Materiali e documenti

Abu Tbeirah Excavations I. Area 1

Last Phase and Building A – Phase 1

edited by
Licia Romano and Franco D'Agostino

University Press
Sapienza Università Editrice

Collana Materiali e documenti 44

Abu Tbeirah Excavations I. Area 1

Last Phase and Building A – Phase 1

edited by

Licia Romano and Franco D'Agostino

SAPIENZA
UNIVERSITÀ EDITRICE
2019

STATE BOARD FOR ANTIQUITIES
& HERITAGE, IRAQ

Sapienza Università Editrice
Piazzale Aldo Moro 5 – 00185 Roma

www.editricesapienza.it
editrice.sapienza@uniroma1.it

Iscrizione Registro Operatori Comunicazione n. 11420

ISBN 978-88-9377-108-5

DOI 10.13.133-9788893771085

Pubblicato giugno 2019

In copertina: Modified satellite imagery of Abu Tbeirah site (courtesy of © DigitalGlobe, distributed by e-GEOSx). Logo: Iraqi SBAH

This book is dedicated to Amir Doshi,
whose friendship is the pillar
of our work at Abu Tbeirah

Table of Contents

CHAPTER 11

AREA 1 POTTERY – PART 2: CLAY, FABRICS AND FIRING TECHNOLOGY

CHAPTER 11
AREA 1 POTTERY – PART 2
CLAY, FABRICS AND FIRING TECHNOLOGY

Giulia Festa
Museo Storico della Fisica
e Centro Studi e Ricerche
"Enrico Fermi"
giulia.festa@centrofermi.it

Licia Romano
Sapienza University of Rome
Department "Institute
of Oriental Studies"
licia.romano@uniroma1.it

Vanessa Forte
University of Cambridge
McDonald Institute
for Archaeological Research
vanz.forte@gmail.com

11.1 Introduction

The study of Abu Tbeirah's pottery fabrics is still ongoing, evolving and enriching on the basis of the new results that each campaign is producing.[1] As a result of our collaboration with the Consortium of Italian Research Infrastructure for Cultural Heritage (CoIRICH), the first analyses on the pottery recovered during the 2012-2014 campaigns have undergone non destructive and non invasive bulk analyses, instead of focusing immediately on classical studies. We decided indeed to use Neutron Diffraction (ND)[2] and Neutron Resonance Capture Analysis (NRCA)[3] in order to have a general picture of the ceramic pastes in use in the excavated phases and a first confirmation of the autoptic subdivision.[4] This work thus presents a non-destructive and non-invasive neutron study of ancient Sumerian pottery fabrics from the 3rd mill. BC and aims at verifying the potentialities and limitation of the information derived from the ND and NRCA. Both techniques have shown their potential in the investigation of complex artefacts of cultural and artistic relevance.[5] Further analyses are already planned and will benefit both from the results of the neutron analyses and the now improved knowledge of Abu Tbeirah's pottery horizon.

The analyses on Abu Tbeirah's pottery were performed thanks to the support of CNR, within the CNR-STFC Agreement 2014-2020 (N. 3420), concerning collaboration in scientific research at the Italian Neutron Experimental Station (INES)[6] and the ISIS Spallation Neutron and Muon Source, located at the Rutherford Appleton Laboratory of the Science and Technology Facilities Council, on the Harwell Science and Innovation Campus in Oxfordshire, United Kingdom.

11.2 Mesopotamian Clay Sources [LR]

Chemical and mineralogical composition of ceramic material from other Mesopotamian sites constitute the necessary base of comparisons for Abu Tbeirah's pottery. Most of the archaeometric studies focused on pottery of the 3rd mill. BC from the Diyala and Hamrin area (Fig. 11.1).[7] Notwithstanding the complete description of the pottery from this area, contemporaneous data from the southernmost part of Mesopotamia are still very few, with the exception of the published research by Mynors and Al Kaissi,[8] and few

[1] Festa is author of §§11.4.1-3; Romano is author of §§11.1-2; Forte is author of §§11.3. §§11.3.1 is common work of Romano and Forte while §§ 11.4.4 and 11.5 are common work of Festa and Romano.

[2] Windsor 1981; Festa *et al.* 2011a; 2011b.

[3] Postma - Schillebeeck 2005; Festa *et al.* 2011a; 2011b.

[4] The pottery analyzed come from Area 1 and other ED III/Akk. contexts.

[5] Festa *et al.* 2008; 2009; Pietropaolo *et al.* 2011; Festa *et al.* 2013; 2015; 2016; Postma - Schillebeeckx 2005. Few are the studies on archaeological pottery in general: Kockelmann - Kirfel 2001; 2006; Imberti *et al.* 2008.

[6] Imberti *et al.* 2008.

[7] Thuesen *et al.* 1982; Mynors 1983; Méry - Schneider 1996; Gibson (ed.) 1990 (also with data from the eastern Farukhabad).

[8] Mynors 1983 (including data from the northern site of al-Usiyeh); Mynors - Al Kaissi 1987. The sample analyzed are less than 40 for the following sites Tell-ed-Der, Jemdet

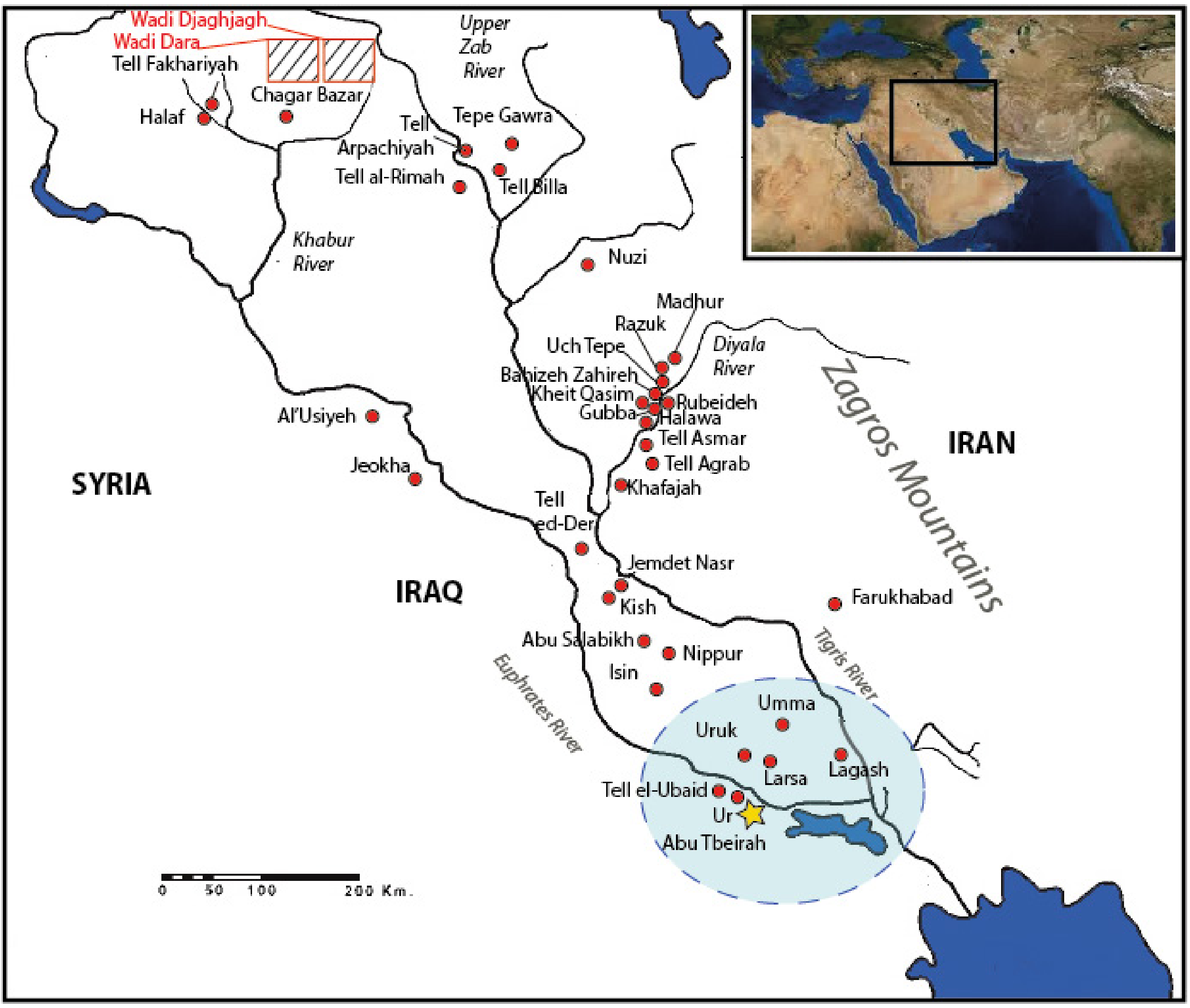

Fig. 11.1 Map with the indications of sites from which pottery samples have been analyzed (Festa *et al.* 2019: Fig. 1).

sparse data presented as comparisons from other contexts.[9] Data from the literature show in general a complete homogeneity of the ceramic pastes used in Mesopotamia. This chemical and mineralogical uniformity of Mesopotamian ceramic pastes' composition,[10] demonstrated by the quoted researches, is due to the nature of the alluvial plain (see § 3).[11] Abu Tbeirah's pottery production was based on the same natural secondary clay from the Mesopotamian alluvial plain and thus dissimilar results from those obtained by previous researches were not expected. Nevertheless, the limited amount of data on 3rd mill. BC southern Mesopotamian pottery make the archaeometric analyses on Abu Tbeirah's pottery compelling, in order to verify and support previous results, clarifying in the light of the new archaeometric analyses the Sumerian potter's technological choices.

Nasr, Nippur, Kish, Abu Salabikh, Fara, Tell al-Wilayah, Uruk, Larsa, Lagash, Obeid, and Ur.

[9] Méry - Schneider 1996.

[10] On the composition of southern Mesopotamian clay see Festa *et al.* 2019.

[11] Armstrong - Gasche 2014: 77.

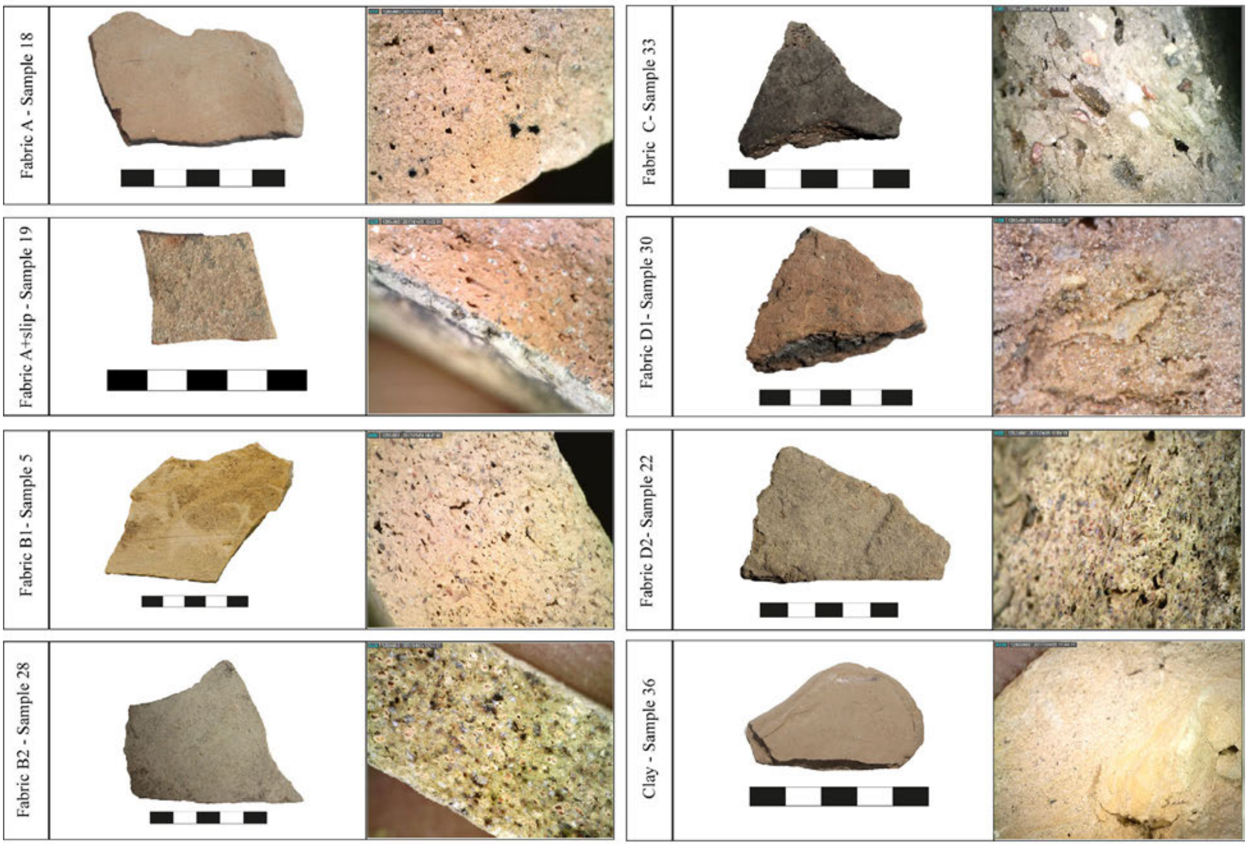

Fig. 11.2 A selection of pottery and clay samples analysed, divided following the fabric classification. Photo of the fragments' sections (not in scale) are acquired through a portable digital microscope (®Dinolite). (Festa *et al.* 2019: Fig. 2).

11.3 Macroscopic Classification of Ceramic Pastes [VF]

The first autoptic analysis allowed us to distinguish four main macroscopic groups (Fig. 11.2).[12]

- **Fabric A**: Fine grained paste with low porosity featured by planar voids. Red-orange colour of paste. Firing mainly in oxidised atmosphere. Sometimes covered with clearer slip (**Fabric A+slip**).
- **Fabric B_1**: Fine grained paste with a low porosity featured by planar voids. Yellow colour of paste. Firing mainly in oxidised atmosphere.
- **Fabric B_2:** Fine grained paste with a low porosity featured by planar voids. Yellow colour of paste with orange inclusions. Firing mainly in oxidised atmosphere.
- **Fabric C:** Coarse grained paste with abundant sedimentary fragments and angular inclusions. High porosity compared to other groups. Firing mainly in oxidised atmosphere.
- **Fabric D_1:** Fine grained paste with abundant straw. Red-orange colour of paste. Firing mainly in oxidised atmosphere.
- **Fabric D_2:** Fine grained paste with abundant straw. Yellow colour of paste with orange inclusions. Firing mainly in oxidised atmosphere.

[12] The different fabrics were distinguished also through the use of a portable microscope (®Dinolite AD 7013MZT; Druc 2015) It is still not clear if the samples 3 and 34, that have larger and very visible inclusions, should be considered as a separate fabric (and thus the result of the intentional adding of sand): this kind of fabric is quite rare and no association with specific shapes was detected up to now. Similarly, the evidence of a reducing firing atmosphere or of a not complete oxidation of the organic material seems to be quite casual and due to the not always complete control of the firing process by Abu Tbeirah potters.

11.3.1 Selected Fragments (VF - LR)

On the basis of the autoptic subdivision of the fabrics, 36 pottery shards were selected,[13] together with one clay sample, coming from the canal running east of Abu Tbeirah.

Sample n. 1 (AbT.13.140.17)
Fabric: B2.
Clay: Outer and inner colour: 2.5Y 8/3 (pale brown); fabric colour: 2.5Y 8/3 (pale brown) to 5Y 6/3 (pale olive).
Thickness (average): 0.6 cm.
Description: jar fragment with red slip? (5YR 6/3 light reddish brown) traces on the internal and external (not uniform) surface.

Sample n. 2 (AbT.12.109.24)
Fabric: A+Slip/Self-Slip.
Clay: Outer colour: 10YR 8/2 (very pale brown); inner colour: 7.5YR 7/3 (pink); fabric colour: 5YR 6/6 (reddish yellow).
Thickness (average): 0.7 cm.
Description: jar fragment with larger and more frequent than usual sand inclusions.

Sample n. 3 (AbT.12.97.47)
Fabric: A+Slip/Self-Slip.
Clay: outer colour: 7.5YR 8/3 (pink); inner colour: 5YR 6/4 (light reddish brown); fabric colour: 5YR 6/4 (light reddish brown) to 10YR 7/4 (very pale brown).
Thickness (average): 0.9 cm.
Description: jar fragment.

Sample n. 4 (AbT.12.84.30)
Fabric: D2.
Clay: outer, inner and fabric colour: 2.5YR 8/2 (pale brown).
Thickness (average): 1.9 cm.
Description: wall fragment of a big container with bitumen traces on the outside.

[13] In the tables and pictures some of the samples numbers are repeated because the analysis was performed on two different pieces of the same vessel.

Sample n. 5 (AbT.12.86.2)
Fabric: B1.
Clay: outer colour: 2.5Y 8/2 (pale brown); inner colour: 10YR 8/4 (very pale brown); fabric colour: 7.5YR 7/4 (pink).
Thickness (average): 0.7 cm.
Description: jar fragment.

Sample n. 6 (AbT.12.71.16)
Fabric: A.
Clay: outer, inner and fabric colour: 5YR 5/6 (yellowish red).
Thickness (average): 0.6 cm.
Description: conical bowl fragment.

Sample n. 7 (AbT.12.71.54)
Fabric: A (incomplete oxidation of the organic material).
Clay: outer colour: 2.5Y 8/3 (pale brown); inner colour: 10YR 6/4 (light yellowish brown); fabric colour: 2.5Y 5/1 (grey).
Thickness (average): 0.8 cm.
Description: jar fragment/cooking pot fragment.

Sample n. 8 (AbT.12.71.54)
Fabric: A (incomplete oxidation of the organic material).
Clay: outer colour: 2.5Y 8/3 (pale brown); inner colour: 10YR 6/4 (light yellowish brown); fabric colour: 2.5Y 5/1 (grey).
Thickness (average): 0.6 cm.
Description: jar fragment/cooking pot fragment (use traces on the exterior).

Sample n. 9 (AbT.12.96.46)
Fabric: A+Slip/Self-Slip.
Clay: outer colour: 10YR 8/3 (very pale brown); inner colour: 7.5YR 6/4 (light brown); fabric colour: 5YR 7/6 (reddish yellow).
Thickness (average): 0.7 cm.
Description: jar fragment.

Sample n. 10 (AbT.13.567.1)
Fabric: B1.
Clay: outer and inner colour: 2.5Y 8/2 (pale brown); fabric colour: 10YR 6/4 (light yellowish brown).
Thickness (average): 0.7 cm.
Description: conical bowl with big intrusive inclusion.

Sample n. 11 (AbT.12.83.8)
Fabric: B2.
Clay: outer colour: 5Y 8/2 (pale yellow); inner colour: 2.5Y 67/4 (pale brown); fabric colour: 5Y 7/1 (light grey).
Thickness (average): 1.2 cm.
Description: straight wall of a coarse vessel.

Sample n. 12 (AbT.12.96.47)
Fabric: A.
Clay: outer, inner and fabric colour: 5YR 5/4 (reddish brown).
Thickness (average): 1 cm.
Description: conical bowl fragment.

Sample n. 13 (AbT.12.56.34)
Fabric: B2 + Reserved-slip like effect.
Clay: outer, inner and fabric colour: 5Y 8/2 (pale yellow), fabric slightly darker.
Thickness (average): 0.5 cm.
Description: jar fragment.

Sample n. 14 (AbT.12.109.25)
Fabric: A.
Clay: outer colour: 10YR 8/2 (very pale brown); inner colour: 7.5YR 6/4 (light brown); fabric colour: 5YR 7/6 (reddish yellow).
Thickness (average): 0.8 cm.
Description: jar fragment.

Sample n. 15 (AbT.12.85.16)
Fabric: A+Slip/Self-Slip.
Clay: outer and inner colour: 2.5Y 8/2 (pale brown); fabric colour: 5YR 6/6 (reddish yellow).
Thickness (average): 0.5 cm.
Description: jar fragment.

Sample n.16
Fabric: clay.
Thickness (average): 1 cm.
Description: clay from Abu Tbeirah's canal.

Sample n. 17 (AbT.13.140.16)
Fabric: A+Slip/Self-Slip.
Clay: outer colour: 7.5YR 8/3 (pink); inner and fabric colour: 5YR 7/4 (pink).
Thickness (average): 0.7 cm.
Description: jar fragment.

Sample n. 18 (Area 1)
Fabric: A.
Clay: outer, inner and fabric colour: 5YR 6/4 (light reddish brown).
Thickness: 0.7 cm.
Description: conical bowl.

Sample n. 19 (Area 1)
Fabric: A+Slip/Self-Slip.

Clay: outer and inner colour: 2.5Y 7/2 (light grey); fabric colour: 2.5YR 6/8 (light red).
Thickness (average): 0.6 cm.
Description: jar fragment.

Sample n. 20 (Area 1)
Fabric: B1.
Clay: outer colour: 2.5Y 8/2 (pale brown); inner colour: 7.5YR 8/4 (pink); fabric colour: 7.5YR 6/4 (light brown).
Thickness (average): 0.7 cm.
Description: jar/bowl fragment.

Sample n. 21 (Area 1)
Fabric: B2.
Clay: outer, inner and fabric colour: 2.5Y 8/3 (pale brown).
Thickness (average): 0.7 cm.
Description: jar fragment.

Sample n. 22 (Area 1)
Fabric: D2.
Colour: outer, inner and fabric colour: 2.5Y 7/3 (pale brown).
Thickness (average): 1.7 cm.
Description: big vat fragment.

Sample n. 23 (Area 1)
Fabric: A.
Colour: outer, inner and fabric colour: 5YR 6/6 (reddish yellow).
Thickness (average): 1 cm.
Description: big bowl wall fragment.

Sample n. 24 (Area 1)
Fabric: B2 (incomplete oxidation of the organic matter)
Clay: outer, inner and fabric colour: 10YR 5/1 (grey).
Thickness (average): 0.7 cm.
Description: jar fragment.

Sample n. 25 (Area 1)
Fabric: B2.
Clay: outer colour: 5Y 8/2 (pale yellow); inner and fabric colour: 10YR 8/3 (very pale brown).
Thickness (average): 0.7 cm.
Description: jar fragment.

Sample n. 26 (Area 1)
Fabric: B2.
Clay: outer, inner and fabric colour: 5Y 8/3 (pale yellow).
Thickness (average): 0.6 cm.
Description: jar fragment.

Sample n. 27 (Area 1)
Fabric: B1.
Clay: outer colour: 2.5Y 8/2 (pale brown); inner colour: 7.5YR 8/2 (pinkish white); fabric colour: 5YR 7/4 (light brown).
Thickness (average): 0.7 cm.
Description: jar fragment.

Sample n. 28 (Area 1)
Fabric: B2.
Clay: outer, inner and fabric colour: 5Y 7/1 (light grey).
Thickness (average): 0.6 cm.
Description: jar fragment.

Sample n. 29 (Area 1)
Fabric: D2.
Clay: outer, inner and fabric colour: 2.5Y 8/2 (pale brown), with reddish areas.
Thickness (average): 2.3 cm.
Description: big coarse vat rim fragment.

Sample n. 30 (Area 1)
Fabric: D1 (incomplete oxidation of the organic matter).
Clay: outer, inner and fabric colour: 7.5YR 7/6 (reddish yellow).
Thickness (average): 1.7 cm.
Description: big vat fragment.

Sample n. 31 (Area 1)
Fabric: A? (reducing atmosphere and use traces?).
Clay: outer and inner colour: 10YR 5/1 (grey); fabric colour: 7.5YR 2.5/1 (black).
Thickness (average): 0.9 cm.
Description: closed vessel fragment.

Sample n. 32 (Area 1)
Fabric: A (incomplete oxidation of the organic material).
Clay: outer and inner colour: 10YR 6/3 (pale brown); fabric colour: 10YR 4/1 (dark grey).
Thickness (average): 1 cm.
Description: cooking pot?

Sample n. 33 (Area 1)
Fabric: C (incomplete oxidation of the organic matter).
Clay: outer, inner and fabric colour: 10YR 5/2 (greyish brown).
Thickness (average): 0.8 cm.
Description: cooking pot?

Sample n. 34 (AbT.14.261.2)
Fabric: A (incomplete oxidation of the organic matter).
Clay: outer colour: 10YR 8/3 (very pale brown); inner colour: 7.5YR 6/6 (reddish yellow); fabric colour: 7.5YR 6/4 (light brown).
Thickness (average): 0.7 cm.
Description: cooking pot?

Sample n. 35 (US 152)
Fabric: B2 (incomplete oxidation of the organic matter).
Clay: outer, inner and fabric colour: 2.5Y 5/1 (grey).
Thickness (average): 0.8 cm.
Description.: big burnished bowl.

Sample n. 36 (US 195)
Fabric: B2.
Clay: outer, inner and fabric colour: 5Y 8/3 (pale yellow).
Thickness (average): 0.8 cm.
Description: jar fragment.

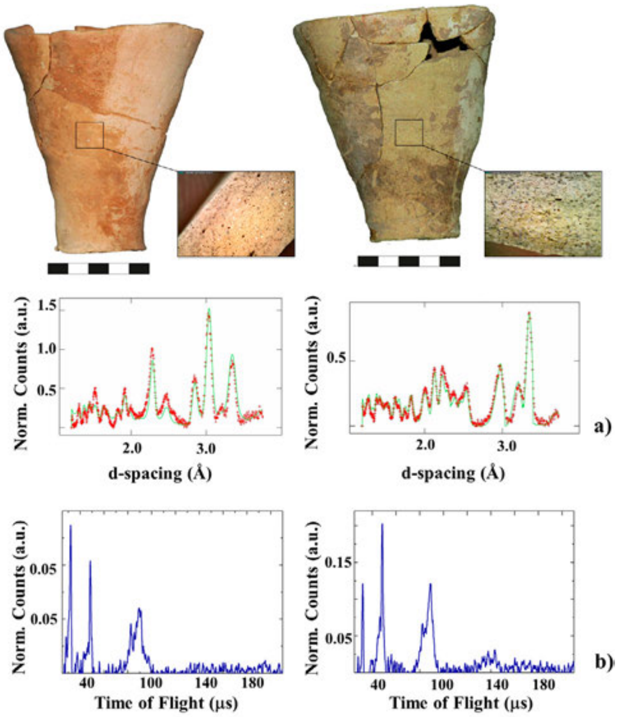

Fig. 11.3 Results of the analyses via neutron techniques on Fabric A (left) and B2 (right). a) Diffraction spectra of two vases are reported (normalized number of counts as a function of the d-spacing). Best fit of the data is also shown in green colour; b) Neutron Resonance Capture Analysis spectra (normalized number of counts as a function of resonant-neutron time of flight) are reported (Festa *et al.* 2019: Fig. 3).

11.4 Neutron Investigations: Results and Discussion [GF]

The samples were analysed through a simultaneous and integrated Time Of Flight (TOF) Neutron Diffraction (ND) and Neutron Resonance Capture Analysis (NRCA),[14] for the characterization of the compositional and microstructural features of Abu Tbeirah's pottery (Fig. 11.3).

11.4.1 Neutron Diffraction [GF]

Measurements were performed at the INES beamline, Rutherford Appleton Laboratory, Oxfordshire. INES[15] is equipped with a general-purpose neutron diffractometer that was built for accommodating large non-standard sample volume in the neutron beam, thus allowing archaeometric measurements. Thanks to the high penetration power of neutrons in ceramics, measurements allow us to determine bulk properties of the samples. Neutron Diffraction on pottery samples gives, thus, information about the crystal phases and compounds formed in the pottery during firing and allows to indirectly trace back the reached firing temperature.

The firing behaviour of clay minerals and inclusions was widely studied and in what follows the discussion will be limited to the transformations highlighted by the analyses. Quartz, the most abundant inclusion in most ceramic bodies, does not undergo significant variations at low temperatures, except for "alpha"-"beta" transition at 573 °C.[16] Although in appropriate conditions primary clay minerals may survive, calcite in the clays is subjected to two main thermic processes: clay de-hydroxylation (ca. 400-600°C) and de-carbonation of the calcareous materials (750-850°C). Higher firing temperatures lead the calcite decomposition products to react with fired clays (montomorillonite and illite/muscovite[17] in Abu Tbeirah samples) and form new calcium silicate phases such as gehlenite, anorthite or diopside (wollastonite). Starting at approximately 850°C, calcite reacts with clay minerals (Al_2O_3, SiO_2 and MgO) forming Ca-silicates as pyroxene (Al-rich diopside, T> 900°C), newly forming Ca-plagioclase feldspar (albite, anorthite over 950°C) or gehlenite (T> 800°C).

According to these trends and using illite/muscovite and diopside as track markers of firing processes, two groups of samples were distinguished in the analysis carried out on Abu Tbeirah material: illite/muscovite is present in the first half of the histogram (Tab. 11.1 and Fig. 11.4), while it disappears in the second half where the newly formed diopside (new calcium silicate phases) is present. On the contrary, quartz shows a constant trend: since quartz grains do not undergo any detectable morphological and chemical transformation until reaching a temperature of 1050°C, this indicates that firing temperature of all pottery under study was below this value. Finally, the trend of calcite reflects the complete

[14] For a detailed report on the analyses carried out see Nardini *et al.* 2018; Festa *et al.* 2019.

[15] Imberti *et al.* 2008.

[16] Rice 2005: 96-97.

[17] Illite is a layered alumino-silicate chemically similar to muscovite and thus not distinguishable in diffraction analyses.

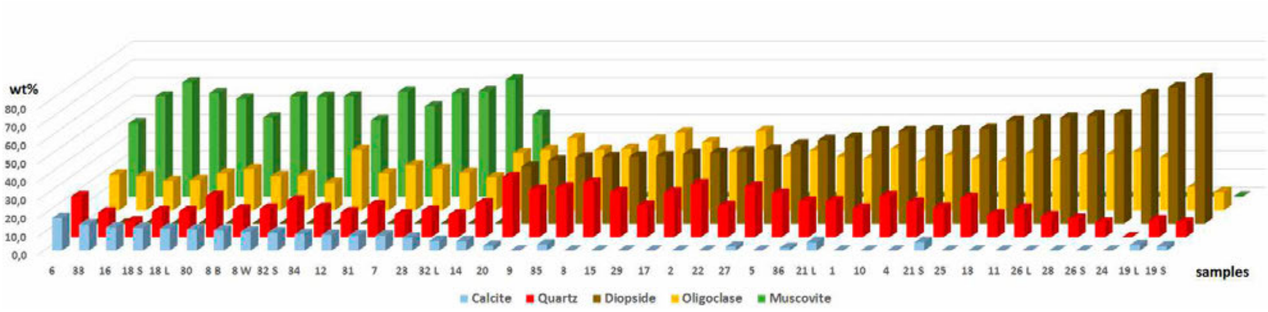

Fig. 11.4 Histogram based on the neutron diffraction quantitative results. The x-axis reports the number of the sample as function of the increasing percentage of diopside. The y-axis reports the weight percentage of the detected crystal phase, z-axis reports the detected phases (yellow - oligoclase, green - muscovite, blue - calcite, orange - quartz, brown - diopside). (Festa *et al.* 2019: Fig. 4).

transformation into carbonate of the original calcium oxide, indicating calcite of secondary formation over 850° C. Moreover, Fig. 11.4 shows that small amounts of calcite increase at high T, to indicate the calcite reaction and so the formation of new Ca-silicates. Taking into account the T stability of the mentioned mineral, the range of firing for the set of samples is between 800-1000°C.

11.4.2 Neutron Resonance Capture Analysis [GF]

Neutron Resonance Capture Analysis (NRCA) is an experimental technique based on the absorption by a nucleus of a neutron with energies in excess of few eV and up to hundreds of keV. This capture events are detected via the prompt gamma-cascade by which the newly formed nucleus de-excites: this resonance energy is characteristic of the element and isotope that produce the gamma. NRCA peak spectra are labelled by means of a comparison with the resonance energies related to (n, γ) processes provided by tables of neutron resonances.[18] The detected main peaks are reported in Tab. 11.2.

The NRCA technique was used to detect main isotopes ^{39}K, ^{56}Fe, ^{55}Mn, ^{23}Na and ^{35}Cl on the entire set of samples. The normalization of NRCA spectra was carried out to perform comparison between different samples through statistical methods; peaks intensities are normalized with respect to the acquisition parameters (incident neutron flux and integrated current), the (n, γ) cross section for each identified isotope and the thickness of each samples. The normalization is carried out through the ®Origin software and results are reported in Tab. 11.2. It should be highlighted that the values presented in Tab. 11.2 are not the quantities of the elements of the sample: they provide a relative indication to compare the content of the respective elements in the various samples through a statistical approach.

11.4.3 Classification of the Samples on the Basis of the ND and NRCA [GF]

The ND and NRCA data results (both phase and elemental analyses) are examined through descriptive statistical approach carrying out correlation analysis (in order to observe the homogeneity of the samples) and Principal Component Analysis (PCA),[19] a method of multivariate statistics that represents a powerful way to classify samples into various possible categories and allows to determine which variables actually contribute to the variation seen in a given data matrix. This technique has been successfully applied in archaeological contexts since the 1970's.[20]

Fig. 11.5 shows six groups of samples with specific tendencies according to T increases. Samples 31 and 5 are found in a border-line position between their group of memberships, described by the diopside and muscovite respectively. Diopside and muscovite appear in opposite position as expected. Samples number 19S+19L and 24 are

[18] Mughabghab *et al.* 1981.

[19] Jolliffe 1986; Husson *et al.* 2009.

[20] See, *e.g.*, Baxter 1994; Husson *et al.* 2009; Scatigno *et al.* 2017.

Sample	Fabric	Calcite	Quartz	Diopside	Oligoclase	Muscovite
1	B2	-	15.7	50.5	33.8	-
2	A	-	29.1	38.9	32.0	-
3	A	-	29.8	36.5	33.7	-
4	D2	-	18.9	51.1	30.0	-
5	B1	-	23.8	43.4	32.8	-
6	A	17.9	22.4	-	19.5	40.2
7	A	8.3	12.4	-	22.6	56.7
8 B	A	11.1	15.2	-	18.7	55.0
8 W	A	10.5	15.7	-	19.0	54.8
9	A	-	26.0	34.6	39.4	-
10	B1	-	22.4	50.7	26.9	-
11	B2	-	15.7	57.1	27.2	-
12	A	8.7	13.7	-	20.1	57.5
13	B2	-	12.4	56.5	31.1	-
14	A	5.3	18.5	-	31.3	44.9
15	A	-	24.6	36.8	38.6	-
16	Modern clay	12.7	8.7	-	16.0	62.6
17	A	-	24.4	38.3	37.3	-
18 L	A	12.1	14.2	-	20.1	53.6
18 S	A	12.5	14.2	-	16.5	56.8
19 L	A	3.2	9.4	74.7	12.7	-
19 S	A	2.4	8.0	79.7	9.9	-
20	B1	2.8	33.0	31.3	32.9	-
21 L	B2	4.7	19.8	47.1	28.4	-
21 S	B2	4.7	16.1	51.2	28.0	-
22	D2	-	17.1	39.5	43.4	-
23	A	7.2	14.5	-	20.5	57.8
24	B2	-	-	71.0	29.0	-
25	B2	-	21.4	51.9	26.7	-
26 L	B2	-	11.6	58.1	30.3	-
26 S	B2	-	8.0	59.8	32.2	-
27	B1	2.3	27.6	40.5	29.6	-
28	B2	-	10.1	59.3	30.6	-
29	D2	-	17.3	37.0	42.5	-
30	D1	11.6	22.5	-	22.5	43.4
31	A	8.3	17.6	-	24.6	49.5
32 L	A	5.5	12.4	-	18.0	64.1
32 S	A	10.0	20.0	-	15.0	55.0
33	C	14.4	13.6	-	18.6	55.0
34	A	9.0	16.0	-	33.0	42.0
35	B2	3.5	27.2	36.3	33.0	-
36	B2	1.6	19.5	45.8	29.3	-

Tab. 11.1 Results of Neutron Diffraction on Abu Tbeirah' samples. The weight percentage [wt (%)] of the detected phases are reported. The errors are ± 0.1 wt (%).

Sample	K	Na	Fe	Cl	Mn
1	0.03	1.76	0.01	1.12	<LOQ
2	0.01	1.35	0.01	0.33	<LOQ
3	0.02	1.08	0.01	0.44	<LOQ
4	0.01	0.72	0.01	0.38	<LOQ
5	0.02	1.27	0.01	0.33	<LOQ
6	-	1.02	0.01	0.60	<LOQ
7	0.02	1.22	0.01	0.66	<LOQ
8 B	0.03	1.94	0.01	0.26	<LOQ
8 W	0.02	1.23	0.01	0.49	<LOQ
9	0.03	1.35	0.01	0.49	<LOQ
10	0.01	0.72	0.01	0.63	<LOQ
11	0.02	1.33	0.01	0.66	<LOQ
12	-	1.89	0.01	1.31	<LOQ
13	0.02	1.01	-	0.93	<LOQ
14	0.03	1.94	0.01	1.11	<LOQ
15	0.02	1.33	0.01	0.31	<LOQ
16	0.01	0.50	0.01	0.13	<LOQ
17	0.02	1.51	0.01	1.20	<LOQ
18 L	0.02	1.28	0.01	0.27	<LOQ
18 S	0.02	1.07	0.01	0.27	<LOQ
19 L	0.01	0.74	-	0.19	<LOQ
19 S	0.01	0.65	-	0.19	<LOQ
20	-	1.15	0.01	0.24	<LOQ
21 L	0.01	0.95	-	0.39	<LOQ
21 S	-	0.99	0.01	0.27	<LOQ
22	-	0.77	0.01	0.21	<LOQ
23	0.01	1.11	0.01	0.27	<LOQ
24	0.01	1.35	0.01	0.33	<LOQ
25	-	0.87	-	0.12	<LOQ
26 L	-	1.11	0.01	0.27	<LOQ
26 S	-	0.89	0.01	0.14	<LOQ
27	-	1.02	0.01	0.21	<LOQ
28	-	1.02	0.01	-	<LOQ
29	-	0.59	0.01	0.32	<LOQ
30	-	0.49	-	0.21	<LOQ
31	-	0.93	0.01	0.59	<LOQ
32 L	-	0.61	0.01	0.18	<LOQ
32 S	-	0.63	-	0.13	<LOQ
33	-	0.80	0.01	0.55	<LOQ
34	-	0.95	0.01	0.88	<LOQ
35	-	0.83	-	0.50	<LOQ
36	-	0.94	-	0.64	<LOQ

Tab. 11.2 Values of the normalised intensity of the main peaks from the Neutron Resonance Capture Analysis spectra for each sample. The normalized intensities (norm. counts) are given with an error of 0.01. It should be highlighted that the reported values are not the quantities of the elements present in the samples, but they are related with their amount; they were used for relative comparison via a statistical approach. LOQ = limit of quantification.

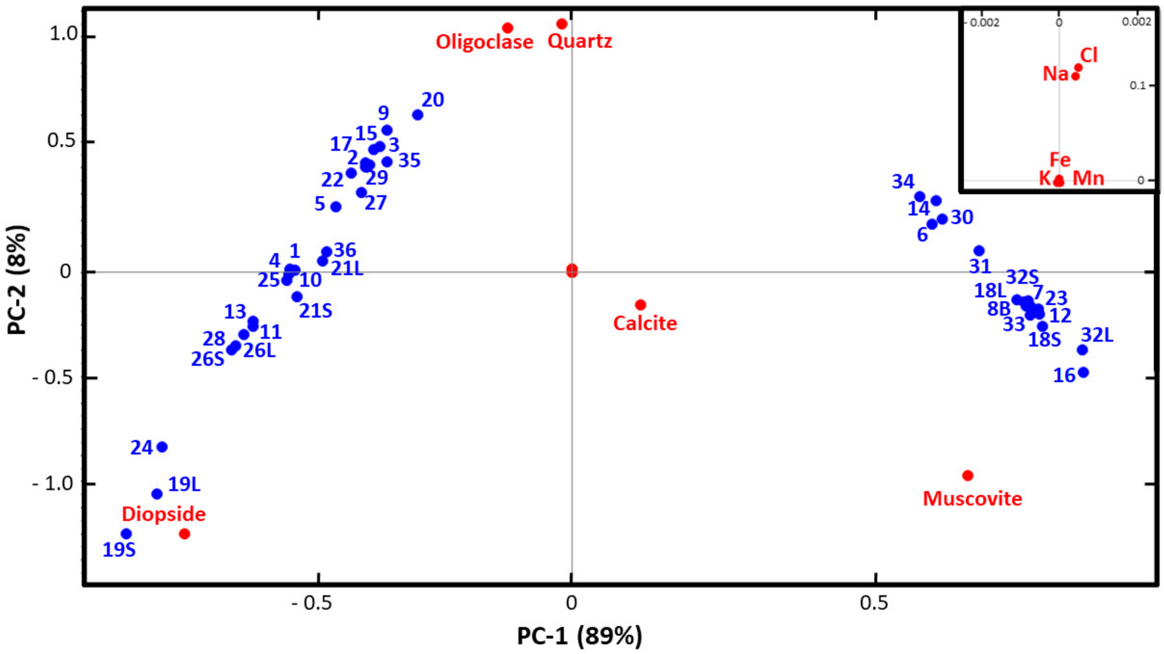

Fig. 11.5 Principal component analysis (PCA) results (Bi-plot) on the investigated AbT samples. The zoom around the origin of the axis is reported in the top right corner. Identified groups are highlighted; PCA Loading plot are reported in blue while PCA Scores plot of the phases and elements are in red. The third component has not been reported because does not improve the model (Festa *et al.* 2019: Fig. 5).

characterized by diopside, to indicate that these samples were fired at T that exceeded 900°C for long time. Muscovite, a mineral naturally present in Abu Tbeirah's clays, characterizes samples n. 16 (the unfired clay) and 32L (Fabric A, not completely oxidized sample): being the Muscovite stable during the firing process up to 900°C, it is clear that the two samples were fired under this limit.[21] Results from the Bi-plot are consistent with the diffraction results reported in Fig. 11.4. Finally, the insect of Fig. 11.5 shows that: Cl and Na are very close in the Bi-plot indicating probable salt intrusion, while Fe, Mn and K are located in the centre of the plot demonstrating that these elements are homogeneously distributed in the investigated sample set. Additionally, these three elements might be linked to pyroxenes, that are present in the metamorphic rocks at the origin of the 40% of heavy mineral composition of the Mesopotamian alluvial sediment.[22]

11.4.4 Autoptic VS Neutron Classification [GF - LR]

The classifications, based on the neutron and autoptic results, show some differences that might be easily explained, though further analysis will confirm or demise these hypotheses. Some features considered in the archaeological classification, such as the presence of vegetal temper mixed to the clay, are not influential from the chemical/compositional point of view (*e.g.*, for samples of Fabrics D_1 and D_2).[23] Moreover, the presence of the slip or self-slip[24] in samples 2, 3, 9, 15, 17, 19 (Fabric A) might justify the higher range of temperature identified by the neutron analysis. The presence of the slip/self-slip (see § 10.4.2) can thus alter the percentage of diopside in the investigated samples: in both the cases the vase surface loses water in a fasterway, reaching easily a temperature higher than 900°, forming crystals not present in the vessel body.[25] This might thus justify the position of the quoted sample in Fig 11.4. The presence of the big intrusive inclusion

[21] Rodriguez-Navarro *et al.* 2003; Rice 2005.
[22] See al-Mukhtar 2015.

[23] Future thin section analyses will help in better defining the ancient "ceramic recipes".
[24] See § 10.4.2 on the surface treatment.
[25] Jordan *et al.* 2009.

in sample 10 (Fabric B1) might have altered the results and thus the position in Fig. 11.5. A similar effect on mean data is observed for Fabric C (sample n. 33) used in the realization of rare cooking pots. Their coarse grained and porous paste, fired at a temperature lower than 900°C can help in withstanding the thermal shock due to the contact with fire:[26] the presence of bigger and angular sedimentary inclusions might be connected to a different preparation of the raw material by the ancient potter, through the addition of sand temper or avoiding the elimination of the bigger inclusions during the clay cleaning process. This Fabric is rare at Abu Tbeirah maybe due to the wider use of tannur and other peculiar firing installations for food processing.

11.5 Insights into the Clay Selection and Firing Process [GF - LR]

As previously demonstrated, clay from Mesopotamia was uniform in all the alluvial plain.[27] Nonetheless, a slight variation between north and south Mesopotamian pottery has been noted for the ED period: southern pottery seems to be characterized by the absence (or a less frequent presence) of pyroxene, biotite, epidote and, in a lesser extent, illite/muscovite.[28] However, on the basis of the limited amount of samples analysed these conclusions should be handled carefully: for example, while the similarity between pottery coming from Ur and from 'Ubaid is interesting and plausible, it is a parallel based only on two samples, one for each site.[29]

Analyses on Abu Tbeirah samples show, indeed, results that are sharply in contrast with this distinction between northern and southern 3rd mill. BC pottery. Comparing the diffraction data with the autoptic results, differences from the set of samples can be attributed mainly to firing temperature and in a less extent to clay preparation, confirming the general uniformity of the clay used in Mesopotamia. The samples analysed are, indeed, distributed in Fig. 11.3 according to an almost continuous temperature gradient.[30] Muscovite is present in the low fired samples and this discrepancy with the previous analyses is clearly due to the extremely reduced number of samples examined: probably the fragments selected simply belonged to vessels fired at higher temperature (thus with illite/muscovite completely transformed). The comparison of pottery samples with the clay gathered form the canal near Abu Tbeirah, seems to show a local origin of the used clay (see the uniformity in the values reported at Tabs 11.1-2 for phases and elements). Future analyses and researches on our samples will verify the possible presence of regional trends, though it appears once again clear that bulk chemical composition is not useful in grouping Mesopotamian clay fabrics on a regional base.[31]

Although Abu Tbeirah pottery presents inconstant use of the same fabric for a vessel typology, general trends can be recognized: a) drinking vessels, such as quickly wheel-thrown or wheel-coiled beakers and conical bowls, are usually realized in Fabric A and fired at a temperature lower than 900°C; b) medium and big closed vessels are instead mainly realized with Fabric B_1-B_2(T >900°C) or Fabric A self-slipped/slip;[32] c) rare cooking pots are realized in low fired (T <900°C) Fabric C; d) big containers, such as trays, vats or coffins are instead always realized in Fabric D_1 (T <900°C)-D_2 (T >900°C).

The thermal gradient, exhibited on and within Abu Tbeirah's vessels, confirms that the main distinction among fabrics is due to the temperature reached during firing: *e.g.* coffins and big coarse vat, on the bases of the firing temperature reached by each part of their body, show a fabric colour that ranges from red (D1) to pale-yellow/greenish (D2). This thermal gradient is, indeed, due to the non-uniform firing temperature to which vessels were exposed: the attribution of a sherd to a

[26] Rice 2005: 229-231. Müller 2016.

[27] Armstrong - Gasche 2014: 87.

[28] Mynors 1983; Mynors - al-Kaissi 1987: 144, 150 and Tab. 2.

[29] Data from the Southernmost part of Mesopotamia, in which Abu Tbeirah is located, are still extremely limited: a total of 40 shards coming from several sites (Tell-ed-Der, Jemdet Nasr, Nippur, Kish, Abu Salabikh, Fara, Tell al-Wilayah, Uruk, Larsa, Lagash, 'Ubaid, and Ur) were analysed (Mynors 1983; Thuesen *et al.* 1982; Mynors - al-Kaissi 1987).

[30] See also the comparable results for ED I-II pottery from the Diyala region in Gibson (ed.) 1990: 65. Here five group, very similar to ours, have been distinguished (with the addition of a grey-ware group).

[31] Gibson (ed.) 1990: 22.

[32] However, never in Fabric C or D (with the exception of ring bases realized always in Fabric D).

specific fabric group depends thus on the part of the vessel preserved or analysed.

As far as the firing technology is concerned, analyses carried out at Abu Tbeirah demonstrates that temperatures reached during their firing never exceeded 1000°C. The highlighted temperature range led us to focus on the firing technology in use for Abu Tbeirah's 3rd mill. BC pottery production. Utilization of kilns has been generally associated to a firing temperature range comprised between 750°C and 1150°C.[33] Kiln firing is considered a more advanced technique compared to open or pit fires, that are usually not considered adequate at reaching these temperatures.[34] Nevertheless, as already highlighted by several scholars, it is incorrect to search a linear technological progression from bone-fire to the adoption of kiln in pottery production.[35] Though pottery kilns are attested in Mesopotamian archaeological record also for earlier periods,[36] little attention was given to the presence of open-firings.[37] Though we do not exclude the use of kilns for Abu Tbeirah's pottery production (or at least for part of it), the sole maximum temperature reached during the firing cannot be taken as proof. Ethnographic comparisons show that temperatures of more than 900° C can be reached in open-firing using dung or palm fronds as fuel,[38] materials largely available in 3rd mill. BC southern Mesopotamia.[39] Besides, several doubts have been raised on the possibility of using firing temperature to determine the firing technology used:[40] ethno-thermometric approach demonstrates that the temperature reached by bone-fire and kiln overlap within the interval of 600-900°.[41] Furthermore, neither the structure, fuels, duration of the firing process, heating rate and soaking time induce thermal characteristics that can be considered as specific for open or kiln firings.[42]

In determining the firing technology in use in an archaeological site a synergy of elements should therefore be considered, together with archaeological data. In the case of Abu Tbeirah, we cannot suppose a kiln firing because of the temperature reached. In addition, some other elements related to Abu Tbeirah's pottery production might point toward the use of open/pit firing. Abu Tbeirah survey documented, especially in the North-Eastern part of the settlement, the presence of areas with pottery production wastes not connected to any visible structure: though the erosion that characterize Abu Tbeirah's surface could have obliterated the original kilns structure, it is not possible to exclude the identification of these areas with open firings.[43] Abu Tbeirah vases, as said, often show traces of a non-uniform firing, a characteristic that is usually connected to open-firing.[44] The dark or black core of some Abu Tbeirah vessels is due to the non-complete control of the firing atmosphere or to an insufficient duration of the firing process. All these elements put together suggest caution in hypothesizing the exclusive use of a specific firing technology: as for the pottery modelling technique (see § 10), the introduction of a new and more performative methodology[45] does not mean the complete abandonment of the old one[46] and both techniques could have been used in synergy for a period longer than expected.

[33] Tite 1969: 140 Tab. 3 (Ubaid pottery; 500-1110°C); Gibson (ed.) 1990: 40 (Tell Razuk; 850-1100°C); Méry - Schneider 1996: 86 (various site with a max. T>1000°C); Armstrong - Gasche 2014: 84; 89 (various sites; 750-1000°C).

[34] Gosselain 1992: 244-245 (open fire temperature ranges 500-900° C).

[35] Moorey 1994: 144; Laneri 2009: 111-114.

[36] Hansen Streily 2001; Laneri 2009: 117.

[37] Moorey 1994: 144-145. For a resumé of the evidence of open firings in the Near East see Laneri 2009: 113-114.

[38] Nicklin 1981: 352; Shepard 1985: 78 Fig. 4; Rice 2005: 157.

[39] Marsh's Arab used to bake their pottery in pits, using dung cakes (350-450 dung patties) and reeds to ignite (see Ochsenschlager 2004: 119-121).

[40] Gosselain 1992; Livingstone Smith 2001.

[41] Gosselain 1992: 257.

[42] Livingstone Smith 2001: 999.

[43] D'Agostino - Romano 2017. In the northern part of the site also a fragment of a potter's wheel was found (Romano 2015).

[44] Rice 2005: 158; Armstrong - Gasche 2014: 83. Nevertheless the results of kiln firing depends also on the potter's ability and on the same properties of the built structures.

[45] Kiln technology has several disadvantages such the necessity of maintenance and repair or the not completely efficient use of fuel (Rice 2005: 162).

[46] The association of the earliest productions with the "more primitive" techniques is a frequent trend in Ancient Near East archaeology, as already noticed by Moorey 1994: 144.

References

Al-Mukhtar, L.E.

2015 Heavy Mineral Analysis of the Quaternary Sediments in the Southern Part of the Mesopotamia Plain, Iraq, *Iraqi Bulletin of Geology and Mining* 11(2): 59-73.

Armstrong, J.A. - Gasche, H.

2014 *Mesopotamian Pottery: A Guide to the Babylonian Tradition in the Second Millennium BC* (=Mesopotamian History and Environment Series II, Memoires VI), Ghent-Chicago.

Baxter, M.J.

1994 *Exploratory Multivariate Analysis in Archaeology*; Edinburgh.

D'Agostino, F. - Romano, L.

2017 Abu Tbeirah's Craft Area NE: A Preliminary Survey, *Ash-Sharq* 1: 131-154.

Druc, I.

2015 *Atlas of Ceramic Pastes. Components, Texture and Technology*, Blue Mounds, WI.

Festa, G. *et al.*

2008 Composition and Corrosion Phases of Etruscan Bronzes from Villanovan Age, *Measurement Science and Technology* 19 (3), 034004 (7 pp.).

2009 A Non-Destructive Stratigraphic and Radiographic Neutron Study of Lorenzo Ghiberti's Reliefs from Paradise and North Doors of Florence Baptistery, *Journal of Applied Physics*, 106 (4). doi.10.1063/1.3204514.

2011a Neutron Diffraction Measurements at the INES Diffractometer Using a Neutron Radiative Capture-Based Counting Technique, *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment* 654 (1) 373-376.

2011b Non Destructive Neutron Diffraction Measurements of Cavities, Inhomogeneities and Residual Strain in Bronzes of Ghiberti's Relief from the Gates of Paradise, *Journal of Applied Physics* 109(6). doi.10.1063/1.3560915.

2013 Simultaneous and Integrated Neutron-Based Techniques for Material Analysis of a Metallic Ancient Flute. *Measurement Science and Technology* 24. doi.10.1088/0957-0233/24/9/095601.

2015 Neutron Resonance Transmission Imaging for 3D Elemental Mapping at the ISIS Spallation Neutron Source, *Journal of Analytical Atomic Spectrometry* 30: 745.

2016 Isotope Identification Capabilities Using Time Resolved Prompt Gamma Emission from Epithermal Neutrons, *Journal of Instrumentation* 11, C03060.

2019 Sumerian Pottery Technology Studied Through Neutron Diffraction and Chemometrics at Abu Tbeirah (Iraq), *Geosciences. Special Issue "Archaeological and Heritage Research"*. Doi: 10.3390/ geosciences9020074

Gibson, McG. (ed.)

1990 *Uch Tepe* II: *Technical Reports*, Chicago.

Gosselain, O.P.

1992 Bonfire of the Enquiries. Pottery Firing Temperatures in Archaeology: What For?, *Journal of Archaeological Science* 19: 243-259.

Hansen Streily, A.

2001 Early Pottery Kilns in the Middle East, *La pyrotehnologie à ses débuts. Évolution despremières industries faisant usage du feu / Early Pyrotechnology. The Evolution of the First Fire-Using Industries. Paléorient* 26(2): 69-81.

Hunt, A.M.W.

2015 *Palace Ware Across the Neo-Assyrian Imperial Landscape: Social Value and Semiotic Meaning* (=Culture and History of the Ancient Near East 78), Leiden.

Husson, F. *et al.*

2009 *Exploratory Multivariate Analysis by Example Using R*, London.

Imberti, S. *et al.*

2008 Neutron Diffractometer INES for Quantitative Phase Analysis of

Archaeological Objects, *Measurement Science and Technology* 19: 1-8.

Jolliffe, I.T.

1986 *Principal Component Analysis*, New York.

Jordan, M.M. *et al.*

2009 Mineralogy and Firing Transformations of Permo-Triassic Clays Used in the Manufacturing of Ceramic Tile Bodies, *Applied Clay Science* 44: 173-177.

Kockelmann, W. - Kirfel, A.

2001 Non-Destructive Phase Analysis of Archaeological Ceramics using TOF Neutron Diffraction, *Journal of Archaeological Science* 28: 213-222.

2006 Neutron Diffraction Imaging of Cultural Heritage Objects, *Archeometriai Műhely* 2006(2): 1-15.

Laneri, N.

2009 *Biografia di un vaso. Tecniche di produzione del vasellame ceramico nel Vicino Oriente antico tra il V e il II millennio a.C.,* Paestum.

Livingstone Smith, A.

2001 Bonfire II: The Return of Pottery Firing Temperatures, *Journal of Archaeological Science* 28: 991-1003.

Méry, S. - Schneider, G.

1996 Mesopotamian Pottery Wares in Eastern Arabia from the 5th to the 2nd Millennium BC: A Contribution of Archaeometry to the Economic History, *Proceedings of the Seminar for Arabian Studies, Vol.* 26, *Papers from the Twenty-Ninth Meeting of the Seminar for Arabian Studies Held in Cambridge, 20th-22nd July, 1995*, Oxford: 79-96.

Moorey, P.R.S.

1994 *Ancient Mesopotamian Materials and Industries: The Archaeological Evidence*, Indiana.

Mughabghab, S.F. *et al.*

1981 *Neutron Resonance Parameters and Thermal Cross Sections*, New York.

Müller, N.

2016 Mechanical and Thermal Properties, Hunt, A. (ed.), *The Oxford Handbook of Archaeological Ceramic Analysis*, Oxford.

Mynors, H.S.

1983 An Examination of Mesopotamian Ceramics Using Petrographic and Neutron Activation Analysis, in Aspinall, A. - Warren, S. (eds), *Proceedings of the 22nd Symposium on Archaeometry Held at the University of Bradford, Bradford, U.K., 30th March - 3rd April 1982*, Bradford: 377-387.

Mynors, H.S. - Al Kaissi, B.

1987 Ceramic Analyses of Mesopotamian Wares in the Early Dynastic Period, *Research on the Antiquities of Saddam Dam Basin Salvage and Other Researches*, Baghdad: 134-154.

Nardini, M. *et al.*

2018 Neutron Resonance Capture Analysis applied to Archaeometry, in *Proceedings of "Neutrons Matter, the 7th edition of the International Workshop on Electron-volt Neutron Spectroscopy", November 2017* (=Journal of Physics: Conference Series 1055): doi :10.1088/1742-6596/1055/1/012005.

Nicklin, K.

1981 Ceramic Pyrometry: Two Ibibio Examples, in Howard, H. - Morris, E.L. (eds), *Production and Distribution: A Ceramic Viewpoint* (=BAR International Series 120), Oxford: 347-359.

Ochsenschlager, E.L.

2004 *Iraq's Marsh Arabs in the Garden of Eden*, Philadelphia.

Pietropaolo, A. *et al.*

2011 A Multitask Neutron Beam Line for Spallation Neutron Sources, *Europhysics Letters* 95. doi.10.1209/0295-5075/95/48007.

Postma, H. - Schillebeeckx, P.

2005 Non-Destructive Analysis of Objects Using Neutron Resonance Capture, *Journal of Radioanalytical and Nuclear Chemistry* 265(2): 297-302.

Rice, P.M.

2005 *Pottery Analysis: A Sourcebook. The Raw Materials of Pottery Making: Perspectives from Chemistry, Geology, and Engineering*, Chicago-London.

Rodriguez-Navarro, C. *et al.*

2003 TEM Study of Mullite Growth after Muscovite Breakdown, *American Mineralogist* 88 (5-6): 713-724.

Romano, L.

2015 A Fragment of a Potter's Wheel from Abu Tbeirah, *Zeitschrift für Assyriologie* 105. doi 10.1515/za-2015-0018.

Scatigno, C. *et al.*

2017 Combination of in Situ Spectroscopy and Chemometric Techniques to Discriminate Different Types of Roman Bricks and the Influence of Microclimate Environment, *Environmental Science and Pollution Research.* doi 10.1007/s11356-017-0938-6.

Shepard, A.O.

1985 *Ceramics for the Archaeologist* (1st edition 1956), Washington, D.C.

Thuesen, I. *et al.*.

1982 Investigation of 5000-Year-Old Pottery from Mesopotamia by Instrumental Neutron Activation Analysis, V. Mejdahl (ed.), *Proceedings of Second Nordic Conference on the Application of Scientific Methods in Archaeology, Vol.* 2, Strasbourg: 375-381.

Tite, M.S.

1969 Determination of the Firing Temperature of Ancient Ceramics by Measurement of Thermal Expansion: A Reassessment, *Archaeometry*: 131-143.

Verhoeven, K.

1998 Geomorphological Research in the Mesopotamian Flood Plain, in Gasche, H. - Tanret, M. (eds) *Changing Watercourses in Babylonia. Towards a Reconstruction of the Ancient Environment in Lower Mesopotamia*, *vol.* I. (=Mesopotamian History and Environment, Series II, Memoirs V), Ghent-Chicago: 159-240.

Windsor, C.G.

1981 *Pulsed Neutron Scattering*, London.