Research Article

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A Multi-Method Study of a Chalcolithic Kiln in the Bora Plain (Iraqi Kurdistan): The Evidence From Excavation, Micromorphological and Pyrotechnological Analyses

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Abstract: Pyrotechnology has always been a core topic in the archaeological debate concerning phases of deep cultural transformations, such as the Chalcolithic period in the Near East (c. 6000–3500 BC). However, previous studies on pyrotechnological installations, such as pottery kilns, pertaining to this period, have often been mainly descriptive, with a limited use of archaeometric investigations. This work presents a multi-method investigation of a Chalcolithic kiln recently discovered in the Bora Plain (part of the larger Peshdar Plain, in Iraqi Kurdistan), which combines stratigraphic analysis, pyrotechnological, micromorphological, and micro-remains analyses. Since this kiln represents the first Chalcolithic architectural feature excavated in the Bora Plain, this work offers precious insights into the pyrotechnology of the period, which is still relatively poorly understood, through the reconstruction of the kiln's use and abandonment processes. The analytical outputs can be used to compare with other Near East kilns from the Chalcolithic and later periods.

Keywords: Iraqi Kurdistan, Chalcolithic kiln, pyrotechnology, micromorphological analysis, XRPD

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1 Introduction

Pyrotechnology is a crucial topic within the study of past societies (e.g. Clark, 2015; Gourdin & Kingery, 1975; Miller, 2017; Wertime, 1973) because mastering pyrotechnological techniques was a vital part of people's daily life. These techniques were imbued with social meanings and were essential in the development and spread of several technological innovations that have marked the history of humankind (e.g. Wertime, 1983).

Whitin the history of pyrotechnology, the development of the technical know-how to produce ceramic objects from fire-hardened clay represents an important step that had a deep repercussion on the lifestyle of the societies that adopted this innovation (e.g. Gibbs, 2015 and literature therein). Therefore, it is not surprising that ceramic objects, which are also among the best-preserved materials in the archaeological record, have been at the focus of several pyrotechnological studies (e.g. Tite, 1995, pp. 37–38 and literature therein). Archaeometric studies that focus on the reconstruction of pottery pyrotechnology are especially concerned with the estimation of firing temperatures through the identification of changes in the pottery microstructure (e.g. porosity, clay matrix, progressive sintering, vitrification and mineralogy) in relation to temperatures and atmospheric conditions applied during the firing process (Gliozzo, 2020; Maniatis & Tite, 1981; Rice, 2015, pp. 376–387). Although less frequently, multidisciplinary experimental studies have also been conducted that combine the analysis of pyrotechnological structures with the study of ceramics (e.g. Barbaro, Forte, Muntoni, & Eramo, 2021). These works illustrate well how pottery firing was a complex procedure due to the large number of variables, which were involved in this process and which cannot be reconstructed only by studying the ceramic objects themselves (Amicone et al., 2021). Hence, the importance of studying pyrotechnological installations such as pottery kilns to gain more insights into ancient pyrotechnological techniques. Kilns for pottery firing are a common feature of many archaeological sites, yet they are seldom the target of systematic and multi-method investigations (Hansen Streily, 2000). Recent studies have shown the potential of combining a detailed stratigraphic investigation of a pottery kiln with geoarchaeological studies to reconstruct the life history of the pyrotechnological installations and acquire more information about their use as well as the kiln's abandonment processes (Karkanas, Berna, Fallu, & Gauß, 2019; Weiner et al., 2020).

In this study, we present a multi-method approach to study a late sixth millennium BC pottery kiln identified in the Bora plain, a subunit of the Peshdar Plain, located in Iraqi Kurdistan, near the border between Iraq and Iran. Micromorphological and micro-remains analyses were applied to the study of the kiln fill to understand the formation processes of this deposit as well as to trace possible remains of the fuel used in the operation of this installation. Ceramic petrography and X-ray diffraction were applied to the study of the kiln lining to identify the composition of raw materials used to produce the structure and have insights on the temperatures at which this installation was exposed. Overall, our results contribute to reconstructing the life history of a Chalcolithic pottery installation in a region relatively poorly understood for that period as well as increasing the understanding of prehistoric ceramic kiln technology. In the next sections, we will provide some archaeological background before moving to describing the results of our analyses.

2 The Chalcolithic Period in the Peshdar and Bora Plains

The Chalcolithic period (c. 6000–3500 BC) in Mesopotamia (largely corresponding to modern Iraq) and West Iran was a period of intense innovation, where societies transitioned from small-scale agricultural villages typical of the Neolithic period (c. 10000–6000 BC) to larger cities and city states that characterized the Bronze Age (c. 3500–1200 BC) (Rothman, 2002). However, the breadth of contact and the interaction between Chalcolithic cultures of northern Mesopotamia with those that developed on the Iranian plateau are still poorly understood (Stein & Alizadeh, 2013; Stein, 2012). The region of Iraqi Kurdistan represents a crucial area to better understand the timeline and the trajectory of this interaction as it is located at the junction between the Mesopotamian plain to the west and the Iranian plateau to the east, separated by the *chaîne magistrale* of the Zagros Mountains (Figure 1).

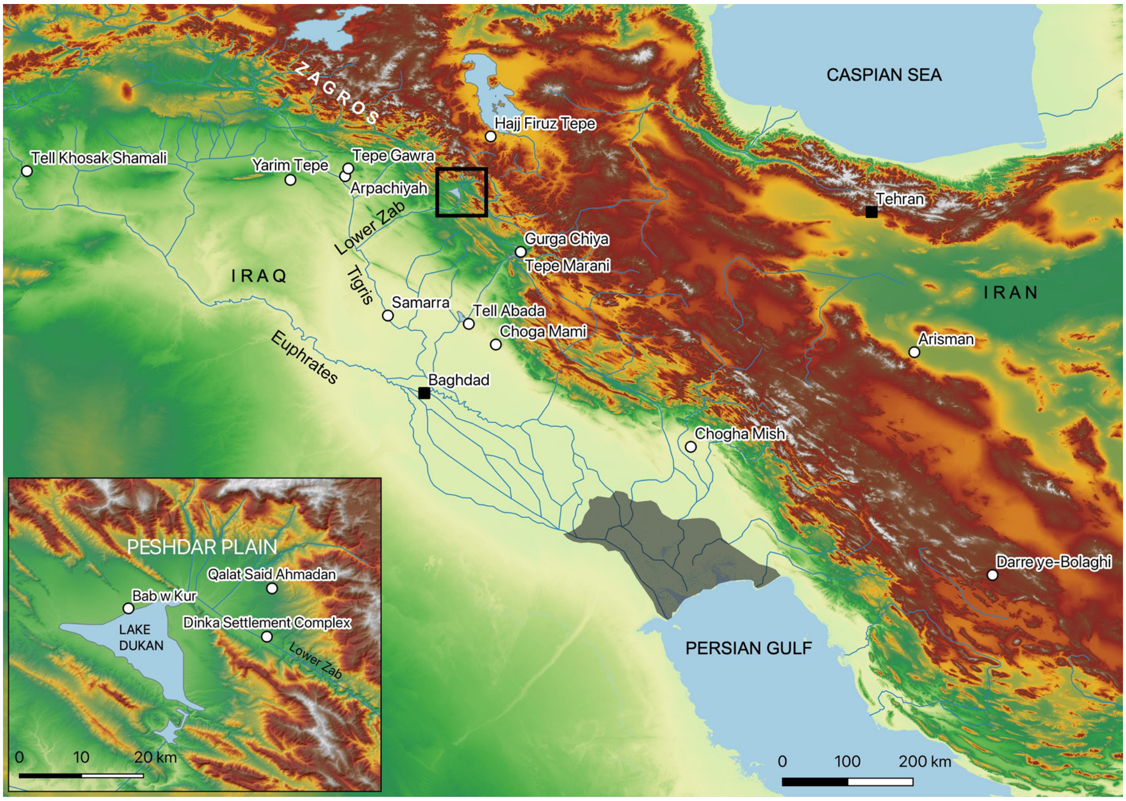

Figure 1: Map showing the sites mentioned in the text (white dots) along with the main modern cities (black squares). The inset shows the location of the Dinka Settlement Complex in the Peshdar Plain. Prepared by Andrea Squitieri.

Recent archaeological work focusing on the Chalcolithic period in Iraqi Kurdistan has been carried out in the Duhok, Erbil, Rania, and Sharizor Plains, as well as in the Chamchamal area (Peyronel & Vacca, 2015; Sconzo, 2019; Skuldbøl & Colantoni, 2016; Stein & Alizadeh, 2013; Vallet, 2017; Wengrow et al., 2016). However, the Peshdar Plain and its subunit the Bora Plain are relatively poorly known for what concerns the Chalcolithic period. Although the archaeological survey conducted by the "Sulaymaniyah Governorate Archaeological Survey" team revealed the presence of Chalcolithic period sites (Baldi, 2018; Giraud, 2016), excavations reaching Chalcolithic period levels have been conducted only at the site of Qalat Said Ahmadan (WGS84/UTM 38N 513191 E, 4008924N), which have yielded a pottery sequence and a very few partially excavated structures (Tsuneki et al., 2015). The Chalcolithic kiln discovered in the Bora Plain (UTM 38N 512258 E, 3999222N), which is the focus of the present article, represents the first architectural feature unearthed in this area belonging to the Chalcolithic period. It was found below the Iron Age (c. 1200–800 BC) remains of the site named Dinka Settlement Complex (Radner, Kreppner, & Squitieri, 2016, 2017, 2018, 2019) (UTM 38N 512710 E, 3999280N) (Figure 2), which has been investigated by the "Peshdar Plain Project" since 2015.[1] In the following sections, we will first provide the excavation results of the pottery kiln, followed by the results of the analysis conducted on its fill, and, finally, we will discuss the implications of our findings.

1 The Peshdar Plain Project (PPP) was initiated in 2015 by Prof. Dr Karen Radner (LMU, Munich) with funds provided by the Alexander von Humboldt Foundation and LMU, Munich. Since 2018, it has been co-directed by Radner and Prof. Dr F. Janoscha Kreppner (WWU Münster). The authors are grateful for the opportunity to study and publish the materials presented in this article. The excavation of the Chalcolithic kiln was carried out within the framework of PPP with additional funding provided by the Rust Family Foundation, with a grant awarded in 2019 to Dr Andrea Squitieri (Heidelberg University) and Dr Mark Altaweel (UCL) (grant no. RFF-2019-95).

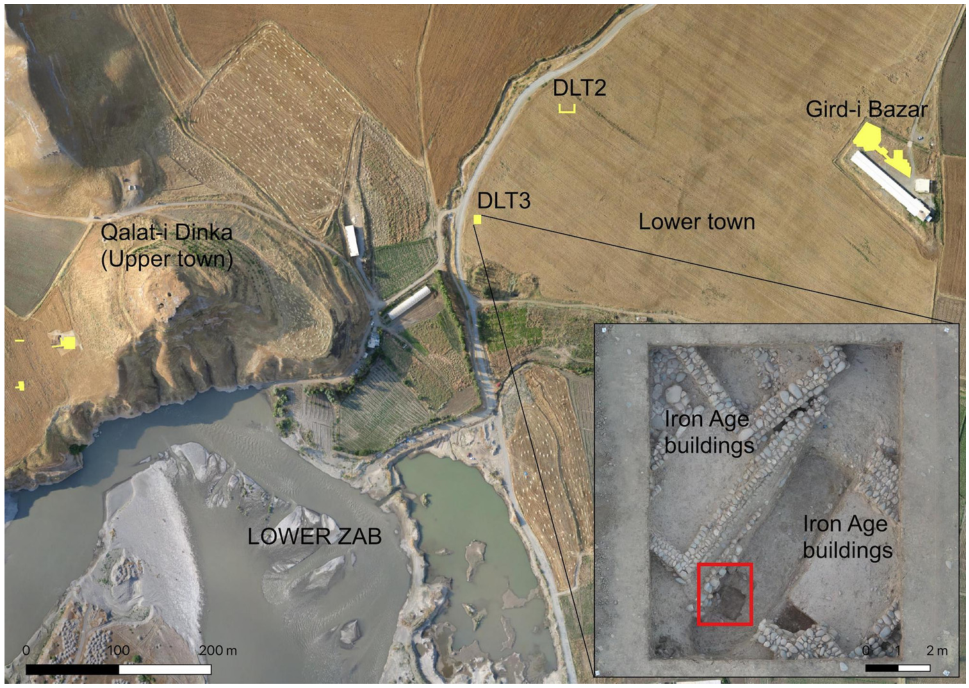

Figure 2: Orthophoto of the Dinka Settlement Complex, with in yellow the excavated areas updated to 2019. The inset shows the operation DLT3, where the Chalcolithic kiln was found (red square) below the Iron Age walls. Orthophoto by ICONEM (courtesy of J. Giraud). Prepared by Andrea Squitieri.

3 The Excavation of the Kiln and Its Structural Comparisons

After initially being found in a geoarchaeological investigation (Altaweel & Marsh, 2016), a full investigation of the pottery kiln did not occur until the spring of 2019 (for a full report, see Squitieri et al., 2020). During this campaign, the portion of the Iron Age wall located right above the kiln was removed to clear the kiln's structures and allow its excavation. The excavations revealed the kiln originally consisted of two parts. The upper part, that is the firing chamber, was directly above ground level, while the other part, the combustion chamber, was sunk into the ground. In between the two parts, a grate existed to support the pots. The upper chamber was fully destroyed by the construction of the Iron Age wall. Only a few tumbled remains of this chamber were found inside the kiln's fill. The lower chamber, dug into the ground, was almost entirely preserved (Figure 3a–c). It was dug into a natural layer of pebbles.

Inside this chamber, at the lowest level, there was a large accumulation of friable, yellowish-brown soil with some pebbles. On the backside of the kiln, the heat of the fire had rubified the soil. The lining of the combustion chamber consisted of heavily fired clay, with a grayish-greenish appearance. Toward the middle of the lower chamber is a central column with a diameter of about 30 cm, with about 90 cm height. The column is made of clayey material with the lining, about 3–4 cm thick, being light grayish in color. The column is broken in the lower half and slightly displaced from its central position as a result of the destructive processes that occurred at the end of the kiln's use. The north-western part of the kiln fill contained architectural elements representing the collapse of the upper chamber, mixed with a dark brown soil (Figure 3d). Among the most striking fragments are elongated plano-convex elements, which were part of a structure that extended radially from the edge of the kiln to the central column. This structure represented the grate of the kiln. They were observed in different sizes and at least one of these was slightly curved. There were also fragments of architectural elements with one flat side with a concave imprint on the reverse, which probably functioned to fix the plano-convex elements between the column and the outer edge. A few fragments with holes were found, which may have been part of the original firing chamber grate. They may have been located at the edges of the grate to provide perforations that enabled the heat to go from the lower into

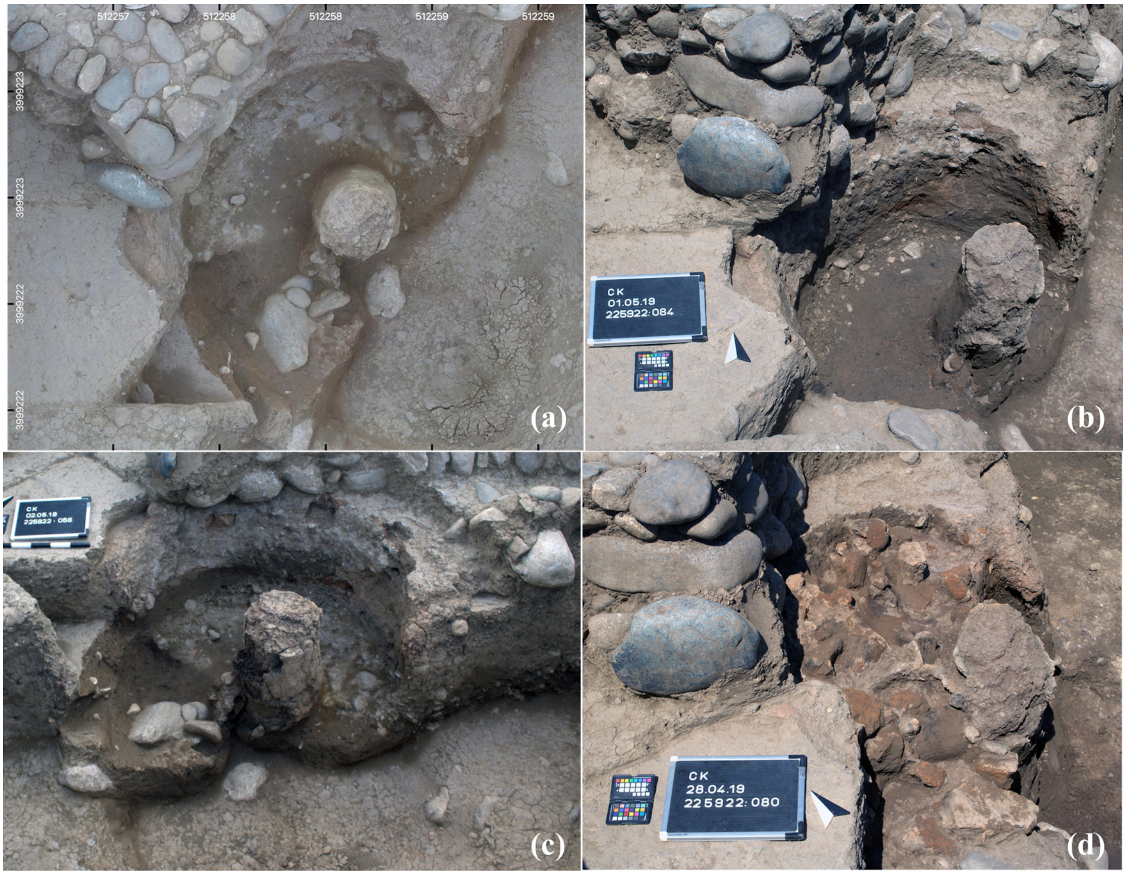

Figure 3: The Chalcolithic kiln at different stages of the excavation. (a) Orthophoto showing the last phase of excavation; (b) field photo showing the last phase of excavation; (c) field photo showing the partially excavated kiln's fill; and (d) field photo showing the collapse of the upper structure in the kiln's fill. Photos by Jens Rohde, Sophie Pietsch, and Andrea Squitieri.

the upper (or firing) chamber. Several other architectural pieces had a rather thick, flattish shape, and they most likely belonged to the exterior casing of the firing chamber. Apart from collapsed architectural elements and some pottery sherds, which will be dealt with below, no other finds were collected from the kiln fill. A charcoal sample collected from the kiln fill was radiocarbon dated to 5218–5024 calBC (95.4% probability), corresponding to the Chalcolithic period in Mesopotamia (also Ubaid 3–4) (Squitieri et al., 2020, p. 201).

Based on the excavation results, it is possible to reconstruct the original form of the kiln as that of an up-draft kiln with a circular shape, having two stacked chambers. The combustion chamber was sunk into the ground and separated from the upper chamber by a grate, which was supported by a central free-standing column (Figure 4a). The kiln grate was made of plano-convex structural elements that extended radially from the kiln's edge to the column (Squitieri et al., 2020, Figure I10). Other structural features of the kiln, such as the stocking channel, or the aperture through which the fuel was inserted could not be found since they had been most likely destroyed by the construction of the Iron Age walls. Moreover, no clear evidence of the upper chamber has been preserved; hence, we can only suggest that the kiln had a dome-shaped cover. Some of the earliest up-draft kilns, with a circular plan, developed in Mesopotamia during the second half of the seventh millennium BC, those from the site of Yarim Tepe I being among the oldest attested (Hansen Streily, 2000). Up-draft kilns dated to the same period as our kiln has been found at various sites from Syria to Iran (see e.g., Tell Kosak Shamali and Choga Mish; Alizadeh, 1985; Nishiaki, 2016). There could be different structural variations in up-draft kilns. Based on the kiln typology proposed by Boroffka and Becker (2004), our kiln belongs to the so-called "Typ V," characterized by the presence of a free-standing central column supporting the kiln grate. Close parallels to our kiln can be found at the site of

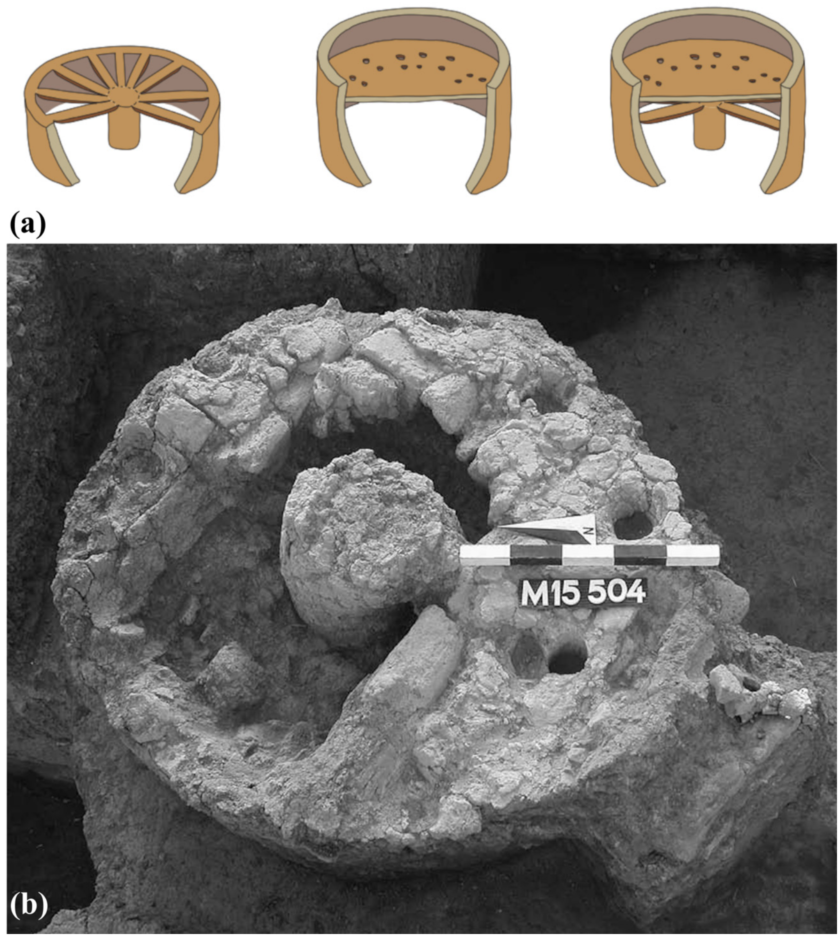

Figure 4: (a) Hypothetical reconstruction of the kiln presented in this article based on the excavation results. Drawings by Jens Rohde; (b) Darre ye-Bolaghi. Kiln 504 (site 131) (Helwing and Seyedin, 2016, Figure 17.8). Image reproduced with permission of the Oriental Institute, University of Chicago.

Darre ye-Bolaghi, in south-western Iran, in the layers dated to the Ubaid 3–4 period (c. 5500–4300 BC; Helwing & Seyedin, 2016) (Figure 4b). More parallels come from the site of Arisman, in central Iran, from the late fourth millennium BC levels (period Sialk III) (Vatandoust, Parzinger, & Helwing, 2011). In the Mesopotamian plain, a close parallel to our kiln comes from the site of Tell Abada, in the Hamrin basin, where a kiln was found, dated to the fifth millennium BC, having a grate supported by a central column (Jasim, 1985, Figure 35a). Later examples of up-draft kilns with a free-standing central column are known from Namazga Depe (Turkmenistan) (Boroffka & Becker, 2004) and Tell el-Far'ah North (West Bank; Medeghini, Sala, De Vito, & Mignardi, 2019), both dated to the Bronze Age (c. 3000–1200 BC). Archaeometric and micromorphological analysis of the fills from this type of kiln have not been carried out, which makes the results from our kiln an important step forward in our understanding of the use and the abandonment of such kilns.

4 Pottery From the Kiln Fill

The study of the pottery coming from the kiln fill is ongoing and will be part of a dedicated study. However, it is worth providing here some preliminary information. Overall, about 30 sherds were collected from the

Figure 5: A selection of pottery sherds from the kiln fill. (a) Diagnostic sherds from a small conical bowl and (b) body sherds from a painted and polished pot. Prepared by Jean-Jacques Herr.

kiln fill (Figure 5). Five sherds belonged to a pot with flared rim and a polished surface on which faint traces of a red painting can be observed. Similar pots were found at the site of Qalat Said Ahmadan (Ubaid layer 1), also located in the Peshdar Plain, and the site of Gurga Chiya, located further to the south, in the Shahrizor Plain (Tsuneki at al., 2016, Figure 2.10.1–2; Wengrow et al., 2016, Figure 12.4–6). Flared-rim pots are also attested at the site of Tepe Gawra (level XII A), to the northwest of the Peshdar Plain (Tobler, 1950, pl. CXXXVIII.291). Thin everted rim fragments were also found in the kiln fill, having an organic tempering (possibly chaff), which have parallels to Tepe Gawra (level XII) (Tobler, 1950, pl. CXXXV.267). Finally, the complete profile of a small conical bowl with a flat base was also recovered from the kiln, made using a plant tempering and fired in a semi-oxidizing atmosphere. Parallels of this bowl come from the sites of Tepe Marani and Gurga Chiya (Wengrow et al., 2016, pp. 263, 273), both in the Sharizor Plain, as well as Hajji Firuz Tepe, in north-western Iran (Voigt, 1983, p. 75). Overall, the chronological horizon of the above-mentioned parallels ranges from the late sixth millennium BC through the fifth millennium BC, thus matching the radiocarbon date obtained from our kiln. The pottery from kiln fill seems to be connected to both the Northern Mesopotamian and Iranian Chalcolithic traditions, especially to the area of the Zagros Mountain range.

5 Scientific Analysis of the Kiln: Materials and Methods

Scientific analyses were carried out on both the kiln lining and the kiln fill. The kiln lining was studied combining ceramic petrography and X-ray powder diffraction analysis (XRPD). Petrographic analysis was applied using a LEICA (2500P) polarizing microscope to characterize the composition of raw materials employed to make the kiln structure (Quinn, 2013, pp. 23–33; Whitbread, 1989). The application of XRPD provides a detailed mineralogical characterization of the structure fragments that helps in the reconstruction of their original firing temperature ("archaeothermometry", see Gliozzo, 2020; Rice, 2015, pp. 99–116 and the references therein). This method makes use of the presence and absence of mineral phases that form or disappear at specific temperatures and atmospheric conditions. The instrument used was a Bruker D8 advance with a Cu-sealed tube (40 kV/20 mA), a Göbel mirror optics, a 0.2 mm divergence slit, a fixed knife edge to suppress air scatter, sample rotation, and a VÅNTEC 1-detector. To identify the crystalline phases, the 2006 Powder Diffraction File database from the International Centre for Diffraction Data-Joint Committee of Powder Diffraction Standards was used.

The kiln fill was studied combining micromorphology and micro-remains analysis. Archaeological, or soil micromorphology, is the study of soils and sediments at the microscopic level through transmitted light microscopy. Utilizing thin sections, micromorphology describes the microstratigraphic structures of sedimentary deposits to understand the syn- and post-depositional processes, which formed the deposits (Goldberg & Aldeias, 2018; Karkanas & Goldberg, 2018). In the investigation of kilns, micromorphology can provide insights into the kilns' use as well as post-abandonment processes (Karkanas et al., 2019). Micro-remains analysis focused on quantifying phytoliths, fecal spherulites, and ash pseudomorphs, which are micro-remains that can be found in either wood ash or animal dung in different concentrations depending on the fuel that was used and various other factors such as animal diet, age, and sex (Canti, 1999; Dalton & Ryan, 2020). This strategy enabled us to differentiate wood and dung fuel types and determine mixtures of them. Phytoliths are silicified plant cells that can give us information on what plant species are present and which anatomical parts were used. Dung spherulites are calcitic radial spheres that form in the gut of different animals, especially ruminants like sheep/goat and cows tend to produce them in relatively high numbers. Their presence is direct evidence for the presence of dung. Ash pseudomorphs, or pseudomorphs after calcium oxalate crystals, are the combustion residues of biominerals that are present in most higher plants and are abundant in woody plants. They are one of the main components of wood ash and, therefore, are indicative for the use of wood as a fuel (Gur-Arieh & Shahack-Gross, 2020).

6 Analysis of the Kiln Structure

6.1 Sampling Strategy

One sample was analyzed through petrographic analysis, while nine samples were analyzed via XRPD analysis. The sample for the petrographic analysis was selected from a well-preserved part of the kiln lining. The samples for XRPD were selected to represent both altered and thermally unaltered specimens. Among them, seven samples were taken from surfaces of the kiln structure which were exposed to heat, the remaining two are control samples taken where the kiln lining was likely not thermally altered. The control samples were taken as a reference to understand the direct impact of heating on the material. Figure 6 shows the locations of all samples in the kiln structure. The control samples are each associated directly with the thermally altered samples. CK2 is directly associated with CK1 (as in Figure 6) and CK8 is associated with CK7. From these samples, we can directly see the impact of thermal alteration on the kiln's material.

6.2 Petrographic and XRPD Analysis Results

Petrographic results show that the paste contains medium-coarse (0.4 mm) to fine (0.15 mm) inclusions with polymodal distributions, suggesting that they are naturally occurring in the sandy clay used to build the pyrotechnological installation. Dominant inclusions are quartz and feldspars (plagioclase), while micritic calcite and metamorphic rocks are common (Figure 7a and b). The latter are foliated and composed of quartz and micas. The clay matrix is non-calcareous and optically inactive to weakly active. Muscovite and amphiboles are also rarely occurring. The sample is also characterized by the presence of abundant

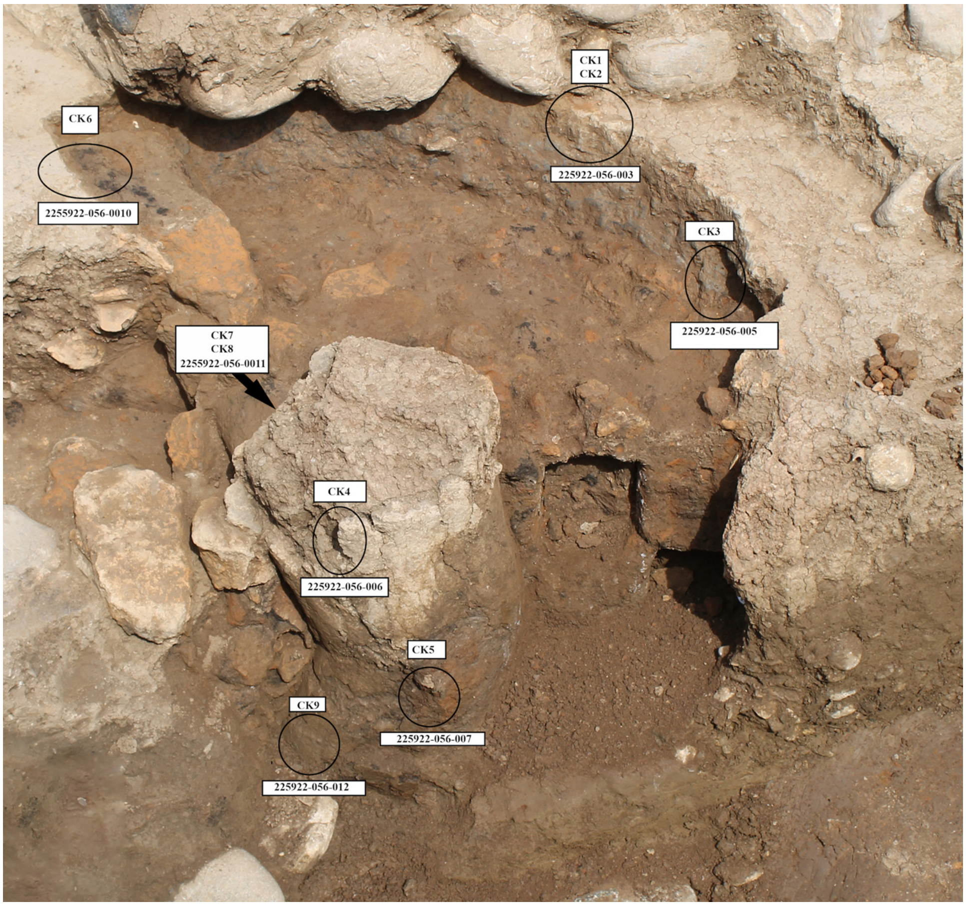

Figure 6: Locations of the samples CK1 to CK9, with indication of the sample IDs. Prepared by Silvia Amicone and Ada Dinckal.

elongate voids occasionally filled with charred material (Figure 7a). The XRPD analysis confirmed that quartz and feldspars are present throughout all samples, in both the reference and the thermally altered specimens (Figure 7c–e). The reference samples represent components of the kilns, which were not thermally altered. These samples are CK2 and CK8 (Figure 7c). They show the presence of calcite, and a weak peak that could be correlated to amphibole. In addition, both specimens show peaks at 6.193° (d = 14.26 Å), which could indicate the presence of chlorite and/or montmorillonite. Another peak at 8.836° (d = 10 Å), as well as at 19.717 (d = 4.49 Å), could suggest the presence of illite and/or muscovite.

Among the thermally altered samples, two show evidence for the chlorite/montmorillonite as well as illite/muscovite and amphibole. These samples are CK5 and CK6 (Figure 7d). CK5 displays the more distinct evidence for these peaks, while in CK6, the peaks for chlorite/muscovite are very weak. Meanwhile sample CK3 also shows peaks at 8.836° (d = 10 Å) and 19.717° (d = 4.49 Å) for illite/muscovite but did not preserve evidence of chlorite/montmorillonite (see Figure 7d). The remaining samples, namely CK1, CK4, CK7, and CK9, show no evidence for chlorite/montmorillonite (Figure 7e). There is potentially evidence for illite/muscovite at 19.717° (d = 4.49 Å); however, the peak at 8.836° (d = 10 Å) is very weak (CK7 and CK9), or absent entirely (CK1 and CK4). Calcite is present and identifiable in all the samples.

6.3 Raw Materials and Heat Alteration of the Structure

The results of the petrographic analysis show that the characteristics of the raw materials used to build the kiln are perfectly compatible with the clay materials available around the site. The Bora Plain is surrounded on the

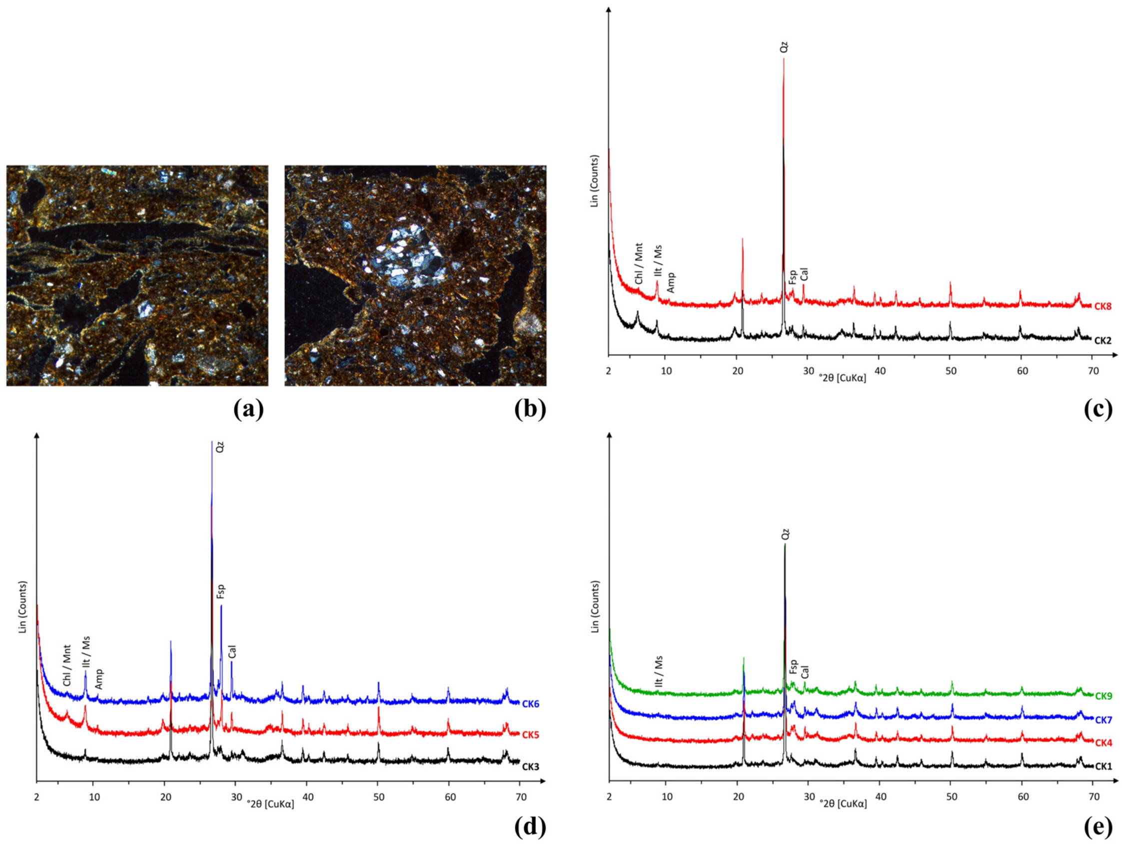

Figure 7: (a and b) Thin section micrographs of selected samples from the kiln structure (field of view 3mm). (c) XRPD diffractograms of samples C2 and C8; (d) XRPD diffractograms of samples C3, C5, and C6; and (e) XRPD diffractograms of samples C1, C4, C7, and C9. Prepared by Silvia Amicone. Mineral abbreviations from Whitney and Evans (2010).

east, north, and west by a ridge of smooth hills formed by alluvial deposits that could have been the results of heavy fluviatile activity or several great flow events during the Pleistocene. Alluvial fan deposits also cover all low-elevation areas of the plain. These contain sandy calcareous and not calcareous clays rich in metamorphic, sedimentary, and less frequently volcanic clasts that derived from the Zagros area (Geiger, 2019). It has been already shown that these deposits were used as clay sources for pottery making (Amicone, 2017).

The presence of abundant inclusions with a polymodal distribution with the sample analyzed suggest that the clay used to build this kiln was minimally processed, in other words there is no evidence for a thorough cleaning (e.g., via sieving and levigation) or addition of mineral tempers (Quinn, 2013). Nevertheless, organic material such as chaff could have been added to as indicated by the presence of abundant elongated voids sometimes filled by charred materials (Figure 7a). The use of an organic, fibrous inclusion as temper is not surprising as this type of inclusion makes the firing structures much lighter, while providing structural support and thermal insulation and it is a widely known tradition across several periods in different parts of the world (Martinón-Torres & Rehren, 2014; Skibo, Schiffer, & Reid, 1989).

Heat alteration can be evaluated by comparing the XRPD results of the two control samples to their heated counterparts. The peaks of chlorite/montmorillonite, illite/muscovite, and the amphibole are clearly weaker in the samples that are heated. In addition, taking this into account and exploring the locations of the samples, patterns can be observed and discussed. To begin with, CK5 which is a thermally altered sample, preserves evidence for the chlorite/montmorillonite and illite/muscovite peaks (see Figure 7d). CK5 is located at the bottom of the kiln's central column. This may indicate that the central column had been exposed to less heat alteration compared to the other structural material. However, it is also possible that the sample CK5 does not represent the original surface of the kiln structure that was exposed to thermal alteration, with that original surface structure having been crumbled into the kiln fill revealing an unheated surface. Comparing CK5 to CK4, which is further up the column (Figure 7d and e), CK4 shows clear evidence of thermal alteration from the absence of the chlorite/montmorillonite, as well as the illite/muscovite peak at 8.836° (d = 10 Å).

CK6 is another thermally altered sample that preserves peaks for chlorite/montmorillonite and illite/muscovite (Figure 7d). The peaks are weaker compared to the control samples; however, they are still distinctively present within the sample. This is similar to CK3, which also shows the presence of some illite/muscovite (Figure 7d). These two samples are found along the outer structural edges of the kiln and could indicate further erosion of the kiln structure's surface revealing surfaces of less thermally altered material. It could also be that these surfaces were generally less impacted by thermal alteration. However, CK1, which is from a level of the kiln structure similar to CK6 and CK3, has no chlorite/montmorillonite and illite/muscovite peaks (Figure 7e). As it is approximately on the same surface as CK6 and CK3, this could indicate that it is more likely that weathering and post-abandonment processes are impacting the results of CK6 and CK3. Similar to CK1, CK4, and CK7, sample CK9 (Figure 7e) has been very thermally altered. It is located in the lowest portion of the kiln. This sample could preserve the most authentic reference for thermal alteration without being impacted by weathering due to its low position in the kiln with the likelihood that it was quickly covered and protected by the kiln's post-abandonment fill. Overall, by combining results from all the samples, the presence of clay minerals in several samples and the absence of mineral phases that are normally found forming at high temperature (e.g., cristobalite, mullite) indicate that the kiln was operated at relatively low temperatures, that is not exceeding c. 900°C. The implications of this result will be discussed below.

7 Micro-Analysis of the Kiln Fill

7.1 Sampling Strategy

Two block samples were collected from the kiln fill (Figure 8). They were taken in the shape of two rectangular blocks, each measuring c. 17 cm × 12 cm × 4 cm. After isolating them from the rest of the fill by means of a deep cut, they were each wrapped into gypsum bands and left to dry for several minutes until it was safe to remove them without compromising their structural integrity. The lower sample, 108, was processed into 2 thin sections (108A, 108B), while the stratigraphically higher sample, 107, was prepared into 3 thin sections (107A, 107B, 107C). Thin section preparation was conducted at Terrascope in France. Samples were prepared into 5 thin sections at 30-μm thickness with dimensions of 6 cm × 9 cm. Thin sections were analyzed under the naked eye and magnified up to ×200 under plane-polarized light (PPL), as well as cross-polarized light (XPL) using LEICA (2500P). High-resolution scans of the thin sections were produced with a flat-bed scanner (Arpin, Mallol, & Goldberg, 2002) while photomicrographs were taken with a digital camera mounted onto the microscopes. Figures were made utilizing Adobe Photoshop. Descriptions of the micromorphological thin sections were conducted as per the protocols of Stoops (2021) and Courty, Goldberg, and Macphail (2009). In addition, from the two block samples 107 and 108, ten loose sub-samples were collected and analyzed to determine the fuel used in the kiln, four sub-samples from block 107, and six from 108. As mentioned above, three micro-remains were quantified per 1 g sediment: phytoliths, dung spherulites and ash pseudomorphs. In this study, the rapid extraction method developed by Katz et al. (2010) was used to quantify the phytolith concentration. Both dung spherulites and ash pseudomorphs were extracted and quantified following the method developed by Gur-Arieh, Mintz, Boaretto, and Shahack-Gross (2013).

7.2 Stratigraphy and Microstratigraphy

The macro-stratigraphy of the kiln fill is composed of a dark-brown soil with embedded architectural elements that collapsed from the kiln's structure (Squitieri et al., 2020, pp. 193–196). This simple description

Figure 8: Location of the micromorphological samples 107 and 108 in the kiln fill. Photo and annotations by Andrea Squitieri.

is mostly reinforced by the micromorphological analysis, as the fine fraction can be described as a dark brownish clay along with frequent organic silts. The coarse fraction consists of ceramic fragments, likely construction material, coarse sand-sized quartz grains as well as similarly sized limestone fragments. This groundmass is consistent throughout the deposit, with the only variation being in the microstructural organization varying from somewhat accommodated moderately separate sub-angular crumbly aggregates (in Figure 9 Somewhat Accommodating Crumbly Microstructure) to smaller unaccommodated porous crumbs (in Figure 9 Porous Crumbly Microstructure). These changes in the groundmass organization are mostly visible through macroscopic analysis of the thin sections. In Figure 9, this variation can be seen with the well-formed accommodating aggregates appearing in the lowest thin section (108B). Above that, along the right edge of 108A, we see the first instances of the porous crumbly microstructure, which recurs and becomes dominant from 107B and higher. These are the two major structural patterns found within the 5 thin sections. One additional structural type occurs at the base of 107C, which has a massive microstructure with no clear aggregate formation and simple channel voids distributed throughout. This change in the microstructure is seemingly driven by the frequency and size of the accompanying ceramic fragments. Where the frequency and size are low or absent, the microstructure consists of the large well-formed aggregates or is massive, as the presence of the ceramic fragments increases the microstructure grades into the highly porous crumb type.

7.3 Features

7.3.1 Ceramic Fragments

The ceramic fragments are generally gravel sized. Figure 9 illustrates the frequency and size of the ceramic fragments. Noteworthy within the fragments are the highly consistent morphology of vegetal voids, as well as the highly frequent presence of sand-sized inclusions of the same nature of those observed in the thin

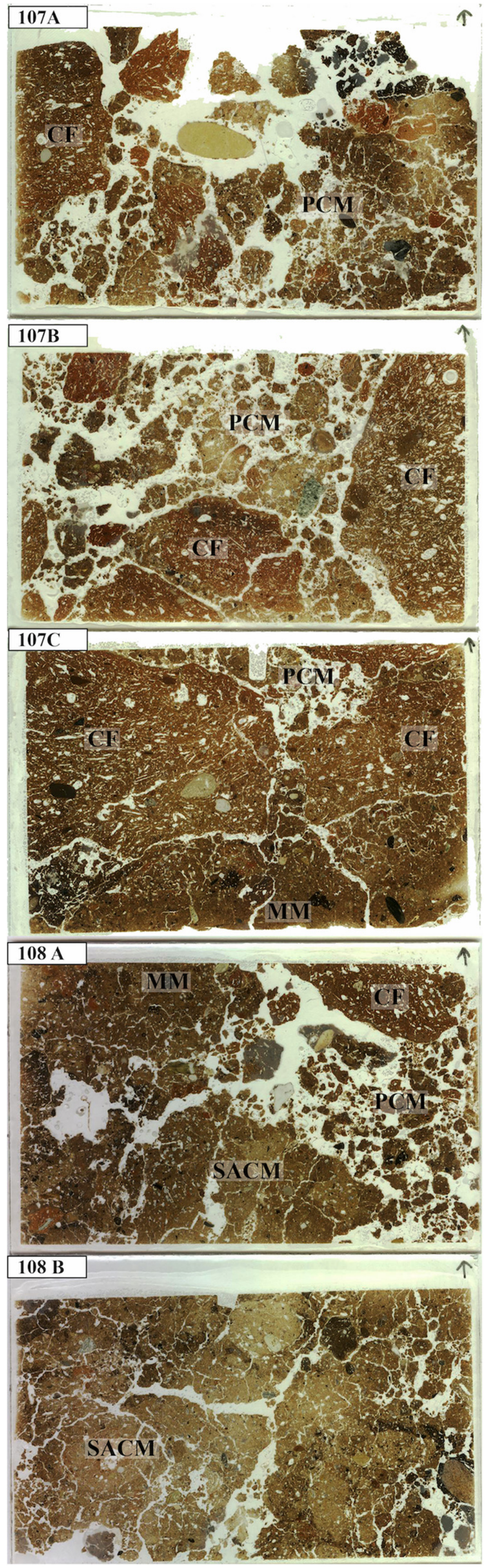

Figure 9: Thin sections aligned to their elevation in the fill. (Note, in actual fill sample 108 is approximately 20 cm to the right of sample 107, see Figure 8). Each thin section is 9 cm long and 6 cm wide. The lowest thin section (108B) is composed of a somewhat accommodating crumbly microstructure (SACM), this microstructure with no ceramic fragments is composed of a well-formed groundmass divided into somewhat accommodating pedological structures. In the next thin section (108A), the SACM transitions into porous crumbly microstructure (PCM) with the introduction of the gravel-sized ceramic fragments (CF). To the top and left of this thin section, the SACM also transitions into a more massive microstructure (MM). The MM is continued in the next thin section (107C) where it transitions into a PCM with the introduction of further gravel-sized CF. A pattern that continues in the next two thin sections (107B and 107A). Prepared by Ada Dinckal.

section of the sample of the kiln structure. As indicated previously, these fragments are likely a component of the kiln structure itself. While present throughout the fill; the ceramic fragments increase in both size and frequency higher up into the kiln's fill.

7.3.2 "Dung" Features

Primarily at the base of the fill, appearing in thin section 108B, but also reoccurring in small fragments in higher regions, potentially in 107A, is what may be dung. This component is composed of highly birefringent dusty clay occurring with fibrous organic fragments along with high density of phytoliths. Of important note is the complete absence of dung spherulites within these features. This typic marker of dung presence is notably absent from all the thin sections, and especially within the "dung" features (see also below). Previous investigations of dung features have shown that spherulites can be absent due to burning in excess of 700°C (Amicone, Morandi, & Gur-Arieh, 2020; Canti & Nicosia, 2018). However, this absence is probably not a product of burning of the dung. At temperatures higher than 500°C, there is clear evidence for the combustion of organic material (Amicone et al., 2020); furthermore, at temperatures between 600 and 900°C, the siliceous material that forms phytoliths tends to deform due to heat (Brönnimann, Ismail-Meyer, Rentzel, Pümpin, & Lisá, 2017). Such evidence for heating is not present within the "dung" features of the present deposit. It is possible that these dung features did not contain any spherulites to begin with (Canti, 1999), or that they had been removed through diagenetic processes. Calcite within the deposits appears to be generally composed of secondary pedofeatures; it is possible that during the original depositions of these dung features, the depositional environment may have been acidic enough to remove the calcareous dung spherulites. The results of the micro-remains analysis presented in Table 1 show that all samples had relatively low-medium concentrations of phytoliths (1.62–4.44 million per 1 g sediment), and no calcitic micro-remains (dung spherulites or ash pseudomorphs) were found. As mentioned above, the absence of dung spherulites does not necessarily mean the absence of dung, yet the relatively low phytolith concentration suggests that wood may have been the main fuel material used to fire the kiln, with some dung additions (Gur-Arieh et al., 2014). Finally, within sample 108, there is also evidence for fragments of bones along with the dung matter.

7.3.3 Slaking Crusts and Other Water Features

Slaking crusts are small fragments of graded bedding occurring from ponded water, such as those that form on muddy surfaces after rain. The fragments are normally evidence that there was a surface somewhat

Table 1: Results of the micro-remains analysis

Sample nr.	Phytoliths in 1 g burnt sediment (10^6)	±30% error	Pseudomorphs in 1 g burnt sediment	±30% error	Spherulites in 1 g burnt sediment	±30% error
107.3	4.44	1.33	—	—	—	—
107.5	2.07	0.50	—	—	—	—
107.8	2.12	0.52	—	—	—	—
107.12	3.30	1.27	—	—	—	—
108.2	1.16	0.35	—	—	—	—
108.4	1.17	0.35	—	—	—	—
108.6	2.02	1.01	—	—	—	—
108.8	2.05	0.49	—	—	—	—
108.10	4.02	1.09	—	—	—	—
108.12	2.03	0.49	—	—	—	—

Prepared by Shira Gur-Arieh.

present at the elevation the crusts appear on (Deak, Gebhardt, Lewis, Usai, & Lee, 2017). While they can be moved around and redeposited, there is no evidence that this is the case here. Rather, the crusts here are found lying horizontally. The presence of slaking crusts only occurs with thin sections 108A and 107B. Other evidence for the influence of water on the deposits includes the presence of "dusty" clay coatings, which likely formed as a result of water percolation through the deposits (Figure 10, right). Furthermore, the presence of secondary micritic calcite as hypocoatings around voids (Figure 10, left; Deak et al., 2017; Miller & Goldberg, 2009) indicates that water had an effect on the post-depositional formation of the deposits.

7.3.4 Discussion

The petrographic analysis showed that the kiln object of this study was made using a locally available clay minimally processed and tempered with organic materials that surely contributed to make the structure lighter and better insulated. The XRPD analysis conducted on the kiln surfaces exposed to heat has shown that the kiln was used at low temperatures, not exceeding 900°C. Mineralogical differences found within the structure could reflect a differentiated distribution of heat within the kiln (Gosselain, 1992; Livingstone Smith, 2001). Tite and Maniantis previously suggested a firing temperature range between 850 and 1,050°C for Chalcolithic kilns, based on the degree of vitrification observed on pottery sherds coming from the sixth millennium BC sites in central Mesopotamia, namely Choga Mami, Samarra, and Arpachiyah (Moorey, 1994, p. 150; Tite & Maniatis, 1975). Our results suggest that firing temperatures toward the lower end of Tite and Maniatis' range may have been more likely for Chalcolithic kilns structurally similar to that analyzed in this article. Concerning the firing temperature, both fuel and fuel management are clearly important aspects to be taken into account. Our analyses have shown the possible presence of dung features, despite the absence of dung spherulites. We suggest that, in our kiln, a mixture of dung and wood, the latter being more dominant, could have been used as a fuel. Dung could have helped reduce the loss of heat as it has the characteristic of burning very quickly; it could be used on top to create a layer of ash that would work as an insulator (Henderson, 2000, p. 141). Moreover, as sample 108 has also yielded evidence for fragments of bones, it is possible that bone fragments may have been used as a flux to stabilize the firing atmosphere (Moorey, 1994, p. 144). Micromorphological identification of dung features and bone are in the context of post-abandonment fill, while associated with the kiln another explanation for their presence could be through secondary discard or non-anthropogenic accumulation.

The results from the micromorphological analysis tend to agree with the overall interpretation that the fill within the kiln is a product of the abandonment of the kiln itself. The lower fill deposits (investigated through sample 108) do not show evidence for combustion – there is no clear evidence for ash and the potential dung features identified within this deposit do not show evidence of having been burned. It is likely that, after the abandonment of the kiln, there was a period where it was left open to environmental or anthropogenic debris being littered into the kiln's lower chamber. The slaking crusts occurring in thin sections 108A and 107C indicate that after abandonment the kiln fill was left exposed for a period of time allowing for water to pond on the deposit and percolate through it. As mentioned above, as we move higher up into the fill, the microstructure changes and there is an increase in the frequency of construction components being fragmented into the deposit. It seems that for a period of time after abandonment, the kiln's structure remained stable, so that the fill deposition was the product of the degradation of gravel sized or smaller structural fragments, along with sedimentary and organic components coming from outside the kiln. After the occurrence of the slaking crusts, the kiln's degradation seemed to have increased with larger structural fragments tumbling in. Whether this was intentionally driven by people or just a factor in the natural degrading of the kiln structure is hard to tell. A long period of abandonment for the kiln, as suggested by microstratigraphic analysis, is also supported by the overall understanding of the site, as no structural evidence has been found so far in the excavation areas indicating intermediate occupations between the Chalcolithic features and the Iron Age buildings of the Dinka Settlement Complex (Radner et al., 2019).

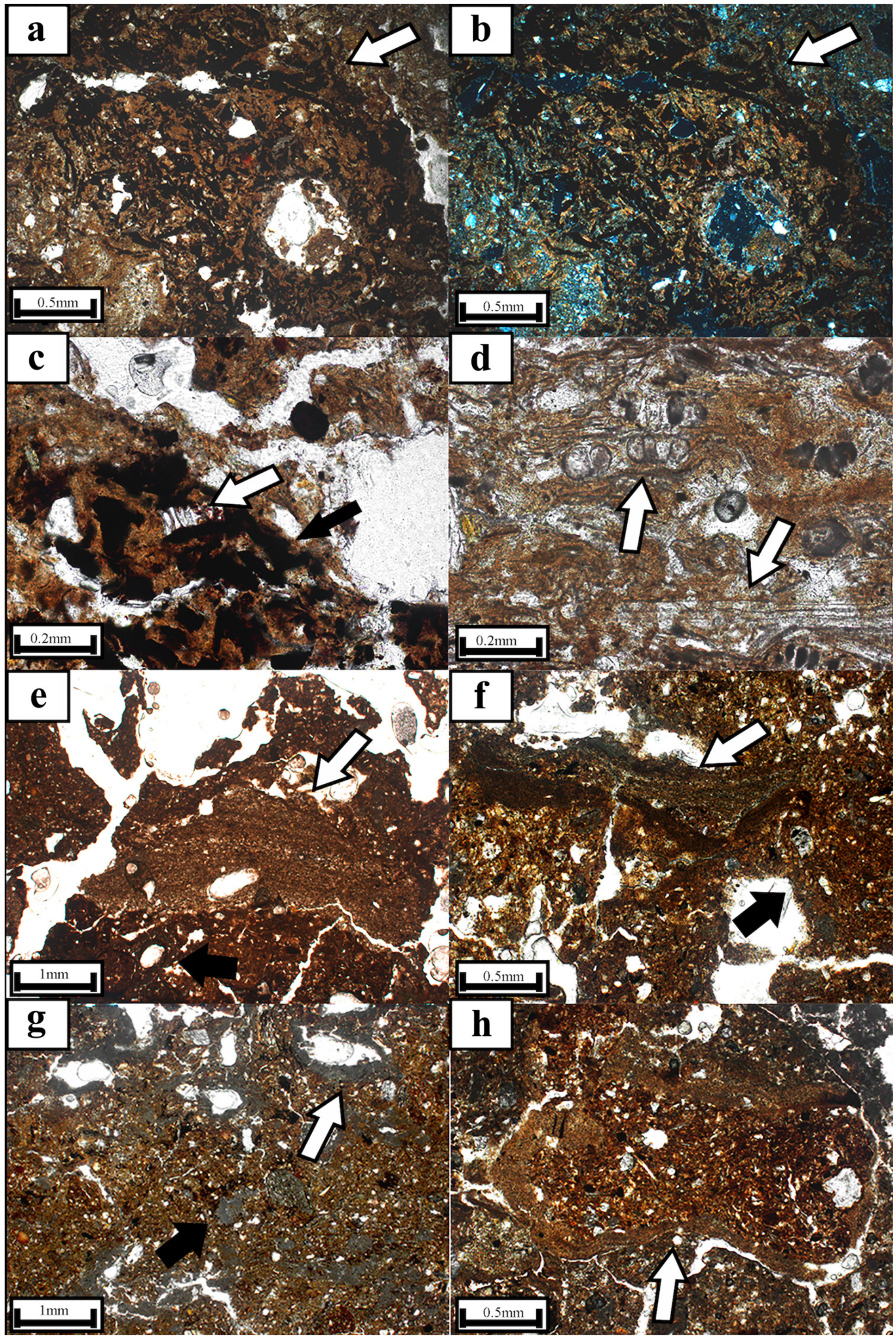

Figure 10: Photomicrographs. (a) PPL and (b) XPL showing the dung features identified in thin section 108B. White arrows identify the rolled-up structure of fibrous organics within a highly birefringent fine material. (c and d) White arrows show phytolith-microscopic silica casts formed inside of plant matter. Black arrows indicate black organic matter. Together, images (a)–(d) show the organic-derived components of the deposit indicating some biogenic origin to the fill. (e and f) White arrows indicate slacking, shallow deposits of ponded sediment with graded bedding. Black arrows indicate "dusty" coatings of void structures that are formed through the percolation of water after the deposition of the deposit. White arrows indicate "dusty" coatings surrounding voids (g) and ceramic fragments (h). Black arrows indicate a void that has been infilled by the same water percolation forming the "dusty" coatings. Images (e)–(h) show that water had a clear influence on the groundmass. Prepared by Ada Dinckal.

8 Conclusions

This article has combined results from excavations, micromorphological and micro-remains analyses, as well as ceramic petrography and X-ray diffraction to gain insights into the use and abandonment processes of a late sixth millennium BC pottery kiln in the region of Iraqi Kurdistan. Until now, the region has been relatively underexplored for what concerns sites dating to the Chalcolithic. The multi-method approach proposed in this work has been seldomly applied to the study of Chalcolithic pottery kilns in the Near East; however, this article has shown its efficacy in providing a wealth of data concerning the kiln's firing temperature, the types of fuel possibly used, and enlightening the kiln's abandonment process. The results are novel for the region and provide demonstration on the efficacy of the techniques presented. Such a multi-method approach has provided details into the pyrotechnology of the time by looking at the entire kiln's life cycle, from use to abandonment, and has offered results that the study of pottery sherds and excavation stratigraphy alone could not have revealed. Future work can build from the results presented by further exploring other regionally located kilns in the Peshdar and Rania plains and by looking at kilns across different periods such as in the Iron Age. This could allow a better understanding of how kilns evolved in this region and how such technologies linked to the broader Near East pyrotechnologies.

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