“Seeing shit”: assessing the visibility of dung tempering in ancient pottery using an experimental approach

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Widespread ethnographic evidence exists for the addition of animal dung to clay during the process of ceramic production. The use of this material was probably very common in antiquity, given its large availability and the advantages resulting from its mixing. Organic-tempered pottery acquires enhanced plasticity, as well as a lighter weight. However, conclusive evidence of dung tempering in archaeological ceramics is relatively rare. The aim of this study is to ascertain whether, and under which conditions, dung tempering of pottery is identifiable. Further investigated is how firing temperature may affect dung visibility. To answer these questions, we assessed whether a combination of micro-particle analysis in loose sediment and thin-section petrography can reveal the addition of dung to the clay paste by focusing on faecal spherulites, ash pseudomorphs, phytoliths and coprophilous fungal spores. We analysed several series of experimentally-produced ceramic briquettes tempered with different types of dung and dung ash, which were fired at a range of increasing temperatures. Our study shows that the identification of dung tempering represents a challenge, and it depends on a number of different factors, among others the original presence of dung markers in the dung used, the manufacturing process, the firing temperatures and the firing atmosphere. Overall, through a multidisciplinary approach, our work brings a significant contribution to the study of this tempering practice and clarifies a variety of issues connected to the identification of dung in ancient pottery, highlighting the role of faecal spherulites as the most promising proxy.

Key words:
Dung tempering
Pottery technology
Faecal spherulites

Dung fungal spores
Phytoliths

1. Introduction

1.1. Dung tempering in pottery production

Animal dung is a multi-functional resource, increasingly employed by humans since at least the beginning of animal domestication. Ethnographic studies have recorded dung use for fuel (e.g. Gosselain 1992; Gur-Arieh et al. 2013; Livingstone Smith 2001a; Portillo et al. 2017; Sillar 2007), manure (Broderick and Wallace 2016; Jones 2012), and construction (e.g. dung added as a temper into soil and lime features or used as plaster; Berna 2017; Boivin 2000; Gur-Arieh et al. 2019). Similar uses have been identified archaeologically from as early as the Pre-Pottery Neolithic Period in southwest Asia, ca. 10,000 years ago (Stiner et al. 2014). Each of these different practices may lead to differential preservation of various dung proxies in the archaeological record, which in turn would affect our ability to identify dung utilisation in the past (Shahack-Gross 2011).

Widespread ethnographic evidence also exists for the addition of animal dung to clay during the process of ceramic production. This includes the use of horse, cattle, sheep, goat, rabbit and donkey dung, which has been directly documented in living societies, e.g. in North Cameroon and in the Sahelian zone, from Senegal to Sudan (e.g. Gosselain 2002; Gosselain and Livingstone Smith 2005; Livingstone Smith 2001b; 2015), central America (Bowen and Moser 1968) and India (Saraswati 1964, 41; 1979, 4). Given its large availability and the advantages resulting from its use in ceramic production, dung could have been employed as a temper also in antiquity. The addition of dung into clay paste improves not only its plasticity, but also the drying and firing process. In addition, as other organic materials (e.g. plant fibers), dung confers on the vessels a lighter weight after the firing, having much of the organic material been subjected to loss on ignition during the firing (London 1981; Rice 2015).

However, dung tempering has rarely been identified with certainty in archaeological pottery (e.g. Biton et al. 2014; Gaimster 1986), and its presence is often argued only on the basis of the macroscopic observation of voids (Bobrinskii 1978) that can be left by the combustions of different types of organic materials and not exclusively by dung (Dumpe and Strivis 2015). This lack of evidence is possibly due to the firing temperatures, which exceed the preservation point of dung micro-proxies such as faecal spherulites (Canti and Nicosia 2018). Therefore, the aim of this study is to ascertain whether and under which conditions dung tempering of pottery is identifiable, and how firing temperature may affect its visibility. For that purpose, we assessed whether a combination of micro-particle analysis (spherulites, ash pseudomorphs, phytoliths, coprophilous fungal spores) in loose sediment and thin-section petrography can reveal the addition of dung to the clay paste. To do so, we analysed five series of experimentally produced ceramic briquettes tempered with different types and quantities of dung and dung ash which were then fired at increasing temperatures. Through a multidisciplinary experimental approach, our work gives a significant contribution to the study of this tempering practice and clarifies a variety of issues connected to the identification of dung in ancient ceramics.

1.2. Potential markers for dung tempering

Faecal spherulites, ash pseudomorphs, phytoliths, and coprophilous fungal spores are all micro-remains that can be found in animal dung in different concentrations depending on various factors of animal diet, age, sex, etc. (Canti 1999; Dalton and Rayn 2020).

Spherulites, mostly produced in the gut of herbivores, are unique to dung, and therefore are unequivocal direct evidence for its presence, along with bile acids and faecal organic compounds (Canti 1999; Linseele et al. 2013). They are radially-forming calcareous spheres ranging in size from 5 to 20 µm that can be identified using a polarised light microscope, based on their typical interference colors and fixed cross of extinction (Canti 1997, 1998; Canti and Brochier 2017). Upon burning at temperatures between 500°C and 700°C, and especially in reducing conditions, spherulites may darken, expand and change their appearance, becoming more discernible under plane-polarised light (PPL) (Canti and Nicosia 2018). Other microscopic look-alikes that may appear to the untrained eye as dung spherulites (e.g. coccoliths), may also be found as part of the paste matrix (Canti 1998; Gur-Arieh and Shahack-Gross 2020; Morandi 2020).

Ash pseudomorphs derive from calcium oxalates crystals (hereafter CaOx), which are biominerals common in higher plants (Franceschi and Horner 1980). The crystals range in size from 10 to 50 µm and are idiomorphic with smooth faces and several common morphologies (Canti 2003; Shahack-Gross and Ayalon 2013). Upon exposure to temperatures between 450–500°C and 740°C, CaOx crystals composition alters to calcite ($CaCO_3$), but they maintain their morphologies, hence the name pseudomorphs after CaOx, or ash pseudomorphs for short (Shahack-Gross and Ayalon 2013).

Phytoliths are hydrated silica (opal- $SiO_2 \cdot nH_2O$) microfossils that form intra- and extra-cellularly in living plants (Piperno 2006). As the phytoliths preserve the shape of the plant cells in which they originate, their presence in the dung can be used to make inferences, to a certain level, on animal diet and the flora in the surrounding roamed environment.

Ash pseudomorphs are abundant in the ash of dicotyledonous plants (e.g. most of the woody plants), while siliceous phytoliths are less common. The opposite occurs in monocotyledonous plants, and especially grasses, where phytoliths are very common and ash pseudomorphs are rare or absent (Gur-Arieh and Shahack-Gross 2020). These two types of plant micro-remains are present in animal dung in different concentrations depending on their diet, but as they can originate from other types of temper (e.g. plant ashes), they cannot be taken alone as a direct marker for the use of dung.

Furthermore, there is a fourth category of micro-remains which is not found within dung but still associated with it, i.e. spores of coprophilous fungi. The fungal kingdom includes thousands of fimicolous species, which tend to be rather cosmopolitan and have a world-wide distribution (Bell 1983; Krug et al. 2004). Herbivore dung forms an excellent substrate for the colonisation by dung fungi, the life cycle of which revolves around spore ingestion/inhalation by the animals and subsequent germination on fresh excreta (Wicklow 1992). A number of genera produce spores which are resistant enough to be preserved in Quaternary and pre-Quaternary deposits, and morphologically distinct enough to enable recognition of their origin from coprophilous taxa (van Asperen et al. 2016; van Geel et al. 2003). The walls of fungal spores mostly consist of a varying percentage of chitin (Ruiz-Herrera 1991), which allows good fossil and sub-fossil preservation. Recent research suggests that spores of coprophilous fungi can withstand firing at low temperatures (<350°C) and can be successfully extracted from low-fired potsherds (Dumpe and Stivrins 2015).

2. Materials and methods

2.1 Pottery briquettes preparation and firing

Five series of briquettes tempered with known amounts of dung or dung ash were produced with slight changes to their recipes, which were adjusted after each experiment in order to address different issues or examine different compositions, as described below and in Tab. 1. The clay used for all five series was the same commercial non-calcareous clay (Carl Jäger type 2). The concentration of the different dung micro-remains inside the raw clay was quantified prior to the experiment, in order to check what the clay contribution would be, if any, to the overall micro-remain concentration.

For the first series, each briquette was made by mixing 30 g of hydrated clay with 3 g (10w%) of ovicaprine (50/50%) droppings (*Capra hircus* and *Ovis aries*) rich in dung spherulites which were collected at the Campus Galli open-air museum (Meßkirch, south Germany) on July 2018. This series allowed us to check the visibility of pottery tempering using a spherulite-rich dung type, at the maximal limit of organic temper addition to clay, before losing its workability. Prior to mixing with clay the dung was dried for 48 hours in a drying cabinet at 110°C and the weighted aliquot was hand-crushed while mixed within the wet clay of the briquette. The second series of briquettes was tempered with dung ash that was produced from the dung used for the first experiment. Although the use of ash tempering was not the main focus of this paper, we aimed to investigate whether the addition of unburnt and burnt dung can be differentiated by our suggested methodology. The dung was fired in a covered crucible using a Nabertherm P 300 furnace in oxidising conditions for four hours at 500°C, with two hours of heating and cooling time. Seven briquettes were produced in each of these experiments, one left unfired and six fired at 100°C intervals between 300°C and 800°C with the same type of furnace (2 hours to reach the maximum temperature, 1 hour at maximum temperature, 2 hours of cooling).

The third series of briquettes was made by mixing 20 g of hydrated clay with 0.5 g (2.5w%) of sheep dung with low spherulite concentration, collected at Campus Galli in September 2018. The dung was ground with a mortar and pestle prior its addition. The purpose of this series was to check the visibility of dung tempering when using a dung with low content of calcitic micro-remains (spherulites and ash pseudomorphs). Originally we planned to add about 2 g of dung to the clay in order to get to 10w% organic temper like in the first series, but the characteristics of the specific dung did not allow us to add as much without losing the workability of the clay, possibly because it was more fibrous. Seven briquettes were produced and fired from this series in the exact same conditions as series 1 and 2. For this series, as we aimed to check the visibility of the dung spherulites when using dung temper with low spherulite concentrations, we only quantified the concentration of the calcitic micro-remains in loose sediment. As a result of the low amount of dung we were able to add to the clay in series 3, we produced series 4 by mixing 20 g of clay with 4 g of dung with low spherulite concentration collected at Elpersheim (Germany) in October 2019 (Tab. 2). Also, in this case the dung was ground in a pestle prior its addition. In order to ensure a homogeneous mixing of the dung inside the clay to improve its visibility, despite the low spherulite concentrations, a different paste preparation technique was employed. For this series, we dried the clay in advance for 48 hours in a drying cabinet at 110°C and ground it using a pestle and a mortar. We then mixed the ground and weighted clay with the ground and weighted dung, finally adding water to create a workable paste. Two briquettes were made, one of which was left unfired, while the other one was fired at 500°C.

Series 5 was prepared only to check the visibility of dung fungal spores in dung-tempered pottery. The seven briquettes were made by mixing 30 g of clay with 1 g (3.3w%) of cow dung (*Bos taurus*) rich in spores but with low concentration of dung spherulites and ash pseudomorphs. This was collected at Somma Lombardo (northern Italy) in February 2019. Seven briquettes were produced and fired from this series in the exact same conditions as series 1 and 2.

2.2. Micro-remains analyses

2.2.1. Calcitic micro-remains quantification

The concentration of faecal spherulites and CaOx/ash pseudomorphs per 1 g of sediments was calculated using the method developed by Gur-Arieh et al. (2013). A piece of each briquette was gently ground using a pestle and mortar, and about 50 mg of the sediment were sieved through a 150 µm mesh sieve. The remaining material was suspended in 500 µL sodium polytungstate (SPT) with 2.4 g/L density and sonicated for 10 minutes to prevent aggregation. The suspension was vortexed and immediately 0.5 µL of it was mounted on a glass slide, covered with a 24X24 mm cover slide and analysed at X400 magnification, first at PPL for CaOx/ash pseudomorphs and then at the same field of view at XPL for spherulites. The number of micro-remains in about 30 fields of view was counted and their concentration per 1 g of sediment was calculated following the formula presented by Gur-Arieh et al. (2013).

2.2.2. Phytoliths quantification

Phytolith concentration per 1 g of sediment was quantified following the rapid method developed by Katz et al. (2007). A piece of each briquette was gently ground using a pestle and mortar, and about 30–50 mg of the sediment were dissolved using 0.5 µL HCl 6 N and suspended in 450 µL sodium polytungstate (SPT) with 2.4 g/L density. The samples were sonicated for 10 minutes and then centrifuged at 5000 rpm for 5 minutes to separate the phytoliths from the heavy residues. 0.5 µL of the supernatant were mounted onto a glass slide, covered with a 24X24 mm cover slide and analysed under plane polarised light (PPL) at X400 magnification using a petrographic microscope (Euromex iScope IS.1153-Pli). The number of phytoliths in about 30 fields of view was counted and phytolith concentration per 1 g of sediment was calculated following the formula of Katz et al. (2007).

2.2.3. Dung fungal spores

A fraction of the briquettes was ground to a fine powder using a pestle. For each briquette 1 g of powder was obtained, mixed with a known amount of *Lycopodium* spores to enable calculation of concentrations and filtered through 200 and 5 µm meshes. The powder was decanted in water multiple times in order to reduce the heavier minerogenic component. The residue was then mounted onto a microscopy slide with a drop of liquid glycerol and examined under light microscopy at X400 and X600 magnifications using a Vickers ML 1300 compound microscope. Palynomorphs were identified following published keys (e.g. van Geel and Aptroot 2006; van Geel et al. 2003) and using reference slides from personal collections.

2.3. Thin-section petrography

For optical analysis, polarized light microscopy (PLM), using a Leica DM 2500 P on thin sections with a thickness of 30 µm, was performed. The thin sections were analysed under PPL and XPL at X50, X100, X200, X400 magnifications. Each briquette was impregnated with epoxy resin. After the resin has hardened, a flat section was cut from each briquette. Each section was polished and pasted over a glass slide using UV glue. The sections were then mounted onto the glass slide that was subsequently ground (with a petrothin) and manually polished to reach a thickness of 30 µm.

With this technique, a detailed inclusion identification is possible. Additionally, information about the particle shape of each individual grain and texture of the whole material can be obtained. Therefore, PLM allows one to gain insights into technological and manufacturing aspects but also helps to define raw material sources, thus providing important information about the provenance of the examined materials (Quinn 2013). Another major aspect where thin section petrography can aid archaeological investigation is the recognition of tempers, i.e. the intentional addition of aplastic materials into clay daub to improve its plasticity.

Considering the aims of the study, the petrographic examination focused on identifying the four dung proxies inside the pottery matrix and the effect the tempering had on the general ceramic microstructure.

3. Results

3.1. Phytolith and calcitic micro-remains concentration

The concentration of phytoliths and calcitic micro-remains in millions per 1 g sediments (M/g sed) from the different experiments are presented in Tab. 2 and Fig. 2. The raw clay used to produce all of the experimental briquettes contained no dung spherulites or CaOx/ash pseudomorphs prior to the addition of the dung, and a very low concentration of phytoliths (0.03 M/g sed). Since it is much easier to quantify micro-remains concentrations in ash, rather than in dung which still contain the organic components, we calculated the micro-remains concentration in the dung from their concentration in the ash based on the weight percentage (W%) of ash produced by each dung sample. To calculate the W% of the ash, an aliquot of each dung sample was burnt at 500°C for four hours after it was dried for at least 48 hours in a drying cabinet. This allowed us to extrapolate the micro-remain concentrations in the fresh dung (Tab. 2). The goat and sheep dung ash used for the first experiment had very high spherulite concentrations (148.03 and 138.39 M/g sed respectively), much lower ash pseudomorph concentrations, pointing to clear dietary preference between the goats (15.34 M/g sed) and the sheep (0.46 M/g sed), and medium phytolith concentrations relative to previous studies (54.71 and 35 M/g sed respectively, Gur-Arieh and Shahack-Gross 2020, Tab. 1). The calculated values for the concentration of micro-remains within the dung are naturally lower (17.37 and 30.6 M/g sed spherulites, 1.8 and 0.1 M/g sed ash pseudomorphs, and 6.42 and 7.74 M/g sed phytoliths for goat and sheep dung respectively). Only the concentrations of the calcitic micro-remains were calculated for the dung used for experimental series 3 and 4, as we observed that these are the most informative in this case for the identification of dung temper. The ash produced from the sheep dung samples, which was used for experimental series 3 and 4, had a generally low concentration of spherulites (32.72 M/g sed and 45.2 M/g sed respectively) and relatively high concentration of ash pseudomorphs in the dung used for series 3, but low concentration in the one used for series 4 (10.83 M/g sed and 1.73 M/g sed respectively). The dung used for producing series 3 had 18.02 ash weight percentage and therefore the values of micro-remain concentration calculated for the dung were 5.89 M/g sed spherulites and 1.95 M/g sed ash pseudomorphs. The dung used for experimental setting 4 had 15.91 ash weight percentage resulting in calculated concentrations of micro-remains of 7.19 M/g sed for spherulites and 0.28 M/g sed for ash pseudomorphs.

For the first experimental series, we calculated how many micro-remains should be present in each briquette if the raw clay was tempered with 10% percent 50/50% mixture of sheep and goat dung. It is clear from the results that the actual concentrations calculated from all the briquettes are lower, even from the unburnt one, that should have shown similar values (Tab. 2; Fig. 2a). In addition to being generally lower than expected, the spherulite concentrations drop significantly around 600–700°C, and they are completely absent at 800°C. Ash pseudomorph concentration was also much lower than their estimated values (0.02 vs. 0.29 M/g sed), and they were not found in any of the briquettes fired above 300°C. Generally, although their concentration was lower than the estimated one, the phytolith concentration was relatively stable in the range between 0.2 to 0.37 M/g sed, regardless of firing

temperature. For the second series of ash-tempered briquettes we have only quantified the calcitic micro-remains (Tab. 2; Fig. 2a). Only in the briquettes fired between 500–700°C very low concentrations of spherulites were detected (0.02–0.05 M/g sed), while ash pseudomorphs were completely absent.
Also, for experimental series 3 and 4, only the calcitic micro-remains were quantified (Fig. 2b). From the third series – those which were tempered with a low weight percentage of dung (2.5w%) with low spherulite content – we identified only low spherulite concentrations in the briquette fired at 500°C (0.07 M/g sed). The fourth experimental series of briquettes was made with sheep dung that contained 7.19 M/g sed spherulites and 0.28 CaOx/ash pseudomorphs respectively (based on these micro remains concentrations in the dung ash which was 45.20 M/g sed and 1.73 M/g sed spherulites and ash pseudomorphs respectively). Although each of the two briquettes that were produced in this set were tempered with 4 g of dung, no spherulites and CaOx/ash pseudomorphs were identified in the quantitative analysis.

3.2. Dung fungal spores

Abundant spores of fungi possibly belonging to the coprophilous family of Sordariaceae were observed in the unfired briquette (Tab. 3). As shown by the graph (Fig. 3), the concentration of these spores decreases dramatically with increasing firing temperatures, so that in raw samples, up to ca. 5000 spores per g occur, along with another ca. 5000 palynomorphs (pollen grains and other NPPs). At 300°C the amount of dung spores already drops by ca. 98.5% of the total, with a reduction to only 70 elements per g. The rate of decrease remains similar at 400°C (64 spores per g), but from 500°C to 900°C a complete disappearance of palynomorphs occurs. Our findings agree with the results by Ghosh et al. (2006), which showed the presence of palynomorphs in experimental pottery only when fired below ca. 350–400°C.

3.3. Thin-section petrography

Our results show that two main types of dung markers are visible in thin section: phytoliths and faecal spherulites. In the samples of the first series of briquettes tempered with ovicaprine dung (Fig. 4 a–f), spherulites were still present up to 700°C. Groups formed by abundant spherulites can be best observed at 500°C and 600°C degrees (Fig. 4c–d). This is probably because only at 500°C the combustion temperature is sufficiently high to completely remove the organic material in which the spherulites are covered. At 700°C the number of spherulites is generally reduced due to their thermal degradation and they tend to occur in smaller groups (3–4 elements; Fig. 4e), although locally bigger assemblages can occasionally be observed. At 800°C, while there is no unequivocal evidence for their presence, some micro-particles may represent highly degraded spherulites. The situation observed petrographically is consistent with the findings reported by Shahack-Gross (2011) and Canti and Nicosia (2018), noting spherulite decomposition around 700°C.

The petrographic analysis also allowed us to observe the original arrangement of the spherulites within their micro-context: they tend to be mostly concentrated along the edges of the voids left by the burning of the organic component of the dung, and more rarely they occur as isolated elements within the clay matrix (Fig. 4 c–d).

Similar observations were made on the ash-tempered series (Fig. 5a–f), where up to 600°C (Fig. 5d) there are particle clusters formed by abundant spherulites. At 700°C (Fig. 5e) their number is generally reduced, and they tend to only occur in smaller groups (3–4 elements) and sporadically in bigger assemblages. Again, at 800°C (Fig. 5f) there is not unequivocal evidence, and only very few micro-particles that may derive from highly degraded spherulites. Very interestingly, as in the dung-tempered series, the spherulites became best visible at 500°C and 600°C degrees. At these temperatures, the remaining organic material that was still present in the ash used to temper the briquettes was completely combusted. However, unlike the dung-tempered briquettes, no voids typical of the combustion of organic matter were observed in this series.

In the third and fourth series tempered with sheep dung and in the fifth series tempered with cow dung, no clear evidence of spherulites was observed (Fig. 6a–f). Even in the third series, some spherulites degraded by the grinding process may be present (Fig. 6a–b). However, all dung-tempered series are characterised by typical vesicular voids left by the combustion of organic materials (Fig. 7a–d). Under the microscope it is possible to see the increasing thermal degradation of dung and plant fibers that are still visible up to 500°C degrees; beyond that temperature, only vesicular voids are visible.

In all series studied, phytoliths were detected by visual inspection of the thin sections (Fig. 7a–d). However, the detection of phytoliths in thin section is challenging as they become visible only when the ceramic fabric is fired to an almost complete oxidising state. Moreover, in thin section phytoliths are observed only in a fixed position and two-dimensional vision, thus hindering their identification (Pető and Vrydaghs 2016 and literature therein). Other micro-remains (ash pseudomorphs and spores) were not visible in thin section.

4. Discussion

4.1. The effect of firing temperature on the preservation of the different dung micro-remains

In this study we have explored the possibility to recognise evidence of dung tempering in ancient pottery through the application of a multi-proxy approach. Since this class of material is produced via thermal alteration of clay, a particular focus has been placed on how the firing temperatures affect the visibility of different microscopic proxies for dung tempering in pottery. Therefore, each of the aforementioned methods will be discussed in detail in this section, considering their resilience to the firing process. In this regard, it is important to bear in mind that in order to produce a functional vessel, a temperature of at least 600°C should be reached, although normally potters tend to fire earthenware and terracotta in a range of temperatures between 700°C and 1000°C (Rice 2015, 166–185). Nevertheless, it should not be forgotten that the length of the firing process, the soaking time (i.e. how long the maximum temperature is maintained), and the amount of oxygen in the firing chamber are all factors that could impact the preservation of these proxies, along with the maximum temperatures reached during the firing.

Our results, (Tab. 1; Fig. 2a) in accordance with previous studies, show that phytoliths are the proxy with the highest resistance to the firing process, as they start to melt only between 750°C and 800°C (Pető and Vrydaghs 2016; Starnini et al. 2007). Yet, although in the first experimental series they were preserved up to 800°C, in all of the briquettes their concentration is lower than expected (Tab. 1; Fig. 2). As these lower concentrations are more or less consistent throughout the entire series, this may result from the preparation process, rather than from the increase in temperature. This procedure can lead to both some material loss and an uneven spread of the dung, as it was crumbled manually and mixed into the clay paste. However, even though phytoliths are preserved in temperatures as high as 800°C, they cannot be attributed exclusively to the use of dung temper, as they can originate from other types of organic tempering. Other possible sources for phytoliths in pottery could be the original sediment used for the vessel's production (Ting and Humphris 2017), or simply from the use of vegetal temper (Delhon et al. 2008). Therefore, the occurrence of phytoliths by themselves, does not necessarily imply that dung has been used as a tempering agent, and they can be linked with dung only when they appear in association with direct proxies such as faecal spherulites (and to a lesser extent, with spores of coprophilous fungi). When phytoliths are correlated to dung their morphologies can be used also to reconstruct animal diet (Dalton and Rayn 2018), but as this latter will depend on animal species, age, season, feeding practice and several other complex variables, phytolith analysis might not be of great help in differentiating between dung and vegetal temper based on phytolith morphologies alone.

In regard to dung fungal spores, as suggested by previous research, palynomorphs survive only in low-fired pottery (Dumpe and Stivrins 2015; Ghosh et al. 2006; Yao et al. 2012), in our case up to ca. 400°C (matching the results by Ghosh et al. 2006) within ceramic fired under oxidising atmospheres (Fig. 3). The destruction of virtually every spore at temperatures above 400°C makes it rather pointless to focus specifically on this proxy for the identification of dung-tempered pottery. If present in high concentrations in the original dung, however, they can be detected in daub or in pottery fired in short firing processes at relatively low temperatures. Moreover, firing under reducing conditions could favor their preservation at higher temperatures (Dumpe and Stivrins 2015).

Nevertheless, it is important to bear in mind that spores of coprophilous fungi do not provide unequivocal evidence of dung, as they may have been transported in the air over a short distance and incorporated into the ceramic paste, due to herbivores grazing in the vicinity (Dumpe and Stivrins 2015). At the same time, as mentioned above, spores are not necessarily present in all excrements, as they are bound to the existence of coprophilous fungi in the area, so that the absence of dung fungal spores does not automatically equal absence of dung.

Finally, our results on the micro-calcitic remains show that while ash pseudomorphs can be detected only in the first series and only up to 300°C, faecal spherulites, constituting a direct dung proxy, survive up to 700°C in pottery fired in oxidising atmospheres, which is also in agreement with previous studies (Canti and Nicosia 2018, 34). Therefore, provided that they are present in the dung used to temper the clay paste, spherulites represent the best proxy for dung tempering. Another factor to bear in mind is that spherulites can also be found in ash derived from the combustion of dung. Therefore, their identification may also indicate tempering with ash. However, if dung ash is added as a temper, the typical vesicular voids left by the combustion of the organic material would not be observed.

It is worth noting that the shape of the voids, observable in ceramic thin sections, is a further important parameter to consider, as it can contribute to support the hypothesis of dung tempering (Fig. 7). As regards the arrangement of spherulites along one edge of the voids, which was frequently observed in experimental dung-tempered samples, it can be accounted for by the disappearance of the organic matter constituting the largest part of the dung fragment. Consequently, spherulites become free-moving within the voids, and are probably forced by gravity toward one of the sides, until they are fixed in place by resin impregnation during the sample preparation. The disappearance of the CaOx/ ash pseudomorphs in this case is more likely to be related to their initial low concentration in the dung, rather than a reaction to the increasing temperatures, as ash pseudomorphs start to disintegrate only in temperatures around 700°C (much like spherulites), but some have been shown to survive even up to 900°C.

4.2. Possible formation and degradation processes affecting the preservation of faecal spherulites in dung-tempered pottery

Our results show that among the dung markers considered in this study, faecal spherulites are the only one that can unequivocally indicate dung tempering in pottery, provided that vessels were fired to temperatures below 800°C. However, our different sets of experiments demonstrate that the visibility of spherulites in ceramics depends on a diverse range of factors. First, as was shown before, not all animal faeces necessarily contain spherulites (Canti 1999; Lancelotti and Madella 2012) and their concentrations may differ considerably, as we have seen in our small sample size. In addition, the concentration of the spherulites in the dung, together with the amount of dung being used as temper, largely affects the possibility of detecting them in the ceramic body, as shown by our third and fourth experiments. In summary, spherulites can be clearly identified within ceramics only if they are present in a very high concentration in the original dung that was used to temper the clay.

Other formation processes that may affect the preservation of dung spherulites in tempered pottery are the way in which the dung is prepared prior to its addition to the paste, the paste itself, and the kneading process. The dung can be minimally processed and not cut at all, simply pulled apart by hand into smaller pieces, or it can be ground (e.g. Bowen and Moser 1968), a process which may introduce not only mechanical degradation, but will also leave the spherulites more exposed to dissolution due to contact with the water in the clay paste. The dung temper can also be added either to dry clay powder and mixed with water or into a pre-made wet paste. Each of these preparation methods might result in a different preservation rate of spherulites which can easily dissolve by water (Canti 1999). While our results seem to suggest that grinding the dung prior to its addition to the clay may result in some degradation of the spherulites, which led to difficulties in their identification, more systematic work is needed on this topic, which is beyond the scope of the current paper.

The duration and intensity of the kneading also result in different degrees of homogenisation of the paste. If the processing is minimal, dung pockets are going to be present and this will provide better chances to preserve micro-remains such as spherulites and ash pseudomorphs. Additionally, an unhomogenised dung-tempered paste may bias the analysis results if the sampling happens to fall on an extraordinarily rich or poor area in the pot. Therefore, this is an issue that needs to be taken into consideration when sampling a vessel for bulk sediment analysis, as it can be easily overcome by either analysing samples from several locations or by homogenising several samples together. An intense preparation of the paste could facilitate the dissolution of calcitic micro-remains, as well as reduce the amount of the typical voids left by the combustion of the organic materials present in the dung pockets due to its integration into the paste. Therefore, even if spherulites can be detected, it would not be possible to discriminate between dung or dung ash tempering.

A further important parameter concerns the firing conditions. These include not only firing temperatures, but as mentioned above also the firing atmosphere (oxidising versus reducing), the soaking time, and the overall duration of the process. In our experiments, we decided to fire our vessels in a purely oxidising atmosphere for 5 hours with 1-hour soaking time. The presence of more reducing conditions could also have an impact in the preservation of spherulites. Firing in a reducing atmosphere, as compared to an oxidising atmosphere, results in the lowering of the temperatures at which the various vitrification structures form by about 50°C (Maniatis and Tite 1981) and in a faster decomposition of calcite. In addition, firing in conditions of limited gaseous exchange at temperatures between 500–700°C may result in the darkening of spherulites (Canti and Nicosia 2018), a phenomenon that would hamper their visibility and make their identification more difficult. On the other hand, the soaking time used in our experiment (1 hour) was relatively long and we cannot exclude the possibility that reducing the duration at which our briquettes were exposed would have resulted in a better spherulite preservation, even when they were exposed to higher temperatures. All these hypotheses need to be tested with further experiments.

5. Conclusions

Our study shows that identification of dung tempering in pottery is not straightforward. The main factors playing a role in the identification of dung are the presence/absence of the dung markers in the faeces used to temper the clay paste, the amount of dung added, the way in which it is processed and, most importantly, the conditions at which the pottery is fired, such as the temperature.

Our contribution confirms that the only unequivocal dung proxy for dung tempering, faecal spherulites, can survive firing temperatures as high as 700°C, and that higher temperatures would destroy them (Canti and Brochier 2017). Phytoliths can withstand higher temperatures, but unfortunately their identification alone is not sufficient evidence for dung tempering. It should be borne in mind that spherulites are not always present in dung or in concentrations that are high enough to be detected in pottery, therefore their absence cannot be taken as a certain indication of no dung temper. Furthermore, they could derive from the addition of dung ash, rather than of dung itself. Therefore, only the presence of spherulites along with the typical voids left by the combustion of organic materials can provide definite proof of dung tempering. Both these features can be easily observed in ceramic thin sections, which is one of the most widely applied techniques to the study of ancient pottery.

Spores of coprophilous fungi are further indirect dung indicators but can be detected only in pottery fired at very low temperatures or daub. Therefore, they are of limited use in the identification of dung-tempered archaeological pottery (Dumpe and Stivrins 2015).

The use of gas chromatographic techniques is unlikely to be effective in better identifying dung tempering, as organic compounds seem to be completely removed during the firing process, even at temperatures as low as 400°C (Reber et al. 2019). However, there are no studies specifically aimed at the detection of faecal bile acids and organic compounds included in ceramics, possibly because these will be destroyed by the firing process. A potential contamination deriving from the use of dung as fuel during the firing could also be a source of complication (Reber et al. 2019). Chemical analysis could be a promising line of investigation, as the use of dung would probably result in a phosphorus enrichment. Nevertheless, the concentration of this element in pottery could also derive from various post-depositional effects (Holliday and Gartner 2007).

All these limitations explain why to date clear and undisputed evidence for dung tempering in ancient pottery is so scant. It is also important to emphasise that macroscopic identification based on the recognition of voids, which is often the most common type of approach used by archaeologists, is not a reliable approach when used alone, and often also the application of different kinds of analytical methods fails to demonstrate evidence of dung tempering. While we cannot rule out that cultural taboos may have played a role in limiting the use of dung as a tempering agent (e.g. Gelbert 2000), considering its wider availability and good plastic properties it may have played a relevant role in antiquity in the manufacturing of ceramics, as suggested by ethnographic studies (e.g. Bowen and Moser 1968; Gosselain and Livingstone Smith 2005; Saraswati 1964, 41; 1979, 4).

Acknowledgements:

We would like to thank Christoph Berthold, Beatrice Boese, Aspen Cooper, Marco Cocuccioni, Jörg Fischer, Carlotta Gardner, Lars Heinze, Eirini Koutouvaki, Rosi Manthey, Arvin Raj Mathur, Marco Müller, Hannes Napierala, Klaus Nickel, Lars-Ole Schmutz, Martin Rogier, Johannes Seidler for their help and useful feedback.

Funding:

This work was supported by the Excellence Initiative of the Eberhard Karls Universität Tübingen and the Ministry for Science, Research, and Art of Baden-Württemberg.

SGA's work has been funded by the European Union's MapDung project. The project has received funding from the EU Horizon 2020 research and innovation programme under the Marie Sklodowska-Curie grant agreement No H2020-MSCA-IF-2017-794823.

Disclosure Statement:

No potential conflict of interest was reported by the author(s)

Author's contribution:

Silvia Amicone: Conceptualisation, Methodology, Formal analysis (petrography), Investigation, Resources, Writing - original draft, Review and Editing, Visualisation (Figures 4–7), Project administration, Funding acquisition.

Lionello F. Morandi: Conceptualisation, Methodology, Formal analysis (dung fungal spores), Investigation, Writing - original draft; Review and Editing, Visualisation (Figure 1 and 3, Table 3).

Shira Gur-Arieh: Conceptualisation, Methodology, Formal analysis (micro-remains analyses), Investigation, Writing - original draft; Review and Editing, Visualisation (Figure 2, Table 1 and 2), Funding acquisition.

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Captions:

Fig. 1. Some stages of the field and lab work: a) View of the penned area sampled at Campus Galli (Meßkirch, south Germany); b) Ovicaprine droppings on the soil surface; c. Sheep and goat dung ash; d. Clay briquettes tempered with dung prior to firing.

Fig. 2. a) Chart showing the concentrations (10^6) of dung spherulites, ash pseudomorphs and phytoliths per 1 g sediment in the raw clay that was used for the different experiments, and in the dung that was used for experiments 1 and 2 together with the results of experiments 1 and 2; b) Chart showing the concentration (10^6) of dung spherulites and ash pseudomorphs in the dung used for experiments 3 and 4 together with their results.

Fig. 3. Plot showing the abundance of dung fungal spores (per gram of pottery) recovered at different firing temperatures.

Fig. 4. Thin-section photomicrographs of the first series of briquettes tempered with dung: a) 300°C; b) 400°C; c) 500°C; d) 600°C; e) 700°C; f) 800°C . Pictures taken under XP; field of view=0.7 mm.

Fig. 5. Thin-section photomicrographs of the second series of briquettes tempered with ash: a) 300°C; b) 400°C; c) 500°C; d) 600°C; e) 700°C; f) 800°C . Pictures taken under XP; field of view=0.7 mm.

Fig. 6. Thin-section photomicrographs of the third (a–b), fourth (c–d) and fifth series (e–f) of briquettes tempered with dung: a) unfired; b) 500°C; c) unfired ; d) 500°C.; e) unfired; f) 500°C. Pictures taken under XP; field of view=0.7 mm.

Fig. 7. Thin-section photomicrographs of the first (a–b) and fifth (c–d) series of briquettes tempered with dung: a) 400°C; b) 800°C; c) 400°C; d) 800°C. Pictures taken under PPL; field of view=6 mm.

Table 1: Details of the experimental protocol used for the various briquette series.

Table 2: Concentration of micro-remains per 1 g sediment in the dung and briquettes fired at different temperatures in the different experiments. *The concentration of micro-remains per 1 g of fresh dung was calculated based on their concentration in the ash and the ash weight percent. **Based on the fact that each brick was tempered with 10% weighted dung (dung was a 50/50% mixture of goat and sheep dung), we estimated the concentrations of micro-remains that should theoretically be present in 1 g of unburnt homogenised brick sediment. This is a very rough estimation.

Table 3: Abundance of dung fungal spores and other palynomorphs (per g of pottery) recovered at different firing temperatures.

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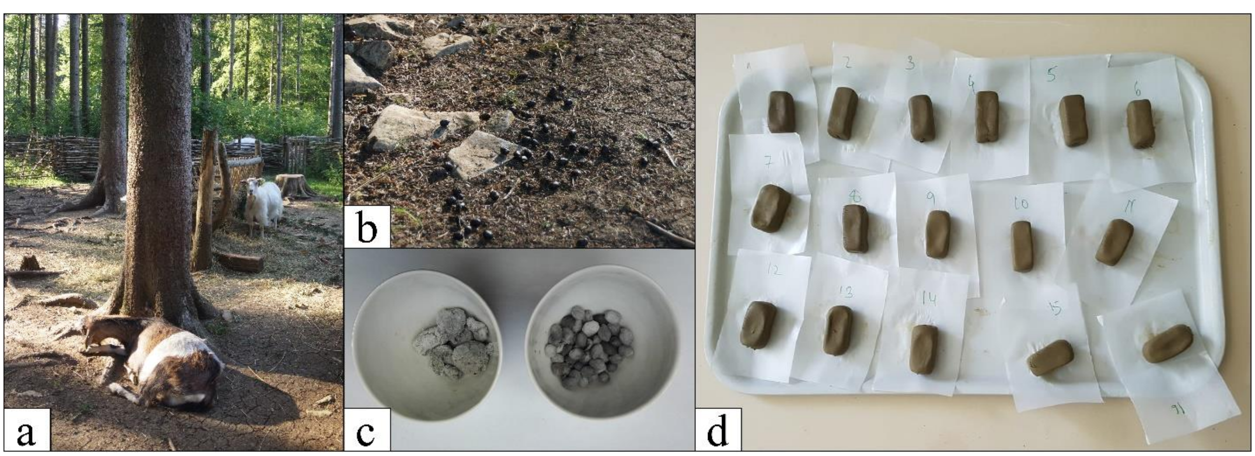

Figure 1

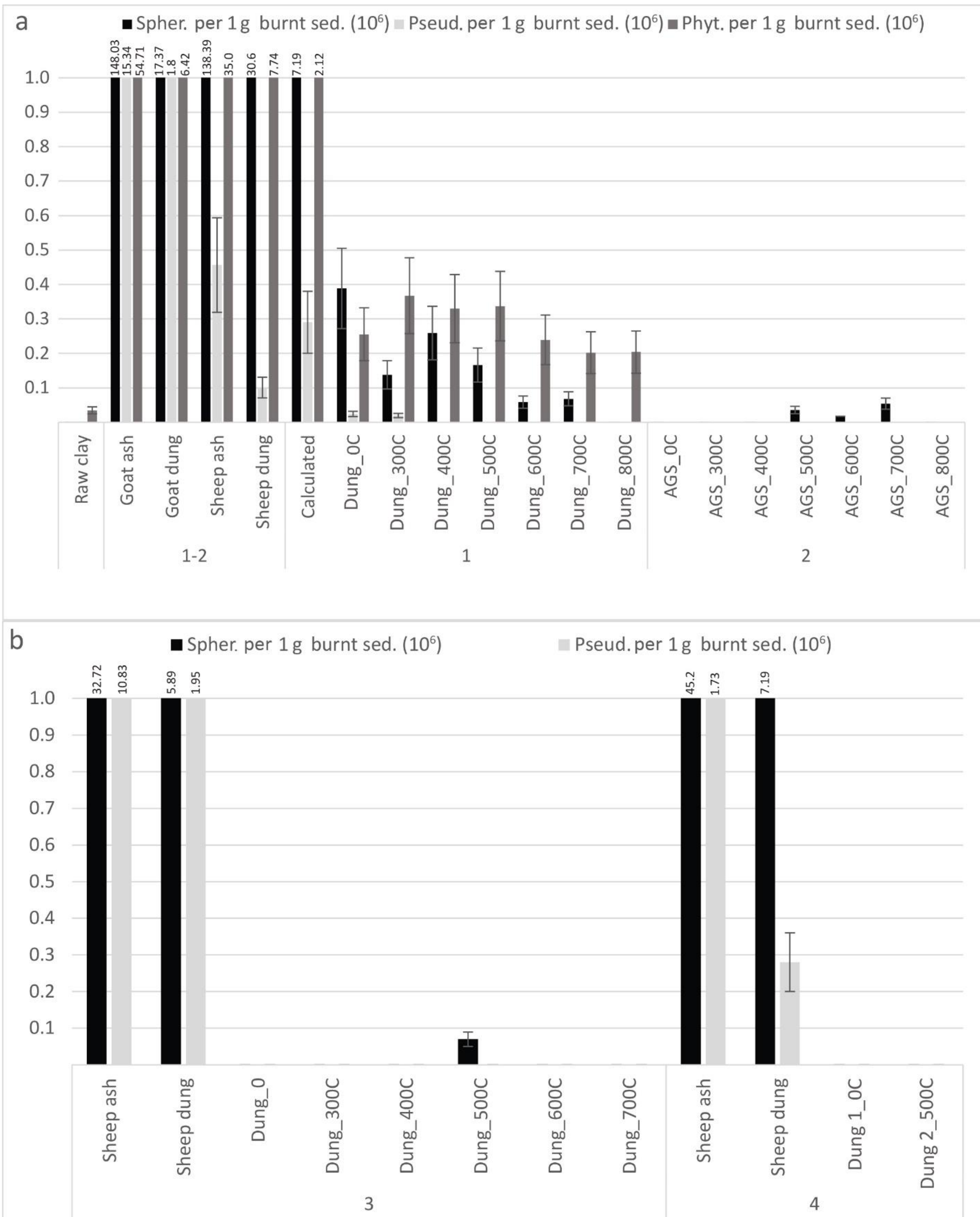
a
Spher. per 1 g burnt sed. (10⁶) Pseud. per 1 g burnt sed. (10⁶) Phyt. per 1 g burnt sed. (10⁶)
148.03 15.34 54.71 17.37 1.8 6.42 138.39 35.0 30.6 7.74 7.19 2.12
1.0 0.9 0.8 0.7 0.6 0.5 0.4 0.3 0.2 0.1
Raw clay
Goat ash
Goat dung
Sheep ash
Sheep dung
Calculated
Dung_0C
Dung_300C
Dung_400C
Dung_500C
Dung_600C
Dung_700C
Dung_800C
AGS_0C
AGS_300C
AGS_400C
AGS_500C
AGS_600C
AGS_700C
AGS_800C
1-2
1
2
b
Spher. per 1 g burnt sed. (10⁶)
Pseud. per 1 g burnt sed. (10⁶)
32.72 10.83 5.89 1.95 45.2 1.73 7.19
1.0 0.9 0.8 0.7 0.6 0.5 0.4 0.3 0.2 0.1
Sheep ash
Sheep dung
Dung_0
Dung_300C
Dung_400C
Dung_500C
Dung_600C
Dung_700C
Sheep ash
Sheep dung
Dung 1_0C
Dung 2_500C
3
4

Figure 2

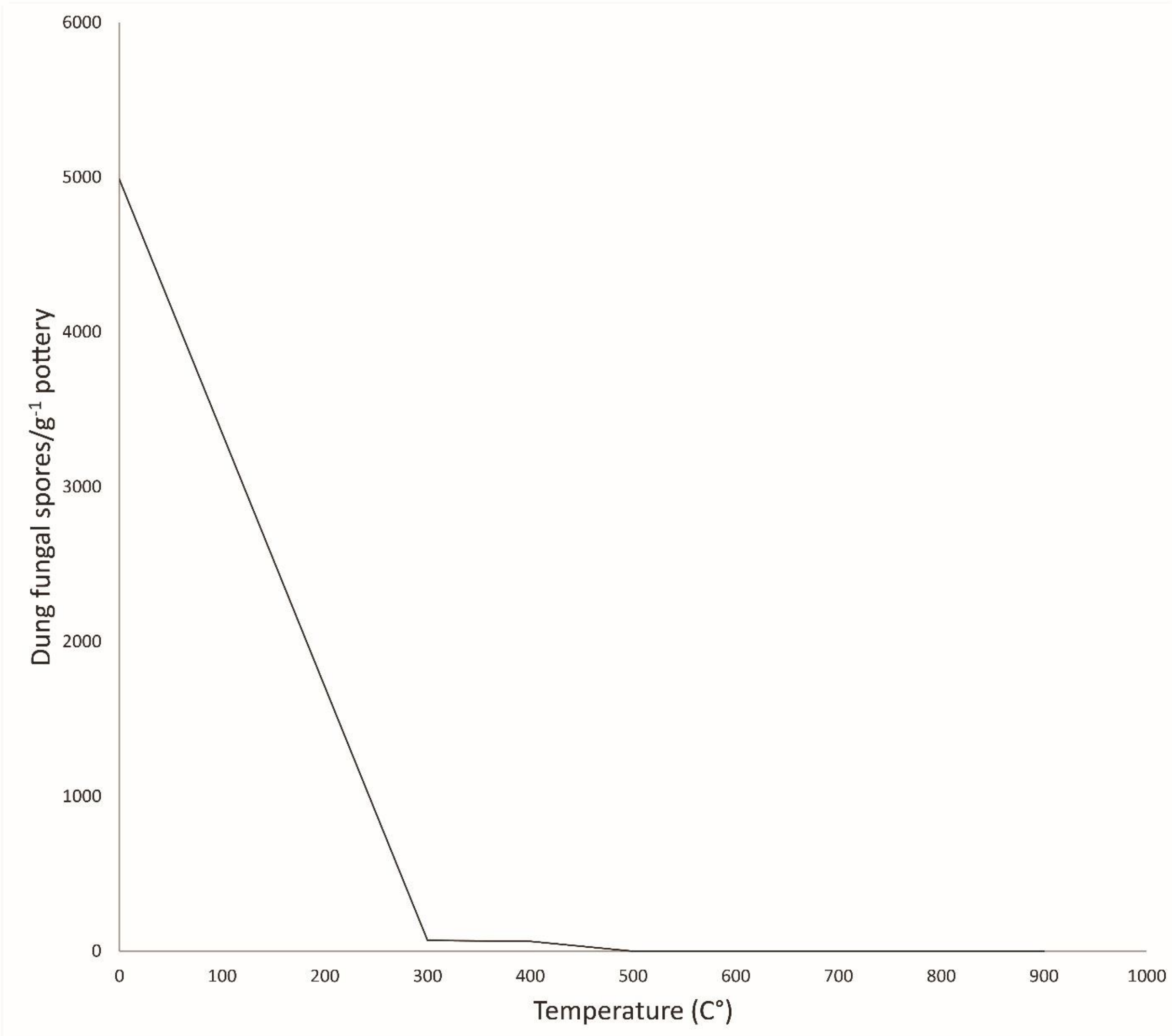
Dung fungal spores/g-1 pottery
6000
5000
4000
3000
2000
1000
0
0
100
200
300
400
500
600
700
800
900
1000
Temperature (C°)

Figure 3

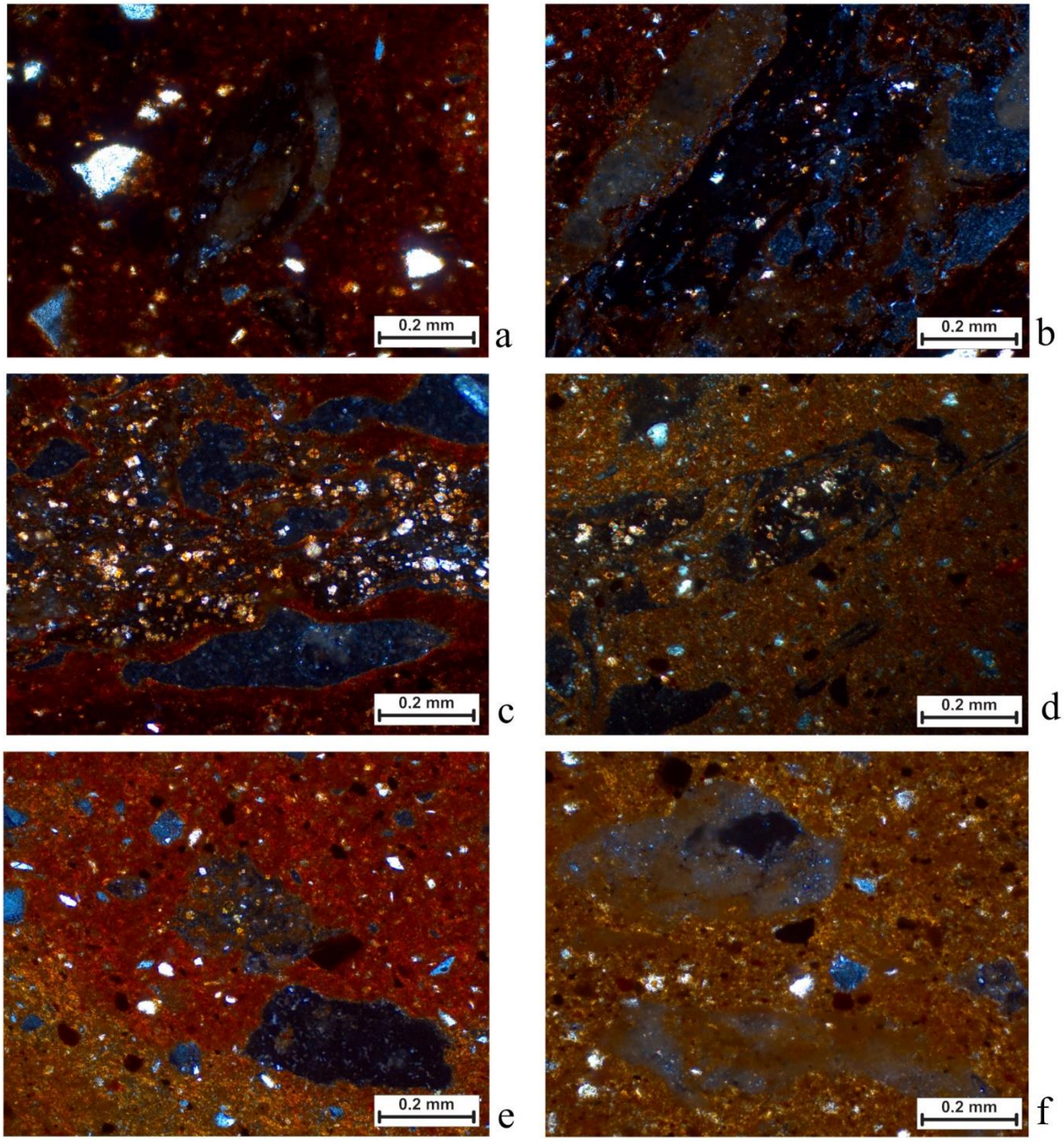

Figure 4

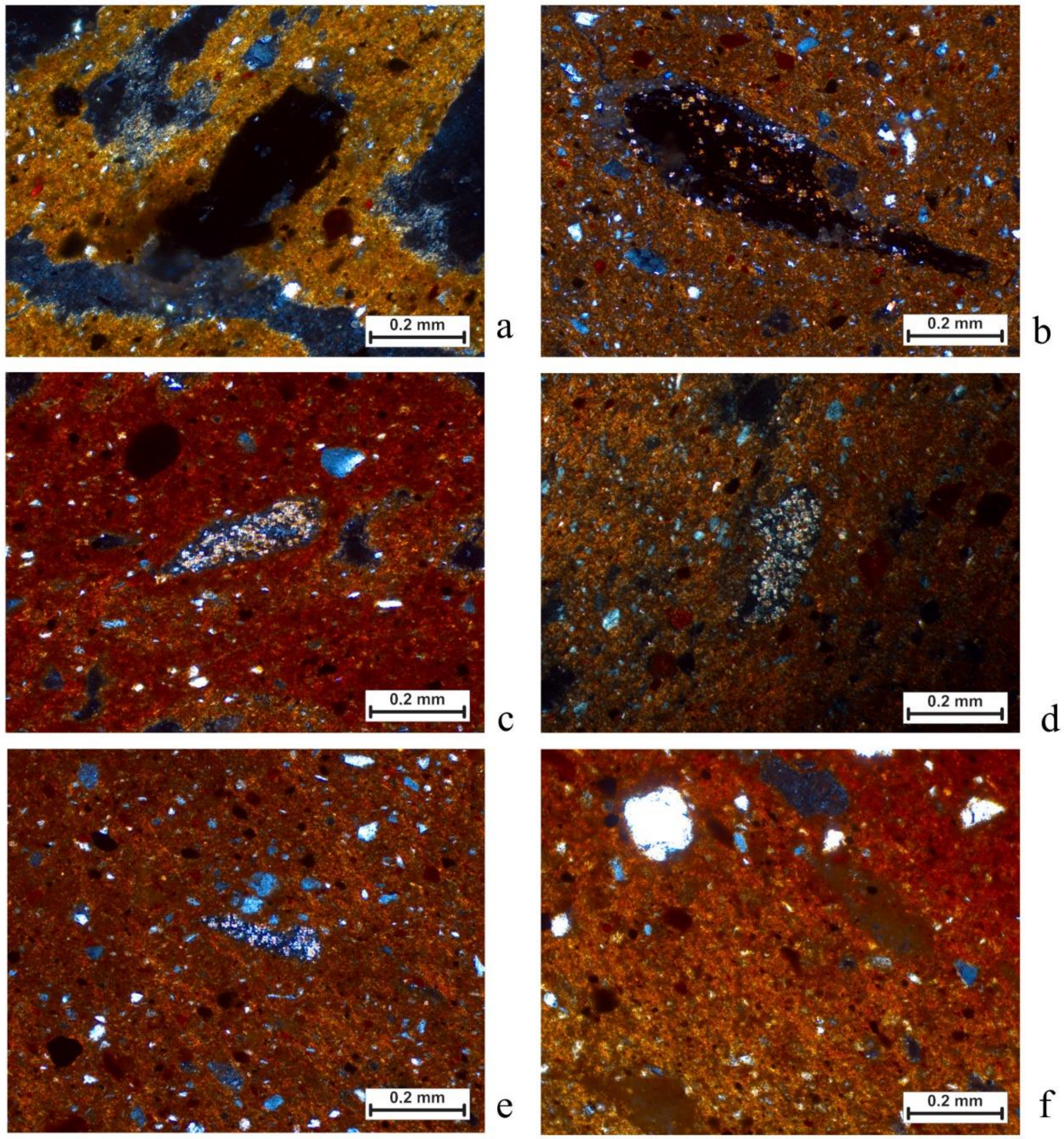

Fig 5

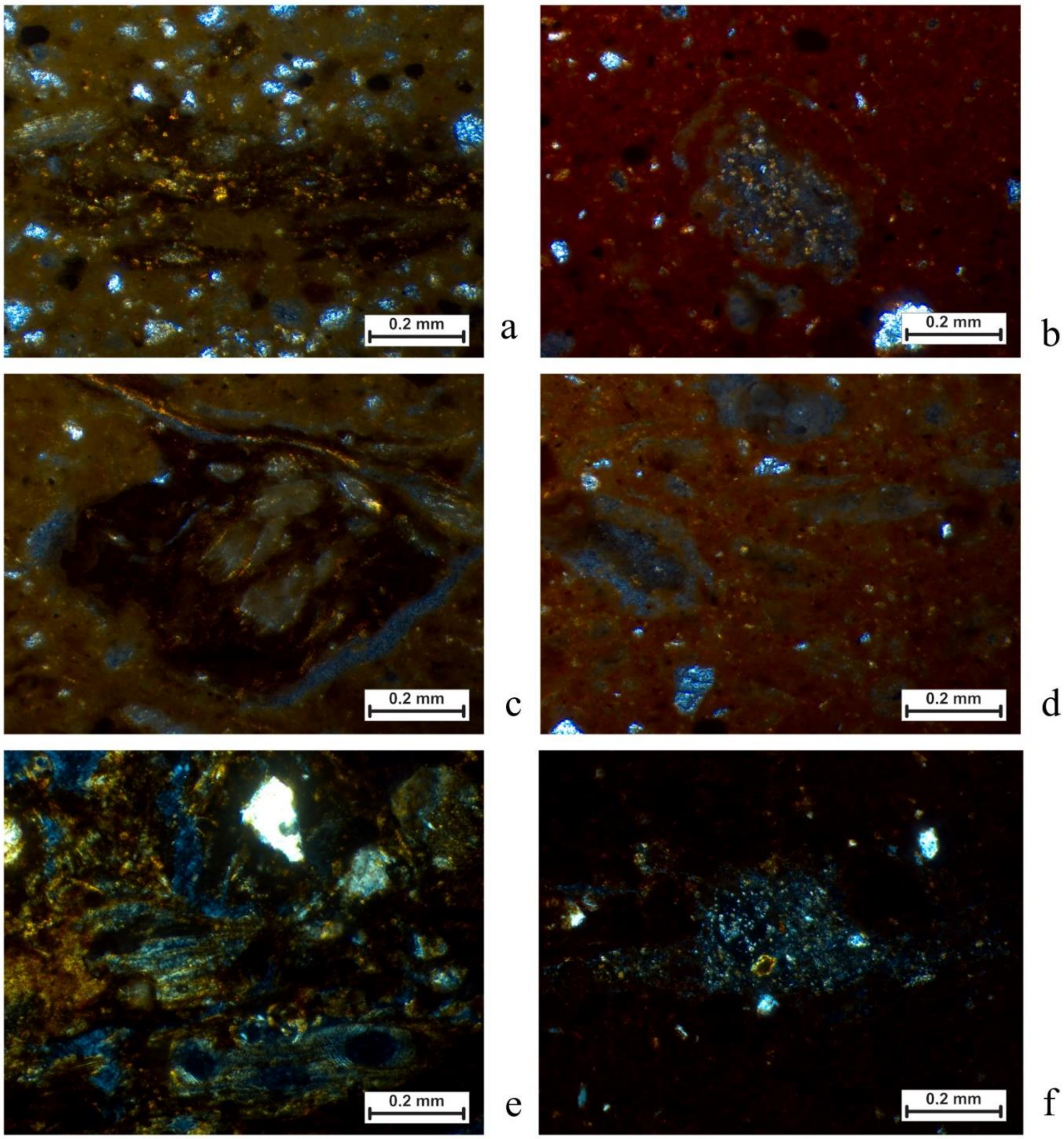

Fig 6

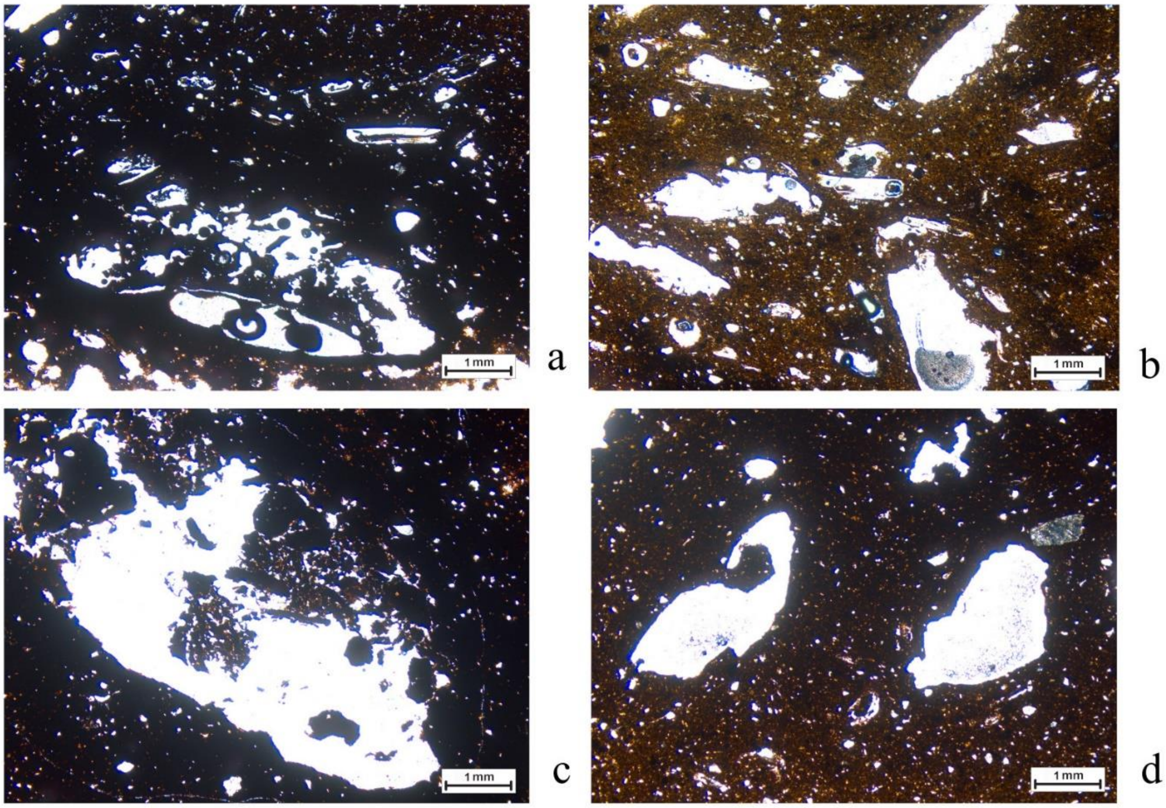

Fig. 7

Series No.	Type of dung	Sampling location	Date of sampling	Dung ash micro-remain concentration (10^6)	Dung pre-treatment	Clay pre-treatment	Briquette composition	Briquette preparation	Procedure	Analysis performed
1	Ovi caprine	Campus Galli, Germany	June 2018	Spherulites: 138/148 Ash pseudomorphs: 0.46/15 Phytoliths: 35/55	48 hours in a drying cabinet at 100 °C	None	30 g clay 1.5 g sheep dung + 1.5 g goat dung (10 wt%) 10 wt% typical amounts of temper added	Weighted aliquots of wet clay were mixed by hand with 3 g of dry dung which was crushed by hand	Seven briquettes, one was not fired and the rest between 300–800°C at 100°C intervals	Quantification of phytoliths and calcitic micro-remains in loose sediments, thin-section petrographic analysis
2	Ovi caprine	Campus Galli, Germany	June 2018	Spherulites: 138/148 Ash pseudomorphs: 0.46/15 Phytoliths: 35/55	Burned in a covered crucible in a muffle furnace to produce dung ash. 2h to reach 500 °C. Temperature kept for 4 h, 2 h cooling	None	30 g clay dung ash (c 2 wt%)	Weighted aliquots of wet clay were mixed by hand with dung ash to create each briquette	Seven briquettes, one was not fired and the rest between 300–800°C at 100°C intervals	Quantification of calcitic micro-remains in loose sediments, thin-section petrographic analysis
3	*Ovis*	Campus Galli, Germany	September 2018	Spherulites: 33 Ash pseudomorphs: 10	48 hours in a drying cabinet at 100°C	None	20 g clay 0.5 g dung (2.5wt%) Impossible to add more than 1 g of dung, otherwise the briquettes would become too hard	Weighted aliquots of wet clay were mixed by hand with 0.5 g of ground dung to create each briquette	Seven briquettes, one was not fired and the rest between 300–800°C at 100°C intervals	Quantification of calcitic micro-remains in loose sediments up to 700°C
4	*Ovis*	Elpersheim, Germany	October 2019	Spherulites: 45 Ash pseudomorphs: 1.7	48 hours in a drying cabinet at 100°C	48 hours in a drying cabinet at 100°C	20 g clay powder 4 g ground dung (20 wt%) In order to test a higher amount.	After mixing well-dried and ground clay powder and ground dung to ensure homogeneity, water was added to create workable paste for each briquette	Two briquettes, one unfired and one fired at 500°C	Quantification of calcitic micro-remains in loose sediments
5	*Bos*	Somma Lombardo, Italy	January 2019	Spherulites: 55 Ash pseudomorphs: 2.4	48 hours in a drying cabinet at 100°C	None	30 g of clay 1 g of Dung (c. 3 %)	Weighted aliquots of wet clay were mixed by hand with 1 g of dry dung which was crushed by hand	Seven briquettes, one was not fired and the rest between 300–800°C at 100°C intervals	Quantification of dung fungal spores in loose sediments, thin-section petrographic analysis

Table 1

Exp. No.	Sample	Spher. in 1 g burnt sed. (10^6)	±30% error	Pseud. in 1 g burnt sed. (10^6)	±30% error	Phytoliths in 1 g burnt sed. (10^6)	±30% error
	Raw clay	0.00	0.00	0.00	0.00	0.03	0.01
1/2	Goat ash	148.03	44.41	15.34	4.60	54.71	16.41
	Goat dung (Ash w%11.7)*	17.37	5.21	1.80	0.54	6.42	1.93
	Sheep ash	138.39	41.52	0.46	0.14	35.00	10.50
	Sheep dung (Ash w%22.1)*	30.60	9.18	0.10	0.03	7.74	2.32
1	Calculation of how much should be in the briquettes**	7.19	2.16	0.29	0.09	2.12	0.64
	Dung_0°C	0.39	0.12	0.02	0.01	0.26	0.08
	Dung_300°C	0.14	0.04	0.02	0.01	0.37	0.11
	Dung_400°C	0.26	0.08	0.00	0.00	0.33	0.10
	Dung_500°C	0.17	0.05	0.00	0.00	0.34	0.10
	Dung_600°C	0.06	0.02	0.00	0.00	0.24	0.07
	Dung_700°C	0.07	0.02	0.00	0.00	0.20	0.06
	Dung_800°C	0.00	0.00	0.00	0.00	0.20	0.06
2	Ash temper_0°C	0.00	0.00	0.00	0.00	NA	NA
	Ash temper_300°C	0.00	0.00	0.00	0.00	NA	NA
	Ash temper_400°C	0.00	0.00	0.00	0.00	NA	NA
	Ash temper_500°C	0.04	0.01	0.00	0.00	NA	NA
	Ash temper_600°C	0.02	0.00	0.00	0.00	NA	NA
	Ash temper_700°C	0.05	0.02	0.00	0.00	NA	NA
	Ash temper_800°C	0.00	0.00	0.00	0.00	NA	NA
3	Sheep dung ash	32.72	9.81	10.83	3.25	NA	NA
	Sheep dung (Ash w%18.02)*	5.89	1.77	1.95	0.59	NA	NA
	Dung_0°C	0.00	0.00	0.00	0.00	NA	NA
	Dung_300°C	0.00	0.00	0.00	0.00	NA	NA
	Dung_400°C	0.00	0.00	0.00	0.00	NA	NA
	Dung_500°C	0.07	0.02	0.00	0.00	NA	NA
	Dung_600°C	0.00	0.00	0.00	0.00	NA	NA
	Dung_700°C	0.00	0.00	0.00	0.00	NA	NA
4	Sheep Elpersheim Ash	45.20	13.56	1.73	0.52	NA	NA
	Sheep Elpersheim dung (Ash w%15.91)*	7.19	2.16	0.28	0.08	NA	NA
	Dung 1_0°C	0.00	0.00	0.00	0.00	NA	NA
	Dung 2_500C	0.00	0.00	0.00	0.00	NA	NA

Table 2

Firing temperature	Dung fungal spores per g of pottery	Pollen grains + other NPPs per g of pottery	Notes
0°C	4988	5456	Abundant fresh plant material
300°C	70	139	Frequent burnt and partially burnt fragmented plant material
400°C	64	128	Rare burnt and fragmented plant material
500°C–900°C	0	0	No plant material observed

Table 3