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Lime Production in the Late Chalcolithic Period: The Case of Arslantepe (Eastern Anatolia)

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Abstract: Plaster and mortar samples from Arslantepe (Turkey) hold potential to provide unique information about the lime production and adhibition during the Late Chalcolithic period (4th millennium BCE). A multi-analytical approach including polarized light microscopy (PLM), X-ray powder diffraction (XRPD), and scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) has been applied to characterize mortar samples from temple C and elite residences dated back to the late Chalcolithic 3–4 (3800–3400 BCE). A marly limestone has been identified as starting raw material for the lime production, probably coming from two different sources (local and brought from a different part of the Malatya plain). Moreover, different aggregate selection and the use of different production techniques were also detected in the samples, which are probably related to the function of the buildings. Evidence of a re-plastering process was also detected in the two elite houses, which probably refers to a routine maintenance process.

Keywords: mortar; lime; plaster; Late Chalcolithic; Anatolia; monumental architecture

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1. Introduction

Since ancient times, plaster has played an important role in human activities. It was used to cement the haft of microlithic tools, contemporarily fulfilling ritual and symbolic purposes, and later as one of the main materials used in prehistoric architecture [1]. Mud was the first material used with this purpose in the Ancient Near East [2], as it still is in many parts of the world today.

Plastering technology spread through the Levant (western regions of the Near East) and Anatolia very early, during the Prepottery Neolithic (10th–9th millennia BCE), wherein early sedentary communities built houses and communal structures using stone, mud, and clay (in various forms as wattle/daub, pies, or adobe)—all naturally available materials [3,4]. Most known and discussed is the massive use of strong plaster floors and walls of Levantine buildings since the 9th millennium BCE. Social and economic implications of lime production have been intensively discussed in this context [1,5]. Gypsum and lime have been later used as binder in plasters, showing higher durability respect to mud and clay.

One of the earliest examples of quicklime production, obtained by burning limestone at temperatures in the range 800–900 °C, was identified at Hayonim Cave (Israel) and dated around 10,400–10,000 BCE [6]. High variability in plaster production has been noticed in Prepottery Neolithic sites in the Levant [5]. At the back of the cave, an area having a 20 cm-thick white layer on the surface was discovered. SEM-EDS analysis defined the $CaCO_3$ composition of the layer and allowed the identification of the microstructure embedding limestone fragments surrounded by rounded pure calcium carbonates. Archaeologists interpreted that as a lime-burning kiln [6]. Indeed, most prehistoric plasters and mortars are not completely pure products and consist of very small amounts of burnt lime mixed with anthropogenic debris, soil, and sediment. However, the cave itself is composed of limestone [7] and some results could be affected by the effect of human fire activity on the natural stone [8].

Later, around 8500 BCE and supported by the development of pyrotechnology, plastering the floors and interior walls of houses with lime became common at most sites in central/southeastern Anatolia and the Levant [9]. Indeed, great strength and durability were the advantages of this new binder. Rare examples of gypsum plaster and marl have been documented in a few Neolithic sites in northern Israel [5], and later at Çatalhöyük (Turkey) [9]. Indeed, marl is not as strong nor durable as mortar, and gypsum used as mortar is relatively weak, with limited adhesive power, and it is strongly affected by water, determining a difficult application in wet climates.

Marl, gypsum, or lime plaster surfaces could be burnished using some stones both to prepare the surfaces for wall painting or simply to make them more resistant and durable [6]. In Anatolia, red paintings on the walls such as vegetal motifs, dots or geometrical patterns, and naturalistic decorations were common and discovered during the Neolithic at Boncuklu [10], Aşıklı Höyük [11] Çayönü [12], Can Hasan III [13], and Çatalhöyük [9]. This painting tradition continued in later phases in Anatolia and is found, amongst others, at Değirmentepe [14] during the Middle Chalcolithic and at Norşuntepe and Arslantepe in the Late Chalcolithic [15–17].

Regarding the history of lime plastering technique in the Near East, the attention has been only given to Neolithic settlements. Very few works have focused on later phases, and some evidence of lime application has been identified in Early Bronze Age and Iron Age. The present work aims at reconstructing the plastering techniques in the Late Chalcolithic at the site of Arslantepe by analyzing plaster samples, some of which have painted layers, from Temple C and two elite residences. Comparison of results from different cultural contexts can shed light on any continuity in technological procedures and knowledge that may have existed. At the same time, correlations between building functions and types of plaster can provide information on the organization of architecture and building construction at the site during the Late Chalcolithic period, a phase of increasing political and economic complexity.

The characterization of mortar samples has been carried out by means of PLM in thin sections and XRPD to investigate the mineral-petrographic composition, whereas SEM-EDS has been applied to define the micromorphological features and the chemical composition.

2. Archaeological Setting

Arslantepe is an artificial mound, called *tell* or *tepe*, located in the Malatya plain (Eastern Anatolia), 15 Km south-west of the river Euphrates (Figure 1). The site was continuously occupied from the 5th millennium BCE to the end of the 8th century BCE. Furthermore, some smaller remnants of a village from Roman times and a Medieval cemetery have been found [15–17].

Figure 1. Panoramic view of the site of Arslantepe where the investigated area is pointed out.

The Late Chalcolithic layers (3800–3100 BCE), corresponding to the VII (3800–3350 BC) and VI A (3350–3100 BCE) periods of the sequence within the site (Table 1), show evidence of social differentiation at the site, with households on the slopes and on the margins of the settlement and elite buildings located on the top of the ancient mound. An imposing tripartite temple (Temple C) of Mesopotamian type occupies the central–west area in which economic, religious, and administrative activities were performed [17]. In the temple, several ceramic objects have been found, supporting the hypothesis of ritual practices involved with the redistribution of goods by the elite [18].

Table 1. Arslantepe chronology.

General Chronological Sequence	Arslantepe Periods	Dendrochronology ^{14}C Calibrated Date
Late Roman and Byzantine Age	I	
Iron Age	II–III	1100–712 BCE
Late Bronze Age II	IV	1600–1200 BCE
Late Bronze Age I	VB	1750–1600 BCE
Middle Bronze	VA	2000–1750 BCE
Early Bronze Age III	VID	2500–2000 BCE
Early Bronze Age II	VIC	2750–2500 BCE
Early Bronze Age I	VI B2–B1	3100–2750 BCE
Late Chalcolithic 5 (Late Uruk)	VIA	3400–3100 BCE
Late Chalcolithic 3–4	VII	3800–3400 BCE
Late Chalcolithic 1–2	VIII	4250–3800 BCE

In addition, pictorial representations dating back to the Chalcolithic 3–4 period were found both in common houses in the north-east sector of the *tell*, and in traces, in the elite residential buildings in the upper portion of the hill.

3. Geological Setting

The site of Arslantepe is located in the south-easternmost part of the Malatya Plain in Eastern Anatolia [19]. A brief description of the geological framework is here reported to evaluate the provenance of materials used in plaster production.

The north-western part of the Plain is a mountainous area made of marbled limestone and basalt. To the south and south-west, Paleozoic soils form the Malatya Dağları with marbled limestones, schists and gneiss, and volcanic rocks are present. Calcareous and clayey outcrops are both in the southern and in the eastern and northeastern areas of the Plain. Cretaceous deposits of white limestone and clay also occur in the same area [20]. Arslantepe is located on a terrain made up of clays incorporated into micritic cement sandstones. In the northern and eastern portion of the Plain, Upper Miocene deposits composed of conglomerates, and Mount Gelincik Tepe of volcanic nature (trachytes and andesites) are also present. On the western side of the deposit a clay outcrop originated by the alteration of volcanic rocks is still used to produce bricks [19,20].

4. Materials and Methods

4.1. Materials

Four plaster fragments and a rock sample from Arslantepe were analyzed. These belonged to the walls of Temple C and of two elite residences, stratigraphically and on the basis of the materials therein, both dated to Period VII, Late Chalcolithic 3–4 (3900–3400 BCE) (Figure 2). In particular, sample A900 RM3 VII 2011/1135 is a hard, whitish, and smooth stone, found with a group of other analogous stones at the base of the southern wall of Temple C. Samples A900 M2 VII 2007/102 and A950 M1 VII 2017/1 include layers of both mortar and plaster; the first was sampled from the interior face of the eastern wall in the central room (A900) of Temple C, whereas the second one comes from the interior face of the northern wall in room A950, a small lateral storage room of Temple C. Sample D7 (3) A1469 M3 VII 2018/257 was sampled from the interior face of the southern wall in an elite residence and includes various layers of plaster, some of which have red paint. Finally, sample D6 (12) A1489 13a VII 2018/108 was taken from the interior face of the northern wall of another elite residence. Traces of red pigment were also present on this sample.

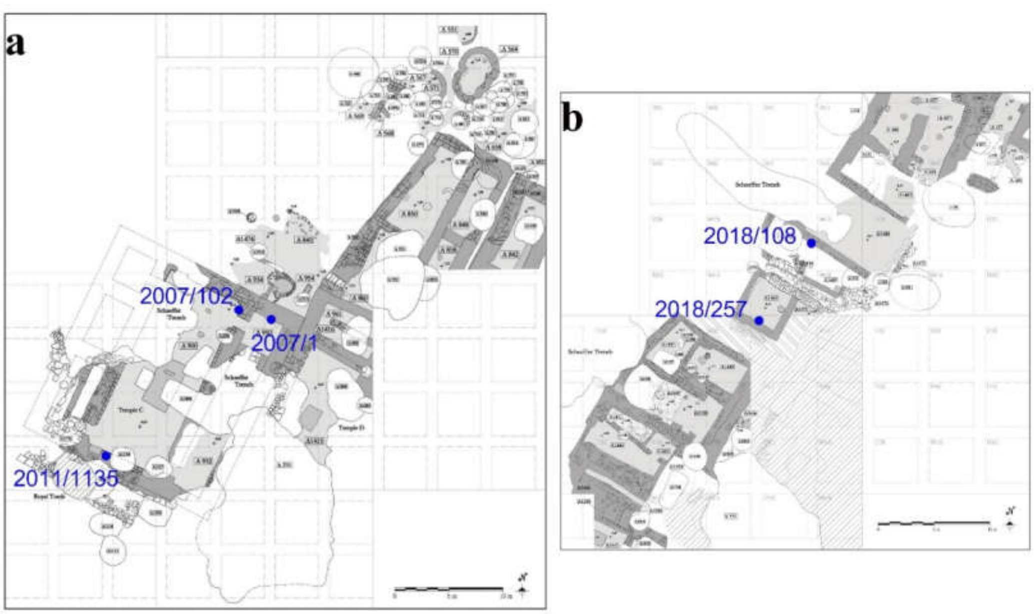

Figure 2. Maps with the localization of the sampling points: (**a**) Temple C; (**b**) elite residences.

4.2. Methods

Thin sections were prepared: each sample was embedded in epoxy resin (HARDROCK 554); once dried, the samples were cut parallel to the surface to characterize the single plaster layer or perpendicularly to the surface, and in the case of stratigraphic sections, to identify the sequence of layers.

Polarized light microscopy analysis via thin section was performed using a Zeiss D-7082 Oberkochen microscope (Department of Earth Sciences, Sapienza University, Rome, Italy) to determine the mineralogical and petrographic compositions of both binder and aggregate. The description of the binder consists in the observation of the structure and texture, mineralogical composition, and interactions with the aggregate also due to the possible addition of materials providing hydraulic characteristics. The description of the aggregate includes granulometry, size, classification, rounding, distribution, orientation, presence of reaction rims, mineralogical composition, and petrographic classification. In addition, the description of lumps and any other additives, binder/aggregate ratio (expressed as a volume percentage of the binder alone with respect to the aggregate only), porosity (shape of pores, their distribution and percentage by volume), and alteration products were also considered [21]. In the case of the decorated samples, a stratigraphic section was made to identify the sequence of layers, their mutual adhesion, the petrographic characteristics, and the pigments.

About 150–200 mg of sample was gently hand-crushed in an agate mortar and analyzed by a Siemens D5000 diffractometer (Department of Earth Sciences, Sapienza University of Rome, Italy) with CuKα radiation, 40 kV and 30 mA, in the range of 3°–60° 2θ, at a speed of 1°/min and 2 s/step, 1° diverging slide, slide receiver of 0.1 mm, and sled anti-scatter of 2°. Data processing, including semi-quantitative analysis based on the "Reference Intensity Ration Method," was performed using XPowderX© software.

Thin sections of samples were metalized with graphite, and SEM investigations were carried out using an electron microscope FEI Quanta 400 (Department of Earth Sciences, Sapienza University of Rome, Italy), operating at 20 kV, coupled with an X-ray energy dispersive spectroscopy system (EDS) to acquire qualitative chemical composition and the morphology of both binder and aggregate.

5. Results

5.1. Polarized Light Microscopy (PLM)

All the mortar samples show similar features that are described in detail in Table 2 and shown in Figure 3. They are mainly composed of an air-hardening, calcium-rich lime binder with an almost homogeneous appearance and micritic texture. Rare, sub-rounded quartz (SiO_2) crystals, with low sphericity and distinct grain size classes and very rare clinopyroxene were identified within the sections. Clay minerals were recognized thanks to the characteristic interference colour, and they correspond to product of feldspar alteration [22]. Some samples show lumps—uncalcined fragments related to the traditional lime production technology. Charcoal residues were also observed in some samples, probably incorporated within the mortar during firing. The binder/aggregate ratio is lower than 1:3. Pores are usually elongated, irregular in shape and distribution, without specific orientation and sometimes with recrystallized calcite ($CaCO_3$) inside. The stratigraphic sections highlight various layers with variable thickness and show some cracks. The outer layers appear finer in grain size and less rich in aggregates then the inner ones, suggesting that the plaster for the finishing layer was expressly made finer.

Finally, the rock fragment (sample A900 RM3 VII 2011/1135) shows an isotropic texture without phenocrystals. Calcite was mainly identified as micritic.

Table 2. Summary of the main microscopic features of plaster samples analyzed by PLM.

Sample	Binder	Aggregate	Porosity	Notes
A900 M2 VII 2007/102	air-hardening calcium-rich lime homogeneous micritic texture	quartz calcite clay minerals	irregular shape no orientation secondary calcite	B/A < 1:3
A950 M1 VII 2017/1	air-hardening calcium-rich lime homogeneous micritic texture	quartz calcite plagioclase clinopyroxene siliceous rock fragments clay minerals uncalcined lumps charcoal fragments	irregular shape no orientation secondary calcite	B/A < 1:3
D7(3) A1469 M3 VII 2018/257	air-hardening calcium-rich lime homogeneous micritic texture	sub-rounded quartz clay minerals pyroxene uncalcined lumps charcoal fragments	irregular shape no orientation secondary calcite	B/A < 1:3 Four layers
D6(12) A1489 13a VII 2018/108	air-hardening calcium-rich lime homogeneous micritic texture	sub-rounded quartz calcite clinopyroxene clay minerals uncalcined lumps	irregular shape no orientation secondary calcite	B/A < 1:3 The external part is less rich in aggregate than the internal one

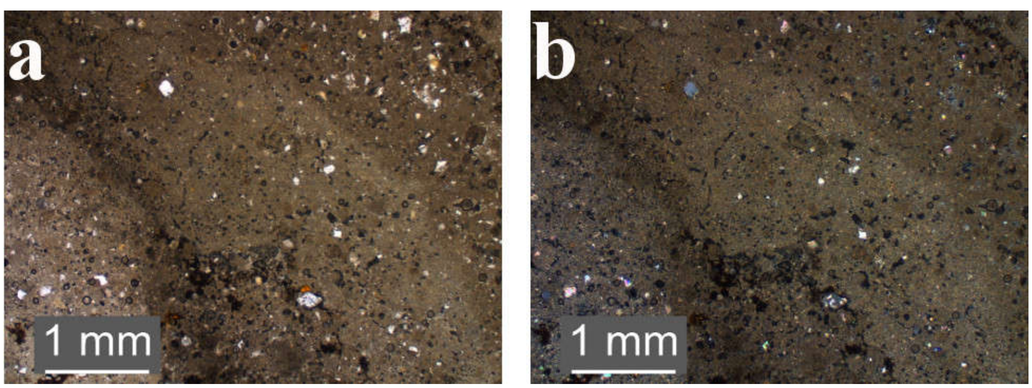

Figure 3. Thin section photographs of a representative sample (D7(3) A1469 M3 VII 2018/257) (**a**) at parallel polarized light, PPL, and (**b**) crossed polarized light, XPL, 2.5× magnification.

5.2. X-ray Powder Diffraction (XRPD)

XRPD analysis gave detailed information on the mineralogical compositions of the samples (Table 3 and Figure 4). In all the plaster samples, calcite was detected as abundant, with variable amounts of quartz and clay minerals (illite–montmorillonite, $K_{0.6}(H_3O)_{0.4}Al_{1.3}Mg_{0.3}Fe^{2+}_{0.1}Si_{3.5}O_{10}(OH)_2{\cdot}(H_2O)$–$Na_{0.2}Ca_{0.1}Al_2Si_4O_{10}(OH)_2(H_2O)_{10}$). In addition to the main minerals, dolomite ($CaMg(CO_3)_2$) (samples A900 M2 VII 2007/102 and D6(12) 13a VII 2018/108), clinopyroxene ($CaMgSi_2O_6$) (samples A950 M1 VII 2017/1 and D7(3) A1469 M3 VII 2018/257), and plagioclase ($Na_{0.5}Ca_{0.5}AlSi_3O_8$) (sample A950 M1 VII 2017/1) were identified in low amounts or traces.

Finally, the rock fragment (sample A900 RM3 VII 2011/1135) was mainly composed of calcite and traces of a ferriferous spinel.

Table 3. XRPD results showing the mineralogical assemblage of each mortar sample. ++++ very abundant 70–50%; +++ abundant 50–30%; ++ present 30–15%; + scarce 15–5%; tr. traces <5%. (Qtz = quartz, Cal = calcite, Pl = plagioclase, Ill–Mnt = illite − montmorillonite, Dol = dolomite, Cpx = clinopyroxene, Sp = spinel).

Sample	Qtz	Cal	Pl	Ill-Mnt	Dol	Cpx	Sp
A900 M2 VII 2007/102	tr	++++		++	tr		
A950 M1 VII 2017/1	++	++++	tr	++		tr	
D7(3) A1469 M3 VII 2018/257	++	++++		+		+	
D6(12) A1489 13a VII 2018/108	+	++++		++	tr		
A900 RM3 VII 2011/1135		++++					+

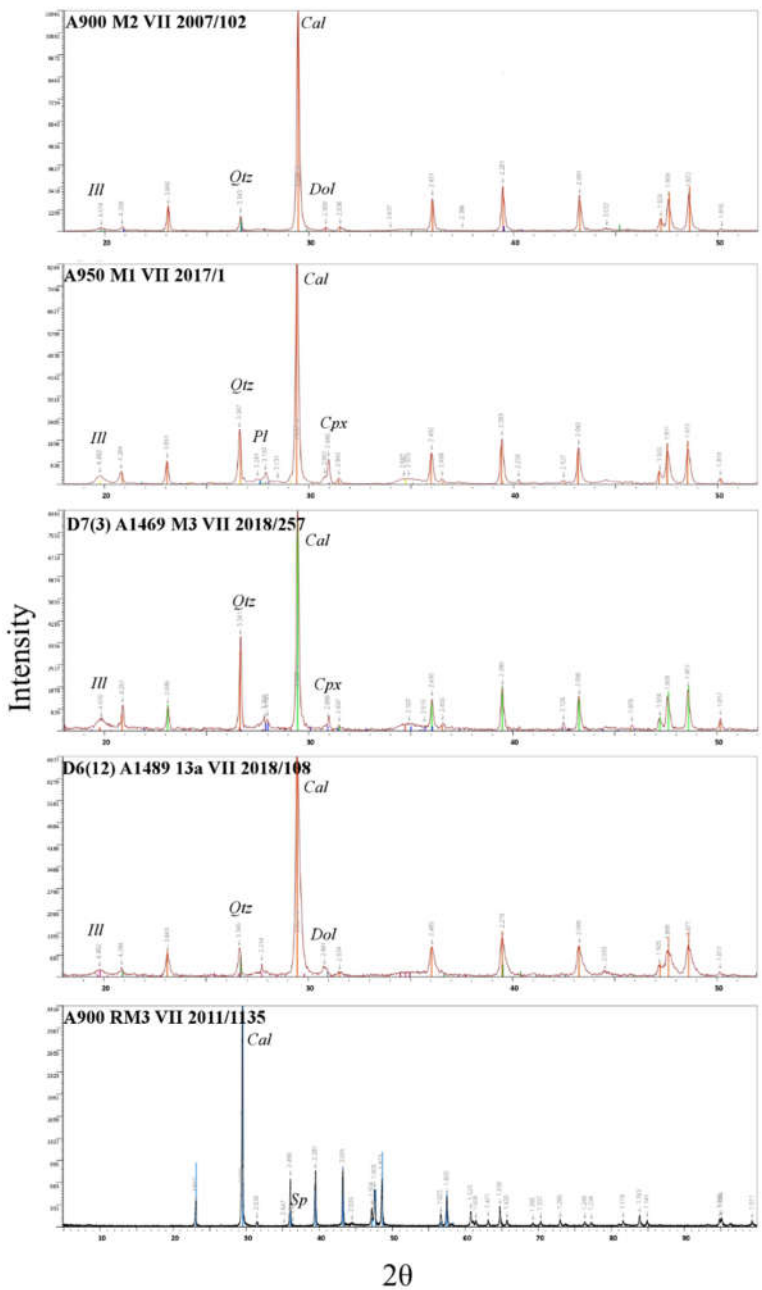

Figure 4. Diffraction patterns of the analyzed samples; main reflections assigned to the various minerals are marked (Cal: calcite, Qtz: quartz, Ill: illite, Dol: dolomite, Pl: plagioclase, Cpx: clinopyroxene, Sp: spinel).

5.3. Scanning Electron Microscopy Coupled with Energy Dispersive Spectroscopy (SEM-EDS)

SEM images of plaster samples confirmed some of the features observed by PLM analysis. The microstructure of the binder, the fine aggregate, and the elongated porosity are evident in the image below (Figure 6). EDS spectra acquired on the binder show that it is mainly composed of calcium (Ca) and silicon (Si) with traces of aluminum (Al) and magnesium (Mg) (Figure 5), whereas a detailed description of the aggregate highlights it is both siliceous and calcareous (Figure 6). Fragments of fossil shells (Figure 7a), bones (Figure 7b), and charcoal (Figure 7c) were also identified.

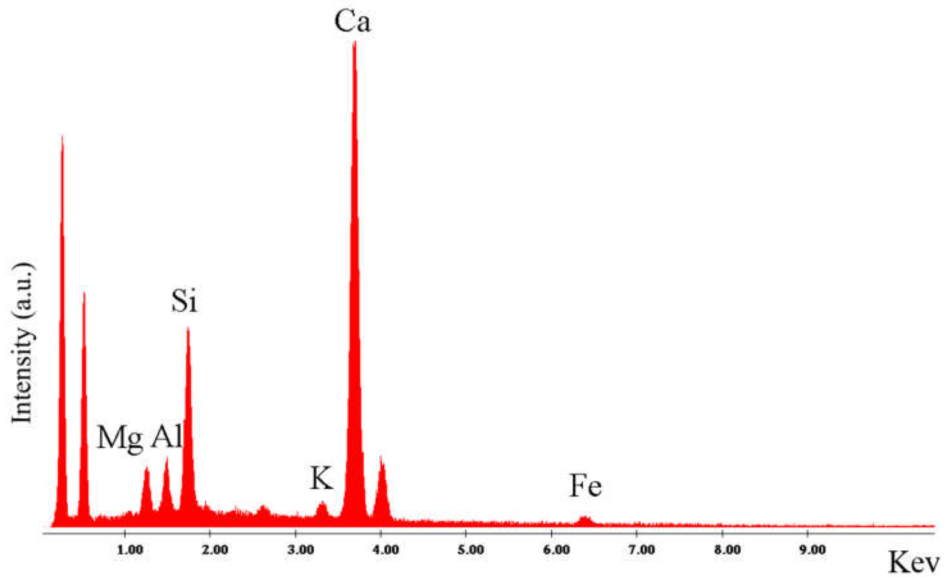

Figure 5. EDS spectrum of representative sample A900 M2 VII 2007/102 EDS showing the chemical composition of the binder.

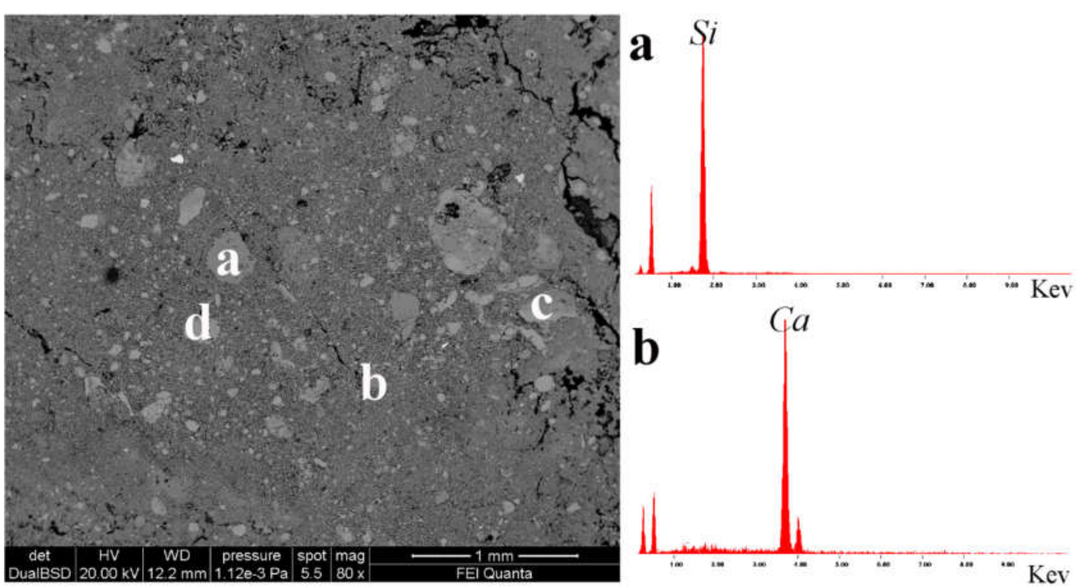

Figure 6. BSD image of sample A900 M2 VII 2007/102 with EDS spectra showing the chemical composition of different point analysis: (**a**) EDS spectrum of a siliceous inclusion (as in points a, c); (**b**) EDS spectrum of a calcareous inclusion (as in points b, d).

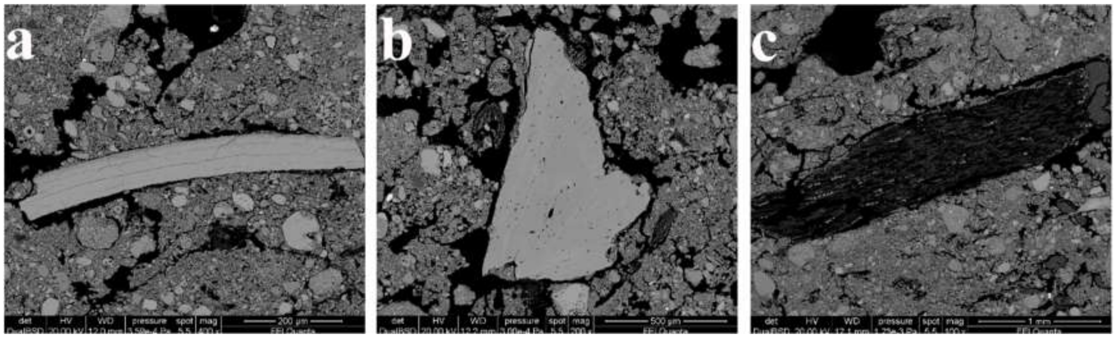

Figure 7. SEM images of inclusions: fragments of (**a**) fossil shell (sample A950 M1 VII 2017/1), (**b**) bone (sample D6(12) A1489 13a), and (**c**) charcoal (sample A950 M1 VII 2017/1).

In samples where both mortar and plaster layers were present, it was possible to highlight the difference in structure and composition between the two. The inner layer is characterized by the presence of numerous inclusions irregularly disposed in the binder, mainly composed of Ca and Si with minor amounts of Al and Mg. The outer layer, more homogeneous and with fewer inclusions, is mainly composed of Si with the presence of Ca, Al, and Mg. Traces of iron (Fe), potassium (K), and sodium (Na) were also identified in both layers (Figure 8).

SEM-EDS analysis also allowed an in-depth study on the paint layers. In particular, EDS spectra defined the presence of an iron-rich earth pigment as responsible for the red colour, mixed with quartz, plagioclase, and calcareous inclusions (Figure 9).

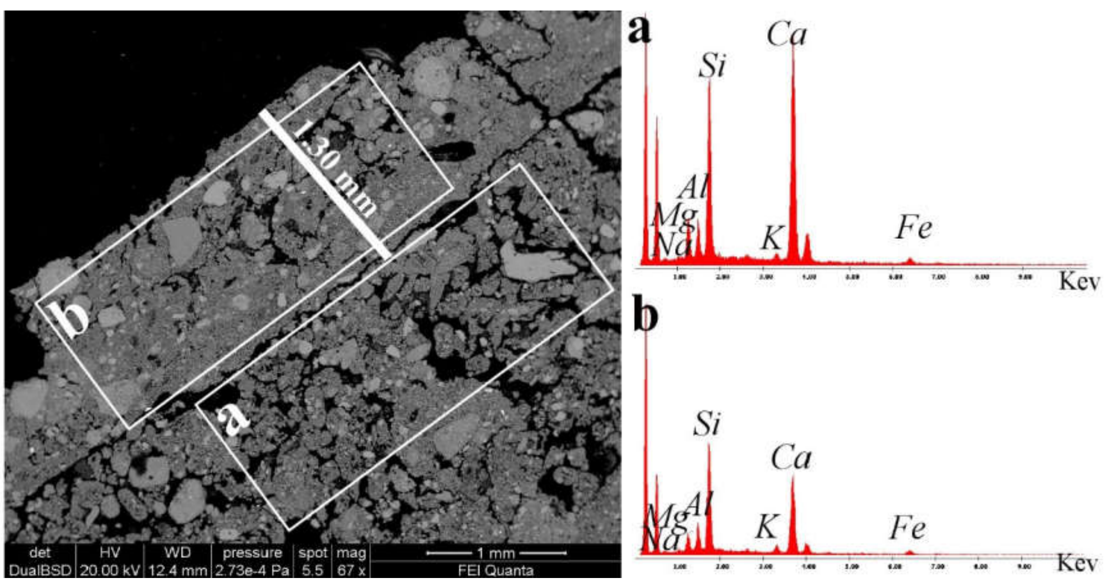

Figure 8. BSD image of sample D6(12) A1489 13a VII 2018/108 and EDS spectra showing the chemical composition of the two layers: (**a**) spectrum of the inner layer; (**b**) spectrum of the outer, more homogeneous layer.

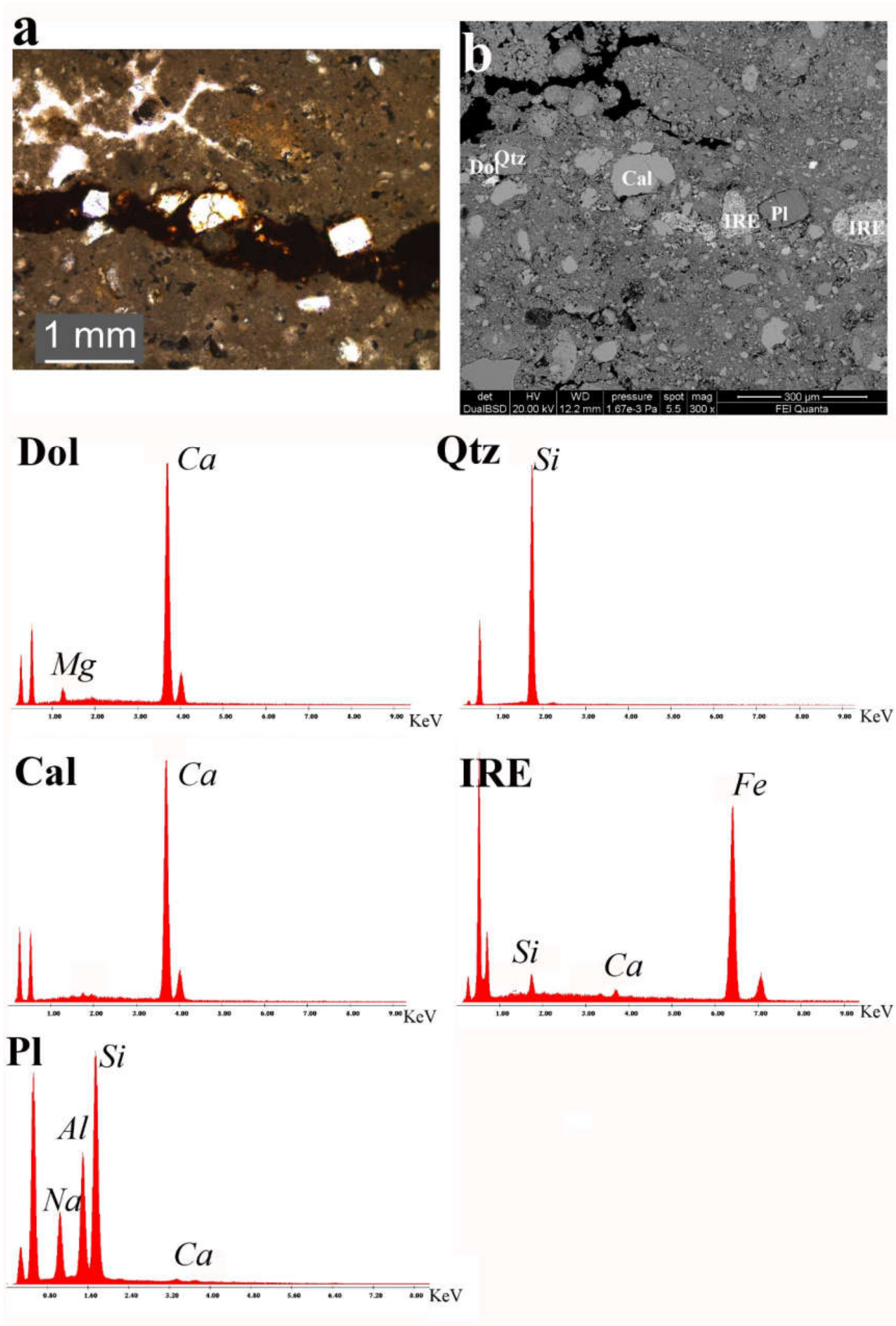

Figure 9. (**a**) PLM image at PPL and (**b**) BSE images of the paint layer (sample D7(3) A1469 M3 VII 2018/257), and EDS spectra of dolomite (Dol), quartz (Qtz), calcite (Cal), iron rich earth pigment (IRE), and plagioclase (Pl).

6. Discussion

The study of prehistoric mortar and plaster focuses on the investigation of raw materials, which normally faces many problems due to the impurities of the samples and the ageing factors related to the weathering processes [23]. It is also useful to understand the technological abilities of the prehistoric society. In this case it is even more interesting, since the Arslantepe period VII is a phase with emerging complexity, in which monumental architecture and craft specialization accompanied labor intensification and control, and resource management [17,24].

The analyses confirmed that the plasters are mainly characterized by an air-hardening, calcium-rich lime binder with a micritic (<5 μm) texture and residual clay minerals. The amount of aggregate is less abundant than the binder and mainly composed of fragments of carbonates, quartz, plagioclase and clinopyroxene. The presence of variable granulometric classes suggests a partial selection, i.e., a minimal levigation process of the aggregate. The low amount of aggregates determines a high binder/aggregate ratio which leads to a finished product which is more friable and prone to cracking [25].

The presence of both uncalcined particles and charcoal residues identified in the binder may be related to an improper quicklime extinguishing process which took place in rudimental kilns [5,26,27]. In particular, the co-occurrence of calcite and clay minerals, observed by XRPD analysis, further supports the hypothesis of marly limestone as starting raw material fired at temperature lower than 800–950 °C. The presence of lime lumps is very common in prehistoric lime and relates to a traditional technology for the production of lime. Causes may be found in strong inhomogeneities in the distribution of temperature in the kilns, lack of adequate sieving of the lime after slaking, possibly done a short time before the mortar was used, and a difficulty in the calcination of the stone because of its marly composition [28,29]. Fossil remains could be useful to identify the provenance of the samples with higher precision, contributing to the recognition of the geological deposits of origin of the raw materials.

The following layers were observed in the present study: a mortar mud layer, at the point of contact with the mud brick wall [18,20], compositionally similar to the bricks; then a plaster calcium-rich layer thicker than the previous one, with rare aggregates; and finally, a smoother and more refined white plaster, poorer in aggregate to finish the surface and/or to be painted. Despite the low binder/aggregate ratio, a good knowledge about the drying and finishing procedures of the material can be noticed. Indeed, it was possible to observe several layers of this fine finishing plaster, sometimes with colour traces. This sequence of layers indicates re-plastering processes taking place in time, a diffuse practice in pre-historic sites related to routine repairing and maintenance and sometimes coinciding with social/ritual events [30]. This re-plastering process might have happened monthly or yearly [9,31,32].

The pigment found on the plaster was identified as red ocher, a widespread natural inorganic pigment [33]. The co-occurrence of other inclusions such as quartz and calcareous rock fragments suggests the absence of purification of the initial raw material, or a low purification process. Since the pictorial surface was found to be resistant to both abrasion and water [16], it is possible to hypothesize that this strength could be related to the lime hardening that incorporates colour particles in a calcium carbonate matrix, or to the presence of an organic binder which retained the pigments as an agglutinant.

A comparison between the materials used in Temple C and those coming from the elite residences shows the structure and materials to be quite similar. In addition, the nature of both the binder and the aggregates of the samples from both contexts seems to suggest a local supply of the raw materials. Indeed, the Arslantepe soils are formed by calcareous clays, sand layers, and calcareous cement [18–20,34].

The rock fragment sample A900 RM3 VII 2011/1135 from the central room in Temple C was analyzed to evaluate whether it could be compatible with the possible raw material used in the production of mortars and plasters. The results allowed the identification of a limestone with micritic cement, which does not appear to be the starting material of lime production due to the total lack of a clayey portion, and instead present in the finished products. However, the mineralogical composition of the limestone supports the hypothesis of a local supply for this material. The smooth surface of the rock fragments found at the base of the wall suggests their probable use for plaster smoothing and polishing, a diffuse procedure already known much earlier at Çatalhöyük [9,35,36]. As starting rock for the production of lime, a marly limestone is therefore assumed, probably coming from the southern or western portion of the plain where limestone and clayey outcrops occur as a result of volcanic rock alteration [19,20,34]. Fragments of fossil shells were identified, probably belonging to the sands used as aggregate.

The incomplete lime burning suggests that it could be a casual, limited activity, and at that time it did not require a huge amount of fuel or an intensive labor [5]. This hypothesis is further supported by ethno-archaeological and anthropological studies, which have proved that lime plaster could be obtained with a small amount of fuel [1]. Hence, it might be expected that even at Arslantepe—where no lime kilns have been yet identified—shallow pits could have been used for lime burning, as was the case during the Neolithic in the southern Levant, at Kfar HaHoresh, and in the (el-Khirbe) Nesher-Ramla quarry [37,38]. According to Toffolo et al. [38], shallow pits could preserve heat more efficiently, stabilize the firing fuel, avoid accidental collapse and offer protection from wind (especially in the case of fuel composed of green wood).

7. Conclusions

A chemical and mineralogical approach (PLM, XRPD, SEM-EDS) was applied to identify the main components of plasters used at Arslantepe during the Late Chalcolithic 3–4. The results suggest that the production of building-related materials in Period VII included the use of lime, despite the lack of full control of the related chemical and physical mechanisms. The cooking of raw materials probably took place inside rudimentary kilns (or pits) that did not allow a complete decarbonation process, fundamental in the production of quicklime.

The decorations were made using natural pigments readily available in the surroundings of the site. Even without a chemically and physically defined true lime, Late Chalcolithic artisans could manage to obtain valuable products thanks to their expertise. An example is the preparation of a wall on which a red decoration was going to be painted, in a way that favored the adhesion of the pigment.

The white rock sample analyzed as a possible starting material was found to be limestone with micritic cement not related to the plaster itself, and the mortars and plasters analyzed were probably produced starting from a marly limestone. Both materials could be related to local outcrops.

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References

1. Rollefson, G. The uses of plaster at Neolithic Ain Ghazal , Jordan. *Archaeomaterials* **1990**, *4*, 33–54.
2. Wright, G.R.H. *Ancient Building Technology. Volume 2, materials*; Brill: Leiden, The Netherlands, 2005.
3. Baird, D. PinarbaŞi. *Anatol. Archaeol.* **2005**, *11*, 12–13.
4. Sagona, A.; Zimansky, P. *Ancient Turkey*; Routledge: London, UK, 2015.
5. Goren, Y.; Goldberg, P. Petrographic thin sections and the development of Neolithic plaster production in northern Israel. *J. F. Archaeol.* **1991**, *18*, 131–140.
6. Kingery, W.D.; Vandiver, P.B.; Prickett, M. The beginnings of pyrotechnology, part II: Production and use of lime and gypsum plaster in the Pre-Pottery Neolithic Near East. *J. F. Archaeol.* **1988**, *15*, 219–243.
7. Mercier, N.; Valladas, H.; Froget, L.; Joron, J.L.; Reyss, J.L.; Weiner, S.; Goldberg, P.; Meignen, L.; Bar-Yosef, O.; Belfer-Cohen, A.; et al. Hayonim Cave: A TL-based chronology for this Levantine Mousterian sequence. *J. Archaeol. Sci.* **2007**, *34*, 1064–1077, doi:10.1016/j.jas.2006.09.021.
8. Carran, D.; Hughes, J.; Leslie, A.; Kennedy, C. A short history of the use of lime as a building material beyond Europe and North America. *Int. J. Archit. Herit.* **2012**, *6*, 117–146, doi:10.1080/15583058.2010.511694.
9. Çamurcuoglu, D.S. *The Wall Paintings of Çatalhöyük (Turkey): Materials, Technologies and Artists*; UCL (University College London): London, UK, 2015.
10. Baird, D. The Boncuklu project : The origins of sedentism, cultivation and herding in central Anatolia. *Anatol. Archaeol.* **2009**, *14*, 11–12.
11. Esin, U.; Harmankaya, S. Aşıklı. In *Neolithic in Turkey: The Cradle of Civilization, New Discoveries*; Özdoğan, M., Başgelen, M., Eds.; Istanbul Arkeoloji ve Sanat Yayinlari: Istanbul, Turkey, 1999; pp. 115–132.
12. Özdoğan, A. Çayönü. In *The Neolithic in Turkey. New Excavations and New Research*; Özdoğan, M., Başgelen, N., Kuniholm, P., Eds.; Archaeology and Art Publications: Istanbul, Turkey, 2011; pp. 185–269.
13. French, D.H. Excavations at Can Hasan III 1969--1970. In *Papers in Economic Prehistory*; Higgs, E.S., Ed.; Cambridge University Press: Cambridge, UK, 1972; pp. 181–190.
14. Gurdil, B. *Architecture and Social Complexity in the Late Ubaid Period: A Study of the Bulk Environment of Değirmentepe in East Anatolia*; University of California: Los Angeles, CA, USA, 2005.
15. Hauptmann, H. Die Grabungen auf dem Norşuntepe. In *Keban Project 1972 Activities*; ODTU: Ankara, Turkey, 1976; pp. 21–78.
16. Frangipane, M.; Fazio, G. Le pitture murali del palazzo Tardo Calcolitico di Arslantepe-Malatya (Turchia). In *Sharing Conservation. Pluralità di Approcci alla Conservazione e al Restauro dei Manufatti Polimaterici*; Pandozy, S., Ed.; Edizioni Musei Vaticani: Fiumicino, Italy, 2014; pp. 529–540.
17. Balossi Restelli, F. *Arslantepe VII. The Development of a Ceremonial/Political Centre in the First Half of the Fourth Millennium BCE (Late Chalcolithic 3–4)*; Abor Sapientiae: Rome, Italy, 2019.
18. Liberotti, G. *Studi o Multidisciplinare Sugli Elementi Costruttivi e Archi Tettonici in Terra Cruda: Il Tardo Calcolitico nel sito Archeologico di Arslantepe (Malatya, Turchia)*; Università degli Studi dell'Aquila: L'Aquila, Italy, 2010.
19. Fragnoli, P.; Palmieri, A.M. Petrographic and geochemical investigations on the pottery production from Arslantepe—Malatya (Eastern Anatolia) from the fourth to the second Millennium BCE: Technological continuity, innovation and cultural change. *Archaeometry* **2017**, *59*, 612–641, doi:10.1111/arcm.12266.
20. Liberotti, G.; Rovero, L.; Stipo, G.; Tonietti, U. Mechanical investigation on adobe samples belonging to the archaeological site of Arslantepe (Malatya , Turkey). *CIAT2015* **2016**, *7*, 3656–3666.
21. Pecchioni, E.; Fratini, F.; Cantisani, E. *Atlas of the Ancient Mortars in Thin Section under Optical Microscope*; Nardini: Firenze, Italy, 2017.
22. MacKenzie, W.S.; Guilford, C. *Atlas of the Rock-Forming Minerals in Thin Section*; Routledge: London, UK, 2014.
23. Affonso, M.T.C. Identification of lime plasters. *Old Potter Alm.* **1996**, *4*, 1–6.
24. Frangipane, M. Riflessioni sui fondamenti delle 'economie politiche' nelle società protostatali del mondo 'mesopotamico.' In *'My Life is like the Summer Rose'. Maurizio Tosi e l'Archeologia Come Modo di Vivere*; Lamberg-Karlovsky, C.C., Genito, B., Cerasetti, B., Eds.; BAR Intern. Series 2690: Oxford, UK, 2014; pp. 251–262.
25. Thirumalini, S.; Ravi, R.; Sekar, S.K.; Nambirajan, M. Knowing from the past—Ingredients and technology of ancient mortar used in Vadakumnathan temple, Tirussur, Kerala, India. *J. Build. Eng.* **2015**, *4*, 101–112, doi:10.1016/j.jobe.2015.09.004.
26. Fusade, L.; Viles, H.; Wood, C.; Burns, C. The effect of wood ash on the properties and durability of lime mortar for repointing damp historic buildings. *Constr. Build. Mater.* **2019**, *212*, 500–513, doi:10.1016/j.conbuildmat.2019.03.326.
27. Pedraza, S.P.; Pineda, Y.; Gutiérrez, O. Influence of the unburned residues in fly ash additives on the mechanical properties of cement mortars. *Procedia Mater. Sci.* **2015**, *9*, 496–503, doi:10.1016/j.mspro.2015.05.022.
28. Bakolas, A.; Biscontin, G.; Moropoulou, A.; Zendri, E. Characterization of the lumps in the mortars of historic masonry. *Thermochim. Acta* **1995**, *269/270*, 809–816.

29. Pesce, G.L.; Ball, R.J.; Quarta, G.; Calcagnile, L. Identification, extraction, and preparation of reliable lime samples for 14C dating of plasters and mortars with the "pure lime lumps" technique. *Radiocarbon* **2012**, *54*, 933–942.
30. Siddall, R.; Çamurcuoglu, D. Plastering the Prehistory: Marl as a unique material to cover, maintain and decorate the Neolithic walls of Catalhöyük. In Proceedings of the 4th Historic Mortars Conference HMC2016, Santorini, Greece, 10–12 October 2016; Papayianni, I., Stefanidou, M., Pachta, V., Eds.; pp. 482–489.
31. Hodder, I. Çatalhöyük in the context of the Middle Eastern Neolithic. *Annu. Rev. Anthr.* **2007**, *36*, 105–120.
32. Matthews, W. Life-cycle and life-course of buildings. In *Çatalhöyük Perspectives: Reports from the 1995–1999 Seasons, Vol. 6*; Hodder, I., Ed.; McDonald Institute for Archaeological Research: Cambridge, UK, 2005; pp. 125–150.
33. Ospitali, F.; Smith, D.C.; Lorblanchet, M. Preliminary investigations by Raman microscopy of prehistoric pigments in the wall-painted cave at Roucadour, Quercy, France. *J. Raman Spectrosc.* **2006**, *37*, 1063–1071, doi:10.1002/jrs.
34. Fragnoli, P. Pottery production in pastoral communities: Archaeometric analysis on the LC3-EBA1 Handmade Burnished Ware from Arslantepe (in the Anatolian Upper Euphrates). *J. Archaeol. Sci. Rep.* **2018**, *18*, 318–332, doi:10.1016/j.jasrep.2018.01.029.
35. Wright, K.; Baysal, A. Cooking, crafts and curation: Ground stone artefacts. In *hanging Materialities at Çatalhöyük: Reports from the 1995–1999 Seasons*; Hodder, I., Ed.; British: London, UK 2005; pp. 307–324.
36. Wright, K.; Tsoraki-Chan, C.; Siddall, R. The ground stone technologies of Çatalhöyük. In *Substantive Technologies from Çatalhöyük: Reports from the 2000–2008 Seasons*; Hodder, I., Ed.; Cotsen Institute of Archaeology, University of California in Los Angeles: Los Angeles, CA, USA, 2013; pp. 355–408.
37. Goren, Y.; Goring-Morris, A.N. Early Pyrotechnology in the Near East : Experimental Lime-Plaster Production at the Pre-Pottery Neolithic B Site of. *Geoarchaeol. Int. J.* **2008**, *23*, 779–798, doi:10.1002/gea.20241.
38. Toffolo, M.B.; Ullman, M.; Caracuta, V.; Weiner, S.; Boaretto, E. A 10,400-year-old sunken lime kiln from the Early Pre-Pottery Neolithic B at the Nesher-Ramla quarry (el-Khirbe), Israel. *J. Archaeol. Sci. Rep.* **2017**, *14*, 353–364, doi:10.1016/j.jasrep.2017.06.014.