent with more managed animals being kept on site (Chapter 7, Figure 7.3). Higher concentrations of faecal spherulites were also identified in plaster fragments recovered from the later Period 2C (~6200-6000 cal. BC) compared with the plaster fragments recovered from Period 2A (8600-7300 cal. BC), Chapter 8. However, as the sample size is small (n=6), the differences may not be related to the time period. Furthermore, as the Period 2C fragments analysed for spherulites (n=2, Chapter 8, Table 8.2, Figure 8.4) were not recovered *in situ*, it is possible that the fragments were reworked from earlier levels (Moore et al., 2000). The concentration of faecal spherulites alone does not necessarily equate to a larger input of dung, as faecal spherulite production and taphonomy can be affected by numerous factors (discussed in Chapter 5). Nonetheless, the increase in spherulites in later samples is worth exploring and provides a potential avenue for future research.

Population and settlement growth is also likely to have increased the demand on raw materials for construction and artefacts. This study has identified animal dung (most likely sheep, goat or cattle) in plaster fragments spanning the Neolithic occupation of the site, periods 2A, 2B and 2C (Chapter 7), indicating it was a highly utilised resource, which may have been used more intensively in later periods. Despite the changes in animal management at Abu Hureyra between periods 2A and 2B, which coincide with (but are not necessarily related to) the 9.2ka event, the continuity in settlement structure, plant-use and material-culture is perhaps even more striking. Houses continued to be built and repaired on the same alignment using primarily the same materials (Moore et al. 2000, Chapter 8). Even the plant-use visible in the macro-fossil record did not undergo significant changes between periods 2A and 2B (de Moulins 2000), particularly compared with the significant shift in the faunal record from a reliance on gazelle to managed caprines (Legge and Rowley-Conwy, 2000). The overall continuity of Abu Hureyra between periods 2A and 2B, combined with some changes in subsistence patterns visible in the charred macrofossil and zooarchaeological records, such as the intensification of managed animals (de Moulins, 2000; Legge and Rowley-Conwy 2000), perhaps suggests the ability of the inhabitants to adapt to internal or external changes or pressures, which created a resilient society which was able to flourish for c. 2500 years.

11.4.2 Abu Hureyra and the development of agriculture and sedentary societies

The Middle Euphrates region is important for understanding the transition to fully agricultural societies (Bar-Yosef and Belfer-Cohen, 1989; Cauvin, 2000; Moore et al., 2000; Akkermans and Schwartz, 2003; Willcox et al., 2009). Sites in the region span from the Epipalaeolithic, predominately hunter-gather communities (van Zeist and Bakker-Heeres, 1986; Moore et al., 2000), to early PPNA settlements with archaeobotanical evidence interpreted as pre-domestication cultivation (Willcox et al., 2008; 2009; Arranz--Otaegui et al., 2016), and then the development of large fully agricultural settlements (Moore et al., 2000; Akkermans et al., 2006; Molist, 2006) (Table 3.3 for sites and key references).

The start of the Neolithic represents a major change in human culture, lifeways, plant-use and subsistence strategies, and understanding the process which led to full agricultural societies has been the focus of much academic research (Hillman and Davies, 1990; Cauvin, 2000; Hillman, 2000; Hillman et al., 2001; Willcox, 2005; 2012; Fuller, 2007; Willcox et al., 2008; Zeder, 2008; 2015; Cohen, 2009; Abbo et al., 2010; Fuller et al., 2010; 2011; Abbo and Gopher, 2017). Equally significant as ascertaining the when and why of plant and animal domestication, is understanding the complex human-plant-animal-environment under which mixed economy agricultural settlements grew and intensified into full scale agricultural communities, as this change in scale and lifeways likely had an unprecedented impact on the management of local resources.

Decades of research at Çatalhöyük, Anatolia has provided key insights into early farming ways of life (Hodder 1996; 2005; 2011; 2012). However, despite many shared cultural, social and economic traits between sites across SW Asia during the Neolithic (Bar-Yosef and Belfer-Cohen, 1989; Cauvin, 1994; 2000; Asouti, 2006), new research is increasingly identifying human selection at a site-specific level and, where the resolution of evidence allows, even household level (Bogaard et al., 2017; Kabukcu et al., 2021). To date, Abu Hureyra represents one of the largest early full-scale farming villages in SW Asia, and even using conservative population estimations, was likely inhabited by several thousand people, and therefore, had the potential to have a heavy impact on the local environment, particularly the fragile steppe ecosystems (Moore et al., 2000, p. 494). The unprecedented size (estimated 11ha) and longevity (two and a half millennia) of Abu Hureyra warrants careful examination to identify the strategies employed by people in the past to sustainably manage resources and adapt to facilitate the growth of the settlement.

Abu Hureyra is a particularly important case study in which to explore sustainable resource management at a local level, as Moore et al. (2000, p. 497) suggest that in terms of subsistence, Abu Hureyra was able to function independently from other sites in the region, based on extensive surveys and vegetation mapping undertaken by Gordan Hillman and colleagues (Moore et al., 2000, pp. 78–80). Due to the construction of the Tabqa dam, the Syrian Department of Antiquities initiated extensive surveys of the part of the Middle Euphrates region where Abu Hureyra is located (Bounni, 1973; Marchetti et al., 2019, pp. 19, table 1). No other contemporary sites of close to the size and capacity of Abu Hureyra have been identified close by, although small scatters of flint in the areas surrounding Abu Hureyra attest the presence of very small, less than 250m^2, peripheral settlements, or possibly base camps used by hunting parties (Wilkinson and Moore, 1978). The closest contemporary sites of a similar scale, Tell Halula and Bouqras, over 70km, and probably several days walk away.

Humans require around 2500 calories per day which is equivalent to 250-300kg of wheat per year (Clark and Haswell, 1967, pp. 18–19, 60) and c. 1 hectare of land cultivated using simple agricultural technology can yield around 1000kg of wheat per year, therefore meeting the caloric requirements of 3 to 4 individuals (Moore et al., 2000, p. 496). Although, depending on the season, inhabitants may have had fluctuating calorific requirements throughout the year. Gender also likely played a role in calorie requirement and energy expenditure, as shown in the skeletal record, men and women likely participated in separate activities (Molleson 2000; Moore and Molleson 2000). Within 2-3 kilometres of Abu Hureyra there would have been an estimated 1000 to 2500 hectares of land suitable for cultivation, based on vegetation models which synthesise modern vegetation, rainfall levels, insolation and temperature, altitude and topography (including aspect), geology and soil type (Chisholm, 1968; Hillman, 1973). Moore et al. conclude that the area surrounding Abu Hureyra would have been sufficient to support the largest population estimations of Abu Hureyra by simple farming, but inhabitants could not subsist in the same area from foraging alone (Moore et al., 2000, pp. 50, 496).

The dense population of Abu Hureyra and absence of other contemporary large sites in the vicinity (Akkermans and Schwartz 2003, p. 58) is relevant to the integral themes of this study, to understand more comprehensively how people managed and interacted with their local environment. The possibility that more mobile or semi-sedentary groups were drawn to the environs of Abu Hureyra, perhaps following the depletion of resources in more marginal areas, such as the dry steppe, perhaps as a result of increased aridity during the 9.2ka event is suggested in section 11.4.1. It's possible that the inhabitants of Abu Hureyra, who relied heavily on their local resource base to maintain their built environment, felt a sense of ownership of local resources. Therefore, immigrants to the region may have been encouraged to join and contribute to the economy and lifeways of the settlement, rather than being allowed to encroach on and exploit the resources on which the site depended, thereby providing a reason why no other large contemporary settlements developed close by.

The small scatters of flint, and potential small or temporary sites identified close to Abu Hureyra (Wilkinson and Moore 1978), could have been deposited by more mobile groups moving through the area. In which case, the scenario proposed above, whereby the people of Abu Hureyra were protective of their local environment would be redundant. However, evidence of small, short-lived settlements, could also represent temporary base camps used by the inhabitants of Abu Hureyra during hunting trips or expeditions to collect resources. Sources of gypsum, for example, may have been a full day walk from the site (Figure 8.1a), and therefore, to collect the significant quantities required for building efficiently, moderate size teams of people may have been away from Abu Hureyra for several days or weeks. At Çatalhöyük, for example, integrated geoarchaeological and phytolith evidence led Roberts and Rosen (2009) to suggest crops such as wheat were grown 13km from the site on dryland soils and propose a seasonal fission-fusion population model whereby groups may spend part of the population away from the site, to explore a broader range of ecological zones and resources (Roberts and Rosen, 2009). This model has been critiqued as it is largely based on the low numbers of silicified wheat phytoliths, suggesting rain fed cultivation rather than flood plain cultivation, as studies have suggested increased silicification of wheat cells with increased irrigation (e.g. Rosen and Weiner, 1994; Jenkins et al., 2016). This evidence is not robust given the broad range of taphonomic factors which may affect the numbers of multi-cells or degree of silicification within a phytolith assemblage (Chapter 2.2, Jenkins, 2009; Jenkins et al., 2016; Shillito, 2011; 2013). However, at Abu Hureyra, the idea of some fission and fusion is plausible given the widespread use of gypsum, and other materials which may have been several days walk away, and not easily "trade" items (Figure 8.1).

Although Abu Hureyra had the potential to subsist independently from other sites in the region, there was contact with groups in the local and wider region. More general traits characteristic of the PPNB are shared between most moderate to large sites of the period, including rectilinear mudbrick architecture, increased plaster manufacturing, burial practices, and, broadly, intensification in the use of domestic plants and animals (Cauvin, 2000; Asouti, 2006). More specifically, in the Middle Euphrates sites, Tell Halula, Bouqras and Abu Hureyra all share some specific lithic technologies including the off-set bi-directional strategy, which was used to produce high quality blades (Borrell and Molist, 2014). Between the same sites there are cultural and economic variations, for example in plaster manufacture. At Tell Halula, lime plaster has been identified both in architectural materials and as a mastic (Molist et al., 2006; Molist, 2013) while at Abu Hureyra all plaster tested so far has been identified as gypsum (Kingery et al., 1988; le Mière, 2000, this study: Chapter 8). As at Abu Hureyra, gypsum plaster was also used in some floor manufacturing at Tell Halula, particularly those with burials under the floors of houses which was common practice at both sites (Moore et al., 2000; Molist, 2013). On the other hand, inhabitants of both Tell Halula and Abu Hureyra placed a high value on making their buildings and lived spaces aesthetically pleasing, through painting designs onto plaster walls and floors (Molist, 1998; Moore et al., 2000, p. 198, fig. 8.9). Moreover, replastering events could have been connected with temporal rhythms, and as has been suggested at Çatalhöyük have high cultural and symbolic significance (Boivin, 2000; Matthews, 2005).

Archaeological evidence from Tell Halula compared with Abu Hureyra suggests that while both sites had access to long distance trade networks, items they sought out differed. Cowrie shells, for example, sourced from the coast, have been recovered from Tell Halula in high densities, often from burials and used as ornaments (Alarashi et al., 2018), but were very rare at Abu Hureyra, where other exotic materials were also relatively rare (Moore et al. 2000). Abu Hureyra's location, in the central fertile crescent, and just a few days walk from the central Levant through the El Kum pass, is favourable for trade, therefore the low densities of exotic materials are likely a cultural choice made by the inhabitants and demonstrates just one of many cultural preferences between sites within the same region.

Gypsum plaster was widely used at Abu Hureyra for construction and utilitarian artefacts such as storage vessels (Chapter 8), but also for covering burials (Moore et al., 2000). This practice was shared with sites spanning a large geographic region and time frame, including PPNA Wadi Faynan in the southern Levant (Mithen et al., 2018) and Kortik Tepe, a PPNA site located on the Tigris in south-eastern Anatolia (Benz et al., 2018). The PPNB is significant as it represents the widespread and intensive use of plaster in construction, which would have required an unprecedented use of resources compared with small quantities required in preceding Natufian and PPNA periods as a mastic and in burials. On the surface, the manufacture and application of plaster for utilitarian functions (e.g. construction material and storage vessels) in the PPNB, may indicate a shift away from conserving plaster, which is energy, resource and labour expensive (section 8.2) for purely special, ritual or symbolic functions. However, at Çatalhöyük, it's been suggested, that wall and floor sequences are intrinsically linked with ritual events, based on the identification of multiple sequences through micromorphology and ethnographic observations from households in rural Rajasthan (Matthews, 1996; Boivin, 2000). Furthermore, also based on archaeological evidence from Çatalhöyük as well as ethnographic observations in SW Asia, Kuijt highlights that ancestors are embedded within the structures, as under-floor burials, and that the spaces therefore held social associations, through multiple generations (Kuijt 2018). Human burials within buildings which are interpreted as households are also a common feature at Abu Hureyra (Molleson 2000; Molleson and Moore 2000). Therefore, the increased use of plaster in the PPNB, may, rather represent the increasing symbolic, cultural and ancestral place of the household, rather than a shift for the use of such a resource intensive material for purely mundane or functional purposes.

The inhabitants of Abu Hureyra likely selected the site for the advantageous access to the range of resources that were available in this vicinity at the border of several ecozones that included riparian valley bottom vegetation, steppe grasslands, park woodland and terebinth woodland steppe (Chapter 3, Figure 3.3, Figure 7.1a). Ecological uncertainty, is therefore unlikely to have been a major concern, however, there may have been some fluctuations in availability caused by the 9.2ka event (discussed above) and growth of the population, although no significant changes are visible in the proxies analysed in this study.

Even relatively minor ecological changes could have provided opportunities for innovation within a panarchy theory framework (Gunderson and Holling, 2001; Holling, 2001), in which the rich resource base available in the environs of Abu Hureyra could have provided opportunities for innovation and experimentation (Zeder, 2012). The variety and abundance of resource availability are likely to have provided at least some buffer against climate and environmental changes which may have influenced local vegetation patterns (Jones et al., 2019). The substantial nature of the settlement combined with ecological evidence from the charred plant remains indicate that Abu Hureyra was likely occupied through the year, and therefore inhabitants were already familiar with adapting their plant resource acquisition strategies between seasons, including preserving and storing food for times of hardship. During Neolithic period 2A, non-migratory species declined (wild cattle, pig, fox, hare), a trend which started in the Epipalaeolithic phase of the site, and has been attributed to hunting pressure of the increasing population (Legge and Rowley-Conwy, 2000, p. 462).

However, despite the diversity of habitats available for animals, the faunal records show very little use of fish, birds or shellfish, throughout the occupation of the site, indicating that inhabitants preferred to focus on large game with high returns during the Neolithic, rather than a broad spectrum of animal protein (Moore et al. 2000, p. 480). This could also suggest that through the sustainable management of resources inhabitants never had a need to broaden their dietary repertoire. In contrast to Abu Hureyra, at Aşıklı Höyük, a broad range of animal resources were utilised prior to the adoption of managed caprines (Itahashi et al., 2021; Stiner et al., 2014). The development of Abu Hureyra close to a major Persian gazelle migration route (Legge and Rowley Conwy 1987) could have facilitated the community's choice of wild larger game over smaller and more diverse prey. Although even once managed caprine became the dominant meat source in Period 2B, gazelle retained some significance for the inhabitants, testified by the very small and intricate carving of a gazelle head from Trench B, phase 10 (Moore et al., 2000, p. 505, fig. 14.6), Figure 11.2. Plants too may have held cultural significance for the people of Abu Hureyra, which in some instances could have encouraged the continued use of the same plant resources, despite opportunities to change.

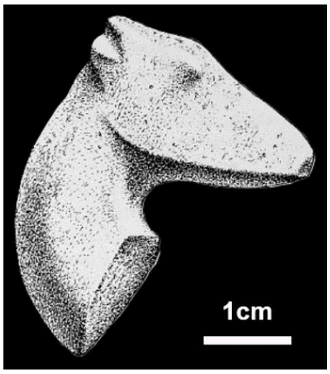


Figure 11.2 Figurine of a female Persian Gazelle worked onto a granite pebble recovered from Trench B, phase 10. Adapted from Moore et al., 2000, p. 505, fig. 14.6

At Aşıklı Höyük*,* changes in the built environment have been attributed to the rise in managed animals, whereby more substantial structures were constructed, for example, an increase in wall heights in response to destructive climbing and trampling by goats (Stiner and Kuhn, 2016). Changes in the built environment are also seen at Abu Hureyra which could have been instigated in response to the increased numbers of managed animals at the site. In Trench E, for example, the plan of the main building constructed in period 2B, phase 4, is respected through 3 further phases of occupation (phases 5, 6 and 7). However, in phase 6, the building is downsized (one less room) which increased the space between buildings, and the external walls are thickened (Moore et al., 2000, p. 236). The channel which ran through the space between buildings in Trench E was hypothesised to have been formed by animals being routinely herded through the site, which has been further substantiated in this study by the particularly high concentrations of spherulites identified in occupation residues from the same area (Figure 7.6). It therefore seems likely, in this instance, that the changes to construction practices, were related to the increased presence of managed animals on site. Increased spacing between buildings is also noted in Trench B during period 2B, and in both trenches this increases even more substantially in the final Neolithic phase of occupation, period 2C (Moore et al., 2000, pp. 203, 208). As the settlement grew, the need for secondary animal products may have increased, for example dung for construction as a material of choice (Sherratt, 1983; Marciniak, 2011), although as hypothesised at Aşıklı Höyük, dung used in buildings could have been a response to a build-up in waste from more animals being kept on and around the site (Stiner and Kuhn, 2016).

At Çatalhöyük, Anatolia, through utilisation of a panarchy theory framework and decades of intensive and precise archaeobotanical research, Bogaard et al. have suggested that flexible cropping regimes were a key factor which promoted the long-term sustainability and resilience to account for the longevity of the site (Bogaard et al., 2017). Long-term trends are clear during the Neolithic occupation of Abu Hureyra which shows remarkable continuity over multiple generations and phases of occupation, whereby houses continue to be built on the same alignment from the same materials, treatment of the dead follows the same rituals and the exploitation of plants does not appear to change significantly, demonstrated by the phytoliths in this study (Chapter 7) and charred macro-fossils (de Moulins, 2000). However, equally significant are the more subtle variations between trenches and households which could suggest that residents of Abu Hureyra may also have benefitted from flexibility which would have enabled small scale innovation, possibly at the household or neighbourhood and community scale (Chapter 8, Chapter 10). The flexibility at a household level, would have contributed towards the site's long-term sustainability, and increased resilience to changes in the balance of resources caused by fluctuations in climate or population.

Chapter 12 Conclusion and Future Directions

12.1 Integrated conclusion

The key aim of this PhD research was to provide new perspectives on plant-use and resource management as agricultural societies developed during the PPNB. This study has provided new information and the types and varieties of plants used at Abu Hureyra through the analysis of phytoliths (Aim 1i). Phytolith analysis of occupation residues identified a range of different vegetation types used throughout the occupation of the Abu Hureyra. In combination with the charred macro fossil record, the phytoliths analysed in this study, suggest the inhabitants of Abu Hureyra made full use of the rich resource base and diversity of ecozones surrounding the site and were able to sustainably manage the resources over the 2500-year occupation of the site (Chapter 3, section 3.2).

The differences between the representation of different plant categories in the phytolith and charred macro fossil records demonstrates the value of adopting a multi-proxy integrated archaeobotanical approach to provide to study plant utilisation and ecology. This study has identified that many occupation deposits at Abu Hureyra likely contained a faecal component, based on the positive identification of faecal spherulites (Aim 2i). The identification of dung in archaeological assemblages is significant for the interpretation of macro and micro remains which can represent a great diversity of plant uses and depositional pathways (Miller, 1996; Spengler, 2018).

The analysis of construction materials, with a focus on floor plasters at Abu Hureyra has provided new information about resource use and possible developments over time (Aim 3). The ubiquitous presence of faecal spherulites in plasters shows that animal dung was a well-used and valuable resource. It remains unclear whether the increasing uses for secondary animal products encouraged the intensification of animal management on the site. Following increasing numbers of animals in and around the site, and therefore, increased animal waste, the inhabitants of Abu Hureyra may have selected animal dung in response to an increasing waste management problem. Variations in composition of plasters, identified by phytolith analysis and pXRF, between those recovered from different buildings, spaces and phases hint at flexible manufacturing practices which may have been adapted at a household level. The selection of different materials likely had both cultural and economic factors which a part in the significant change in human-plant-animal-environment dynamics as agricultural societies developed.

12.2 Future directions

12.2.1 Methodological considerations

As shown in the increasing range of phytolith analyses at Epipalaeolithic and Neolithic sites across SW Asia (e.g. Rosen, 1992; 2005; Jenkins and Rosen 2007; Portillo et al., 2010; 2014; Nadel et al., 2013; Shillito and Elliott, 2013; Shillito et al., 2013b; Power et al., 2014; Ramsey et al., 2016; Elliott et al., 2020), phytoliths provide a valuable medium through which to identify diverse uses of plants in archaeological environments. Archived bulk sediment samples have enabled phytoliths from Abu Hureyra to contribute to the growing body of phytolith research, at a site which is no longer accessible. However, stronger, more specific interpretation could be made if the contextual resolution of sampling was higher. Further studies of phytoliths during new archaeological excavations could use statistical analysis to assess how the phytolith assemblage from a sub sample, retrieved from a bulk sample recovered for flotation compares with a higher spatial resolution of sampling specifically for phytoliths to provide a better interpretative framework for archived samples. The recovery of blocks for micromorphological analysis would also help to identify and clarify specific relationships between diverse plant materials and parts and other materials (Matthews, 2005; 2010; Roe, 2007; Shillito et al., 2011; Matthews et al., 2013; Banerjea et al., 2015; Elliott et al., 2020). An initial aim of this study was to analyse micromorphology thin sections from fire installations and other ashy or burnt deposits, with paired sub samples for phytoliths and spherulites, to explore plant-use, particularly as fuel, phytolith taphonomy and the representation of different plant types. These new analyses could help to further clarify the diverse uses of phytoliths extracted from ashy bulk samples.

The identification of faecal spherulites can be conducted rapidly with minimal equipment (Matthews, 2010; Portillo and Matthews, 2020), and, providing preservation conditions are favourable, provides a rapid indicator of the presence of ruminant dung (Canti, 1999), which could be utilised more widely in archaeological research. This study has demonstrated that new research is needed to build a better analytical and interpretative framework for spherulite quantification which can vary considerably between different methods. Significantly fewer spherulites were observed following processing methods, such as density separation to remove heavy particles which may obstruct counting, and experimental work on rates of faecal spherulite dissolution may also help to understand differences between methods (Aim 4, Chapter 6).

Aspects of faecal spherulite formation and taphonomy require further research to provide a better interpretive framework. This research builds on previous ethnographic work and demonstrates the variability between proportions of darkened spherulites in different dung samples, even when subjected to the same burning temperatures or from the same species (Canti and Nicosia, 2018; Portillo et al., 2020b). New ethnographic data sets will provide some clarification about how species, diet and environment affect the production and preservation of faecal spherulites. This study has demonstrated the potential of using the proportions of darkened spherulites as a screening method for GC-MS. Further work with an increased number of samples and experimental burning temperatures, would help to refine our understanding of how darkened spherulites may be used to screen for techniques where burning temperature may have affected preservation.

pXRF is a rapid and non-destructive method which has the potential to inform on different material types and activity areas in archaeology (Wilke et al., 2016; Jenkins et al., 2017; Cannell et al., 2018; Vos et al., 2018; Williams et al., 2020). As a screening method, pXRF has the potential to aid the selection of materials for more detailed, time consuming and possibly destructive analysis (Shillito, 2017; Elliott et al., 2020). This study has highlighted some of the complications in using pXRF as a screening method for bulk samples of mixed material, likely deposited by a range of human activities, as no correlations were identified between specific elements and the concentration of phytoliths or spherulites. The geochemical characterisation of plasters by pXRF was more promising, with positive relationships identified between key elements (P and Si) and concentrations of spherulites and phytoliths respectively. Elemental data generated through pXRF can also provide the basis for analyses to compare similarities and subtle variations not apparent at a micro or macro scale, demonstrated in this study by the PCA analysis of plaster fragments. However, new research to generate more archaeological reference materials would increase the interpretative value of pXRF.

12.2.2 Future directions for the Abu Hureyra archives

The archive of material from Abu Hureyra continues to provide a valuable resource through which to explore emerging themes. This study has established moderate to good preservation of phytoliths in sediments and plasters materials. A more detailed study of the phytolith composition of different materials (white ware vessels, different plasters, clay balls) could provide new information about the selection of different plant types for different purposes. Given the ubiquity of faecal spherulites identified in this study, it is suggested that all further studies scan a smear slide (as described in Chapter 5) in cross polarising light under a microscope to identify if dung is a potential component of the material.

An original aim of this research was the analysis of phytoliths from ground stone tools, which it was not possible to take forward due to the COVID-19 pandemic and restricted access to collections. If preserved, the analysis of phytoliths from ground stone tools has the potential to inform more specifically on consumption practices (Portillo and Albert, 2014). The dental calculus of human and animal teeth, also has the potential to inform more specifically on diet (Henry et al., 2011; Juhola et al., 2019), compared with the phytoliths analysed from the predominately ashy occupation residues analysed in this study. The analysis of phytoliths from materials such as ground stone tools and teeth which span both the Epipalaeolithic and Neolithic periods of occupation of the site provide exciting new avenues of research to assess the continuity and changes in human economy between these key periods of human history (Arranz-Otaegui et al., 2016; 2017; 2018; Bar-Yosef, 2017).

The analysis of charcoal fragments, as a sub-category of the charred macro-fossil assemblage, from bulk samples from the Neolithic period of occupation at Abu Hureyra would provide new information about the uses of plants, particularly the selection of fuel (Asouti, 2005). Changes in the uses of different types of wood may hint at changes in environmental conditions (or absence of change) at a site-specific level where there are no palaeoenvironmental archives (Shackleton and Prins, 1992; Asouti and Austin, 2005; Roberts et al., 2018). Charcoal from Abu Hureyra 1 has been analysed and published (Roitel and Willcox, 2000), however, to date, charcoal from the Neolithic period of occupation at Abu Hureyra has not been published. The analysis of Neolithic charcoal would also provide a new dimension through which to compare the Epipalaeolithic and Neolithic periods of occupation at Abu Hureyra. This is important as the Epipalaeolithic and Neolithic phases of the site are inhabited under different climatic conditions which would have impacted the availability of plant resources (Moore and Hillman, 1992; Moore et al., 2000; van der Plicht et al., 2004; Maher et al., 2011; Flohr et al., 2016). Therefore, comparing the two periods of occupation would inform on the management practices of resources during different or changing environmental conditions (Hillman et al., 2001).

This study piloted a rapid and cost-effective way to assess construction material through integrated phytolith, spherulite and pXRF analysis (Chapter 8). Further clarification could be provided into the specific techniques through the analysis of thin sections and SEM-EDX of the plaster fragments, which have the analytical potential to inform how dung was incorporated into the plaster matrix (Matthews, 1995; Matthews et al., 1996, 2013). Further plaster analysis would benefit greatly from ethnographic observations and experimental plaster manufacturing to provide a more robust baseline for archaeological interpretations (Gur-Arieh et al., 2019; Amicone et al., 2021).

Cultural heritage is currently threatened by a multitude of different factors including climate change, dam construction, conflict and loss of funding (Marchetti et al., 2019). Dam construction has resulted in the loss of numerous discovered and unknown archaeological sites (Cunliffe et al., 2012).

For this reason, archaeological archives are a valuable resource which hold the potential to continue research to address current questions in key areas of the world.

Furthermore, as new archaeological research methods are developed and new material stimulates new archaeological theories and frameworks to understand the complex interactions between people, plants, animals and the environment, the Abu Hureyra archives, housed in museums and institutions across three continents provide valuable research material.

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Appendices

Appendix 1. Abu Hureyra database of samples

1A: Soil and occupation residues

1B: Plasters and miscellaneous materials

Appendix 2. Abu Hureyra radiocarbon dates

2A: Calibrated radiocarbon dates for Abu Hureyra

2B: Dates for Trench specific phases

Appendix 3. (Chapter 5, SI 1) Quantitative spherulite results

Appendix 4. Phytolith Data

4A: Quantitative phytolith and spherulites data

4B: Phytolith morphotype data

4C: Phytolith vegetation types and plant parts

Appendix 5. pXRF data for soil samples

Appendix 6. Principal Component Analysis data for soil pXRF

Appendix 7. Other bio microfossils and graphs for phytolith types in Trenches A and C

Appendix 8. (Chapter 8, SI 1)

8A: Close up images of plaster a-d

8B: Images of other material types analysed

Appendix 9. (Chapter 8, SI 2)

9A: Context information and spherulite counts for all non-floor plasters

9B: Context and sample info for material used in PCA (include PPM)

9C: Phytolith morphotype analysis (%) data

9D: Proportions of grasses, dicots and wetland derived phytoliths

Appendix 10. GC-MS data

10A: Sterols

10B: Bile Acids

Appendices are available electronically via the following link:

https://livereadingac-my.sharepoint.com/:f:/g/personal/cj804455_student_reading_ac_uk/EkVAvG0y9eVNmai_A3V7_IABgflOJy5lqetQuepvK-p4gQ?e=Be3Ieg