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Dung detective! A multi-scalar, multi-method approach to identification and analysis of ancient faecal material

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ABSTRACT

Ancient faecal material is becoming a highly valuable more frequently investigated proxy with which to address a wide range of research questions. With advancing scientific methodologies it is becoming easier to identify and to analyse. The aim of this paper is to use a set of archaeological and ethnographic case studies to illustrate and evaluate the range of methods that can be used in conjunction with each other to aid investigation of archaeological faecal material. This multi-scalar and multi-method approach uses portable x-ray fluorescence (pXRF), spot sampling and smear slide analysis, micromorphology, gas chromatography mass spectrometry (GC-MS), environmental scanning microscopy with energy dispersive x-ray spectroscopy (ESEM-EDX), and phytolith analysis. The case studies presented here focus on the Neolithic because of this unique opportunity to examine concentrations of animal dung from managed or early domesticated herds. This research illustrates a range of the methods that can be used in conjunction with each other to locate, identify and analyse faecal material. The results demonstrate that an integrated, multi-scalar, multi-methodological approach enables detection, identification, and investigation of a range of faecal attributes and provides new insights into key issues and themes on environment, animal management, diet, health, the built environment and energy sources. This integrated methodology and pilot study highlights two main recommendations. Firstly, modern faecal comparative material should always be consulted within the study region as a baseline for identifying and classifying different types of faecal material. Secondly, micromorphology and GC-MS samples are always vital proxies in further investigations to confirm the nature and identity of the dung sources once potential sample locations have been identified.

1. Introduction

Animals produce copious amounts of faecal material which can be detected in archaeological sediments using appropriate recovery techniques, and is now a highly valuable more frequently investigated proxy with which to address a wide range of research questions by diverse projects for many archaeological periods (Charles, 1998; Adams et al., 2004; Katz et al., 2007; Ghosh et al., 2008; Mlekuz, 2009; Portillo and Albert, 2011; Huffman et al., 2013; Portillo et al., 2020; Amicone et al., 2021; Fuks and Dunseth, 2021; Laugier et al., 2021; Proctor et al., 2022). The investigation of faecal material contents and the specific contexts in which this material is detected can be used to explore research questions related to animal management/domestication, secondary product use, fuel selection, animal diet, grazing and browsing practices, environment and ecology. Many archaeological projects are beginning to integrate the analysis of faecal material within large inter-disciplinary studies (e.g. Portillo et al., 2012; Stiner et al., 2014).

The study of faecal material is not novel, but with advancing scientific methodologies it is becoming a proxy which is now easier to identify and to analyse. Some of the earliest studies of coprolites were conducted in the 1930s (Laudermilk and Munz, 1934, 1938). In early dung research the potential for identification of faecal material in archaeological deposits was recognised, but it was acknowledged that "… basic research still needs to be done on processes of deposition and post-depositional geochemical changes of animal dung" (Chang and Koster, 1986). Recent research has placed a continued emphasis on the importance of integrating physical, chemical and organic methods such as macrobotanical remains, geochemical signatures and molecular genetics (Barker, 2006; Colledge et al., 2013).

The aim of this paper is to use a set of archaeological and

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ethnographic case studies to illustrate and evaluate a range of methods that can be used in conjunction with each other to locate, identify and analyse faecal type, source, and contents, context and taphonomy. Individually these methods have a long history in archaeology. However, the integration of these to specifically target archaeological faecal material is novel.

2. Methods in dung studies: A brief review

In the past, the examination of faecal material has been referred to in some studies as circumstantial, secondary or an indirect line of analysis (Gonalons et al., 2006, p.230; Olsen, 2006, p.256; Zeder et al., 2006, pp.176, 178). However, the integration of analyses of faecal material in a range of studies has often been highlighted as important in addressing a range of questions regarding animals and animal husbandry in particular early animal management and domestication (Zeder, 2006, p.110; Stiner et al., 2014). This has been reviewed in depth in Shahack-Gross (2011). This section aims to evaluate the most routinely applied methods in the study of faecal material and ways in which some of the challenges in this can be addressed to enable wider adoption and integration of faecal material analyses in archaeological fieldwork and research.

With regard to some of the challenges, dung is difficult to identify macroscopically in the field and in bulk or spot samples, and there is currently no standardised definitive protocol or criteria for the identification of animal dung. The nature and components of dung vary between types (Lancelotti and Madella, 2012) and faecal deposits are highly variable (Shillito et al., 2011a). In the field identification is problematic and often proved to be difficult or erroneous (Shillito et al., 2011c). Therefore, in order to identify and analyse faecal material there is a sustained requirement to develop an integrated and systematic field and laboratory methodology (Shahack-Gross, 2011). Previously, standardised macroscopic morphological descriptions of faecal material was attempted (Jouy-Avantin et al., 2003). However, these characteristics are not always recognisable in archaeological faecal remains, they have only been recognised in a small range of specific contexts such as faecal material in cave deposits (e.g. Shillito et al., 2020), or penning deposits (e.g. Matthews, 2005). Frequently, however, animal dung may be transformed and mixed by soil fauna with natural or anthropogenic sediments so that the original shape or bedding is no longer macroscopically recognisable (Courty et al., 1989). Many of the faecal remains from archaeological deposits are only detectable and identifiable microscopically and/or geochemically using biomolecular techniques, or integrated with archaeobotanical studies (Fuks and Dunseth, 2021). Therefore, in the face of difficulties in macroscopic visual characteristics we must also use multi-scalar, multi-method approaches to identification and characterisation of faecal material.

The research presented in this article highlights the lack of comprehensive and extensive reference material. While reference material does exist, collections are small and regionally specific (e.g. Brochier et al., 1992; Canti, 1997; Anderson and Ertug-Yaras, 1998; Canti, 1998; Goren, 1999; Portillo et al., 2017; Portillo et al., 2021). Comparisons between faecal material examining numbers of faecal spherulites extracted from modern dung samples for the same animals in different regions has produced markedly different results. For example with regard to cattle dung from Iraqi Kurdistan, Syria, Greece and Iceland (Portillo et al. 2012, 2014; Milek and Roberts, 2013; Elliott et al., 2015), some studies found high numbers of faecal spherulites, in others faecal spherulites were absent. Pilot data previously published for spherulites extracted from cow dung collected in Iraqi Kurdistan in 2012 (Elliott et al., 2015) highlights differences in cow dung from the same area collected in 2017 (Portillo et al., 2021). Other studies have identified variability in faecal spherulite numbers between different droppings from the same species in the same group of animals (Goren, 1999). There is clearly a requirement for further experimental work, and more investigation into faecal spherulite production. For example, wild animals are rarely studied. It should also be recognised by researchers that faecal spherulites can be easily dissolved and/or leached away in moist environments (e.g. temperate climates), or in conditions susceptible to throughflow (e.g. deposits with a high percentage of voids or sandy sediments with larger particle size). Therefore, the absence of faecal spherulites can also be attributed to post-depositional phenomenon rather than production by any given animal. There also needs to be wider study and understanding of dung components regionally and inter-regionally, and variation in relation to diet, geology and domestication status in order to support comparative analysis of intra- and inter-site variations.

Microscopically, dung can be identified archaeologically by the presence of microscopic faecal spherulites, phytoliths and specific microstratigraphic features of the groundmass and microstructure. Ruminant and omnivore dung can be distinguished microscopically if there is good preservation of the faecal material. This distinction is based on a range of features such as colour, spherulite and phytolith content, and the presence or absence of small fragments of bone, as well as orientation, distribution and comminution of contents, type of fine material and birefringence (Courty et al., 1989; Matthews, 2010). Herbivore dung is generally brown with high phytolith and spherulite content whereas omnivore dungs are orange/yellow, with low phytolith and spherulite content but often include small fragments of bone (Shillito and Matthews, 2013; Brönnimann et al., 2017a; Brönnimann et al., 2017b; Portillo et al., 2020 and pers. observation).

Specific models for dung identification have previously been proposed based on the presence of spherulites, phytolith content and geochemistry (e.g. see Lancelotti and Madella, 2012). Lancelotti and Madella (2012) prioritize identification of faecal spherulites, but in conjunction with phytoliths and chemical elements. Faecal spherulites form in the digestive tract of animals during digestion and pass into the faeces (Brochier et al., 1992; Canti, 1999) (Fig. 1). Faecal spherulites from dung can occur in archaeological sediments singularly, in patches or in layers. This is when dung has accumulated, and the depositional environment is suitable for preservation (Canti, 1998, p.435). Spherulites, however are not always identified in herbivore faecal material (Brochier et al., 1992) and are known to be low to absent in omnivore dungs (Canti, 1999; Brönnimann et al., 2017b). Therefore, the absence of faecal spherulites cannot be taken as absence of faecal material. Investigations into the species which produce faecal spherulites has previously been researched, indicating that feeding and digestive strategy are important to faecal spherulite production (Canti, 1999; Dalton and Ryan, 2020). The highest numbers of spherulites appear to be produced in the ruminants (sheep, cow, goat, deer); low numbers are produced by omnivorous and carnivorous (pig, man, badger, dog, cat, fox) (Canti, 1999). Some studies conclude that spherulites are absent from the caecal digesters (horse, rabbit, hare) (Canti, 1999). However other studies found faecal spherulites to be present in horses (Goren, 1999). Beyond the identification of faecal spherulites many other approaches rely on identification of charred dung in the form of either preservation of

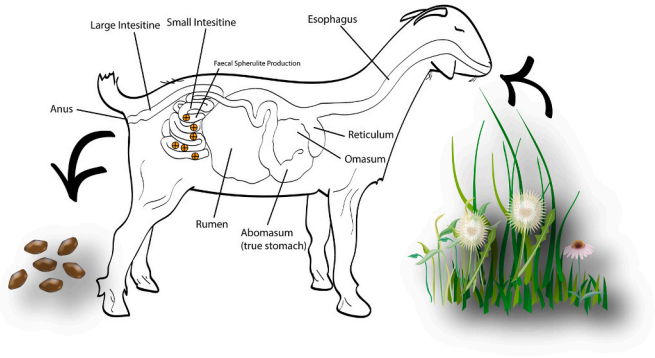


Fig. 1. Location of the formation of faecal spherulites.

whole pellets (Charles, 1998; Fuks and Dunseth, 2021) or identification of macroscopic plant remains likely to be derived from dung remains (Miller and Smart, 1984; Miller, 1996; Charles, 1998).

Once dung has provisionally been identified, the next step is to confirm the identification. A range of methods can be applied for confirming the presence of dung and also distinguishing between species, such as gas chromatography mass-spectrometry (GC-MS) and morphological distinction (Shillito et al., 2011a). Dung deposits can be conclusively identified to genus with the analysis of bile acid molecules and coprostanols by the application of GC and GC-MS analysis (Bull et al., 1999b; Shillito et al., 2011b).

Many animal corrals and pens have been confirmed by identification of the linear compacted fibrous structure of the dung and fodder/bedding in micromorphological thin sections (Courty et al., 1989; Matthews et al., 1996; Akeret and Rentsel, 2001; Bull et al., 2005; Matthews, 2005; Portillo et al., 2019). The structure of the dung remains has in some cases been linked to diet, specifically this linear fibrous structure being a product of a grass rich diet and presence often of compacted phytoliths (Shahack-Gross, 2011). Therefore, the absence of a linear fibrous structure does not specifically represent an area without animal dung, it could represent a mixed deposit, or dicotyledonous dominated diet (e.g. browsing on trees and shrubs). Spherulites identified in compacted dung layers are often used to interpret animal management and domestication (Portillo et al. 2019, 2020; Burguet-Coca et al., 2020; Nicosia et al., 2022). However, this interpretation needs to take into account the type of site being analysed. For example, caution should be taken when interpreting cave deposits. These are contexts where wild animals could shelter and therefore similar micro-stratigraphic features could form as a result of this natural sheltering of wild animals rather than herding of domesticated animals (Goren, 1999). In the case of open-air sites, if compacted faecal material is identified in micromorphological thin section then this can be interpreted as a clear indicator of corralling animals.

The identification of faecal material in archaeological deposits is also influenced by post-depositional preservation conditions. Faecal material on exposed and open-air archaeological sites could be subject to degradation in the form of erosion or bioturbation and therefore may be difficult to identify due to taphonomic changes. While individual faecal spherulites may still be identifiable, the macroscopic structure of the dung remains may be taphonomically affected. The decomposition and degradation of dung has been recorded within 30 years of deposition in open air abandoned modern communities (Shahack-Gross et al., 2003). Low pH conditions (<7.7) will affect the preservation of faecal spherulites and therefore are an important factor in the identification of faecal material in archaeological deposits (Canti, 1999; Albert et al., 2008). Spherulites are also subject to dissolution by leaching and high throughflow (Canti, 1999), which will adversely affect areas with little vegetation cover, sandy soils and an absence of later cultural layers which would seal and protect the earlier deposits. Silica phytoliths which are a significant component in herbivore dungs are also subject to dissolution in soils with a high pH (>8.5).

Although there are clearly difficulties with identifying faecal material, as briefly outlined here, the combined methods proposed here enable the most likely probability of identifying and interpreting faecal materials, as well as providing explanations for their possible absence (see Matthews, 2005, p.390).

3. Integrated methodological approach

This paper presents an integrated, multi-scalar, multi-method approach to the detection, identification and investigation of faecal material (Fig. 2) that was tested and applied to Neolithic sites in the Zagros region to examine a range of key important topics in Neolithic research between 2008 and 2015, and which is ongoing (Matthews et al., 2020). The combined methodological approach presented in this paper seeks to develop a multidisciplinary approach that integrates

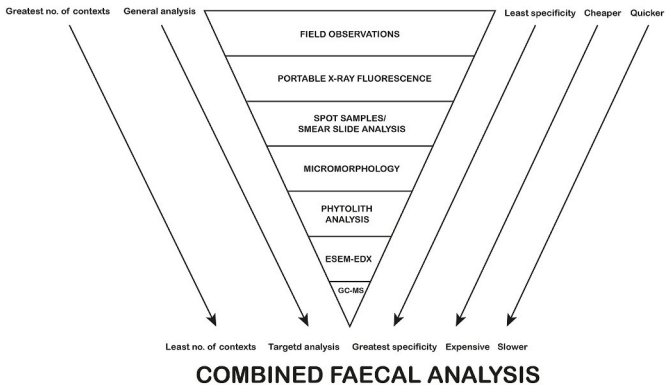


Fig. 2. Integrated, multi-scalar, multi-method approach utilized to detect, identify and investigate faecal material.

field, microscopic and chemical analyses of dung with use of modern ethnoarchaeological samples as comparative reference/control data. It also aims to evaluate the effectiveness of some methods which are not routinely used in dung analysis (e.g. pXRF and ESEM-EDX).

The research presented here expands on existing methodological approaches for the identification and analysis of faecal material and considers how they can be adapted to take into account the type and size of archaeological site. This research also expands on methods already being used for the identification and confirmation of ancient faecal material by using a combination of techniques: smear slide analysis, portable x-ray fluorescence (pXRF), micromorphology, phytolith analysis, GC-MS and Scanning Electron Microscopy with EDS (SEM-EDS), alongside ethnoarchaeological comparanda. The methods tested and presented in this research aimed to provide the highest potential to locate, identify, confirm faecal material and, once identified, to examine the specific research questions that can then be applied to faecal deposits. The overarching aim of this research is to develop a new and robust methodological research framework that establishes field and laboratory criteria for identification of faecal material by interdisciplinary analysis of dung type, contents and context. A key objective was to develop a 'tool-kit' for identification of dung remains in the field, so that interdisciplinary analyses in the laboratory could be effectively targeted on probable faecal materials to inform on their type, content and context and provide direct evidence on interactions and relations between animals, plants, humans and environments.

4. Case studies

The methodological approach evaluated in this research is based on samples from early Neolithic sites in the Zagros foothills or 'Hilly Flanks' (Fig. 3). A brief description of the three sites is provided below and the details of the excavations and other aspects of the research can be found in (Matthews et al. 2013a, 2020).

4.1. Sheikh e-Abad

The Neolithic mound of Sheikh-e Abad in the Iranian Zagros (Fig. 3) is more than 1 ha is size and located on the plains of the Dinavar region at 1430m asl (Matthews et al., 2013b). The mound represents substantial early Neolithic settlement from c.9800 to 7600 BC (Matthews et al., 2013b). Excavated layers are associated with ash, burning, cooking, architectural remains, storage spaces, midden areas and animal penning deposits (Matthews et al., 2013b).

4.2. Jani

Tepe Jani is located at a lower elevation in the Iranian Zagros mountains (Fig. 3) at 1280m asl near to Humeyl village and is similar in

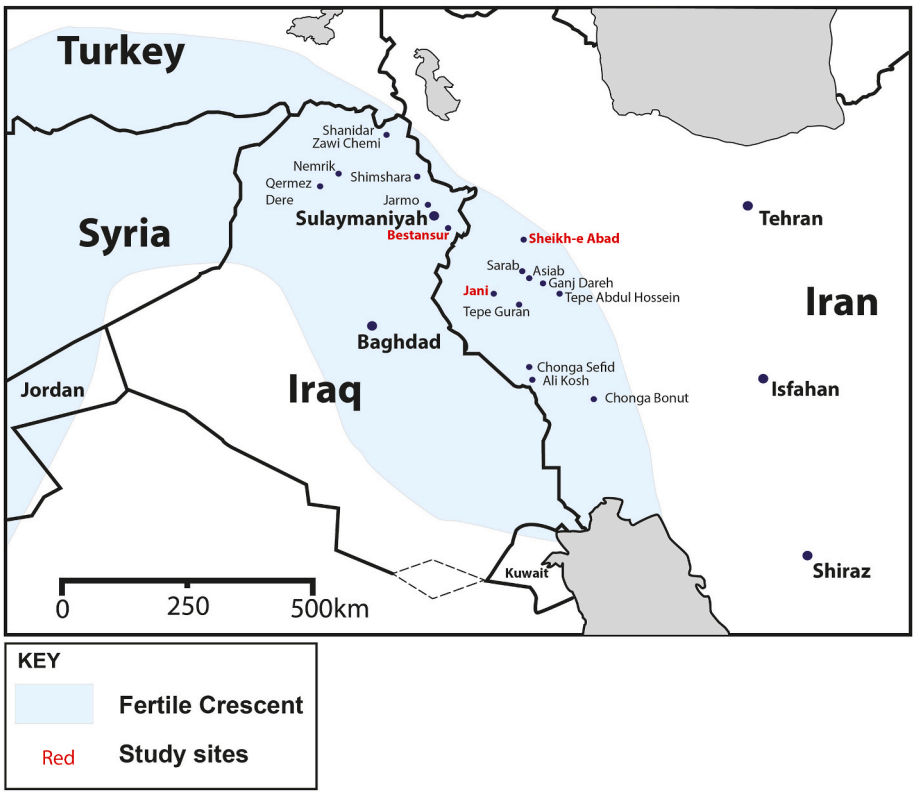


Fig. 3. Location of three case studies used in this research (highlighted in red).

size to Sheikh-e Abad (Matthews et al., 2013c). The main period of occupation is similar to Sheikh-e Abad dating to c.8160-7950 BC, although the earliest levels have not been dated (Matthews et al., 2013c). The investigated layers at Tepe Jani include a sequence of open-areas, middens, pits and architectural features spanning a depth of 8 m (Matthews et al., 2013c).

4.3. Bestansur

The Neolithic mound of Bestansur is a large site located in the Iraqi Kurdish Zagros Mountains (Fig. 3), and Neolithic architecture is preserved across an area which could be up to 4 ha, and is at a lower elevation than the Iranian Zagros sites at 559m asl overlooking a nearby spring (Richardson et al., 2020). The earliest levels at Bestansur are contemporaneous with levels at Sheikh-e Abad, and current C14 dates at Bestansur date to c. 7640-7170 BC (Flohr et al., 2020), however the earliest levels at Bestansur situated underneath the main mound have not yet been excavated or dated. The excavations have revealed a range or architecture, open areas, midden deposits, human burials and burnt materials (Richardson et al., 2020).

5. Methods and rationale

A range of methods was tested to locate, identify, confirm and then analyse faecal deposits within the range of sites in the Central Zagros Archaeological Project (CZAP). The methods and justification are described, and the protocols used are outlined below and in (Matthews et al., 2013d; Elliott, 2015; Elliott et al., 2020b). The multi-scalar, multi-method approach utilized quick, cheap analysis on a large number of contexts in the field to enable the selection of fewer more targeted contexts to implement the more time consuming and costly methods. See Fig. 2 for diagrammatic representation of overall archaeological approach.

5.1. Modern reference material rationale

A pilot ethnoarchaeological research programme was carried out to explore possible traces of livestock presence and management, and in particular signatures for animal dung (see Elliott et al., 2015; Bendrey et al., 2016). A small set of modern dung samples were collected with the associated contextual information for analysis (Elliott et al., 2015). The modern comparative dung reference collection was designed to aid archaeological interpretations of potential ancient dung deposits. A range of information was recorded from the analyses of modern faecal material including numbers of faecal spherulites and numbers and types of phytoliths in order to relate these to known animal diet. Herbivore and omnivore dung were also analysed in resin-impregnated thin-section samples to examine the microscopic visual properties.

5.1.1. Dung collecting, interviewing and analytical method

Samples were collected from modern animal pens and directly from known species, for integrated field characterisation and multi-method analyses, outlined in Elliott et al. (2015). Some of the samples from the animal pens were collected from mixed sheep and goat pens and therefore represent herbivores more generally rather than specific species. Specific samples of dung from sheep, goat and cow were also collected while monitoring the animals on their daily grazing routes; samples were collected directly after defaecation was observed. Pig coprolites were also analysed (supplied by Marta Portillo from Greece), but not collected from the study area because pig meat was not part of the local diet due to local customs and cultural practices.

The interview questions and observations were designed to collect information on the modern diet of herds. This enabled the information collected about grazing and foddering practices to be directly related back to the modern dung samples and analyses. To analyse faecal and phytolith content in the laboratory, the dung samples were combusted at 550 °C for 4 h to remove organic matter, then mounted onto glass slides with Entellan. Faecal spherulite and phytolith content was analysed

using a similar method to Katz et al. (2010) for counting phytoliths. This involved counting the spherulites and phytoliths in 20 Fields of View (500 μm per FoV). The spherulites and phytolith assemblages were then compared to the knowledge of animal diets and management.

One example of herbivore (sheep/goat) dung and one example of omnivore dung (pig) was impregnated with resin, cut, ground and mounted (Guilloré, 1985; Courty et al., 1989) onto a microscope slide for analysis including examination of colour, voids, texture, as well as the size and articulation of silica phytoliths, following internationally standardized descriptions (Bullock et al., 1985; Courty et al., 1989; Stoops, 2010), which were adapted to analysis of dung as outlined below.

5.2. Portable X-ray fluorescence. Rationale to identify phosphorus

The development of the miniaturized XRF tube has led to an explosion in new applications of portable X-ray fluorescence (pXRF) devices in the past decade (Conrey et al., 2014, p.291). Detection of elevated phosphorus levels can successfully identify faecal material in archaeological deposits, however because phosphorus is an indicator of other anthropogenic activities this method alone is often not an adequate field indicator of the presence of animal dung in archaeological deposits.

5.2.1. pXRF field method

At Bestansur in Iraqi Kurdistan (~2.5–4 ha) pXRF analysis was systematically applied in the field during excavation using a standardized procedure. The analyses were conducted using a Niton XL3t GOLDD + analyser. Because the aim was primarily rapidly to detect elevated phosphorus levels across the site, rather than precise concentrations, a short analytical duration was selected to maximize the number of locations that could be analysed on site. Each location selected was recorded and then analysed for 60 s, with 30 s fon the light filter (which detects phosphorus) (Fig. 4). In addition to on-site archaeological locations being tested, six off-site locations with no clear archaeological material were analysed as a baseline for detecting elevated phosphorus levels. The number and location of analyses at Bestansur are summarized in Table 1.

5.3. Spot sampling and smear slide analysis rationale

Spot sampling and smear slide analysis in the field are pivotal elements of the overall methodology in this research. The aim of these rapid and cost effective field methods was to test if elevated phosphorus levels relate to the presence of animal dung by investigating whether deposits contained faecal spherulites, which are a strong indicator of animal dung and are formed in the guts of animals during digestion (Canti,

Fig. 4. pXRF and smear slide analysis in the field at Bestansur, Iraqi Kurdistan.

Table 1
Number of pXRF analysis by trench, Bestansur, Iraqi Kurdistan.

	Number of analyses
Total Bestansur pXRF analyses	**279**
Trench 1	20
Trench 4	20
Trench 7	91
Trench 8	18
Trench 9	79
Trench 10	20
Trench 12	31

1997), as well as to gauge phytolith concentrations and key morphotypes. Smear slide analysis of spot samples in the field laboratories could also be applied without the initial use of pXRF analyses.

The most rapid method for detection of the presence of calcareous spherulites is by the analysis of spot samples from unprepared deposits in smear slides using an optical polarising microscope. Faecal spherulites produced in the animals gut during digestion and passed into the faeces can be identified by the cross of extinction and birefringent properties (Canti, 1997, 1998, 1999) (Fig. 4). Locations containing elevated phosphorus and probable faecal spherulites could then be targeted for further detailed laboratory analysis.

5.3.1. Spot sampling and smear slide field method

A range of the spot samples were examined at Bestansur, samples were selected for analysis based on elevated phosphorus levels and a small set of comparative samples (Table 2). These spot samples taken from the excavations were processed for smear slide analysis in the field laboratory. Smear slides were prepared by placing small sediment aggregates (approximately 300–400 mg) onto a labelled microscope slide with five drops of clove oil which was then mixed to disaggregate the sediment and spread the temporary mounting medium to the approximate area of the coverslip, the coverslip was lowered and then the slide was analysed on a Leica DMEP microscope and at x400 in crossed polarised light (XPL) (Fig. 4). Random fields of view were observed from the centre and periphery of the coverslip to identify faecal spherulites. Because this was a rapid assessment method in the field the sediment weight mounted onto the smear slides was not recorded and therefore not accurately standardised between samples (only a visual estimate of sediment quantity was applied), therefore quantification was not possible. Smear slides were recorded with either presence of absence of faecal spherulites and phytoliths.

5.4. Micromorphology rationale

Micromorphology samples for integrated analysis of faecal materials from Bestansur were selected from specific targeted locations identified by screening for elevated phosphorus using pXRF analysis and spherulite content through spot smear slide analysis. Micromorphological samples from the sites of Sheikh-e Abad and Jani were selected based on specialist observations, and in consultation with experienced excavators in the field.

Table 2
Number of samples analysed by smear slide analysis by trench, Bestansur, Iraqi Kurdistan.

	Number of samples selected with elevated P for analysis
Trench 1	6
Trench 4	1
Trench 7	20
Trench 8	3
Trench 9	21
Trench 10	6
Trench 12	11
Total Smear Slides Bestansur	**68**

5.4.1. Micromorphology method

Micromorphological samples were cut from sections using a Swiss army knife following standard sampling procedures (Bullock et al., 1985; Courty et al., 1989; Goldberg and Macphail, 2006) (Fig. 5), then wrapped tightly with laboratory tissue and clear tape and packaged for transportation (Fig. 5). Samples were opened in the laboratory and initially sub-sampled at 1 cm intervals; where possible ~1–5g was sub-sampled and stored for potential further analyses before being impregnated with resin, cut, mounted and ground to a thickness of 30 μm then coverslipped (Fig. 5) (Guilloré, 1985; Courty et al., 1989).

For each identifiable stratigraphic layer and boundary in the samples, detailed observation and description was carried out in plane polarised and cross polarised light (PPL and XPL) at both low and high magnifications (x40, x100, x200 and x400). The attributes were identified and recorded following standardised published terminology and descriptive criteria (Bullock et al., 1985; Courty et al., 1989; Stoops, 2010). To obtain the maximum information about the faecal material each specific faecal deposit identified in the microstratigraphic units within the micromorphology samples were recorded in detail by documenting 36 characteristics and attributes that are usually routinely applied to the microstratigraphic units as a whole (Bullock et al., 1985; Courty et al., 1989; Stoops, 2010). All criteria applied in the description of identified faecal material therefore follows the standard protocol for micromorphological description. However, instead of recording these attributes for the entire microstratigraphic layer, these attributes are recorded for each individual faecal deposit. The relevance of these characteristics in dung studies are detailed in Table 3.

5.5. Environmental scanning electron microscopy with energy dispersive x-ray spectrometry (ESEM-EDX) rationale

A variety of microscopic spherulitic particles including calcareous faecal spherulites exhibit a permanent cross of extinction under cross-polarised light (XPL) (Canti, 1998, 1999) (see SM table). If you have a polarizing microscope with a waveplate (also known as a lambda (λ) plate) this can be used to confirm faecal spherulites as the bulk of true faecal spherulites turn from white, to blue and yellow, in opposite quadrants always displaying a pseudo-uniaxial negative result (Canti, 1998). However, a lot of microscopic analysis for the identification of phytoliths, spherulites and even micromorphology samples can routinely be conducted on a biological microscope with the addition of a polarizing accessory/adapter. Biological microscopes do not have a lambda plate. It is possible for the analyst to identify these different types of micro particles on both 'true' polarizing microscopes as well as a biological microscope with an adapter. Analysts can work on a biological microscope which can be a fraction of the cost of the true polarizing microscope, but can be used in both plain and crossed polarized light with the adapter. This study has also used an ESEM microscope with EDX to establish whether particles identified as faecal spherulites could also be confirmed using an ESEM-EDX method, should the analyst have access to this equipment.

The majority of the other particles can also be optically differentiated from faecal spherulites either by size or the form of the polarization cross. Calcium oxalates and avian uric acid spheres, which are more likely found in association with faecal material from plant material and urine, are distinguished by size; calcium oxalates are much bigger (20–50 μm) and uric acid spheres being smaller (2–8 μm) (Canti, 1998) (see SM table). Unlike faecal spherulites, uric acid spheres also have extinction crosses which are often irregular, asymmetric and also move in unpredictable ways under crossed polarised light (Canti, 1998). The most challenging spherulitic particles to distinguish from faecal spherulites using normal optical microscopy are coccoliths which are produced in deep sea sediments and are similar in size to faecal spherulites.

Fig. 5. Sampling, packaging, sub-sampling and production of micromorphology slides.

Table 3
Micromorphological characteristics of dung types and taphonomy and their environmental, biological and contextual significance. Many of the characteristics are those used in internationally standardized micromorphological classifications, which can be applied irrespective of genesis, as based on morphology and geometric relationships. In this study and from Bullock et al., 1985), Courty et al., (1989), Stoops (2010).

Characteristic	Example	Significance for Interpreting Animal Dung Deposits
Dung Classification		Main allocated type of faecal material
Dung location		Specific location of dung deposit in samples relating back to sample description
Dung 'layer' thickness	(only applicable only when dung deposits form a layer)	Inferences about duration of animal occupation/penning. For example cattle pen occupied for 10–15 years resulted in 20 cm of dung after degradation (Shahack-Gross et al., 2003), and experimental work has shown that 1m of dung results in a 2–3 cm layer (Shahack-Gross et al., 2003).
Overall unique diagnostic		Used during analysis to highlight difference in forming dung classifications
Colour		Often indicates a clear distinction between herbivore and omnivore dung
Phytolith %		Concentration will vary depending on diet. Herbivores generally have a very high percentages compared with omnivores. Omnivores have a mixed diet compared with herbivores which solely consume on plants
Phytolith type	Monocots, Dicots, Multicells identified to genus	Type will vary according to diet. Therefore phytolith remains are indicative of animal diet, seasonality of grazing, foddering regimes. Grass rich and grass poor diets can be identified.
Phytolith size	Small, medium, large	May reflect taphonomy and preservation
Phytolith Articulation	Single celled or multicelled/conjoined	May reflect taphonomy and preservation, for example bioturbation. Herbivore dung contains more conjoined phytolith forms in comparison to omnivore dung which contains more single celled forms. Conjouined phytoliths may form a linear/parallel layer in penning contexts.
Phytolith preservation	Pitting, etching, dissolved, burnt, melted	Indication of overall preservation of dung deposit, may be the reason for low percentages in specific deposits. Melted silica could represent dung fuel
Spherulite %		High percentage of spherulites produced by Herbivores and low percentage by Omnivores. Although absence needs to take into consideration dissolution of spherulites due to high pH (above 6/7) or high temperatures (650–700 °C). Spherulite numbers are higher in modern dung samples compared to archaeological deposits (Albert et al., 2008).
Spherulite details	Single, grouped, faint	Could indicate preservation, bioturbation, masking by deposits and indicate Herbivore vs. Omnivore (see above)
Spherulite size	<5μ, 5μ, 10μ, >10μ	Could potentially link to species. More research needed on spherulite morphology. This parameter could become useful in the future. Generally spherulites are beween 5 and 15 μ
Visibility of Spherulites and phytoliths	Masked, clear	Indicates how easily identified dung deposits are. Dung could be present but visible poor to locate
Quick reference Matrix details	Dense with planes and laminated or spherulites not within dung matrix	Used during analysis To highlight difference in forming dung classifications. Deposits without dung matrix could indicate mixed deposits or fully combusted dung
Observational visibility-light (PPL/XPL)	Spherulite outline visible in PPL and cross of extinction visible in XPL	Indicates how easily identified dung deposits are. Dung could be present but visible poor to locate
Microstructure (and voids)	Vughy/spongy, planes/ channels/chambers	Size, shape and arrangement of grains/aggregates/voids within dung deposits could vary between dung types and certain voids could indicate trample and compaction during penning (e.g. channels and planes, indicating microlamination from trampling)
Related distribution	Embedded, linked and coated, coated, intergrain aggregate	The relationship between coarse and fine material may represent significant differences between dung types or may represent post depositional alterations such as compaction/trampling
Orientation	Strongly orientated parallel	Inclusions such as phytoliths could be distributed parallel indicating trample and compaction during penning. Microlaminations orientated parallel further indicates trampling. The perpendicular arrangement of inclusions and microlaminations represents the direction of force from above
Distribution	Linear	Inclusions such as phytoliths could be arranged linear to the boundary indicating trample and compaction during penning
Particle size	Silty clay, silt loam	There may be significant variation in particle size between different dung types. Particle size may also reflect the presence of micro particles and how susceptible these are to movement within a dung deposit or specific micro unit. Also particle size

(*continued on next page*)

Table 3 (*continued*)

Characteristic	Example	Significance for Interpreting Animal Dung Deposits
		may affect the degree of post-depositional alteration of dung deposits for example by water drainage, or bioturbation by small roots
Coarse/fine ratio	10:90	Ratios may differ between types of dung
Coarse/fine ratio limit	Fine particle limit-10 μm, beyond 10 μm = coarse material	Additional details given to coarse/fine ratio
Sorting	Well sorted, unsorted	Indicates degree of variability or uniformity within the dung deposit
Fine material (less than 20μ)	Organic or mineral	A higher percentage of organic material is indicative of herbivore dung. Higher mineral content may be indicative of omnivore dung, specifically pig or wild boar which are prone to rooting for food rather than grazing. Alternatively fine mineral material in addition to fine organic material may represent a mixed deposit. A defined dung deposit may contain only organic fine material.
Fine material colour	Brown, orange, grey	Gives the deposit the overall appearance of colour and can generally be related back to species
Birefringence fabric, and minerals	Undifferentiated, Crystallitic, Isotropic	Omnivore dungs generally have an undifferentiated/ isotropic birefringence compared to herbivore dungs which are generally crystallitic
Other plant remains, type	Charred, calcitic ashes	Plant burning at low temperatures (charred plant remains) or high temperatures (calcitic ash). When associated with spherulites indicates use of dung as fuel. Charred plant remains may originate from dung or input of other fuel such as wood.
Other plant remains, %		Could distinguish between dung fuel (lower percentage of charred plant remains and higher percentage of ash) and perhaps duel fuel; wood and dung (equal ash to charred plant remains). Also indicative of burning temperatures. Charred plant remains only represent burning at low temperatures
Other plant remains, size	Small, medium, large	Could be indicative of preservation, bioturbation, fragmentation, taphonomy
Other inclusions, type	Bone, molluscs	Presence of bone in dung indicates omnivore dung. Omnivore diet includes meat, bones, carrion etc.
Other inclusions, %		Could distinguish between different dung deposits, a large percentage of bone inclusions could represent dog, pig or wild boar as opposed to human
Other inclusions, size	Small, medium, large	Could be indicative of preservation, bioturbation, fragmentation, taphonomy. Small, fragmentary and partially digested bone is indicative of omnivore dung
Microbiological inclusions	Parasites and coprophilous fungi	Could specifically be analysed in the future. The presence of coprophilous fungi, is a further dung indicator-it is known as 'dung-loving' fungi. Specific parasites may be able to be related back to species
Microbiological %		A high percentage of parasites in the absence of other dung markers (such as spherulites) could further help identify and confirm dung locations
Post depositional features	Bioturbation, compaction, shrink/swell	Could be factor affecting preservation, mixing of deposits or movement of micro particles such as spherulites and phytoliths. Should be taken into consideration during analysis

Like faecal spherulites, coccoliths are composed of calcium carbonate and exhibit the same permanent cross of extinction (Canti, 1998). While coccoliths are not common in archaeological settings (Young, 2020) and are less likely to be present compared with other microparticles due to the distance from seawater, they could enter through erosion of limestone (Morandi, 2020) where they have been commonly observed (Fischer et al., 2017). Coccoliths have also been observed in Neolithic lime plaster (Grissom, 1996). The lower foothills of the Zagros mountain range contains Upper Triassic well bedded limestone deposits (Maran and Stevanovic, 2009) therefore it is a possibility for coccoliths to be present in these samples. Under scanning electron microscopy (SEM) their form and structure are very distinctive (see SM Table).

Starch grains can usually be distinguished optically, alongside the dark cross of extinction they primarily have a low order white birefringence, meaning that they appear white under reflected light rather than yellow/orange which is a crucial observation in order to rule out misidentification between starch and faecal spherulites (Haslam, 2006, pp.115-116). Additionally when viewed in crossed polarized light when the stage is rotated the arms of the polarization cross shifts and waves (in the majority of cases see Ramsey and Nadel, 2021, p.5 for exceptions) whereas on faecal spherulites the polarization cross will not move (Ramsey and Nadel, 2021). This is because starch grains are not generally spherical or radially symmetrical which results in an irregular cross which is distorted because of grain structure (Ramsey and Nadel, 2021). Starch grains are best differentiated in a liquid mount so that individual starch grains can be rotated to view all aspects of the granule (Canti, 1998; Yeung et al., 2015, p.527) because the cross of extinction on starch grains is not always clear. In this study the archaeological material from the case studies is observed in resin impregnated thin section so the microparticles cannot be rotated to aid identification and therefore rule out the possibility that they are starch not faecal spherulites. Therefore, energy dispersive x-ray spectrometry (EDX) was also applied to see if it was possible to distinguish between faecal spherulites and starches based on their varying chemical composition. Unlike faecal spherulites which are composed of calcium carbonate ($CaCO_3$), starch grains are composed of glucose ($C_6H_{12}O_6$).

5.5.1. ESEM-EDX method

Samples were analysed to confirm the presence of faecal spherulites;

the small crystalline calcium carbonate particles usually 5–15 μm (Canti, 1999). A small sub-set of faecal material was selected for ESEM-EDX analysis from two of the study sites; three samples from Sheikh-e Abad, and one from Jani (Table 4). Well-defined faecal deposits were targeted and samples were selected where dense suspected faecal spherulites were identified by optical microscopy during micromorphological thin section analysis. The spherulites identified in all of these layers had a yellow to orange birefringence in XPL when viewed in samples ground to a thickness of 30-40μ. Of the four archaeological locations selected for ESEM-EDX analysis, there were two loose samples removed from the blocks prior to impregnation (Table 4, S3 & S4). These samples in addition to one location of in situ faecal material (Table 4, S7) were from Sheikh-e Abad thin section sample 804.01, an internal area of the site identified as being repetitively used for animal penning. The fourth location analysed was also and in situ faecal rich deposits from white plaster floors with probable faecal spherulites from Tepe Jani, thin section S6 (Table 4, S6). The two locations analysed directly in the micromorphological thin sections (S6 & S7) were selected in order to analyse the faecal material within the stratigraphic integrity.

Three comparative modern dung samples were also selected for analysis to provide control samples and references of known faecal spherulites for comparison to Neolithic samples (Table 4, S1, 2 & 5). Half of a raw modern sheep/goat dung pellet was examined (Table 4, S1) to enable in situ observation of the shape and appearance of the spherulites for comparison to the archaeological samples. Two loose burnt samples of modern sheep/goat dung were also analysed (Table 4, S2 & S5); these samples were burnt to reduce the organic matter and remove the organic coatings in order to facilitate visibility of faecal spherulites.

Loose sediments and the comparative modern dung material were placed directly on sticky carbon tabs fixed to SEM stubs. The targeted layers in micromorphological slides were etched directly onto the glass with a diamond pen and thin metal rods adhered to either side of the targeted layer; to enable the layers to be located at high resolution in the in the ESEM.

The samples were analysed on a FEI Quanta FEG 600 environmental scanning electron microscope. The energy dispersive x-ray spectrometry was carried out using an Oxford Instruments system and INCA software when potential faecal spherulites were located in the samples. Individual spherulites were pin pointed and analysed for 50 s and results for elemental concentrations produced via the INCA software as peaks based on weight % and atomic %, these were then normalised to provide a relative concentration of elements in each sample (see SM). This method is used to help distinguish between calcium carbonate and glucose (element mass, molecular mass and weight % details in SM). Hydrogen cannot be detected with EDX analysis, and carbon and oxygen are present in both calcium carbonate ($CaCO_3$) and glucose ($C_6H_{12}O_6$). Therefore, the important element to recognise which can be attributed to faecal spherulites is calcium.

5.6. Phytolith analysis rationale

Phytoliths are particularly abundant in herbivore dung particularly where diet is largely based on monocotyledonous plants. The aim in these case studies was to extract silica phytoliths from samples taken directly from significant dung layers or locations which had been identified in the micromorphological thin sections to contain substantial faecal material. Samples targeted for phytolith extraction were also selected from spot samples from the block of deposits from the animal pen identified at Sheikh-e Abad that corresponded with the micromorphological thin-sections and spherulite samples, prior to impregnation, to enable comparison of these integrated methods.

When phytoliths are located in situ in micromorphological samples they cannot be rotated to aid identification, can often be masked by other material or layered with multiple phytoliths on top of each other, and often they cannot be counted to a statistically quantifiable value for interpretation (Ball et al. 1996, 1999; Albert and Weiner, 2001; Piperno, 2006; Strömberg, 2009). Micromorphology therefore can confirm presence of a phytolith type, but the proportions of these types cannot be compared and interpreted between samples with certainty. Therefore a combination of micromorphology and phytolith analysis enables high-resolution micro-contextual analysis of faecal material and contents as well as statistical quantification of the phytolith assemblage (Shillito et al., 2008).

5.6.1. Phytolith analysis method

Twenty-seven samples have been included from the case studies; 13 from Sheikh-e Abad, 13 from Bestansur and one from Tepe Jani. The samples from Sheikh-e Abad were from compacted faecal layers identified as animal penning areas (Shillito and Elliott, 2013; Elliott, 2015), and the samples from Bestansur were from hearths to study fuel type, and an external midden area identified with faecal layers (Elliott et al., 2020b). The sample from Tepe Jani was from the dung tempered plaster floor (Shillito and Elliott, 2013). The samples for phytolith analyses were processed following the protocol developed by Rosen (1999). The

Table 4
Samples selected for ESEM-EDX, descriptions and EDX results for C, O and Ca.

Allocated sample number for ESEM-EDX	Modern or Archaeological	Description	Site and Thin section number	C			O			Ca		
				Wt %	Atomic %	Normalised Wt%	Wt %	Atomic %	Normalised Wt%	Wt %	Atomic %	Normalised Wt%
Sample 1	**Modern Dung**	**Sheep/goat dung pellet unprocessed**	n/a	0.4	33.5	24	0.7	48.9	46	0.0	0.6	1
Sample 2	**Modern Dung**	**Sheep/goat dung ashed**	n/a	3.0	45.9	34	3.6	41.7	41	1.4	6.5	16
Sample 3	**Loose archaeological sample**	**Defined dung layer**	Sheikh-e Abad 804.1	1.0	42.8	29	1.2	38.3	34	0.9	12.0	27
Sample 4	**Loose archaeological sample**	**Defined dung layer**	Sheikh-e Abad 804.1	3.2	34.2	24	6.2	49.2	46	0.5	1.5	4
Sample 5	**Modern Dung**	**Sheep/goat dung ashed**	n/a	1.0	39.9	24	1.2	35.6	29	1.6	19.5	40
Sample 6	**In situ archaeological unit**	**Dung tempered floor plaster**	Tepe Jani TJ S4 (Unit 2)	2.6	41.3	29	3.6	43.5	40	2.2	10.5	24
Sample 7	**In situ archaeological unit**	**Defined dung layer**	Sheikh-e Abad 804.01	4.1	53.5	42	3.6	34.8	36	0.3	1.1	3

samples were screened through a 0.5 mm mesh to remove coarse sized particles; then calcium carbonates were dissolved using a dilution of 10% hydrochloric acid; clay was removed using a settling procedure and sodium hexametaphosphate; samples were placed in a muffle furnace for 2 h at 500 °C to remove organic matter; phytoliths were then separated from the remaining material using sodium polytungstate (SPT) calibrated to a specific gravity of 2.3; phytoliths were then mounted onto microscope slides, using the mounting agent Entellan.

Microscope slides were examined under a Leica DMEP transmitted light microscope at magnifications ranging from x200 to x400. Full counts were attained by counting a minimum of 250 identifiable phytoliths. Identification of phytoliths was based on reference guides (Wang and Lyu, 1992), University of Reading comparative modern reference collections and online databases (Fuller et al., 2007; Albert et al., 2014).

5.7. Analysis of faecal sterols and bile acids: Gas chromatography, and gas chromatography mass spectrometry (GC & GC-MS) rationale

GC-MS analysis of the bile acids and coprostanols can detect whether biomolecular traces of dung are present. This method is therefore utilized to confirm the presence of faecal material and to ascertain whether the allocations of ruminant and omnivore dungs visually in the micromorphological thin sections is correct. A further aim was to find out whether the deposits categorized as omnivore originate from pig or humans. This differentiation cannot be identified by micromorphological analysis alone as they exhibit similar morphology.

Faecal sterols are a sub-group of steroids and the sterol fraction of faecal residues contain a range of biomarkers found in the faeces of several species. The presence of molecules in varying proportions can be specific to certain species (Elhmmali et al., 1997; Bull et al. 1999b, 2002, 2005; Bull and Evershed, 2012; Shillito et al., 2013). Therefore, GC-MS can be used to confirm the both microscopic identifications of dung, and also ascertain whether these traces may indicate which genus of animal produced the dung. Distinction between ruminant and omnivore coprolites is therefore possible. Using stanols alone does not enable a distinction between pig and human faeces; however, this is possible by analysis of bile-acids. Pig faeces contains predominantly hyodeoxycholic acid (3α,6α-dihydroxy-5β-cholanoic acid) with an absence of deoxycholic acid (3α,12α-dihydroxy-5β-cholanoic acid). Human faeces contains lithocholic acid (3α-hydroxy-5β-cholanoic acid) and is dominated by deoxycholic acid (3α,12α-dihydroxy-5β-cholanoic acid) (Bull et al., 1999b, pp.87-90).

5.7.1. GC-MS method

Twelve sub-samples from Bestansur were selected based on the identification of faecal material in the micromorphological thin sections and were processed at the Life Science Mass Spectrometry Facility (NERC LSMSF, Application No. LSMSBRIS049) using the LSMSF standard protocol based on Bull et al. (1999a, pp.538-541) for sterol biomarkers, and a modified version of the methodology proposed by Elhmmali et al. (1997, pp.3663-3665) for bile acids (see SM for details).

The GC and GC-MS analyses were conducted on both the neutral and acid fraction using a Thermoquest Trace MS s operated in election ionisation (IE) mode and has a CTC A200S autosampler. The samples were ionised then the mass analyser separated the positively charged ions according to mass properties. The ions then passed through a detector which sends information to a PC which then converted the signals into a visual output. The GC-MS peak assignments were made by comparison with known mass spectra and comparing retention times of authentic compounds followed by co-injection (Shillito, 2011, pp.30-31).

6. Results and discussion: Applying the methods to case studies

6.1. Ethnoarchaeology, Bestansur, Iraqi Kurdistan

A small pilot study of modern dung samples has previously been published for the spherulite and phytolith results of the sheep/goat and cow dung (Elliott et al. 2015, 2020a). The results from additional species-specific dung (sheep, goat and pig) alongside the micromorphological thin sections are presented here to compare to the already published ethnoarchaeological data.

The highest faecal spherulite numbers were counted in the sheep dung, and lowest numbers in the pig and cow dung (Fig. 6). One sheep dung sample from a juvenile sheep also had low numbers of faecal spherulites. This result could be a result of differential spherulite production in immature animals as young animals do not produce high numbers of faecal spherulites (Brochier et al., 1992).

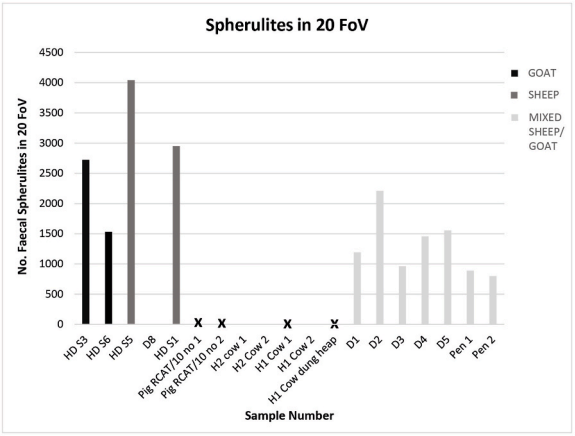
Overall, we can discern a clear general pattern in the characteristics of these modern baseline dung samples. Faecal spherulites are present in high numbers in mature sheep and goats (>798 spherulites in 20 fields of view (FoV)), but are low to absent in the dung produced by young sheep (only 4 spherulites identified in 20 FoV), pigs (3–7 in 20 FoV) and cows (0–2 in 20 FoV) (Fig. 6). Both of these patterns correspond with observations by Canti (1997, 1999). Although previous research has indicated that ruminants produce high concentrations of spherulites, the cow dung included in this study from Bestansur had only a few. This absence/scarcity of faecal spherulites in cow dung is in contrast to a later study from Bestansur in which faecal spherulites were detected in cow dung (Portillo et al., 2021) as well as a study of cow dung studied from modern samples in Syria (Portillo et al., 2014) but corresponds with absence of faecal spherulites in cow dung from Iceland (Milek, 2012). This highlights the potential for regional and/or seasonal or age-related variations in the production of spherulites and the need for more extensive modern reference collections and dung studies. Geology, diet, age and post-depositional conditions, as well as other factors, could affect the production and the preservation of faecal spherulites.

The phytolith data has previously been published (Elliott et al. 2015, 2020a), but is summarized here. All dung samples were dominated by monocotyledon phytoliths indicating a diet consisting of mainly grasses. The goat dung contained 11% dicot phytoliths in both samples. The remaining two sheep dung samples contained 1–3% dicot phytoliths. A range of phytolith types were identified in the dung samples including *Triticum* (wheat), *Hordeum* (Barley), *Phragmites* (reeds), poaceae leaf/-stems, poaceae awns, C4 chloridoideae grasses, C4 panicoid grasses, C3 pooideae grasses and dicotyledonous shrubs/trees. The majority of the samples are dominated by grass leaf/stem phytoliths.

The micromorphological analyses of dung pellets in thin-section revealed clear microscopic differences between samples (Figs. 7 and 8). The sheep/goat dung was brown in colour and contained high abundance/concentrations of both spherulites and phytoliths. Phytoliths constituted 70–80% of the field of view (singular and multi-celled) in plain polarised light (PPL) and the spherulites constituted 60–70% of the field of view in crossed polarised light (XPL) (Fig. 7). The spherulites are masked by organic matter, phytoliths and sediment but clearly identifiable in thin section. The samples were dominated by leaf/stem conjoined phytoliths. Comparatively the pig coprolite was distinctively orange in colour and contained significant concentrations of phytoliths, but fewer spherulites, at only 1% (Fig. 8). There were fewer phytoliths in comparison to the sheep/goat dung samples, representing only 50–60% of the field of view; the phytoliths were single and multi-cells dominated by leaf/stem forms. The phytoliths were more masked by organic remains in the pig coprolite which hindered visibility.

6.2. Portable x-ray fluorescence, Bestansur, Iraqi Kurdistan

Two hundred and seventy-nine pXRF readings were taken at Bestansur, which took approximately five consistent hours of analysis time. Of the readings taken, 161 of these had elevated phosphorus levels (i.e. phosphorus was detected by the analyser) and the remaining were less than the limit of detection for phosphorus (<LOD). The lowest elevated phosphorus value was 612 ppm and the highest was 4508 ppm (values > 4000 ppm corrected for calibration issues) (Fig. 9). The mean,



Species	Sample Number	Number of spherulites
Goat	HD S3	2723
Goat	HD S6	1530
Sheep	HD S5	4041
Sheep (young)	D8	4
Sheep	HD S1	2950
Pig	Pig RCAT/10 no 1	3
Pig	Pig RCAT/10 no 2	7
Cow	H2 cow 1	0
Cow	H2 Cow 2	0
Cow	H1 Cow 1	2
Cow	H1 Cow 2	0
Cow	H1 Cow dung heap	1
Sheep/Goat	D1	1194
Sheep/Goat	D2	2210
Sheep/Goat	D3	962
Sheep/Goat	D4	1456
Sheep/Goat	D5	1554
Sheep/Goat	Pen 1	890
Sheep/Goat	Pen 2	798

Fig. 6. Numbers of faecal spherulites in modern dung samples, crosses indicate values <10 (see adjacent table for details).

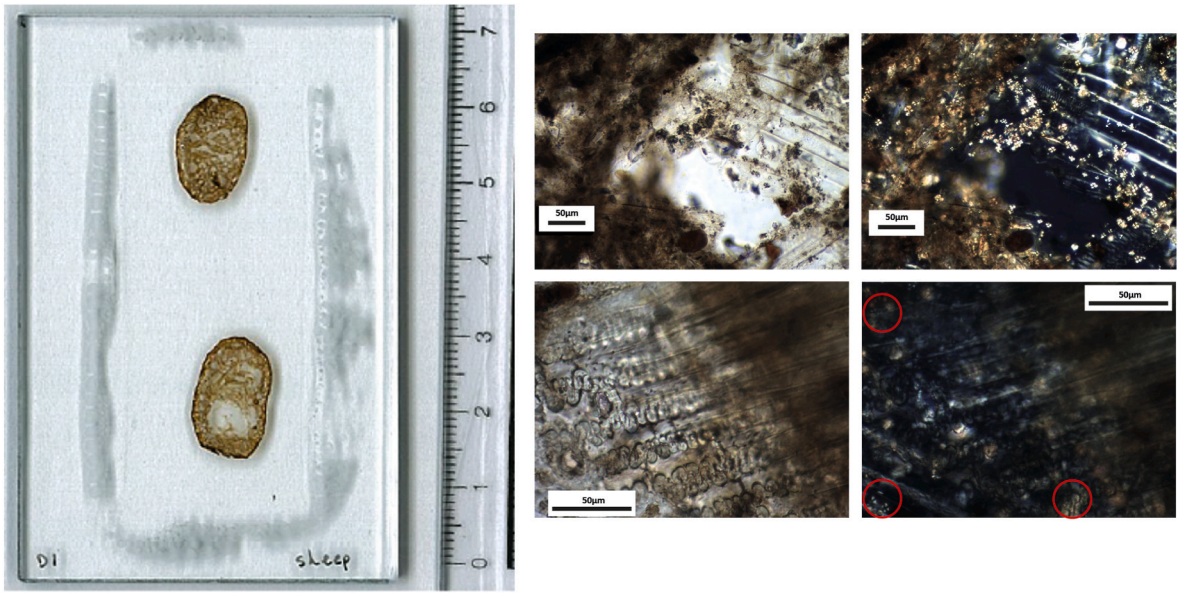


Fig. 7. Thin section through sheep/goat dung with photomicrographs showing brown groundmass, poaceae multicelled phytoliths and numerous faecal spherulites.

minimum and maximum values for the 161 samples with elevated phosphorus are presented in Fig. 9. The comparative off site control samples were <LOD for phosphorus.

6.3. Spot sampling and smear slide analysis, Bestansur, Iraqi Kurdistan

Sixty-eight of the spot samples with elevated phosphorus were analysed in smear slides, in addition to 18 with <LOD phosphorus which were randomly selected. In all 68 samples with elevated phosphorus probable faecal spherulites were also identified (see Fig. 4). These faecal spherulites were spherical particles with a cross of extinction with birefringent properties like those identified in the modern dung samples. However, probable faecal spherulites in low concentrations were also identified in 15 of the 18 samples examined under the microscope with <LOD for phosphorus.

6.4. Micromorphology; Sheikh-e Abad, Jani and Bestansur

Faecal material was identified in the resin-impregnated thin sections from all three case studies in this pilot study. For a full description of the results see Elliott et al. (2020b). A summary of the main faecal types identified which are relevant to the additional high-resolution analyses is summarized here.

At Sheikh-e Abad both omnivore and herbivore dung were identified (Matthews et al., 2013d) in a range of contexts including midden deposits, penning deposits, external areas and some limited evidence of faecal material associated with burning (Elliott, 2015). The most significant faecal deposits are repetitive, compacted, micro-laminated, inter-bedded faecal layers located in three areas of the site, indicating penning layers. In one small room 25 faecal lenses were identified; 16 as herbivore, three as omnivore, and six as mixed herbivore/omnivore (Elliott, 2015). Some of these faecal lenses from the three penning areas were targeted for the phytolith and ESEM-EDX analysis, results discussed below.

At Jani the most significant faecal deposits were identified from two internal rooms as a component of floor plasters; these faecal deposits were characterised as herbivore dung based on the microscopic analyses (Elliott, 2015). Differences were observed between the floor deposits in the East and West building. The floors in the West building had clearly discernible faecal material and a higher component of ash and charred material. While in the east building dung tempering of floor plaster was identified by presence of faecal material with more limited ash and burnt material. There was also some evidence of use of dung as fuel in fire deposits but limited evidence for faecal material in discarded

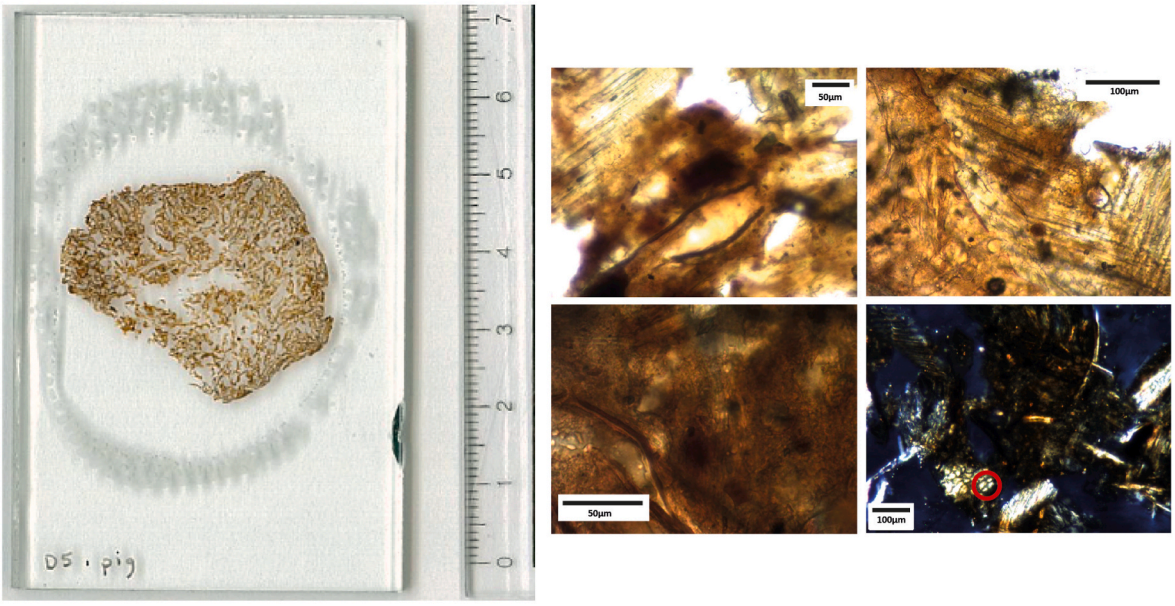


Fig. 8. Thin section through pig dung with photomicrographs showing orange groundmass, poaceae multicelled phytoliths and singular faecal spherulites.

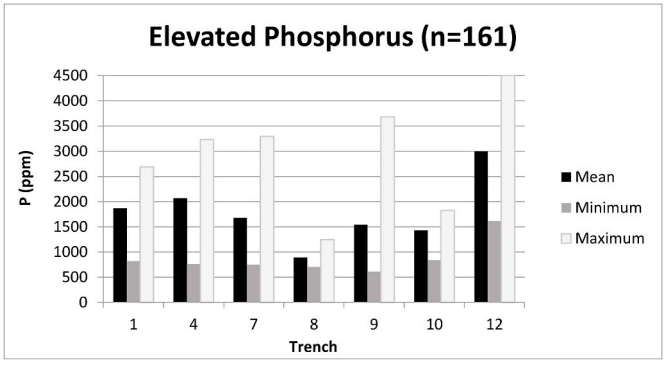


Fig. 9. pXRF results for all readings with phosphorus > LOD at Bestansur by Trench showing mean, minimum and maximum values for phosphorus in parts per million (ppm).

midden deposits. The faecal tempered floor plasters from Jani were targeted for additional ESEM-EDX analysis, results discussed below.

At Bestansur a large number of thin sections was examined for this study, 33 of these included identifiable faecal material. The faecal material at Bestansur identified was dominated by: deposits characterised as omnivore, particularly in the northern area of the site in repetitive discarded deposits; and burnt/ashy deposits with both herbivore and omnivore faecal material, which were most abundant in the southern area of the site (Elliott, 2015). The remainder of the site had only low levels of sporadic singular faecal spherulites which were identified as low-level faecal material of uncertain origin.

6.5. Phytolith analysis, Sheikh-e Abad, Iran and Bestansur, Iraqi Kurdistan

Phytolith analysis was only applied to discreet faecal layers and concentrated faecal deposits to ensure that the phytolith content related to faecal material. Processing of samples from mixed deposits or non-discreet faecal deposits was avoided to ensure no interference of background signatures. Thirteen samples from Sheikh-e Abad faecal layers are summarized here. The data from five of these samples have previously been published in Shillito and Elliott (2013) and an additional eight samples presented in Elliott (2015). Thirteen samples are also presented here from Bestansur, published in full in Elliott et al. (2020b). The comparative findings are summarized below for each site.

Twelve of the samples analysed from Sheikh-e Abad are dominated by grass/herb phytoliths (monocotyledons), and the thirteenth sample contains only tree/shrub (dicotyledons) phytoliths (804.01 ss2, Fig. 10). In the 12 samples dominated by monocots, multicelled phytolith forms which are more readily identifiable to genus were identified. These comprise wheat, barley, sedge and reed phytolith morphotypes. However, poaceae leave/stem multicells dominate these samples (Elliott, 2015) with one sample showing more elevated poaceae husk phytoliths (S804.02 ss3, Fig. 10).

All 13 samples from Bestansur comprised a mixed monocot/dicot assemblage dominated by monocots with phytoliths from both leaf/stem and inflorescence (see Elliott et al., 2020b). The faecal samples with elevated dicots (>10%) are in deposits allocated to omnivore faecal types. Of the multicelled phytoliths, wheat, barley and reed phytolith morphotypes were identified (Elliott et al., 2020b).

6.6. ESEM-EDX, Sheikh-e Abad and Jani, Iran

ESEM and ESEM-EDX analysis was conducted on spherulites from modern dung samples, and samples from Sheikh-e Abad and Jani to evaluate this as a method for characterizing faecal spherulites and confirm or refute their identification. In particular, these methods were used to try and distinguish faecal spherulites from potential starch grains (ESEM-EDX) and coccoliths (ESEM) (see section 5.5). All spherulites examined were between 5 and 15 µm in size. While there is an overlap at the lower end in size for uric acid, and at the larger end in size for calcium oxalates, the size of the microparticles with a cross of extinctions observed in these samples are typically larger than uric acid and typically smaller than calcium oxalates. Calcium oxalates are also visually very different under SEM (see SM), and uric acid spheres have extinction crosses which are often irregular, asymmetric and also move in unpredictable ways under crossed polarised light (Canti, 1998). For these reasons uric acid and calcium oxalates were already discounted. Visually coccoliths were subsequently discounted using ESEM based on their visual morphology, and then the remaining observed spherical particles were analysed using ESEM-EDX analysis. The element of interest when distinguishing starch from faecal spherulites is calcium because ESEM-EDX cannot detect hydrogen (elements < atomic number 6 are not detected using EDX) and is unreliable for oxygen (EDX is only

Fig. 10. Results of the phytolith analysis for single celled forms from Sheikh-e Abad.

reliable at atomic number 11 and above). Carbon is present (but in different weight percents) in both faecal spherulites (calcium carbonate) and starch (glucose), therefore calcium was the important element for recognising faecal spherulites. While uric acid had already been discounted based on particle size, the majority of uric acid spheres being 5 μm (Canti, 1998, p.442) it was not possible to use ESEM-EDX to further support this exclusion because nitrogen detection is also unreliable using EDX analysis because of its low atomic number (EDX is only reliable at atomic number 11 and above).

All of the geochemical EDX results are presented in SM. In all modern and archaeological samples, the proportions of elements, represented by the normalised weight per cent (i.e. the relative concentration of the element in the sample) do not indicate a pure composition of either calcium carbonate or glucose, and therefore incorporate background 'noise' detected by the EDX analysis, whether from the mounting stubs, or other components of the faecal material and sediments. The two samples analysed in resin (part of the micromorphological production) may be affected by the presence of resin in the voids. These elements include aluminium, silica, magnesium, phosphorus, potassium and iron. The discussion of results below therefore focuses on the presence of calcium as this will enable a distinction between starch and faecal spherulites.

The ESEM optical results demonstrate that all the spherulitic particles observed by ESEM in the modern and archaeological samples are

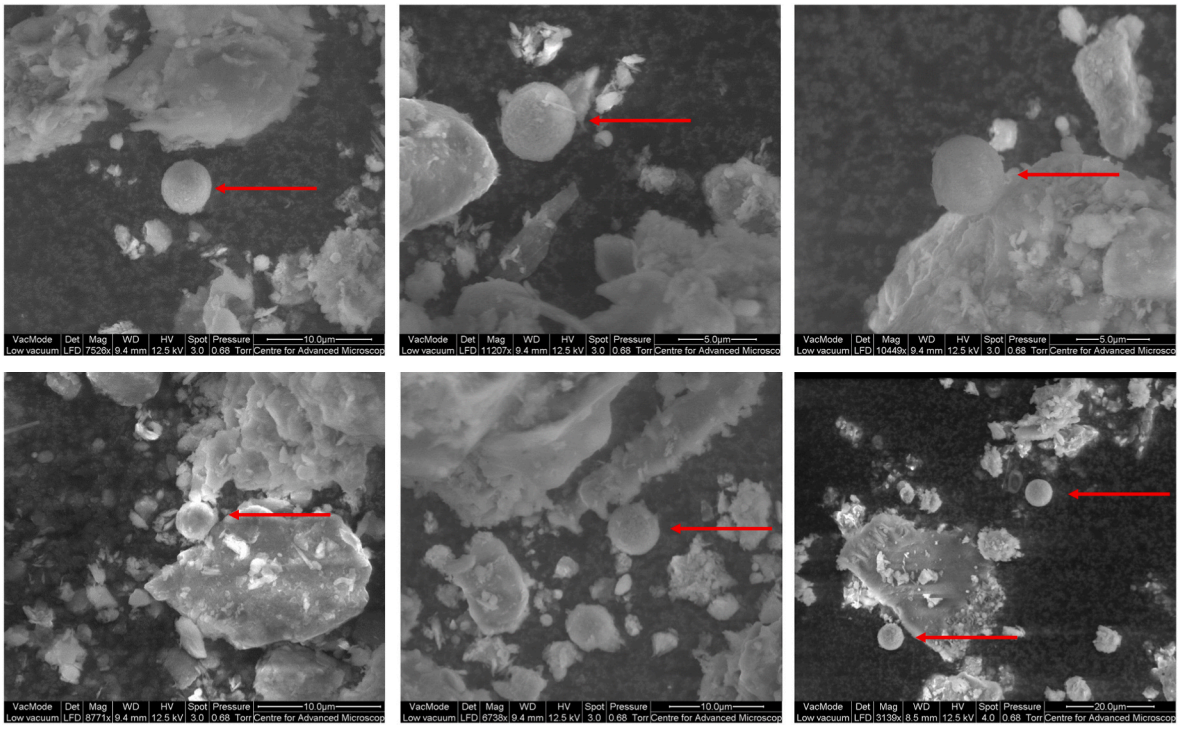


Fig. 11. Examples of the particles observed using SEM. Spherical particles resembling faecal spherulites. Spherulites observed by SEM analysis from archaeological dung, analytical Samples S3 (top three pictures) and S4 (bottom three pictures). Bottom middle, spherulite next to single celled phytoliths (bulliform and long celled phytolith).

spherical with an organic coating and do not resemble coccoliths (e.g. Fig. 11). The spherulites observed in the modern dung samples provide a good baseline data set for comparison to the archaeological dung samples. All of the spherulitic particles observed with ESEM were similar when examined by optical microscopy. The argument that the spherulitic particles in the dung deposits at all sites may be coccoliths, therefore, is unlikely.

The EDX results for all of the samples analysed, both the modern and the archaeological, are not conclusive due to the presence of background 'noise'. Calcium, however, was identified in all samples, ~1–5% in 3 samples, ~15–27% in 2 samples and ~40% in the final sample (S5) (see SM). On their own, these EDX results cannot be used to distinguish between faecal spherulites and starch because the elements do not represent the correct weight percent for each element for each molecular mass (see SM). The results are likely to represent a mixture of the spherulite and a background signal. However, the combination of the ESEM, EDX, and the microscopic micromorphological observations provides irrefutable evidence that these spherulitic particles are faecal spherulites and not starch or coccoliths. This conclusion is based on the presence of calcium (EDX results), SEM spherical morphology and the yellow birefringence (optical microscopy), neither of which are present in starch.

6.7. GC and GC-MS, Bestansur

Eleven samples from Bestansur were analysed by GC-MS to verify whether the dung deposits identified microscopically are of faecal origin of and to aid identification of producer genus by examination of coprostanols and bile acid molecules. The likely origin of faecal sterols was calculated by identification and comparison of the ratios of these different biomolecular markers. Results are published in detail in (Elliott et al., 2020b), a summary of the results is presented below.

From the analysis of coprastanols no faecal component was evident in four samples. the remaining seven did have a faecal component; two attributed to ruminant and five as omnivore (Elliott et al., 2020b). These GC-MS attributions correspond with and confirm the identifications of faecal material type in the micromorphological analyses of dung in thin-sections (i.e. if allocated as herbivore microscopically, confirmed as herbivore by GC-MS). Four of the five omnivore origin samples were analysed for bile acids to distinguish between wild boar/pig and human origin; three were identified as human faecal material and the fourth as originating from wild boar/pig (Elliott et al., 2020b).

7. Discussion and conclusions

Ancient faecal material is becoming a highly valuable more frequently used proxy to address a wide range of research questions in archaeology, but as yet there is currently no standardised definitive protocol or criteria for the identification and analysis of animal dung. As diverse lines of research can be pursued through investigation and analysis of faecal material (Shahack-Gross, 2011; Lancelotti and Madella, 2012; Fuks and Dunseth, 2021, and references therein) it is perhaps not surprising that researchers who study faecal material have not established a standardised methodology. Furthermore, the selection of an appropriate methodology is often related to the specific research questions that each project is investigating. Previous studies have illustrated that most if not all of the proxies can be unreliable especially when used singularly (Lancelotti and Madella, 2012) and has highlighted the need for a combination of techniques in dung studies (Shahack-Gross, 2011; Lancelotti and Madella, 2012; Portillo et al., 2012; Portillo and Albert, 2014).

In a summary paper looking at the legacy of Eric O. Callen, who was regarded in the 1970's as the leading authority on ancient human faecal material, Bryant and Dean (2006) refer to the study of coprolitic material, and specifically people who analyse it, as "jacks of all trades and masters of none". They highlight the need for people who study coprolites (and the same can be applied to the study of faecal material more generally) to be broadly trained and required to have expertise in many fields, including but not exclusive to: archaeology, anthropology, botany, zoology, palynology, entomology, parasitology, genetics, chemistry, microscopy (Bryant and Dean, 2006). In particular we would add geoarchaeology and the many specialisms under this umbrella to this list.

This research here has illustrated a range of the methods which can be used in conjunction with each other to locate, identify and analyse faecal material. These results demonstrate that an integrated, multi-scalar, multi-methodological approach enables detection, identification, and investigation of a range of attributes and implications in the study of faecal material. In this paper, a combination of methods (Fig. 2) has been applied to one modern and three Neolithic case studies in the Zagros region. While many of these methods are already used in dung studies, and some in conjunction with each other, the combination and integration presented here has not been applied before, and is evaluated, discussed and reviewed here, and where relevant the results are related to key research questions.

7.1. Field methods

This research presents the first experimental use of a portable geochemical method (pXRF) to detect phosphorus in the field during excavation as a rapid screening method for identification of potential faecal material. The constraints and limitations of pXRF have been recognised in studies of soils, ceramics and minerals (Frahm, 2012; Goodale et al., 2012; Conrey et al., 2014). The specific aims in applying pXRF analysis in this research was to detect elevated levels of phosphorus as a potential indicator of faecal material; thus identifying potential presence or absence rather than specific identification or recording of exact quantifiable values. Therefore, the advantages of the in situ pXRF analysis outweigh the reduced accuracy of the elemental results for this specific purpose. Nearly 300 analyses were taken in the field in approximately 5 h direct analysis time. Therefore, for the purposes of this research pXRF was a valid method. However, more rigorous testing, on more case studies is required to fully establish the applicability of this approach. Also, sites from different archaeological periods should also be examined using pXRF analysis.

Because phosphorus may be an indicator of a wide range of materials and activities (Akyol and Demirce, 2005; Holliday and Gartner, 2007) in addition to faecal material, areas with elevated levels of phosphorus detected by pXRF analysis were then tested by microscopic smear slide analysis. This was conducted in the field laboratory to identify potential faecal spherulites. This approach has been previously utilized (Matthews et al., 2013a), but not in conjunction with pXRF analysis.

For the Bestansur pilot case study, the locations that were selected for smear slide analysis which had elevated phosphorus (68 locations) all contained faecal spherulites and phytoliths. However, as a control, 18 locations with <LOD phosphorus levels were also examined for faecal spherulites, and 15 of these also contained potential faecal spherulites. Only three samples which had < LOD for phosphorus did not contain faecal spherulites. In the 15 locations where the potential faecal material was identified by the smear slide analysis, the phosphorus is likely to be too low to be detected by the portable analyser, but faecal material is present in these locations in low amounts. This highlights a disadvantage of the portable analyser in that the analyser has lower detection limits than laboratory based XRFs or ICP analysis. One of the main conclusions from the integrated methods used at Bestansur (Elliott, 2015) is that many areas of the site had ubiquitous low levels of faecal material.

The presence of dung at Bestansur was concentrated in some types of contexts, for example: ash/burning/hearth deposits, as well as layered discarded materials. But faecal material was also identified in many external contexts in low levels. However, based on these results, the next step to test this pXRF method as a more routinely applied field approach to target faecal material would be on a site with more defined areas

where animal occupation was kept separate from human activity, and therefore elevated phosphorus and identification of potential faecal spherulites in smear slides could be better evaluated as a combined approach. We could argue that the analyst would aim to target areas for smear slide analysis (and any further analyses) with the higher elevations of phosphorus which would represent more concentrated faecal material, not the low levels of faecal material. The lower levels of phosphorus, or levels technically < LOD would likely be discounted because background faecal material is unlikely to answer specific research questions such as identifying animal management through penning deposits, or identifying dung used as fuel. Therefore this result does not necessarily provide a negative outcome of this field approach. At Bestansur, these more elevated levels of phosphorus occurred in the more concentrated faecal areas such as the burnt herbivore dung from hearths and the layered human and pig faecal material in an external area of the site (Elliott, 2015).

7.2. Laboratory methods

The combination of micromorphological thin section analysis with modern ethnographic reference materials, ESEM-EDX, GC-MS and phytolith analysis enabled not only confirmation of faecal material presence and identification of producer source, but also the specific microstratigraphic context and dietary components of a wide range of faecal deposits. The examination of modern faecal comparanda for faecal spherulite and phytolith content as well as the micromorphological characteristics is vital in identifying and interpreting archaeological faecal material. As with all environmental proxies, the analyst should always utilise modern reference material/collections as comparative baselines for identification and contextual analysis. The modern reference collection used in this pilot study was small (Elliott et al. 2015, 2020a) but vital for examination and characterisation of the colour, contents and fabric of different dung types.

7.2.1. Faecal deposits microscopically; confirmation and context

Faecal materials and deposits from different contexts were examined and identified microscopically from all three case studies presented here. The specific characteristics of modern and published faecal material were used to classify the herbivore and omnivore faecal types. The context of the most concentrated faecal deposits from the different sites varied. At Jani and Bestansur, the faecal material analysed was predominantly from secondary product use. At Jani this included use of dung ash as temper (Matthews et al., 2013d), and at Bestansur use of dung fuel, as well as presence of human and suid coprolites in refuse deposits in an open area. The samples from Sheikh-e Abad were predominantly from faecal material in penning deposits and discard areas. Many of the samples from Bestansur indicate low level background dung signatures. The more concentrated deposits of faecal material were targeted for further analysis here.

Micromorphological analysis has also been applied to identify and investigate animal management and domestication through detection and analysis of compacted, laminated layers of herbivore dung in pens and open areas (Shahack-Gross et al., 2003; Matthews, 2005; Matthews et al., 2013d). Faunal material provides a method to investigate early domestication, but like any scientific methodology, the archaeozoological approach has limitations including: delayed onset of morphological changes by up to 1000 years (Zeder, 2011, 2015); limited assemblages from certain regions, long timescales of archaeological analysis, loss of biological information due to taphonomic processes or extreme fragmentation from either pre- or post-depositional alterations (Vigne, 2011, p.172). The identification of compacted microlaminated dung layers in micromorphological analysis can provide direct evidence for animal management or domestication. This type of deposit was identified in micromorphological samples taken from multiple locations in the latest Neolithic deposits from Sheikh-e Abad and represented both herbivore and omnivore faecal material and were targeted for additional analyses in this current study (phytoliths and ESEM-EDX). In one area of the site at Bestansur layered omnivore faecal material was also identified in the micromorphology and these deposits were targeted for further analyses in this current study (GC-MS).

Dung is often an under investigated secondary product which can be utilized as fuel, as a temper, in construction, or as a fertiliser. 'Manure is clearly a secondary product of domestic animals, as defined by Andrew Sherratt (1981), being a useable resource which does not result in the death of the animal; but it was never specifically mentioned in his paradigm generating paper' (Broderick and Wallace, 2016). Dung has a wide range of applications including as a slow-burning fuel (Anderson and Ertug-Yaras, 1998; Matthews, 2016). The selection of dung as a fuel is likely to relate to its excellent burning properties; burning consistently at temperatures of 800–1000 °C (Matthews, 2010: 106). At Bestansur, every hearth, burnt and ashy deposit examined in micromorphological samples contained identifiable faecal material, notably as high numbers of faecal spherulites and phytoliths. These results confirm the use of dung as a fuel at Bestansur, c. 7660 BC.

Dung can be used as a temper in floor or wall plasters, mud bricks or as a pottery temper (Goodman-Elgar, 2008; Nodarou et al., 2008; Lancelotti and Madella, 2012). Dung could be added raw or potentially as dung ash. In modern societies today dung is used as a temper in construction, for example in Rajasthan the main component of the plasters used on the floors is cow dung (Boivin, 2000). In the micromorphology samples from Jani dung ash was identified as a source material and or/a temper in white plastered carbonate floor deposits in two buildings.

Additional analyses were conducted in order to target specific research questions. A discussion of the locations in the micromorphological samples which were targeted for further laboratory analysis will be examined below in relation to these research questions.

7.2.1.1. Confirming faecal origin and species. Confirmation of faecal material using a combination of ESEM-EDX and GC-MS analysis are valuable laboratory methods and can be used alongside the lambda (λ) plate on an optical microscope for confirming faecal origin. Only seven spherulites were analysed by ESEM-EDX analysis; three modern and four archaeological (from Jani and Sheikh-e Abad). This provides a preliminary study that could be expanded in the future. It was relatively easy but time consuming to identify the spherulitic particles using ESEM analysis, and to disregard the possibility that these were coccoliths. Distinguishing between starch and faecal spherulites using the EDX analysis posed difficulties with background signatures from the other components of the dung (organic material, phytoliths etc.) in addition to components of the matrix (silt/sand/clay) as well as the mounting stub used for ESEM-EDX analysis. This large amount of background 'noise' could possibly be reduced in future analyses by increasing the magnification so that the EDX analysis was carried out closer to the surface of the spherulites. However, based on a combination of the calcium that was identified from the EDX results, alongside the SEM spherical morphology, in addition to the yellow interference colour (optical microscopy), it is highly likely that these spherulitic particles were not starch grains. The ESEM-EDX analysis, therefore, can be used successfully and effectively, to identify and categorise the spherulitic particles as faecal spherulites rather than 'probable faecal spherulites'.

The GC-MS results confirmed faecal material by the quantification of sterols in seven of the locations from Bestansur; two attributed as herbivore and five as omnivore. The remaining locations analysed correlated with presence of faecal spherulites in the smear slides and the micromorphological thin sections, although the biomolecular component of the faecal material did not survive (or was not present in the first instance). A number of other environmental factors influence organic residue preservation, including temperature, light exposure, degree of waterlogging, and redox conditions (Eglinton and Logan, 1991; Evershed, 2008). The contexts where a faecal component was not identified by GC-MS were from ashy hearth deposits identified with

numerous probable faecal spherulites and phytoliths, and therefore it is likely that the burning temperature affected the preservation of the biomolecular component of the faecal material. Extremes of waterlogging and desiccation are conducive to the survival of organic residues, however alternating wetting and drying appears to be detrimental to residue survival (Evershed, 2008), and this has been noted from Bestansur in other proxies (Bendrey et al., 2020). Therefore, the combination of burning and fluctuating water table could account for the lack of faecal signature in these locations.

The five omnivore faecal deposits at Bestansur that were confirmed by the analysis of the sterols were further classified by the bile acids; four were human in origin and one wild boar/pig (Elliott et al., 2020b). All these samples originated from the same area of the site, and the results suggest that it may have been used for discard from a range of activities and from or as a latrine, but also at one point has wild boar/pigs were in this area of the site. Therefore, wild boar or pig were congregating in this area, if not purposefully corralled. This wild boar/pig faecal material was present as thin compacted horizontal layers, with small fragments of bone, low faecal spherulites and minimal phytoliths. These animals were likely to be attracted to this area of the site because of the human waste.

Previously GC-MS analyses was conducted from samples at Sheikh-e Abad and Jani as part of an additional pilot project (Shillito et al., 2013). The recovery of faecal residues was variable, in 16 out of 21 samples faecal material was confirmed, but overall, there were low levels of bile acids recovered in the samples. At the time of publishing these included the earliest sterol residues recovered in the world, dated to ~10, 100–9300 BC (Shillito et al., 2013). The results show that penning deposits and human latrine areas are also identified at Sheikh-e Abad.

7.2.1.2. Animal diet and seasonality. Animals consume plants, and therefore micro-remains identified in animal dung can help to identify and inform on: animal diet, environment, seasonality, ecology, habitat, as well as foddering, grazing and browsing and management regimes. Indicators of seasonality could be inferred through the identification of plant parts, which are easily identifiable using phytolith analysis. The presence of grass-husks may reflect spring-summer grazing (Power et al., 2014). Faecal remains characterized by a high proportion of multi-cellular inflorescence phytoliths has been interpreted in previous studies to represent an early-summer grass-rich diet (Portillo et al., 2012). There are, however, alternate explanations for this phytolith assemblage. By-products of cereal cultivation could have been collected as fodder in spring-early summer (Portillo et al., 2012: 93) and fed to the animals in other seasons. Therefore, each phytolith assemblage must be considered and interpreted with caution. The ideal faecal remains targeted for analysis to provide dietary information on the vegetation consumed are from animal pens and enclosures due to the concentration of dung deposited in these features that enables high-precision sampling and reduction in background signals (Chang and Koster, 1986; Schepers and Van Haaster, 2014). The combination of micromorphological analysis and targeted phytolith analysis enables the investigation of Neolithic animal diet. Without the identification of concentrated and defined faecal material, through microscopic micromorphological analysis, the phytoliths extracted for quantification from sub-samples may incorporate diverse phytoliths from a range of activities and represent a combination of animal and human diet, or other practices of plant deposition (fuel, bedding, matting, etc.). In these case studies substantial layers of faecal material were selected; the penning deposits from Sheikh-e Abad and the latrine/layered omnivore faecal material from Bestansur was targeted.

In the dung layers at Sheikh-e Abad there was temporal variation between the faecal layers. Evidence for foddering and seasonality can be inferred when comparing percentages of phytoliths from the leaf/stems and the husks of grasses. Husks are only present surrounding the seeds in the spring and summer before the harvest when they are flowering. When phytoliths from the husks are identified in animal dung this indicates either spring or summer grazing or foddering using stored cereal waste in the winter. Foddering in the winter would likely utilise stored cereal including the inflorescence bracts removed during crop processing (Harvey and Fuller, 2005) of leafy fodder from shrubs or trees (Halstead and Tierney, 1998). The identification of increased numbers of husks in one of the dung samples from Sheikh-e Abad could suggest stored fodder (cereal waste) given to animals during the winter months and therefore provides an additional indicator of domestication and seasonal foddering. This layer of faecal material from Sheikh-e Abad indicates a combination of grazing, browsing and foddering; represented by the increase in phytoliths from the husks in comparison to the normal pattern in other dung samples analysed with lower husk phytoliths. In another faecal layer analysed from Sheikh-e Abad a signature indicative of foddering without any grazing was identified, this is represented by a diet consisting solely of dicotyledonous plants (shrubs and trees), also often used today for over-wintering (Halstead and Tierney, 1998).

The phytolith signatures from the omnivore faecal material at Bestansur in the northern area of the site indicated a different dietary signature in comparison to the animal pens at Sheikh-e Abad, with these Bestansur omnivore faecal deposits showing an increased dicotyledonous component in the phytolith assemblage. As initially assumed, this could suggest a more varied diet, in the wild boar/pig and humans in comparison to the herbivores which are overwhelmingly dominated by grasses and herbs (monocotyledons) (Elliott, 2015).

8. Conclusions

For the first time in the Neolithic when humans were semi-sedentary and eventually became fully-sedentary, animals, and thereby also their dung, were increasingly managed and concentrated for longer periods in particular localities. This development of much closer proximity, therefore, presents a unique opportunity at Neolithic archaeological sites to look for and investigate faecal deposits, as concentrations of dung can be readily detected and analysed. The case studies presented here focus on the Neolithic because of this unique opportunity to examine concentrations of animal dung from managed/early domesticated herds. The methods presented here, can, however, be applied to a wide range of archaeological sites in earlier and later periods to study traces of wild animals sheltering in caves for example, or later domesticates.

The selection of the specific combined, integrated approach should always be dependent on the projects research questions. The screening and detection techniques presented as a pilot study here, combining pXRF and spot sampling/smear slide analysis, have shown that at Bestansur, in locations with elevated phosphorus, faecal spherulites are also present and identifiable. However, areas with phosphorus below the limit of detection also contained faecal spherulites in smear slides (15 of the 18 examined) and therefore highlight a limitation in detection by a pXRF analyser. However, this combined application is promising as a screening and preliminary detection method, and can be especially useful on large sites where the analyst wants to target the locations for micromorphological sampling. This method needs to be applied to more pilot studies/sites to rigorously test this integrated approach.

Overall, the integration of methods in this study has produced two main recommendations. Firstly, modern faecal comparative material should always be consulted within the study region as a comparative reference and baseline for identifying and classifying different types of faecal material. Secondly, micromorphology and GC-MS samples are always vital proxies in further investigations to confirm identity of dung source once potential sample locations have been identified. The micromorphology samples can be sub-sampled prior to resin impregnation, and the faecal material can be visually categorized at high resolution within micro-stratigraphic units in thin-sections and these locations targeted for further analysis such as ESEM-EDX, GC-MS and phytoliths. By targeting known faecal deposits, more timely and

expensive laboratory techniques can be utilized on the greatest number of samples to their fullest potential (Fig. 2). Once faecal remains have been successfully located using the field methods, these interdisciplinary analyses in the laboratory based on the micromorphology results can inform on faecal type, content and context and provide more robust high-resolution evidence on interactions and relations between animals. plants, humans and environments. These pilot studies have identified evidence for animal penning, human latrine areas and dung being used in multiple contexts as a secondary product for fuel and construction.

These analyses provide a wide range of complimentary results which enable: a) confirmation of faecal material, b) characterisation of faecal material visually into faecal types by comparing against modern material, c) targeting of specific faecal material for further analysis, d) confirmation of faecal spherulite morphology, e) investigation of animal diet, management and environment, and f) identification of which species were present at each site.

Although this integrated methodology was tested across multiple sites and multiple field seasons, specific methods do need further exploration. For example, more sites could be explored using pXRF, including sites where dung is less common and concentrated to certain areas of the site. However, the results presented here show the potential and limitations of each respective method alone, as well as the wider and more robust opportunities for analysis when integrated, enabling fuller identification of the type, nature, context, content and taphonomy of faecal matter. Future analyses at these and other sites can also build on new research on aDNA, proteomic and urine salt studies developed in exciting new research (Abell et al., 2019; Massilani et al., 2022).

Author contributions

Sarah Elliott: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Project administration, Writing-Original draft, Visualization. Wendy Matthews: Conceptualization, Methodology, Resources, Supervision, Funding acquisition, Writing- Review & Editing.

Data availability

Datasets related to this article can be found in the appendices and at https://rdg.ent.sirsidynix.net.uk/client/en_GB/library/search/detailnonmodal/ent:$002f$002fSD_ILS$002f0$002fSD_ILS:1707802/one?qu=Investigating+early+animal+management+in+the+Zagros+Mountains+of+Iran+and+Iraq%3A+Integrating+field+and+laboratory+methods+for+the+identification+and+analysis+of+ancient+faecal+material&lm=EXCL_LR2, the University of Reading library catalogue.

The excavation data are available on Open Access: Central Zagros Archaeological Project (2022) The Central Zagros Archaeological Project 2012 to 2017 [data-set]. York: Archaeology Data Service [distributor] https://doi.org/10.5284/1090506.

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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. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.quaint.2023.02.005.

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