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Reconstructing agro-pastoral practice in the Mesopotamian-Zagros borderlands: Insights from phytolith and FTIR analysis of a dung-rich deposit

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A B S T R A C T

Understanding everyday agro-pastoral practice is critical for reconstructing the formation and maintenance of ancient societies. The ancient Near East (Southwest Asia) has one of the longest histories of agro-pastoral practice and one of the richest textual datasets anywhere on the globe. Yet, our knowledge of local, day-to-day agro-pastoral management strategies remains conjectural in many regions of Southwest Asia during the Bronze Age (late 4th–2nd millennium BCE). In this study we used phytoliths, dung spherulites, and Fourier Transform Infrared (FTIR) spectroscopy to identify and examine dung-rich sediments from Khani Masi, a mid-second millennium BCE Kassite site located in the Kurdish Region of Iraq. While micro-remain and geochemical approaches have not yet been widely applied in Mesopotamia (Ancient Iraq), they have the potential to shed light on the production systems supporting its Bronze Age cities, states, and empires. Our aim was to investigate (1) the range of local pastoral management strategies, (2) the degree of integration between agricultural and pastoral practice, and (3) the presence of signals related to the local ecology, seasonality, and environmental change and continuity.

Phytolith results indicate that sheep-goat herds were primarily free grazed on wild grasses. The dominance of wild grass inflorescences, a potentially strong seasonality indicator, may suggest transhumant pastoralism. However, further evidence, including occasional foddering with cereal chaff, a diverse range of crop types, and significant accumulation of burnt dung within the site, collectively suggests a closely linked local agro-pastoral subsistence economy. This study provides much-needed empirical botanical data as well as productive insights for future application of phytolith studies in the Mesopotamian region, and sheds new light on agro-pastoral practice in the Zagros foothills during the second millennium BCE Kassite period.

1. Introduction

Reconstructing agro-pastoral practice is critical for understanding the formation and maintenance of ancient societies—from everyday practice to statecraft. In the ancient Near East (Southwest Asia), most archaeologists agree that Bronze Age subsistence and political economies were largely based on flexible mixtures of cereal cultivation and animal husbandry that could vary considerably between historical periods and across the region's diverse social and environmental landscapes. However, archaeologists continue to actively debate to what degree sheep-goat pastoralism was site-based, transhumant, or specialized (e.g., Alizadeh, 2010; Arbuckle and Hammer, 2019; Cribb, 1991; Porter, 2012; Potts, 2014; Riehl, 2006; Sallaberger, 2014; Wilkinson et al., 2014; Wossink, 2009) with significant effects on models of Bronze Age subsistence, economies, environmental resilience, and socio-political relationships. Despite widespread scholarly interest in these questions, archaeologists largely lack robust and integrated eco-factual data regarding pastoral practices (cf. Arbuckle and Hammer, 2019;

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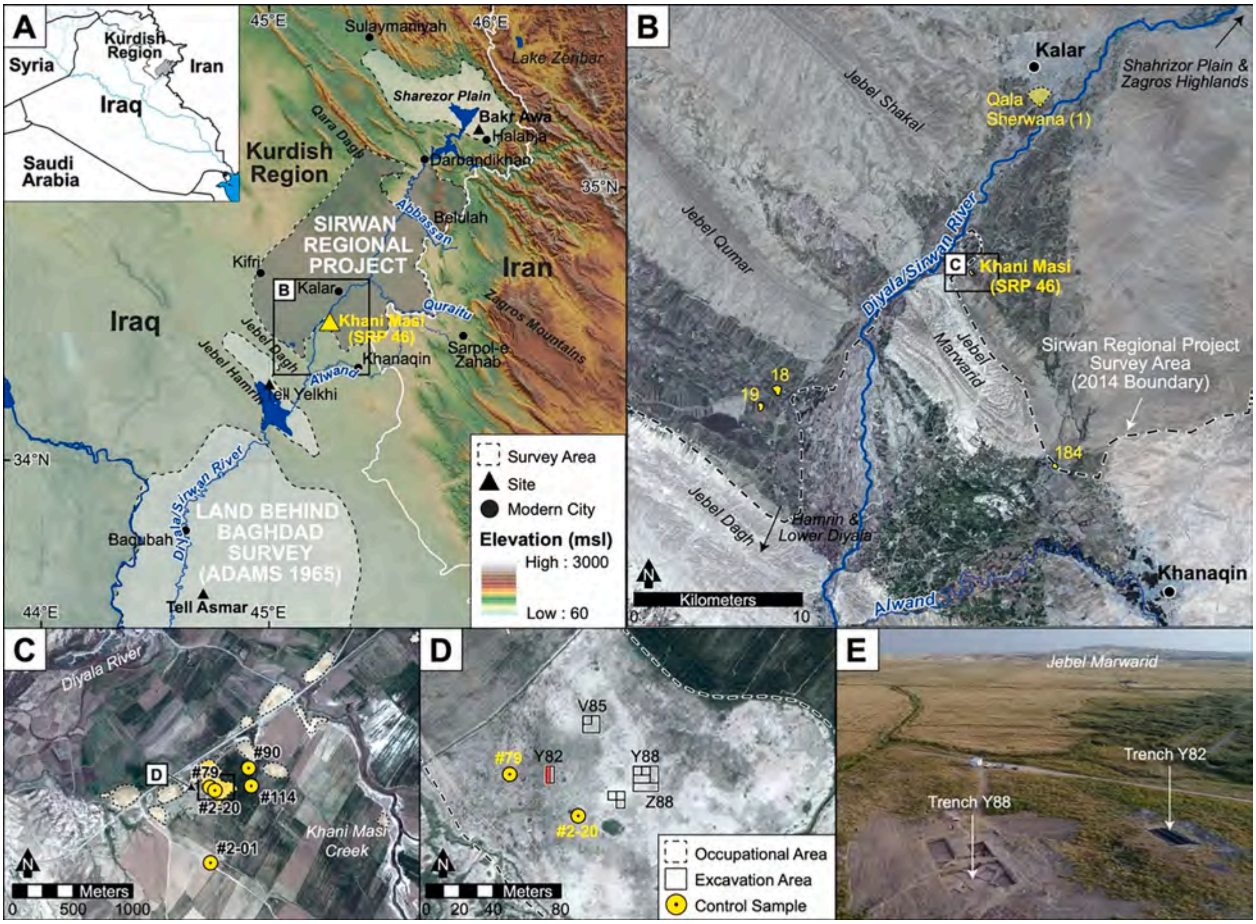

Fig. 1. (A) Map of the Sirwan (Upper Diyala) River Region in Northern Iraq. (B) Location of Khani Masi and other Kassite period sites (Glatz et al., 2019; Glatz and Casana, 2016). (C) Map of the Khani Masi site cluster indicating local perennial water sources and locations of control samples (yellow circles) (modified from Casana and Glatz 2016, 2011 GeoEye, © DigitalGlobe 2015). (D) Location of Y82 and surface control samples relative to other excavation areas (after Glatz et al. 2019). (E) Aerial photo of Y82 facing southeast from May 2019. Imagery and basemap sources for A-B: Esri, DigitalGlobe, GeoEye, i-cubed, USDA FSA, USGS, AEX, Getmapping, Aerogrid, IGN, IGP, swisstopo, and the GIS User Community. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

but see Miller, 2013; Miller et al., 2009; Riehl, 2006; Smith and Munro, 2009).

Several emerging analytical methods, including isotopic analysis of dental collagen (e.g., Makarewicz and Tuross, 2012; Makarewicz, 2014a; Makarewicz and Sealy, 2015) and multiproxy dung analysis (e.g., Dunseth et al., 2019; Shahack-Gross et al., 2014) are beginning to offer insights into past human and animal diet, mobility and transhumant movement, and pastoral integration into agrarian economies. Animal dung, in particular, offers a robust tool for investigating agro-pastoral practice, and a number of recent studies have demonstrated the power of multiproxy and microscopic dung analyses to shed light on animal management strategies (i.e., animal diet, foddering, pasturing, penning, fuel use, and seasonality), plant-animal-human relationships, and local landscape and environmental conditions (see recent reviews by Fuks and Dunseth, 2020; Gur-Arieh and Shahack-Gross, 2020; Portillo et al., 2020b; Smith et al., 2019; Spengler, 2019). In Southwest Asia, dung was likely a significant source of fuel (Gur-Arieh et al., 2014; Miller and Smart, 1984; Miller, 1996, 1984a, 1984b, 1982; Miller and Marston, 2012; Smith et al., 2019) as well as fertilizer (Wilkinson, 1989, 1982), but many archaeologists continue to overlook dung fuel as an essential secondary product and as a primary source of information for examining agro-pastoral practice (Lancelotti and Madella, 2012).

Excavations at Khani Masi, a mid-second millennium BCE site located along the Upper Diyala/Sirwan River in the Kurdistan Region of northern Iraq, recently uncovered a large dung-rich deposit that provides an opportunity to investigate local agro-pastoral practice in the Zagros foothills during a period when the region may have been incorporated into Kassite imperial networks (Fig. 1). The Kassites, widely believed to have been an ethnic group originating somewhere in the Zagros Mountains, ruled Southern Mesopotamia and surrounding regions from around 1550–1150 BCE (Liverani, 2014; Sassmannshausen, 1999; Sommerfeld, 1995; Stol, 1976). Despite being one of the major powers in Southwest Asia for nearly 400 years, we know surprisingly little about Kassite culture, political economy, or subsistence practices (Brinkman, 2017; Paulus, 2013; 2011), particularly in the Zagros piedmont steppe zone: the interface between the Mesopotamian lowlands and the Zagros highlands (Fuchs, 2017; Glatz et al., 2019). Prior to recent work at Khani Masi, we could only speculate the range of agro-pastoral strategies in this region, which might range from highly specialized pastoral mobility (i.e., Alizadeh, 2010; Porter, 2012) to highly integrated site-based herding (i.e., Arbuckle and Hammer, 2019; Potts, 2014), or how strategies might be affected by Kassite imperial networks (Rosenzweig and Marston, 2018; Scott, 1985).

In this study we use phytolith analysis, dung spherulites, and Fourier Transform Infrared (FTIR) spectroscopy to identify and examine the dung-rich sediments at Khani Masi. Our goal was to use micro-remain and geochemical analyses to answer fundamental questions about agro-pastoralism in Bronze Age Mesopotamia: (1) What is the range of local pastoral management strategies? Were animals grazed or foddered? (2) What is the degree of integration between agricultural and

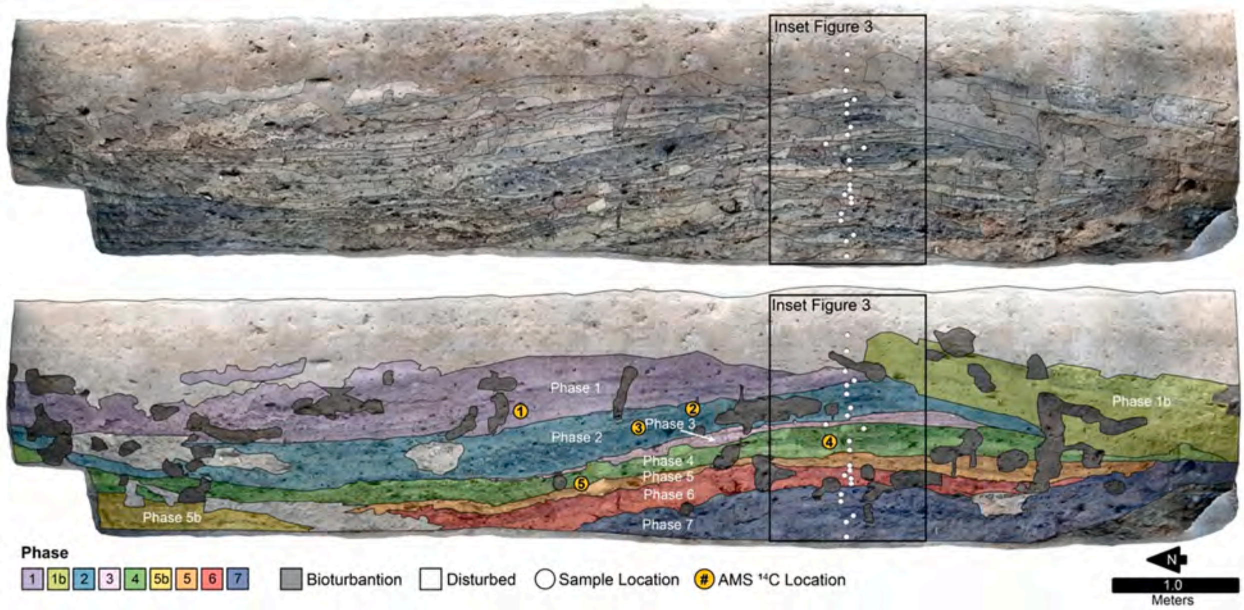

Fig. 2. Eastern profile of trench Y82 west indicating stratigraphic layers (upper) and phases (lower). White circles indicate sediment sample location for this study. Yellow circles indicate the approximate locations of excavated AMS radiocarbon samples (Table 1). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

pastoral practice? Was local pastoralism site-based, transhumant, or variable? (3) Do phytoliths capture signals related to the local ecology, seasonality, and environmental change and continuity?

Our results indicate that local herds were primarily grazed on wild grass and only occasionally foddered with agricultural byproducts. Phytoliths from dung rich layers also exhibit a strong seasonality signature and higher proportions of C_4 grasses than surrounding fill layers indicating an abundance and diversity of local pastureland. Finally, the large size, location in the interior of the site, and burned status of the dung deposit at Khani Masi highlights the local importance of dung fuel and suggests that pastoralism was highly integrated into the local economy. Results from this study provide much-needed empirical (ecofactual) data for the region, offer productive insights for future regional phytolith studies, and shed new light on agro-pastoral practice in the Mesopotamian-Zagros borderlands during the second millennium BCE Kassite period.

2. Background and site description

2.1. Previous phytolith and micro-remain studies

Phytoliths are microscopic inorganic opaline silica (SiO_2) molds of plant cells and intercellular spaces that form from monosilicic acid (H_4SiO_4) in ground water (Piperno, 2006). In contrast to macrobotanical remains, inorganic phytoliths are typically well preserved and ubiquitous within archaeological sites (Katz et al., 2010). Phytolith concentrations in ashed sheep-goat dung range in the tens of millions per gram: orders of magnitude higher than non-archaeological sediments (e.g., Dunseth et al., 2019; Gur-Arieh et al., 2013; Portillo et al., 2020a). While there are multiple approaches for identifying animal dung, the presence of dung spherulites is the strongest single, unequivocal indicator for the presence of ruminant dung (Gur-Arieh and Shahack-Gross, 2020). Dung spherulites are microscopic calcite spheres (5–20 μm) produced in the intestines of many ruminant species but most abundantly in sheep and goats (Brochier, 1983; Canti, 1997; Shahack-Gross, 2011).

Integrated microbotanical and geochemical methods are well suited for investigating agro-pastoral lifeways (e.g., Albert et al., 2008; Burguet-Coca et al., 2020; Cabanes et al., 2009; Portillo et al., 2019; Shahack-Gross et al., 2014; Tsartsidou et al., 2009). However, there are only a handful of published phytolith studies from Iraq, and they primarily focus on the Neolithic and earlier periods (Asouti et al., 2020; Cummings et al., 2018; Elliott et al., 2020b; Matthews et al., 2020; but see Marsh (2015a) unpublished dissertation), ethnographic work (Elliott et al., 2015, 2020a; Portillo et al., 2020a), or off-site geoarchaeological sequences (Altaweel et al., 2019; Marsh et al., 2018; Rabbani et al., 2020). Studies from regions adjacent to Mesopotamia (i.e., Syria, Turkey, and Iran) also primarily focus on the Chalcolithic and earlier (e.g., Hart, 2014; Matthews et al., 2013; Portillo et al., 2014; Shillito and Elliott, 2013). These approaches have the potential to fundamentally transform our understanding of the relationship between Mesopotamian Bronze Age economies and environments (Marston, 2021). To our knowledge, this study represents the first integrated phytolith, dung spherulite, and FTIR analysis of a Bronze Age deposit from the Kurdish Region of Iraq.

2.2. Site description: Khani Masi

This study focuses on Khani Masi, an archaeological site located along the Diyala/Sirwan River in the Zagros piedmont zone of the Kurdish Region of Iraq, characterized by a cluster of variably mounded occupation areas covering >50 ha (Fig. 1A-C). Although the site has minor evidence of both earlier and later settlement, the majority of Khani Masi was occupied during the second millennium BCE, with its most extensive settlement during the Kassite period (1550–1150 BCE). The site cluster is situated on the eastern bank of the Diyala/Sirwan River at the nexus of the Diyala River terrace, the Khani Masi agricultural plain, and the Jebel Marwarid—one of a series of NW-SE anticline foothills in the Zagros front range (~190 m.a.s.l; Fig. 1B).

Climatically, the Khani Masi region is situated in a narrow strip (~120 km) of hot arid steppe (BSh climate zone; Kottek et al., 2006) near the precipitation limit for reliable agriculture (~350 ± 130 mm/year) (Schneider et al., 2020; Wilkinson, 2000). Local vegetation is categorized within the Mesopotamian dry steppe sub-region of the Irano-Turanian system (Zohary, 1973). Like much of Northern Iraq, the steep environmental gradient and complex local topography along the Diyala/Sirwan River create an ecological mosaic ideal for a range of mixed agro-pastoral subsistence strategies. The hillsides are covered in local wild grasses (e.g., *Poa bulbosa s.l.*, *Aegilops* speltoides, and *Hordeum*

Table 1
Accelerator mass spectrometer radiocarbon dates of charcoal samples from Khani Masi (SRP 46). Dates were calibrated using OxCal v4.4.2 (Bronk Ramsey 2020) using Reimer et al. (2020) atmospheric data. Study number corresponds to sample locations (yellow circles) in Fig. 2.

Lab No.	Provenience (Field No.)	Study Number	Phase	Material Type	Carbon Yield (%)	$\delta^{13}C$ (‰)	^{14}C Age (uncal. BP)	Calibrated Range B.C.E. ± 1σ (68.2%)	Calibrated Range B.C.E. ± 2σ (95.4%)	Period
AA114853 / X36458	Area Y82, locus 9/lot 2	1	1	charcoal	54.0	−26.2	3113 ± 30	1426–1311	1446–1285	Middle Babylonian
AA114851 / X36456	Area Y82, locus 7/lot 3	2	2	charcoal	43.5	−26.3	3043 ± 41	1387–1229	1416–1133	Middle Babylonian
AA114866 / X36471	Area Y82, locus 9/lot 3	3	2	charcoal	45.4	−27.1	3077 ± 30	1403–1296	1421–1261	Middle Babylonian
AA114856 / X36461	Area Y82, locus 18/lot 2	4	4	charcoal	54.3	−27.8	3150 ± 27	1492–1403	1499–1320	Middle Babylonian
AA114857 / X36462	Area Y82, locus 20/lot 4	5	4	charcoal	54.1	−26.7	3118 ± 29	1430–1313	1488–1292	Middle Babylonian

bulbosum; interspersed with herbaceous and flowering plants) (Ghazanfar and McDaniel, 2016) suitable for grazing flocks of sheep and goats while the agricultural plains are fed by irrigation from the Diyala/Sirwan River as well as a number of perennial springs (Casana and Glatz, 2017).

Khani Masi was first investigated by the Sirwan (Upper Diyala) Regional Project (SRP) in 2013, with geophysical surveys and excavation between 2014 and 2019. Results show that the region had close cultural connections to Kassite Babylonia (Casana and Glatz, 2017; Glatz et al., 2019; Glatz and Casana, 2016; Perruchini et al., 2018) and that Khani Masi was one of a series of sites that may have formed the northern perimeter of Kassite control (Fig. 1B) (Glatz et al., 2019). In 2019, excavations in Khani Masi Area Y82 uncovered a large (>10 × 5 m^2), ~2m deep dung-rich deposit consisting of alternating layers of organic rich, black-gray layers and brown, orange-red, and white sediments (layers slope from SW down to the NE; Fig. 2). The deposit closely resembles a midden or *fumier* (burned animal pen accumulation; Angelucci et al., 2009). Both on-site animal pens and middens with substantial dung accumulations have parallels across Western Asia including numerous sites in the Konya Plain, Turkey (Matthews, 2005; Portillo et al., 2019; Shillito and Matthews, 2013; Shillito and Ryan, 2013), the Khabur Basin, Syria (McCorriston, 1995; McCorriston and Weisberg, 2002), Southern Iraq (Stone, 1987), and Israel (e.g., Albert et al., 2008; Butler et al., 2020; Shahack-Gross et al., 2009; Bar-Ozet al., 2019).

Macro-stratigraphic interpretation of the Y82 profile indicates seven major depositional layer packages, or phases, of varying thickness (Fig. 2). For each of these phases, foundational leveling layers can be traced continuously across the section. Layers of debris appear to have accumulated (and compacted) on each foundational leveling fill before being truncated by the subsequent phase's leveling activity. Only phase 4 (Fig. 2) appears to preserve an active outdoor surface as evidenced by "bedding structures", "fire-spots", and installation-like mudbrick features visible in the profile (Shillito and Ryan, 2013). Five radiocarbon dates place the deposit firmly in the mid- to late second millennium BCE, or Kassite Period (Middle Babylonian) (Table 1).

3. Materials and methods

3.1. Materials: Sample collection

We collected a total of 27 loose sediment samples for this study. Fig. 1C-D shows the location of the 5 control samples as well as the location of excavation trench Y82. We recorded control sample locations and excavation data using an Emlid RS+ RTK GNSS system. Fig. 2 indicates the locations of the 22 samples taken from the eastern section of trench Y82. We collected bulk sediment samples from a freshly cleaned section and placed them in individual plastic bags. Sampling carefully avoided bioturbated areas. Sediment samples were exported to the US with the permission of the General Directorate of Antiquities of the Kurdistan Region of Iraq as well as the Garmian Department of Antiquities. Each sediment sample was desiccated following USDA guidelines before analysis at Rutgers University Anthropology Lab for Micro-Archaeology (ALMA).

3.2. FTIR (Fourier Transform Infrared) spectroscopy

FTIR analyses were performed using a Thermo Scientific Nicolet iS5 FT-IR spectrometer. Approximately 1 mg of sample was mixed with 80 mg of KBr in an agate mill. We obtained infrared spectra between 4000 and 400 cm^{-1} by scanning the samples 32 times at 4 cm^{-1} resolution. We determined the main mineral components of each sample using the wavelengths of the strongest absorption peaks (Weiner, 2010) and referenced the standards from the Kimmel Center for Archaeological Science, Weizmann Institute of Science. We determined whether calcite was primarily geogenic, biogenic, or anthropogenic (i.e., ash) using the grinding curve method established by Regev et al. (2010). We also used the reference generated by Regev et al. (2015) to approximate the percent phosphate concentration.

For this study, we generated new thermal alteration references of local clay and sediment as recommended by Berna et al. (2007) (see details in Appendix A). Finally, we interpreted whether clays in this study were subject to high temperatures following Berna et al. (2007) as well as the unique absorptions in the clay spectrum from the new local thermal alteration references.

3.3. Organic and carbonate content

We assessed total organic content (TOC) using the loss-on-ignition method (Dean, 1974). Approximately 1 g of sample weighted using a Sartorius MSA225S100DI Cubis semi-micro balance (sd: 0.01 mg) and placed into a ceramic crucible. Then the samples were burned with a closed lid at 550 °C for 2 h. After samples returned to room temperature, they were weighed again, and weight lost was calculated as percent soil organic carbon content. We processed ten samples in triplicate to determine TOC sample variability and measurement error. Following Heiri et al. (2001), each of the 27 samples was also heated to 550 °C for 4 h to check additional errors.

Following the TOC procedure, we treated samples with 3N HCl following Albert and Weiner (2001) to determine the acid insoluble fraction (AIF). Weight lost in the acid treatment provides a measure of the percent soluble minerals (hereafter: carbonates) present in the samples including carbonates, phosphates, gypsum, and calcitic dung spherulites. We processed twelve samples three times to determine AIF sample variability and measurement error.

3.4. Microscopy

3.4.1. Dung spherulite concentrations

Using approximately 20 mg of original sediment, we extracted dung

Table 2
Comparison of sediment sample location, minerology, sediment color, and facies type. Main mineral components are ordered by relative peak height in each FTIR spectrum.

Sample	Location	Phase	Elevation	Main Mineral Components*	Calcite Type	Sediment Color Range	Facies
SRP_78	Plough Zone - Y82	Plough Zone	190.47	Cl, Ca, Qz	Anthropogenic (Ash)	Brown-Orange	(Type B)
SRP_77	Plough Zone - Y82	Plough Zone	190.34	Ca, Cl , Qz	Anthropogenic (Ash)	Brown-Orange	(Type B)
SRP_76	Y82	1	190.19	Cl, Gy, Ca	Anthropogenic (Ash)	Brown-Orange	Type B
SRP_75	Y82	1	190.11	Gy, Cl(a), Ca	n/a (Gypsum)	Dark Gray	Type A
SRP_74	Y82	2	190.05	Ca, Gy, Cl(a)	n/a (Gypsum)	Dark Gray	Type A
SRP_73	Y82	2	190.01	Cl(i), Gy, Ca	n/a (Gypsum)	Dark Gray	Type A
SRP_72	Y82	2	189.90	Gy, Cl(a), Ca	Anthropogenic (Ash)	Dark Gray	Type A
SRP_71	Y82	2	189.82	Cl, Ca, Qz	Anthropogenic (Ash)	Brown-Orange	Type B
SRP_70	Y82	3	189.75	Cl, Qz, Ca	Geogenic	Brown-Orange	Type B
SRP_69	Y82	4	189.72	Cl(a), Ca, Qz + P	Anthropogenic (Ash)	Dark Gray	Type A
SRP_68	Y82	4	189.62	Cl(a), Ca, Qz + P + Gy	Anthropogenic (Ash)	Dark Gray	Type A
SRP_67	Y82	4	189.56	Cl(i), Qz, Ca	Geogenic	Brown-Orange	Type B
SRP_66	Y82	5	189.43	Cl, Ca, Qz + Gy	Geogenic	Brown-Orange	Type B
SRP_65	Y82	6	189.38	Ca, Cl(i), Qz	Anthropogenic (Ash)	Dark Gray	Type A
SRP_64	Y82	6	189.35	Ca, Cl, Qz	Anthropogenic (Ash)	White	Type B
SRP_63	Y82	6	189.32	Ca, Cl(i), Qz	Anthropogenic (Ash)	Brown-Orange	Type B
SRP_62	Y82	6	189.27	Cl, Qz, Ca + Gy	Geogenic	Brown-Orange	Type B
SRP_61	Y82	7	189.20	Ca, Cl(a), Qz + P + Gy	Anthropogenic (Ash)	Dark Gray	Type A
SRP_60	Y82	7	189.14	Cl, Qz, Ca	Anthropogenic (Ash)	Brown-Orange	Type B
SRP_59	Y82	7	189.02	Cl(a), Qz, Ca + P	Anthropogenic (Ash)	Dark Gray	Type A
SRP_58	Y82	7	188.98	Cl, Ca, Qz	Geogenic	Brown-Orange	Type B
SRP_57	Y82	7	188.86	Cl, Qz, Ca	Geogenic	Brown-Orange	Type B
SRP_79^C	Control (Fig. 1D)	NA	Surface	Cl, Qz, Ca	Geogenic	Brown-Orange	(Type B)
SRP_90^C	Control (Fig. 1C)	NA	Surface	Cl, Ca, Qz	Geogenic	Brown-Orange	(Type B)
SRP_114^C	Control (Fig. 1C)	NA	Surface	Cl, Qz, Ca	Geogenic	Brown-Orange	(Type B)
SRP_2-01^C	Control (Fig. 1C)	NA	Surface	Cl, Ca, Qz	Geogenic	Brown-Orange	(Type B)
SRP_2-20^C	Control (Fig. 1D)	NA	Surface	Cl, Ca, Qz	Geogenic	Brown-Orange	(Type B)

*Ca, calcite; Cl, Clay (a = altered, i = indeterminate); Gy, Gypsum; Qz, quartz, P, phosphate, + minor presence; ^{C} = Control sample; ^{C*} = controls not included; NA, Not applicable.

spherulites following the procedure outlined by Gur-Arieh et al. (2013). Spherulites were identified and counted in 16 random fields under cross-polarized light at 400x magnification. Spherulites per 1 g of sediment were calculated following Gur-Arieh et al. (2013). Ten samples were counted three times to determine sample variability and measurement error.

3.4.2. *Phytolith concentrations and morphologies*

We extracted phytoliths from approximately 10–20 mg of AIF sediment (section 3.3) using the Katz et al. (2010) Rapid Extraction Method. Phytolith concentrations and morphological identifications were carried out using a Nikon eclipse LV100N POL petrographic microscope at 200x and 400x magnification, respectively. We counted phytoliths in 16 fields (24x24mm coverslip) and calculated concentrations following Katz et al. (2010). Calculating phytolith concentrations per 1 g of AIF allows independent comparisons between samples with different mineral compositions and different levels of diagenesis (Albert et al., 2003, 2000, 1999; Albert and Weiner, 2001; Cabanes et al., 2009; Karkanas et al., 2000). We also identified fresh water siliceous microorganisms, including diatoms and chrysophyte cysts (algae), and calculated their combined concentrations per gram of AIF. We counted eight samples in triplicate to determine sample variability and measurement error.

Morphological identification followed the standard literature (Madella et al., 2005; Piperno, 2006; 1988;; Rapp and Mulholland, 1992; Twiss et al., 1969) using the International Code for Phytolith Nomenclature (ICPN) 2.0 when possible (Neumann et al., 2019). We identified >250 individual phytoliths per sample where possible to ensure morphotypes were accurately represented (Albert and Weiner, 2001; Zurro, 2018). Individual phytoliths in anatomical connection (multicellular structures, silica skeletons) were identified and counted, and the phytolith composition of these structures was recorded (Cabanes, 2020). The percentage of phytoliths recovered in anatomical connection and their size (i.e., number of individual phytolith within a structure) serve as a metric for the preservation state of each sample (Cabanes et al., 2011, 2009).

3.5. *Statistical tests*

We performed a k-means cluster analysis in R to verify if facies types existed for samples 57–76 in Y82 based on percent organic content, percent carbonate content, phytolith concentrations, and spherulites concentrations (Hartigan and Wong, 1979; Kassambara and Mundt, 2020; Maechler et al., 2021). The cluster analysis excluded control samples and samples from disturbed contexts (#77–78). We verified the optimal number of clusters using the Average Silhouette and gap statistic methods (Tibshirani et al., 2001). We tested for differences between cluster (facies) means using the non-parametric Wilcoxon rank sum test. To test relationships between variables (independent of facies type) and trends by elevation, we performed a non-parametric correlation test to calculate spearman's rho correlation coefficient (Kassambara, 2020; Appendix B). We chose the non-parametric test due to the study's small sample size.

4. Results

4.1. *FTIR spectroscopy*

Clay, calcite, and quartz are the main mineral components in all samples (Table 2). Five samples contain gypsum as one of their highest absorption peaks and three samples contain a minor presence of gypsum. Following the relative peak heights provided by Regev et al. (2015, Fig. 5), four samples indicate the minor presence (<3%) of the authigenic phosphate mineral carbonated hydroxylapatitie (dahllite) (Regev et al., 2015); although, samples with high gypsum content may mask low percentages (<5%) of dahllite because gypsum and dahllite both have an absorption peak near 602 cm^{-1}. Grinding curves indicate calcites are geogenic or anthropogenic (i.e., pyrotechnic ash). We could not determine the calcite types in three samples with high gypsum content due to the strength of the gypsum absorption peak.

Results from the heat alteration experiments are presented in Appendix A. We categorized seven samples as containing thermally altered

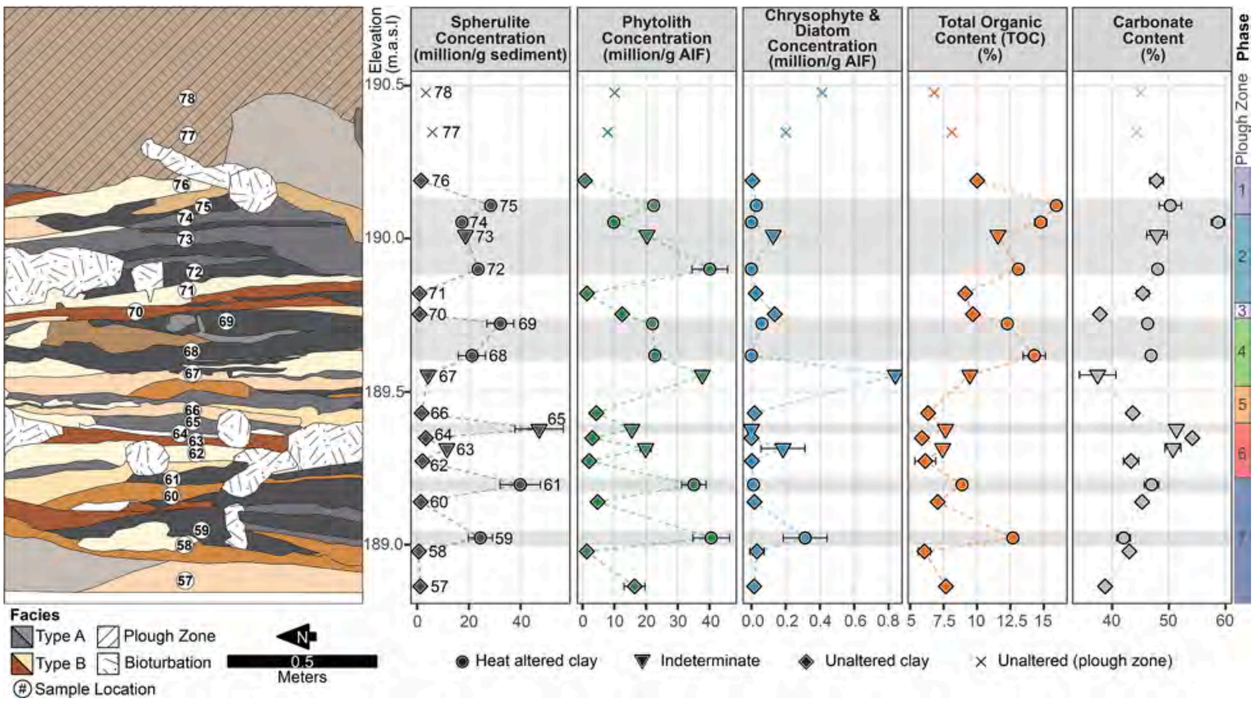

Fig. 3. Inset of stratigraphy and sample locations (white circles) from Fig. 2 (left). Main results of micro-remain concentrations (dung spherulites, phytoliths, and chrysophytes/diatoms), percent organic content, and carbonate (inorganic solubles) by elevation (right). Semi-transparent horizontal gray bars highlight spherulite, or dung-rich, samples. Shapes indicate clay alteration status determined by FTIR.

clays and four samples as containing indeterminately altered clay because these spectra contain conflicting absorption peaks: both weak absorption peaks in the 3700–3500 cm^{-1} range (i.e., unheated clay <500°C) and reduced absorptions in the 520–510 cm^{-1} range indicating heating > 500 °C (i.e., heat altered clay >500°C) (Table 2).

The four samples that produced indeterminate or atypical spectra (#63, 65, 67, and 73) are similar to those reported by Shahack-Gross et al. (2009, p. 178) from a midden deposit at Tel Megiddo. Shahack-Gross et al. (2009, p. 178) attributes these mixed signals to either mixtures of opal and clay or different clay types. The latter is most likely for the Y82 samples. Here, atypical spectra may result from (a) undetected micro-laminations of heat altered sediments or (b) sediments heated at temperatures lower than 500°C for more than four hours. Further experimental FTIR work or an additional test for burnt phytoliths (Elbaum et al., 2003) are needed to determine the heated status of these spectra.

4.2. Organic and carbonate content

Fig. 3 and supplementary table B.1 contain the results of the total organic content (TOC) and acid insoluble fraction (AIF) procedures. Sediment from Y82 (#57–76) contains an average of 9.8 ± 3.2% organic content (range: 5.5–16.3%). Triplicate samples of organic content produced a mean standard deviation of ± 0.5% and a standard error of ± 0.3%. There was no difference between samples heated for 2 h and those heated for 4 h. TOC content increases with increasing elevation ($\rho = 0.65$, $p < 0.05$, $n = 20$), but it is the only overall significant trend by elevation in this study (Supplementary Material Fig. B.1). This trend is likely driven by the concentration of organic-rich layers higher in the excavation profile. Average carbonate content is 46.3 ± 5.3% (range: 34.0–59.6%) with a mean standard deviation of ± 1.4% and a standard error of ± 0.8%.

4.3. Microscopy

4.3.1. Dung spherulite concentrations

Dung spherulite concentrations are displayed by elevation in Fig. 3. Based on 10 samples counted in triplicate, the precision for spherulite concentration in this study shows a ± 33.6% percent error meaning all reported values may vary up to ± 3.2 million/gram of sediment. This is in line with the proposed measurement error for this method which is 30% (Gur-Arieh et al., 2013). Spherulite concentrations in Y82 (#57–76) range between 0.3 and 57.5 million per gram of sediment with a median value of 7.9 million/g. Although, sheep, goats, and cattle are currently grazed on and around Khani Masi, surface and external control samples do not contain concentrations>1.3 million per gram of sediment and some contain no spherulites (supplementary table B.1). Samples 76, 71, 70, 66, 60, 58, and 57 contain spherulite concentrations in the same ranges as the controls (<2 million/gram of sediment).

4.3.2. Phytolith concentrations

Phytolith concentrations are displayed by elevation in Fig. 3. Based on eight samples counted in triplicate, the precision for phytolith concentration in this study shows a ± 18.5% percent measurement error meaning all reported values may vary up to ± 2.4 million/gram of AIF. This is well below the reported error for the Katz et al. (2010) method which is ~ 30%. Phytolith concentrations in Y82 range from 0.4 to 45.9 million/gram of AIF with a median value of 15.9 million/gram of AIF (samples 57–76; see also section 4.4.1). Control sample concentrations range from 1.5 to 0.17 million phytoliths per gram of AIF (supplementary table B.1).

The median value for diatoms and chrysophyte cyst concentrations in Y82 is 0.02 million/g of AIF. Sample 67 notably has a diatom and chrysophyte cyst concentration of 0.84 million/g of AIF—significantly more than any other sample in this study. The high concentration in sealed context #59 is also notable (0.31 ± 0.12 million/g of AIF). Higher concentrations in #77 and #78 are likely due to mixing with surface soil which can also have relatively high concentrations (control sample 2–20: 0.2 million/g of AIF).

Table 3
Results of phytolith morphological analysis related to taphonomic criteria.

Sample	Weathered (%)	Phytoliths in Anatomical Connection (%)	Average Multicell Size	Delicate Morphotypes (%)	Long to Short Cell Ratio	Morphological Richness (n)
SRP_78	3.1	16.2	3.0	20.2	1.5	27
SRP_77	3.4	9.6	3.1	21.1	1.2	29
SRP_76	5.6	22.8	4.0	26.4	2.3	26
SRP_75	4.0	20.3	3.3	22.3	1.6	27
SRP_74	4.9	25.5	3.5	22.2	1.4	29
SRP_73	0.3	25.8	3.0	35.1	2.0	28
SRP_72	1.9	46.2	3.9	36.4	3.0	28
SRP_71	10.1	5.1	2.5	15.7	2.3	22
SRP_70	2.3	2.6	2.7	28.0	1.4	21
SRP_69	0.4	54.3	4.2	53.3	4.6	19
SRP_68	7.4	29.9	3.5	17.5	1.5	26
SRP_67	0.6	12.4	5.4	13.8	1.8	23
SRP_66	4.6	14.7	3.6	26.4	1.9	26
SRP_65	2.7	36.0	4.0	27.9	1.7	29
SRP_64	7.0	35.7	6.1	20.6	2.0	32
SRP_63	1.9	16.7	5.2	20.5	1.8	25
SRP_62	4.4	14.3	3.1	23.4	1.4	27
SRP_61	0.8	47.7	3.9	27.6	2.8	31
SRP_60	1.4	6.8	3.1	30.6	2.2	27
SRP_59	0.9	34.7	3.1	27.4	2.3	28
SRP_58	4.9	8.5	3.7	15.0	1.4	28
SRP_57	1.3	1.7	2.5	15.0	1.7	20
SRP_2-20[C]	8.2	1.1	2.0	11.9	1.2	24
Descriptive Statistics						
Median	2.9	18.5	3.5	22.8	1.8	27.0
SD	2.6	15.2	0.9	8.9	0.7	3.4
Range	0.3–10.1	1.7–54.3	2.5–6.1	13.8–53.3	1.2–4.6	19–32

[C] = Control sample, not included in summary statistics; SD = standard deviation

4.3.3. Assessing phytolith assemblage integrity

All phytolith taphonomic metrics suggest overall good preservation in the Y82 samples (Table 3). Among the control samples, only sample 2–20 had sufficient phytolith counts for reliable morphological comparison (i.e., >250) (Albert and Weiner, 2001; Zurro, 2018). Across the profile (#57–76), samples have relatively low percentages of weathered phytoliths (range: 0.3–10.1%), high percentages of phytoliths in anatomical connection (PAC) (median 21.6%, range: 1.7–54.3%), relatively stable average PAC sizes, and higher than control level percentages of delicate morphologies. Long cells and delicate morphologies are most susceptible to post-depositional dissolution, so the additional presence of delicate morphologies and higher ratios of long to short cells than surface samples also suggest good preservation (Cabanes et al., 2011; Cabanes and Shahack-Gross, 2015; Madella and Lancelotti, 2012). Additionally, none of the above taphonomic metrics show trends based on elevation (supplementary Fig. B.1). Finally, morphological richness (number of unique morphologies) is not correlated with phytolith concentrations further suggesting good phytolith preservation across the assemblage (supplementary Fig. B.2) (Madella and Lancelotti, 2012).

4.3.4. Phytolith morphotypes

Phytoliths from monocotyledonous plants dominate the Khani Masi assemblages (samples 57–76: 84.7 ± 8.7%) (Figs. 4 and 5A). Sedges (*Cyperaceae*-type) appear in only 7 samples in very low percentages (<2.1%). Palm phytoliths were not identified. Thus, the majority of monocotyledonous phytoliths likely derive from grasses. We identified dicotyledonous plants in very low percentages (2.7 ± 1.8%; range: <6.1%) and derive almost exclusively from wood. Samples 60–61 and 64–65 are notable for their high wood percentages (4.4–6.1%) compared to background values (2.8%). Given that grasses produce 20 times more phytoliths than woody species (Albert and Weiner, 2001), the proportion of wood in these samples may be substantial.

Overall, Y82 grass phytoliths tend toward notably higher proportions of inflorescence phytoliths than leaf and stem phytoliths (leaf-stem to inflorescence ratio < 1.2; 0.7 ± 0.6) (Fig. 5B). Modern whole plant ratios fall between 1.2 and 3.2, so ratios lower than 1.2 indicate higher proportions of inflorescences and ratios <0.5 are dominated by inflorescences (Regev et al., 2015). According to Albert et al. (2008), both wild grasses and domesticated cereals produce ELONGATE DENDRITIC phytoliths (dendritic long cells, ICPN 1.0), but phytolith assemblages derived from cereals (wheat and barley) will contain dendritic phytoliths above 7–8%. Shahack-Gross et al. (2014) add that in agro-pastoral systems, assemblages above 3% dendritics likely indicate some level of foddering with cereal byproducts. Here, sample 69 contains the most inflorescence phytoliths of any sample (leaf-stem to inflorescence ratio: 0.1) and, at 13.2% ELONGATE DENDRITIC phytoliths, is the only sample clearly containing cereal inflorescences (Fig. 5C). Samples 72 and 73 have >3% ELONGATE DENDRITIC phytoliths and may contain a mixture of cereal inflorescences from occasional foddering. Notably, a few samples (#61, 65, 67, 69, and 70) each contained 1–2 ELONGATE DENTATE phytoliths similar to *Avena* sp. (oats) (Fig. 4F) (Albert et al., 2016; Portillo et al., 2006). All other samples are dominated by wild grasses.

Across all samples, Grass Silica Short Cell Phytoliths (GSSCP) are dominated by temperate Pooid (C_3; RONDEL and TRAPEZIFORM short cells) grasses with 74.2 ± 12.1% of the GSSCP and an average C_3 to C_4 ratio >1 (average: 3.9 ± 2.6) except for #59 (0.96) (Fig. 6A-B). These ranges are expected given the site's latitude and location in an agricultural plain (Twiss, 1992). GSSCPs were also compared to two climate indices: the Ic climate index, which reports the proportion of C_3 grasses compared to all grass types (Barboni et al., 2007; Twiss, 1992), and the Iph humidity-aridity index, which indicates local aridity by reporting the percentage of Chloridoideae within C_4 grasses (Bremond et al., 2005; Diester-Haass et al., 1973). In Y82, most Ic climate index values are >60%, which also indicate a C_3 dominated local environment (Fig. 6C). Three samples, 59, 61 and 68, are in the 40–60% Ic index range indicating a mix of C_3 and warm-C_4 grasses. All Iph aridity index values are >20–40% indicating that when C_4 grass types are present, they are dominated by chloridoideae grasses (warm and arid; SADDLE short cells), not panicoideae grasses (warm and humid; BILOBATE and POLYLOBATE short cells) (Fig. 6C).

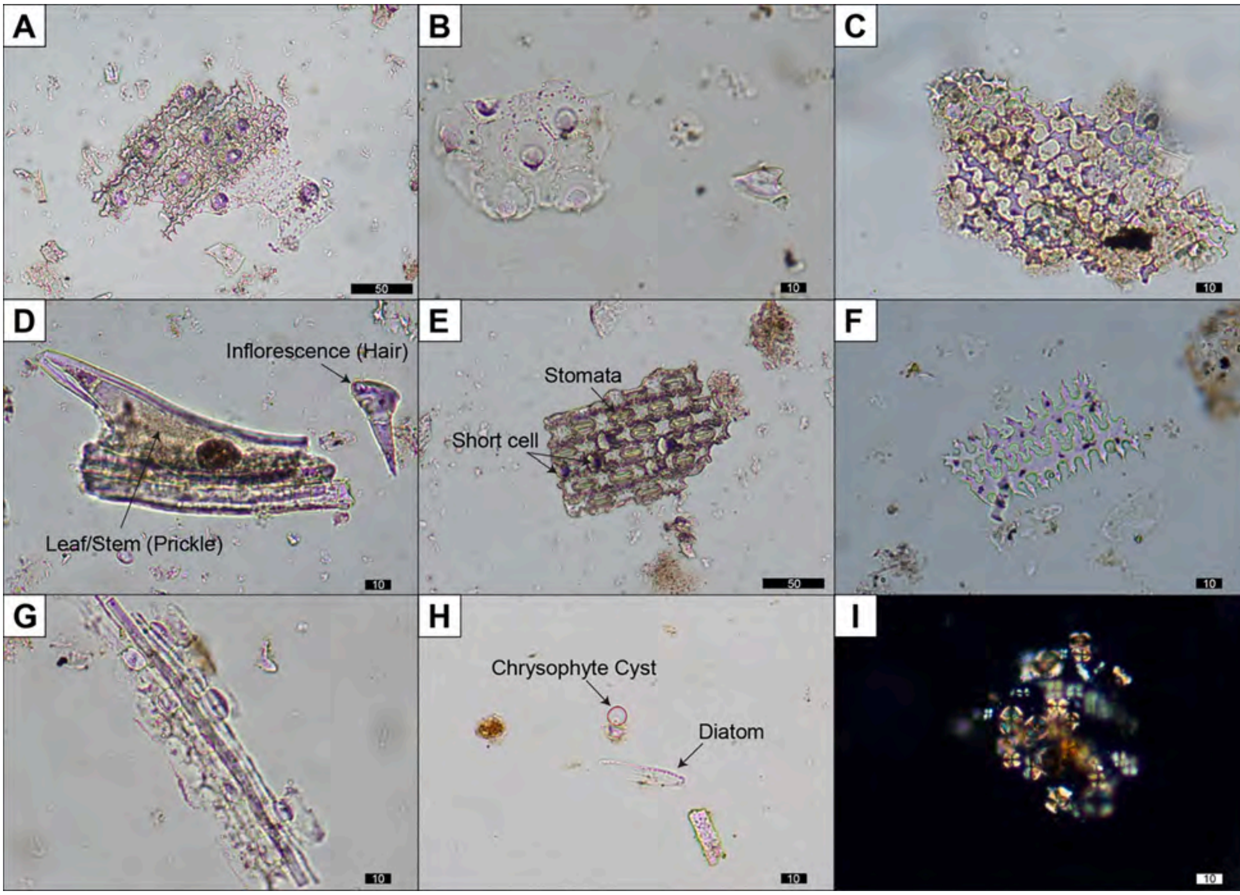

Fig. 4. Examples of prevalent phytoliths and other micro-remains. (A) Anatomically connected phytolith (PAC), or articulated multicell, composed of ELONGATE DENDRITIC, ELONGATE SINUATE (wavy long cell in ICPN 1.0), and PAPILLATE phytoliths from cereal inflorescences; (B) oblique view of articulated (left) and single (right) PAPILLATE phytoliths from grass inflorescences; (C) articulated ELONGATE DENTATE phytoliths from wild grass inflorescences; (D) ACUTE BULBOSUS phytoliths from both the grass leaf/stem and inflorescence; (E) articulated grass leaf multicell composed of stomata, short cell, and ELONGATE SINUATE phytoliths; (F) ELONGATE DENTATE phytoliths similar to *Avena* sp. (oats) (Albert et al., 2016; Portillo et al., 2006); (G) articulated SADDLE short cell phytoliths distinctive of chloridoid (C_4) grasses; (H) chrysophyte cyst and diatom; (I) clump of dung spherulites under cross-polarized light. Scale bars are in μm.

4.4. Defining facies

4.4.1. Cluster analysis (TOC, carbonates, spherulite concentrations, and phytolith concentrations)

Results from the cluster analysis indicate that samples from trench Y82 at Khani Masi optimally cluster into two main groups: (1) samples with high micro-remain concentrations and relatively high organic (and carbonate) content (cluster 1: facies A); and (2) samples with relatively low micro-remain concentrations and lower percentages of organic and inorganic solubles (cluster 2: facies B) (Fig. 7; Table 2). In fact, facies A sediments contain significantly higher concentrations of spherulites (28.1 ± 10.0 million/g sediment), phytoliths, and organic content than facies B ($p < 0.05$; Fig. 8). Qualitatively, facies A are also exclusively dark gray sediments while type B facies have a variety of sediment colors including browns, oranges, reds, and even white (Table 2). The reduced organic content in the facies A dung-rich samples (12.4 ± 2.7%) compared to fresh dung (Shahack-Gross et al., 2003, >55%; Shahack-Grosset al., 2004b) and the lack of preserved dung pellets indicates that Y82 is categorized as an organic-poor dung deposit (Shahack-Gross, 2011). It is notable that sample 67 (facies B), a likely outdoor surface with high phytolith concentrations, clusters with facies A when percent carbonate content is not included in the cluster parameters. Control samples cluster with type B facies when controls are included in the analysis, and control values are often in the same ranges as Y82 facies B sediments (supplementary table B.1). Facies groups are additionally supported by the FTIR and phytolith morphology results presented below.

4.4.2. FTIR

The main mineral components (i.e., clay, calcite, and quartz) and calcite types did not cleanly group by facies type; however, facies can be generally distinguished by thermal alteration, some calcite types, and the presence of phosphate. In facies A, 7 out of 9 samples are thermally altered while 9 of the 11 samples in facies B are not thermally altered. The two remaining samples in both facies type are indeterminately heated. Only facies A contains authigenic phosphate (dahllite) and only facies B are composed of geogenic calcite. Both facies types contain anthropogenic calcite (ash). We did not statistically compare sediment contents and micro-remains by calcite type or main mineral component because of the low sample sizes.

4.4.3. Phytolith facies comparisons

4.4.3.1. Assessing phytolith assemblage integrity between facies types. When we compared sediments by facies type, we found no differences between percent weathered phytoliths, average PAC size, morphological richness, or ratio of long to short cells ($p > 0.05$) (supplementary Fig. B.3). However, we did find type A facies contain significantly higher percentages of PACs, delicate morphologies, and phytolith concentrations ($p < 0.05$) (Fig. 8; supplementary Fig. B.3).

The difference between facies types is unambiguous in most samples in Y82. Although we carefully avoided bioturbated areas during

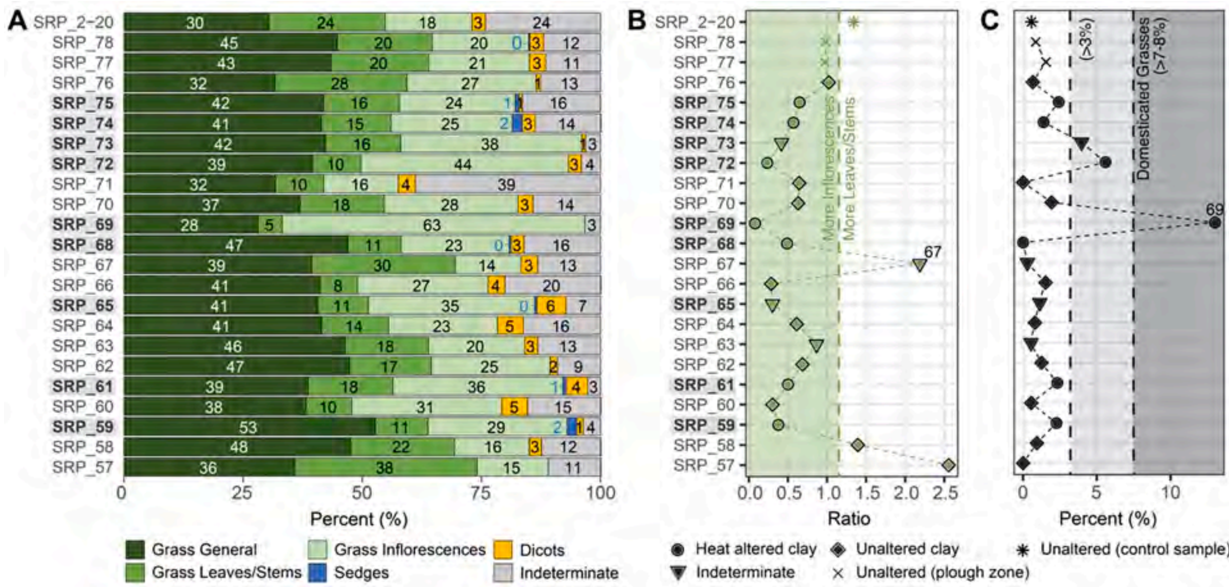

Fig. 5. (A) Phytolith morphologies as percent of sample assemblages arranged by elevation. Bold and highlighted (gray) sample names contain high concentrations of dung spherulites. (B) Ratio of leaves and stems to inflorescence phytoliths in each sample compared to the range of whole modern wild and cultivated grasses (ratio 1.2–3.2; Regev et al. 2015). Ratio values below 1.2 (dotted green line) indicate higher proportions of grass inflorescences. (C) Percentages of dendritic long cell phytoliths in each sample. Assemblages with percentages above 7–8% (dark gray area) derive from domesticated cereals (Albert et al. 2008) while percentages above 3% (left dotted black line; light gray area) may indicate inputs of cereal byproducts (i.e., agro-pastoral foddering with chaff; Shahack-Gross et al. 2014). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

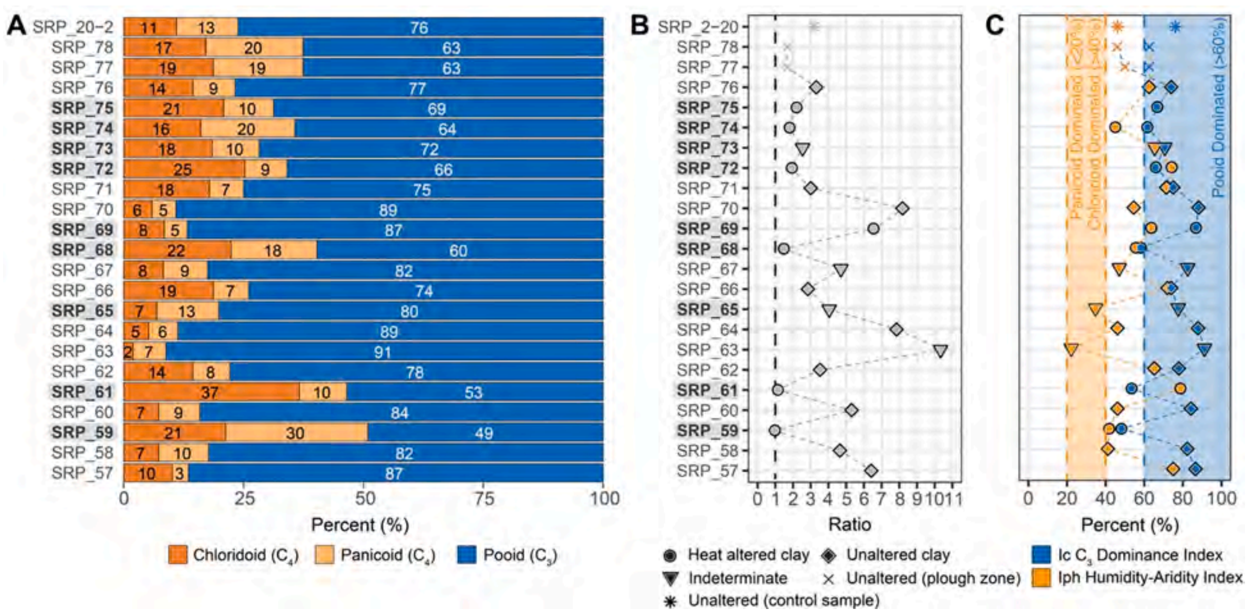

Fig. 6. (A) Phytolith short cell types as percent of total sample grass silica short cell phytoliths (GSSCPs) arranged by elevation. Bold and highlighted (gray) sample names contain high concentrations of dung spherulites. (B) Ratio of C_3 to C_4 short cells (Pooid: Chloridoid + Panicoid) in each sample. Ratios above 1 (dashed line) are dominated by C_3 grasses. (C) Short cell climate Ic and Iph indices reported as percentages. Iph humidity-aridity index percentages (orange) above 40% or below 20% (orange dashed lines) are dominated by Chloridoid or Panicoid grasses, respectively. Percentages between 20 and 40% (orange area) have equal proportions of each C_4 grass type. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

sampling, we do note that some micro-remain translocation, especially from dung-rich layers, is possible and may be responsible for slightly elevated concentrations of dung spherulites and grass inflorescences in fill layer samples. These results emphasize the importance of micro-sampling (Lancelotti and Madella, 2012) and integrating micromorphological analysis because we suspect our bulk sampling strategy mixed together microlaminations in at least two samples masking their clear facies signals (i.e., #63 and 67). Mixed microlaminations is also a potential cause of the indeterminate clay heat alteration spectra from these fill samples.

4.4.3.2. Facies comparison of phytolith morphotypes. Comparing phytolith morphotypes between facies, we found that type A facies are dominated by grass inflorescences (leaf-stem to inflorescence ratio: 0.4 ± 0.2) and have significantly less leaf and stem phytoliths ($p < 0.05$) than type B facies (1.0 ± 0.7) (Fig. 9). Type A facies also have

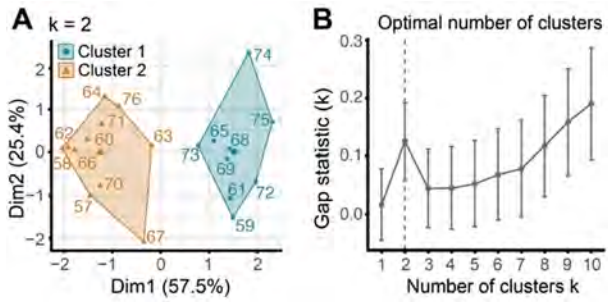

Fig. 7. Results of cluster analysis based on phytolith concentrations, spherulite concentrations, percent carbonate content, and percent organic content. (A) Cluster 1 (teal fill) groups together samples with high percent organic content, high carbonate content, and high micro-remain concentrations. Cluster 2 (brown fill) groups samples with generally lower values in all categories. Cluster 1 and 2 are mutually exclusive with facies A dung-rich sample and facies B samples of fill layers, respectively. (B) Two is the optimal number of clusters according to the gap statistic method (Tibshirani et al., 2001).

significantly more ELONGATE DENDRITIC cells than type B facies ($p < 0.05$; Fig. 9) and only Facies A sediments contain dendritic percentages >3%. However, the median value for facies A is low, 2.3%, indicating that most facies A sediments are dominated by wild grasses. Sedges are also predominately found in type A facies. Wood phytoliths, percent grass leaves and stems, and chrysophyte and diatom concentrations do not differ between the facies types ($p > 0.05$; supplementary Fig. B.3).

All percentages and metrics based on grass silica short cell phytoliths (GSSCPs) are significantly different between facies types except the Iph humidity-aridity index (Fig. 9; supplementary Fig. B.3). Compared to facies B, facies A has significantly lower C_3 to C_4 ratios, that is, higher percentages of total C_4 grasses. Facies A also has significantly higher percentages of each C_4 subtype, Panicoideae (warm and humid; e.g., reeds) and Chloridoideae (warm and arid). However, the lack of difference in the Iph (%) aridity-humidity index between facies types indicates that Chloridoideae C_4 types dominate compared to Panicoideae in both facies types. Several GSSCP metrics appear to exhibit notable opposite trends by elevation between facies A and B but none are significant (supplementary Fig. B.1).

It should be noted that several environmental niches surrounding the site contain notable C_3 confuser taxa that produce C_4-type short cells (BILOBATE or SADDLE), including *Phragmites* sp. (common reeds; saddles), *Arundo donax* (giant reed; bilobates), and two genera from the grass subfamily *Danthonia* (desert-steppe oatgrass; *Schismus* sp. and *Asthenatherum* sp.; bilobates) (Bor, 1968; Brown, 1984; Mulholland, 1989; Ollendorf et al., 1988). This study did not differentiate bilobate short cell sub-types, so panicoid percentages may include a combination of desert grasses and *A. donax* reeds in addition to the expected environmental sources: riparian vegetation growing along the Sirwan/Diyala River and vegetation near the region's many perennial springs (see Fig. 1C). Goats, and sometimes sheep, are known to consume reeds (Postgate and Powell, 1993). However, the low proportions of leaves and the notably low percentages of sedge (*Cyperaceae*) phytoliths suggests that confuser desert grasses may be the primary sources of panicoid short cells. For

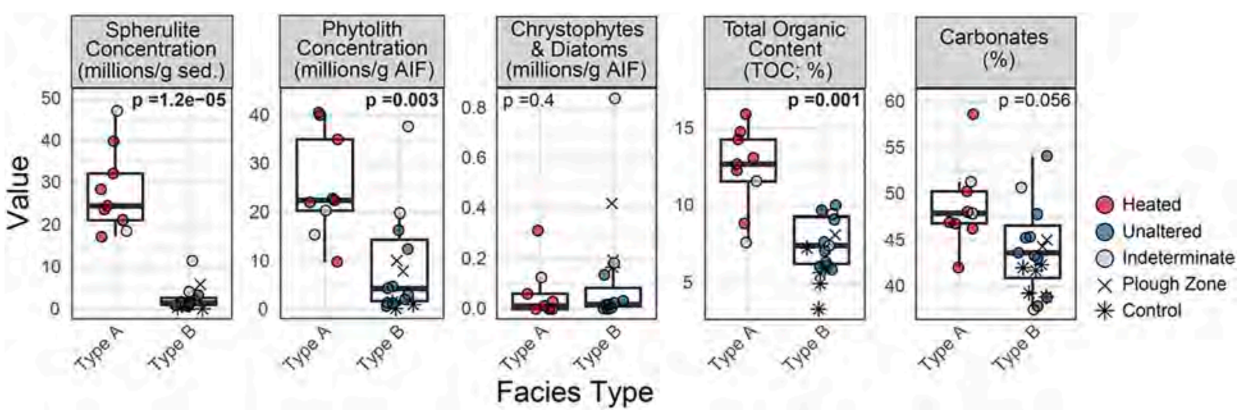

Fig. 8. Boxplots of micro-remain concentrations, organic content, and carbonate (inorganic soluble) content by facies type. Type A facies are dung-rich samples ($n = 9$), and Type B facies are sampled fill layers ($n = 11$). Significant p-values are in bold. Circle fill colors indicate FTIR derived clay heat alteration status. Plough zone and control samples are displayed for reference only.

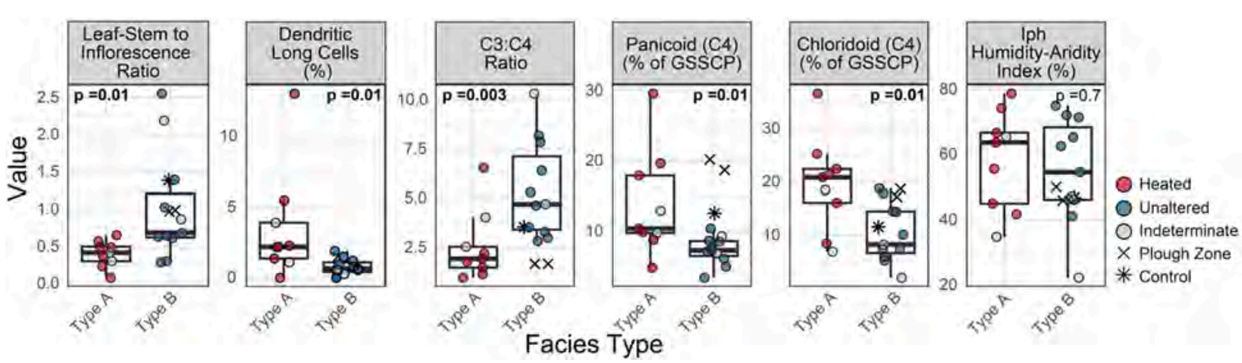

Fig. 9. Boxplots of phytolith morphology metrics by facies type. Type A facies are dung-rich samples ($n = 9$), and Type B facies are samples from fill layers ($n = 11$). Type A facies have significantly more inflorescence phytoliths (leaf-stem to inflorescence ratios < 0.5; higher percent dendritic long cells) than Type B facies. Type A facies also contain significantly more C_4 grass short cells, both panicoid and chloridoids short cells, than Type B facies, but facies types are similarly arid according to the Iph humidity-aridity index (Bremond et al., 2005; Diester-Haass et al., 1973). Significant p-values are in bold. Circle fill colors indicate FTIR derived clay heat alteration status. Plough zone and control samples are displayed for reference only.

Table 4
Interpretation of the major formation process, post-depositional process, and animal diet for dung-rich samples in each phase of Y82.

Phase	Sample (#)	FTIR	Post-depositional process	Formation Process	Animal Diet
1	**75**	Gy	Exposed (Desiccation)	Discarded dung fuel ash (midden)	Grazing
2	**74**	Gy	Exposed (Desiccation)	Discarded dung fuel ash (midden)	Grazing
	73	Gy	Exposed (Desiccation)	Discarded dung fuel ash (midden)	Grazing/ Mixed
	72	Gy	Exposed (Desiccation)	Discarded dung fuel ash (midden)	Grazing/ Mixed
3	–	–	–	–	–
4	**69**	P	Quickly buried (diagenesis)	Burned (*in situ*) pen accumulation	Foddering
	68	P + gy	Quickly buried (diagenesis)	Burned (*in situ*) pen accumulation	Grazing
	67	–	–	Outdoor surface	–
5	–	–	–	–	–
6	**65**	Absent	None	Discarded dung fuel ash (midden)	Grazing
7	**61**	P + gy	Quickly buried (diagenesis)	Burned (*in situ*) pen accumulation	Grazing
	59	P	Quickly buried (diagenesis)	Burned (*in situ*) pen accumulation	Grazing

Gy: gypsum is a main mineral component; gy: gypsum present; P: phosphate present.

chloridoid grasses, we did note that saddle short cells are predominately the "squat" or square saddle type typical of chloridoid grasses, not the "trapeziform saddle" or "plateaued saddles" distinctive of *Phragmites* sp. reeds (Fig. 4G; Gu et al., 2008; Novello et al., 2012; Ollendorf et al., 1988; Piperno and Pearsall, 1998). Therefore, the chloridoid short cells in dung-rich samples most likely derive from expected sources such as weedy species growing along disturbed agricultural field edges and from wild grasses growing on the hillsides surrounding the Khani Masi plain.

5. Interpreting Y82 facies and phase formation processes

Based on the FTIR, organic content, and micro-remain results, it is clear that Y82 contains two facies types: burned dung-rich sediments (facies A) and fill sediment (facies B). Dung-rich sediments are all dark gray, likely due to high organic content, and composed of heat altered clays and anthropogenic ashes with high concentrations of dung spherulites, phytoliths, and organic content. Fill sediments, on the other hand, are composed of unaltered clays, both geogenic or anthropogenic ash calcites, and contain comparatively less organic and micro-remain content. Given the overall good preservation for phytolith samples in Y82, the differences between the facies types are best interpreted as the result of different site formation processes and represent real contextual differences, rather than taphonomic bias.

For the facies in Y82 we propose three major depositional processes: (1) discarding of refuse and dung fuel, (2) burning of *in situ* animal pen accumulations, and (3) periodic leveling. Animal penning is often readily distinguished from midden accumulations by the presence of trampling microlaminations in micromorphological data (Shahack-Gross et al., 2003; Shahack-Grosset al., 2004b). Although we lack micromorphological data, by coupling several lines of evidence including FTIR, micro-remain, and stratigraphic data, we are able to characterize likely depositional processes for each phase in Y82 (Table 4). Dung layers containing traces of both gypsum and phosphate (samples 59, 61, 68, and 69) are best interpreted as burned and quickly buried pen accumulations while burned and desiccated dung layers are likely composed of discarded dung fuel ash (samples 65 and 72–75). Additionally, based on the visible stratigraphy and micro-remains, sample 67 in phase 4 likely captures an outdoor surface on which phase 4 dung and refuse accumulated.

5.1. FTIR – Heat altered clays

FTIR spectra indicate that clays in dung-rich sediments (facies A) could only have been briefly heated to a maximum temperature of 700°C. The abundance of dung spherulites, which begin to dissolve between 650 and 700°C, also support a maximum temperature of <700°C (Shahack-Gross, 2011). These temperature estimates are consistent with open "domestic" fires (as opposed to industrial fires, >1000°C; Berna et al. 2007, p. 368), dung or mixed fuel tanur cooking fires (Gur-Arieh et al., 2013), or experimentally combusted manure heaps (maximum 630°C: Shahack-Gross et al., 2005; 450-800°C: Vergès et al., 2016). Thus, based on temperature alone, burned dung-rich sediments could either represent discarded dung fuel (i.e., midden heaps) or dung accumulations that were burned *in situ* (i.e., animal pens). Both activities are archaeologically and ethnographically attested but can only be definitively distinguished with micromorphological analysis (Shahack-Gross et al., 2003, 2004b).

5.2. FTIR: Insights from gypsum and phosphates

The presence of gypsum in multiple samples offers an additional means for interpreting facies formation processes. Gypsum ($CaSO_4 \cdot 2H_2O$) has multiple pathways into the archaeological record and is not an uncommon mineral in archaeological contexts (Goldberg and Macphail, 2006). In general, gypsum sources are primarily geogenic (bedrock), pyrotechnic (anthropogenic plaster; Tamarisk wood ash), or authigenic (*in situ* evaporate; secondary precipitate) (Karkanas and Goldberg, 2019). In the absence of micromorphological analysis, distinguishing these gypsum-specific pathways is necessarily tenuous. For this study, by coupling gypsum data with other FTIR, micro-remain, and stratigraphic data, we are able to limit and discuss the multiple possible formation processes for the phases in Y82.

The origin of the gypsum in the Y82 leveling fills, captured by samples 62, 66, and 76, is most likely geogenic (i.e., gypsum present in the sediment prior to deposition. Geologic gypsum occurs in bands of local bedrock across Northern Iraq including near Khani Masi (Sissakian and Fouad, 2015; Smith and Robertson, 1962). It is unlikely that the gypsum in these fill layers formed *in situ* after deposition through evaporation because, despite the arid environment, gypsum is not present in the uppermost layers in Y82 (#77–78), in other intermediate geogenic layers of Y82, or in any of the control samples at Khani Masi. The calcite type in these samples is also mostly geogenic. An additional, potential non-authigenic source of gypsum in archaeological sediments is Tamarix wood ash (*Tamarix aphylla*) (Shahack-Gross and Finkelstein, 2008), but these trees are only common in the southern desert regions of Iraq and are an unlikely source of gypsum at Khani Masi.

For the dung-rich sediments, we propose three depositional scenarios based on two distinct authigenic formation processes of gypsum: evaporation and biochemical precipitation. In each of these respective scenarios, dung-rich sediments were either (1) desiccated through exposure for extended periods (#72–75; phases 1–2), (2) quickly buried (#59, 61, 68, and 69; phases 4 and 7), or (3) deposited as dung ash with low organic and moisture content (#65; phase 6) (Table 4).

(1) *In situ* evaporation is a common source of gypsum in arid environments (Goldberg and Macphail, 2006) and gypsum is known to form in exposed and desiccating dung and other decomposing

organic materials under arid conditions (i.e., drying out versus rapid accumulation) (Cabanes and Albert, 2011; Shillito and Ryan, 2013). The four uppermost dung-rich samples in Y82, #72–75, contain gypsum as one of their main mineral components. The high levels of gypsum in these samples are most likely due to gypsum crystal formation during extended exposure of these dung layers.

(2) The presence of both gypsum and phosphate (dahllite) in dung-rich samples 61 and 68 indicates that these minerals most likely formed together as a product of the in-situ decay of organic-rich dung (biochemical precipitation), not evaporation. Authigenic gypsum is known to form in decaying animal dung in conjunction with phosphates (dahllite) (e.g., Brochier et al., 1992; Cabanes and Albert, 2011; Shahack-Grosset al., 2004a). The presence of phosphates indicates the in-place decay of organic matter or bone in sediments, but phosphate-rich minerals are particularly common within dung deposits (Shahack-Gross, 2011; Weiner, 2010).

(3) It is notable that sample 65 is the only dung-rich sample that contains neither gypsum nor phosphates. In this case, we propose that the burning of sample 65 sediment (dung-rich anthropogenic ash) removed the organic matter or moisture necessary to form authigenic minerals.

5.3. Exterior spaces and discard at Khani Masi

The phases in Y82 have different depositional pathways and thus reveal different uses of the space through time. The layers associated with phase 4, #67–69, appear to capture material from an outdoor surface (#67), the subsequent buildup of refuse in the space (#68–69), and provide the best opportunity in Y82 to consider primary use of space in this part of the ancient settlement.

Stratigraphically, the thin laminations in phase 4 contrast with the thick disordered layers in phase 1–2, which we interpret as midden deposits. The foundational layer for phase 4, captured by sample 67, extends across much of the length of the Y82 profile and appears to have served as a leveling fill. To the right and between the #67 and #68 sampling locations, there are alternating laminations of dung and fill layers distinctive of trampling, by people or animals, in high traffic areas. Sample 67 contains the highest concentration of phytoliths in any sampled fill layer and more phytoliths than most dung-rich samples (43.6 million/g of AIF). The #67 phytolith assemblage is also notable for containing a predominance of leaves and stems (Fig. 5B). The high concentrations of diatoms and chrysophytes in #67 indicate good preservation, high moisture, and may represent diatoms that accumulated on surfaces exposed to the elements in antiquity or through deposition from freshwater drinking animals (Brochier et al., 1992; Portillo et al., 2019; Shahack-Gross, 2011). Sample 68 captures a burnt and quickly buried dung-rich layer. The feature associated with sample 69 (phase 4) may be a fire pit with *in situ* dung burning (excavators noted this layer contained burned bone and fire cracked rock).

Together, “fire-spots,” dung lamination structures, and diatom and chrysophyte micro-remains indicate exposure, trampling, and other outdoor activities in the area (Shillito and Ryan, 2013, p. 692). The artifacts present in phase 4 layers may be associated with these outdoor activities. However, abundant archaeological and ethnographic research on similar deposits indicates that artifacts are more likely associated with the buildup of refuse in the space, post-use dumping activities, or secondary animal penning (McCorriston, 1995, p. 37; Shahack-Gross et al., 2005).

5.4. Phytoliths as a reflection of animal diet

The dung-rich layers in Y82 derive from both burned animal pen accumulations and discarded dung fuel. In both cases, phytolith assemblages may reflect not only animal diet, but could also include plant matter introduced during dung cake preparation and use (i.e., tempering, kindling, and drying substrate), cooking spills, mixing with pen bedding (if dung was scooped out to make dung fuel cakes), or mixing with other midden refuse (Lancelotti and Madella, 2012; Miller, 1996; Shahack-Gross, 2011; Smith et al., 2019). When plant material is added as temper, kindling, pen bedding, or fodder, it most often consists of reeds, hay, or the agricultural byproducts of winnowing and threshing (i.e., straw, chaff; Hillman, 1984; Willcox, 1992).

In Y82, the phytolith assemblages suggest that plant remains from dung, even those used as fuel, primarily reflect animal diet because of the high proportions of wild grass inflorescences. If dung contained inclusions from other sources (i.e., straw, hay, or reeds), assemblages would show much greater leaf and stem-to-inflorescence ratios or higher percentages of dendritic long cells (e.g., chaff temper or kindling) beyond the values reflected by most phytolith assemblages in Y82. Samples 69, 72, and 73 could reflect an addition of cereal chaff temper for dung fuel (ELONGATE DENDRITIC), but the selective addition of wild grass inflorescences (ELONGATE DENTATE) in other layers is extremely unlikely.

To address dung cake production in particular, additional plant material is not necessary to make dung fuel cakes and may be avoided since it can reduce the quality of the fuel by increasing smoke and decreasing burn times (Anderson and Ertug-Yaras, 1998; Reddy, 1999). Lancelotti and Madella (2012) also found that the intentional addition of temper to cattle dung fuel cakes did not significantly affect phytolith concentrations or morphological percentages. Given the high phytolith concentrations in ashed mixed sheep-goat dung (46.0 million/g; average of values reported by Dunseth et al., 2019; Gur-Arieh et al., 2013; Portillo et al., 2020a) relative to the concentrations in plant material (1.5 million and 0.5 million per gram of dried material for whole wild plants and wheat and barley inflorescences, respectively; Albert et al., 2008), the minimal impact of temper should also be mirrored in sheep-goat dung cakes. Thus, the high percentage of cereal inflorescences (13.2% ELONGATE DENDRITIC) in sample 69 most likely reflect chaff or grain foddering.

6. Discussion

6.1. Agro-pastoralism

The phytolith assemblages from the dung-rich layers in Y82 shed light on agro-pastoral management, fuel use and discard, and the local environment in the Khani Masi region. Dung-rich sediments appear to primarily reflect animal diet and indicate that animals were mostly grazed on wild grasses but were also, at times, foddered with cereal chaff. Phytoliths may also preserve a strong seasonality signature. The large on-site dung accumulations reveal the close relationship between animals and the residents of Khani Masi, a relationship that was likely necessitated by the need for dung fuel—a vital secondary product that may be underappreciated outside archaeobotanical discussions of Mesopotamian agro-pastoralism (Miller, 1996; Charles, 1998; Lancelotti and Madella, 2012).

6.1.1. Foddering vs. Grazing

Our results indicate that sheep and goat herds at Khani Masi were primarily grazed on wild grasses with only occasional foddering with chaff produced from grain processing. They did not consume post-harvest field stubble or straw fodder. The choice to graze or fodder animals relies on a complex combination of social, economic, and environmental factors (Marston, 2011; Miller and Makarewicz, 2019; Miller, 1997; Miller and Marston, 2012). At Khani Masi, the predominance of grazing indicates at least a reliable abundance of grazing land, and perhaps suggests an emphasis toward pastoralism within the local economy. Together, a high proportion of sheep-goat faunal remains compared to other animals and a large wild seed to cereal ratio would also confirm an emphasis toward pastoralism at the site (Miller, 1997; Miller and Marston, 2012).

Throughout Mesopotamian history, both textual and archaeobotanical data from Mesopotamia suggest that sheep and goats were sometimes foddered with agricultural (by-)products, especially barley (e.g., Charles, 1998; Ellison, 1978, p. 94; Miller, 1997). However, explicit evidence for foddering or fattening with barley grain is limited both in textual sources (Sallaberger, 2004; van Driel, 1993; Wiggermann, 2000) and in the archaeobotanical record because plant material may not survive both ruminant digestion and subsequent charring (Hillman, 1981; Valamoti and Charles, 2005). In most cases, it is unclear what percentage of sheep and goats were foddered (or "from the fattening pen/shed") and in what proportions they were fed barley grain, chaff, or straw. Texts from third millennium BCE Tell Beydar in Northern Syria indicate that archaeological evidence for foddering may also be limited because only lambing ewes and animals intended for slaughter were fed grain while the majority were left to graze away from cities (Sallaberger, 2004; Van Lerberghe, 2001). Given that foddering was practiced infrequently at Khani Masi, it is also likely that foddering was limited to lambing season, fattening for slaughter, and perhaps times of reduced pasture (e.g., seasonally, bad years).

6.1.2. Seasonality

The high percentages of wild grass inflorescences at Khani Masi seemingly preserve a strong seasonality marker for free grazing during the late spring through early summer (Dunseth et al., 2019; Shahack-Gross et al., 2014). Wild grasses grow on the foothills surrounding the Khani Masi plain throughout the winter and spring rainy season (November-April) and quickly mature in late spring–early summer (April–May). However, three scenarios could account for the strong seasonality signature.

First, ethnographic work indicates that dung fuel cake preparation can be a seasonal activity in which a year's supply of dung cakes are made exclusively in the late spring or summer months (Anderson and Ertug-Yaras, 1998, p. 101; Kramer, 1982; Watson, 1979, p. 37, p. 89). In this case, discarded dung fuel will reflect the season of preparation. Second, phytolith assemblages at Khani Masi could reflect transhumant pastoral mobility where flocks are grazed locally in the spring, penned for breeding and the collection of dung fuel or manure, and then moved up to summer pastures (Miller, 2013). Third, Burguet-Coca et al. (2020) have shown that wild grass inflorescences can remain attached to stems throughout the year, greatly complicating phytolith seasonality signatures. They suggest that in areas with abundant pastureland, small local flocks could selectively graze on inflorescences throughout the year without exhausting inflorescence availability.

From the available evidence, we argue that the third scenario is the most likely for the Sirwan/Upper Diyala region—small, local household-scale size flocks grazing on an abundance of local wild grasses throughout the year. Although the foothills in the SRP region are seemingly devoid of vegetation during the summer-autumn months (see Fig. 1B), they are, in fact, blanketed in dried wild grasses that herds can consume. During the mid- to late second millennium BCE, the amount of settled area on the east bank of the Sirwan/Diyala River was relatively small compared to available pastureland (Casana and Glatz, 2017, Fig. 5), and only extremely large herd sizes would exhaust pasture availability. The third scenario is also in line with recent reviews of pastoralism in the ancient Near East that suggest pastoralism was primarily site-based and highly integrated into local agro-pastoral strategies, and that most interpretations of transhumant pastoralism are likely anachronistic projections of modern ethnographic research onto the distant past (Arbuckle and Hammer, 2019; Potts, 2014).

6.1.3. Grazing ecology and environmental continuity

In general, the elevated proportions of C_4 grasses (chloridoids and panicoids) in dung-rich sediments indicate selective grazing in ecological niches beyond agricultural fields. Areas with heightened C_4 grass species include nearby hillsides (chloridoids) and areas along irrigation canals and close to perennial water sources (panicoids; although sedges (*Cyperaceae*) are notably rare in the Khani Masi assembledge). A lack of significant elevational trends for C_3 and C_4 grasses ($p > 0.05$) suggests continuity in local climatic aridity (facies B), anthropogenic disturbance (facies B), and pasture availability (facies A) (Marston, 2011, 2015b; Marston and Miller, 2014; Miller, 1997). Additionally, the lack of evidence for sheep and goat grazing in agricultural areas suggests that local agriculture may not have relied on sheep-goat dung fertilizer—a point Charles (1988) argues for lowland Mesopotamia. In this case, sheep-goat dung may also have been reserved or preferred for fuel (Gur-Arieh et al., 2013).

6.2. Towards an integrated model of agro-pastoral practice in the Mesopotamian-Zagros borderlands during the Kassite period

Together, the micro-remain and geochemical results from Khani Masi reveal a range of previously unverified agro-pastoral practices that shed new light on daily life in the understudied Kassite period. Results indicate a highly integrated local agro-pastoral economy in the Kassite borderlands. Khani Masi residents penned ruminate herds on-site and burned dung for fuel. They primarily grazed animals on local wild grasses but could, at times, fodder herds with agricultural (by-)products (i.e., cereal grains or chaff).

The lack of temporal trends likely signifies continuity in aridity, anthropogenic disturbance, and pasture quality and availability. From an animal management perspective, long-term continuity coupled with the seasonality signature could be interpreted as a concerted effort to maintain ecological health through transhumance, but it is more likely that resilience was achieved through herd size management. Manageable herd sizes may be a function of both small regional populations and local ecological knowledge that was undisturbed by the formation of Kassite networks in the Zagros foothills region. Unlike the large, transhumant herds managed by Uruk and some large Bronze Age institutions to support regional textile economies and provision urban centers (Lawrence et al., 2015; McCorriston, 1997), evidence from Khani Masi suggests a diversified economy in the Kassite borderlands where flocks were resiliently small and likely locally grazed year-round. These results closely align with interpretations of Kassite interests in the area as military or administrative outposts intended to control trade routes rather than in the intensive exploitation of the local agro-pastoral potential (Fuchs, 2017).

In (wood) fuel-poor regions like the Upper Diyala/Sirwan River valley, integrated agro-pastoral strategies were at least partially driven by fuel needs. The size of the large dung deposit at Khani Masi highlights the importance of dung fuel as a major secondary product in addition to meat, milk, and wool, and animals were likely penned on site to expedite dung fuel collection (Reddy, 1999). Local herds need not be large to support local dung fuel needs and the collection of other secondary products. Sheep and goats produce an average of 500 and 300 pellets per animal per day, respectively (Valamoti and Charles, 2005; Wallace and Charles, 2013), so even small herds can generate large quantities of dung. Recent zooarchaeological research has called the text-based and institutionally-focused "pastoral bias" in Mesopotamia into question (Grossman and Paulette, 2020; Price et al., 2017). These studies provide important critiques of institutional narratives but elide how the day-to-day necessity of dung fuel would have kept flocks of sheep and goats tied to household life.

Finally, the large on-site dung accumulation reveals a close relationship between people, animals, and bio-waste with implications for human health, use of space, demographic estimates, and site formation processes (Albert et al., 2008). Khani Masi is an important reminder that portions of Mesopotamian settlements must have included unoccupied open areas, animal pens, and areas dedicated to multiple types of waste disposal (Anvari et al., 2017, p. 12). Detailed consideration of these contexts and their micro-remains is essential for revealing the range and intensity of agro-pastoral practice in the past.

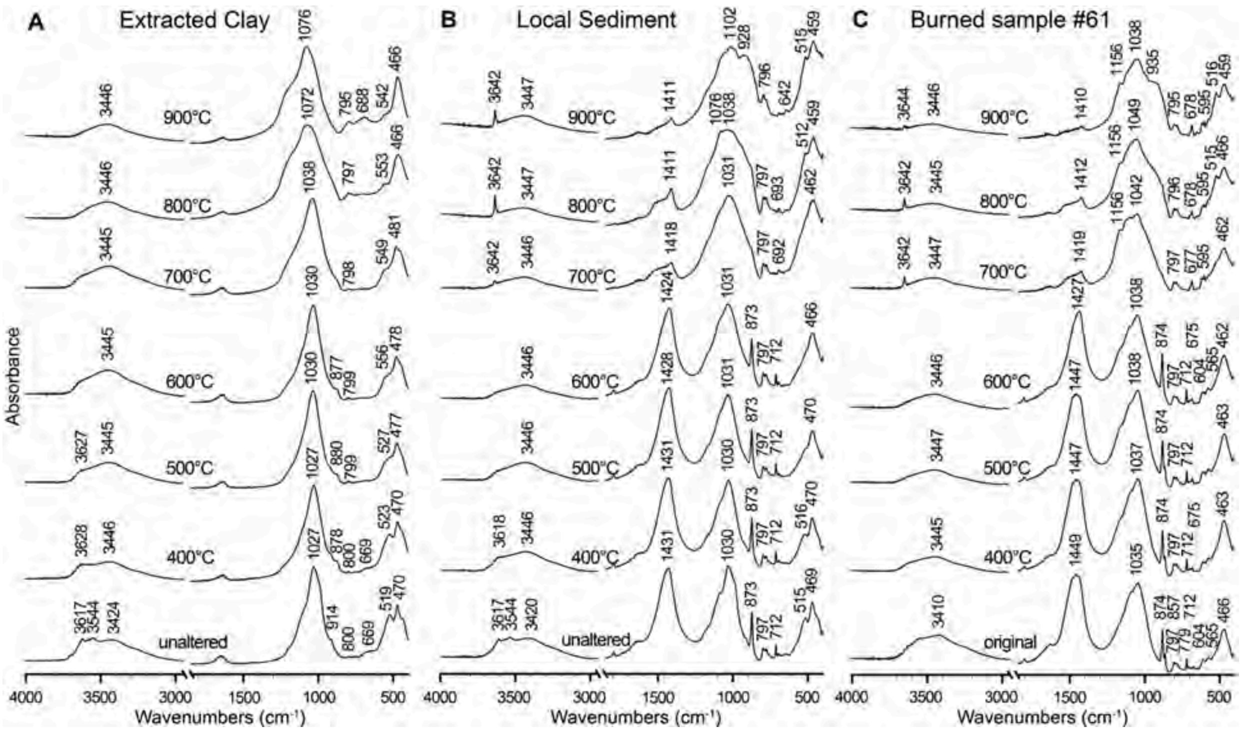

Fig. A.1. FTIR spectra of the experimentally heated sediments. (A) Major changes in the clay spectra occur at 500°C with the loss of the hydroxyl group peaks around 3628 cm^{-1}, the movement of the main clay peak from 1027 to 1030 cm^{-1}, and the change in the 523 cm^{-1} from a distinct peak to a shoulder. Major changes at 700°C and above include the continued broadening and movement of the main clay peak to higher wavelengths (1038–1072 cm^{-1}) and the weakening and loss of absorptions in the ~870 and ~550 cm^{-1} ranges. (B) Local sediment. The first notable changes are at 500°C with the loss of the 3628 cm^{-1} peak and the weakening of the 516 cm^{-1} peak to a shoulder. At 600°C, the 516 cm^{-1} peak disappears altogether. The most distinctive spectral changes in the local sediment occur at 700°C with the appearance (formation) of the calcium hydroxide peak (3642 cm^{-1}) and the disappearance of the main calcite peak (~1430 cm^{-1}). The main clay peak only changes significantly at 800°C and above. (C) Sample 61. Major changes occur at 700°C with the disappearance of the calcite peak (1449 cm-^{1}) and the appearance of the calcium hydroxide peak (3642 cm^{-1}).

7. Conclusion

Archaeologists debate the degree of integration between the agricultural and pastoral components of local economies across Southwest Asia, but models generally lack robust ecofactual data (Arbuckle and Hammer, 2019). This study provides new data and insights into local agro-pastoral management strategies at Khani Masi, a second millennium BCE Kassite site located along the Upper Diyala/Sirwan River in Northern Iraq. Micro-remain and geochemical approaches revealed the range of local animal management strategies which included animal diet, penning, and fuel use and discard. Animals were primarily pasture grazed across a diversity of ecological zones and periodically foddered with agricultural (by-)products. While more work on phytolith seasonality signatures is needed, we interpret the strong seasonality signature as reflecting small, site-based herds grazing year-round on an abundance of pastureland rather than reflecting transhumant pastoralism. Phytoliths also indicate that animal diets were ecologically diverse and reflect continuity through time.

This study demonstrated that micro-remain and geochemical analyses offer a wealth of information for answering fundamental questions about agro-pastoralism in Bronze Age Mesopotamia. Micro-remains, in particular, offer unique but underutilized sources of information on Mesopotamia's Bronze Age economies and ecologies, and this study highlights the potential of using FTIR and micro-remains together to study a) site formation processes, b) use of space, c) local ecology, and d) pastoral strategies.

In future, isotope analysis of ovicaprid remains could confirm that herds at Khani Masi were grazed locally throughout the year (e.g., Makarewicz and Tuross, 2012; Makarewicz, 2014b; Makarewicz and Sealy, 2015)). Dunseth and colleagues (2019; Fuks and Dunseth, 2020; see also Riehl, 2006, p. 122) also advocate leveraging whole dung pellets where they are preserved; although, pellet preservation is low except in the most arid regions of Mesopotamia (Charles, 1998, p. 119). Additional multi-proxy data sources would have greatly improved the interpretive power of this study. In the future we recommend the routine collection of micromorphological blocks and a sampling strategy that includes micro-sampling as well as paired phytolith and macrobotanical samples. Future studies in the region would greatly benefit from the development of a modern phytolith reference collection of local plants and sediments, additional ethnographic research with a particular emphasis on deciphering seasonality indicators, and an increase in Bronze Age case studies for both synchronic and diachronic comparison.

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CRediT authorship contribution statement

Elise Jakoby Laugier: Conceptualization, Validation, Formal analysis, Investigation, Writing - original draft, Writing - review & editing, Funding acquisition. **Jesse Casana:** Supervision, Resources, Writing - review & editing, Funding acquisition. **Claudia Glatz:** Writing - review & editing, Funding acquisition. **Salih Mohammed Sameen:** Project administration. **Dan Cabanes:** Conceptualization, Validation, Resources, Supervision, Writing - original draft, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A: FTIR analysis of experimentally heated local clays

FTIR Thermal Alteration References

For this study, we generated new thermal alteration references of local clay, local sediment, and archaeological sediment as recommended by Berna et al. (2007). We selected three references: (A) the clay size fraction from control sample 2-01 (local sediment) (Fig. 1C), (B) the original local sediment from control sample 2-01, and (C) sample 61, the most heat altered archaeological sediment from the Y82 section (Fig. 3).

Materials and methods

Extracting clay size fraction. We first treated control sample 2-01 sediment with 3N HCl to remove all carbonate content (Albert and Weiner, 2001). After fully washing and drying the acid insoluble fraction, we then extracted the clay size fraction using centrifugation following Poppe et al. (2001) and placed the sample in an oven at 50°C until dry.

Heating Experiments. Small amounts of clay or sediment ($<$0.25 g) in closed ceramic crucibles were heated for 4 hours in an oven at 100°C intervals between 400 and 900°C (Nabertherm LT 9/12/C450). We then analyzed heat altered sediments using FTIR spectroscopy as outlined in section 3.2. Finally, we interpreted whether clays in this study were subject to high temperatures following Berna et al. (2007) and the relative absorptions in the clay spectrum from the new local thermal alteration references.

Results and discussion

Results of the local clay and sediment heating experiments are presented in Fig. A.1. Major changes in the local sediment and clay size fraction (Fig. A.1 A-B) occur at 500°C with the loss of the 3628 cm^{-1} peak and changes to the 520-530 cm^{-1} absorption range. The two notable spectral differences between the local sediment and clay-sized fraction are (1) the loss of the 520-530 cm^{-1} shoulder 100–200°C earlier in the local sediment and (2) the major increase in the main clay peak (1030 cm^{-1}) of the local sediment 100°C later than the clay size fraction. Overall, the main clay peak was less informative for this study because there were no major movements until 700–800°C. Instead, we considered clays from archaeological samples in this study to be thermally altered if FTIR spectra had lost distinct hydroxyl peaks at ~3620 cm^{-1} and contained a distinct clay peak in the ~470 cm^{-1} range.

In antiquity, sample 61 (Fig. A.1 C) was heated below 700°C as evidenced by the presence of a strong calcite peak (1449 cm^{-1}) and the absence of the calcium hydroxide peak (3642 cm^{-1}). However, the movement of the main clay peak after 4 h at 400°C suggests that sample 61 could have been heated to a far higher temperature for a much shorter duration or to a lower temperature for much longer duration. Thus, the lower temperature limit is unknown and requires further heating experiments at shorter time intervals (e.g., 1-3 h).

Appendix B. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jasrep.2021.103106.

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