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Production technology of Nabataean painted pottery compared with that of Roman *Terra Sigillata*

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ABSTRACT The Nabataeans, who founded the city of Petra (southern Jordan) in the late first millennium BC, are noted for the production of a distinctive very fine pottery with painted decoration and a wall thickness sometimes as little as 1.5 mm; this pottery appears largely locally made and not widely circulated. Using a combination of OM, SEM with attached EDS, surface XRF, and XRD, it is shown that the Nabataean fine pottery bodies were produced using semi-calcareous clays which were fired to temperatures of about 950°C. In contrast, published data indicates that contemporary and in many ways apparently functionally equivalent Roman *terra sigillata*, which was traded throughout the Roman Empire, was produced using fully-calcareous clays which were fired to temperatures in the range 1000-1100°C. Furthermore, the high gloss slip applied to Roman *terra sigillata* is fully vitrified whereas the red-painted decoration applied to the Nabataean pottery is unvitrified. The more robust Roman *terra sigillata* is therefore better suited as tableware for serving and consuming food than would be the case for Nabataean fine pottery, and would be a more successful export material.

KEYWORDS: POTTERY, *TERRA SIGILLATA*, SLIPS, NABATAEAN, ROMAN, CALCAREOUS CLAYS, FIRING TEMPERATURES, OM, SEM-EDS, XRF, XRD

INTRODUCTION

The Nabataeans, who were originally nomads, founded the city of Petra (southern Jordan) in the late first millennium BC as their royal capital and management centre for trade. It is famed for its rock-cut tombs and temples intricately carved into the pinkish sandstone, giving it its epithet – the 'Rose Red City'. The importance of Petra as a trading centre increased to reach a peak in the last two centuries BC and first two AD; following the Roman annexation of the Nabataean kingdom in 106 AD, the commercial role of Petra shifted and eventually declined. During their peak period, the Nabataeans produced, for domestic and religious use, fine pottery with wall thickness sometimes as little as 1.5 mm. Although production of such pottery continued post-annexation, the quality declined towards the end of the 2nd century**C**

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AD, with the pottery becoming coarser and thicker. Typical were shallow open bowls which would have required considerable skill to produce on a potter's wheel. This pottery had red bodies which were decorated with dark red to brown to sometimes near black flower and leaf designs. The flower and leaf designs are often found with geometric motifs such as dots and lattices which connect the floral motifs.

The main emphasis of previous research on Nabataean fine pottery has been directed at identifying and distinguishing between the different raw material sources used in its production (Amr 1987, Daszkiewicz et al. 2013), and at replicating the forming, decorating and firing procedures (Mason and Amr 1990, Mason and Amr 1995, Amr et al. 2005). The aim of the present study has been to extend the available analytical data on the raw materials and to investigate the firing temperatures used in the production of Nabataean fine pottery.

A small group of Nabataean sherds from the excavations by Brown University of the Great Temple at Petra (Joukowsky 1998) have been examined using optical microscopy (OM), scanning electron microscopy with attached energy-dispersive spectrometer (SEM-EDS), surface X-ray fluorescence analysis (XRF), and X-ray diffraction (XRD). The results obtained are then compared with corresponding published data for Roman *terra sigillata* which was traded throughout the Roman Empire, and which was in many respects a ceramic akin in usage to Nabataean fine pottery.

EXPERIMENTAL PROCEDURES

Samples

Fourteen Nabataean sherds from the Brown University Great Temple excavations at Petra were selected for examination (figure 1). The majority were open bowl fragments from two separate days of excavation of the Shrine Room and Baroque Room (SRBR) which has been assigned a date range of 1st century BC to 1st century AD. On the basis of the style of decoration, sherds from 24/VI/02 (MS3-4, MS6, MS9-10) were possibly slightly later in date than those from 29/VI/02 (MS1-2, MS11-13, MS15-16). In addition, a few sherds (MS18-19) of similar date range but of varying forms were selected from the excavation of the Corridor between the Settling Tank and Residential Quarter (CSTRQ). It should be noted that this type of pottery is not only found in contexts across the city center, but also commonly in its regional hinterland; it does not, however, appear to have been widely traded beyond (Alcock and Knodell 2012).

Analytical procedures

Polished cross-sections were prepared from all fourteen sherds. Of these, seven polished cross-sections were examined at Brown University under OM using a Mitutoyo Ultraplan FS110 microscope at magnifications up to 200x, and seven were examined at Oxford and Cranfield Universities with SEM-EDS. The SEM-EDS systems used were a JEOL SEM (JSM-5910) with Oxford Instruments EDS (INCA 300 System) at Oxford University, and a JEOL SEM (JSM-840A) with EDAX EDS at Cranfield University. Both SEM-EDS systems were operated at 20kV with 120s measuring times for the EDS analyses. Backscattered electron (BSE) images were obtained in order to study the microstructures of cross-sections, and the EDS systems were operated in the standardless analytical mode. Surface XRF analyses were also make on the slip and body surfaces of four sherds using a benchtop XRF

(Seiko SEA600X) run at 50kV with a 10 mm working distance and 1.5 mm analytical area, and operated in air**.**

XRD measurements were made on the slip and body surfaces of two sherds (MS12, MS16) at Universitat Politècnica de Catalunya using a conventional diffractometer, Bruker D8 with monochromatic Cu-Kα (1.5606Å) radiation with 4-70 degrees two theta range, the penetration depth of the X-rays being less than about 100 μm. Powder XRD measurements were taken from the body of nine other sherds (MS 1, 3-7, 9-10, 14) on a Bruker AXS-Powder X-ray Diffractometer at Clark University (Worcester, MA, USA) with Cu-Kα radiation with a two theta range of 15-60^{o}. Identification of the compounds has been performed based on the Powder Diffraction File (PDF) database from the International Centre for Diffraction Data (ICDD).

Samples of two sherds (MS12 and MS16) were refired in an electric furnace to 950°C and 1050°C, with a heating rate of 300°C/hr and then 1hr at maximum temperature. These samples were then re-examined in the Oxford SEM, and re-analysed by surface XRD.

RESULTS

The microstructures of the Nabataean pottery bodies as observed in the SEM reveal open networks of fine, partially vitrified clay filaments containing varying amounts of non-plastic inclusions, the majority of which are quartz and feldspar (figure 2). On the basis of the abundance and size of the inclusions, as observed in both SEM and OM, the sherds can be divided into two fairly distinct groups. In sherds MS1-2, MS11-13 and MS16, the inclusions are fairly sparse and small in size (about 20-30μm or less across) (figure 2a). In contrast, in sherds MS3, MS 10, MS15 and MS18-19, the inclusions are more abundant and vary in size from 20μm to about 100μm across (figure 2b).

The body compositions for the Nabataean pottery as determined by SEM-EDS are presented in table 1 from which it is seen that the body clays are characterised by comparatively high iron oxide contents (5-8 wt%), and intermediate alumina (19-23 wt%) and lime (5-10 wt%) contents. Comparison of the compositions for the same sherd (MS12) as determined at Cranfield and Oxford are in good agreement with each other, and also, generally in agreement with the compositions of three Nabataean sherds from Petra assigned to Period III (circa 20 AD to early 2nd century AD) (Schmid 1996, Amr et al. 2005), as determined by wavelength dispersive-XRF and reported by Daszkiewicz et al. (2013) (Table 1).

As observed in cross-section in the SEM, the red painted decoration on sherd MS11 is associated with a very thin, fragmented and unvitrified layer about 10 μm thick and containing a scatter of iron oxide particles about 2 μm across (Figure 3a). Its composition is characterised by significantly higher iron oxide content (17.9 wt% *cf.* 7.7 wt%), higher potash content (4.5 wt% *cf.* 2.9 wt%), and lower lime content (2.2 wt% *cf.* 8.6 wt%) than that of the body (table 1). Red paint layers were not observed in any of the other sherd samples examined in the SEM. However, for sherds MS12-13 and MS15, subtraction of the surface XRF spectra of the red painted decoration from those for the unpainted bodies confirmed that the red painted decoration is consistently higher in iron oxide and potash, and lower in lime as compared to the corresponding body (figure 4).

As observed in cross-section in the SEM, the white slip applied to the outer surface of sherd MS19 is associated with a compacted but unvitrified layer about 30 µm thick (figure 3b) which is lime-based containing some 85 wt% CaO (table 1).

Surface X-ray diffraction measurements on sherds MS12 and MS16, undertaken at Universitat Politècnica de Catalunya, showed that the principal mineral phases present in the bodies are quartz, haematite (αFe_2O_3), Ca-Na feldspar ($(Ca,Na)AlSi_3O_8$), K-Na feldspar ($(K,Na)AlSi_3O_8$), akermanite ($Ca_2MgSi_2O_7$), and diopside ($CaMgSi_2O_6$) (figure 5a) (table 2). Essentially the same range of mineral phases were detected in the bodies of a further nine sherds using powder XRD measurements at Clark University (Worcester, MA, USA. In contrast, because of its very low lime content, the only significant phases detected by surface XRD measurements in the red painted decoration on sherd MS16 are quartz, haematite, and K-Na feldspar (figure 5a) (table2).

The examination of the refired samples of sherds MS12 and MS16 in the SEM showed that the bodies fired to 950°C exhibited similar open networks of fine, partially vitrified, clay filaments to those of the as-received sherds with at most a very slight increase in vitrification (figure 6). In contrast, the bodies fired to 1050°C exhibited continuous vitrification with bloating pores less than about 50 µm across. Similarly, XRD measurements indicated that the range and amounts of the different mineral phases present after refiring to 950°C was essentially unchanged, but after firing to 1050°C, only haematite, Ca-Na feldspar and quartz remained with mullite being formed in sherd MS16 (figure 5b) (table 2). It is therefore suggested that the firing temperature employed in the production of the Nabataean fine pottery was about 950°C.

COMPARISON WITH ROMAN *terra sigillata*

Roman *terra sigillata* was first produced in Italy from mid-1st century BC. Production then moved to southern Gaul where the most important workshop was at La Graufesenque (Aveyron) and from where *terra sigillata* was traded throughout the Roman Empire, including around the entire Mediteranean (Sciau et al. 2006).

As observed in the SEM, the microstructures of the *terra sigillata* bodies, which are thicker (3-4 mm) than those of the Nabataean fine pottery, again reveal an open network of fine, partially vitrified clay filaments containing a scatter of quartz and other non-plastic inclusions (Tite et al. 1982a, Sciau et al. 2006) (figure 7). In contrast to the Nabataean red painted decoration, the high gloss *terra sigillata* surface slip layer, which is typically 10-20 µm thick, appears vitrified and impermeable with no visible clay structure but a high concentration of iron oxide particles, typically less that 0.5 µm in diameter.

The compositions of the *terra sigillata* bodies (table 3) are very similar to those of the Nabataean fine pottery except for somewhat higher lime contents (10-17 wt% *cf.* 5-10 wt%) and slightly lower iron oxide contents (5-6 wt% *cf.* 5-8 wt%). The *terra sigillata* slips again have higher iron oxide (10-12 wt% *cf.* 5-6 wt%), potash (7-10 wt% *cf.* 1-4 wt%), and alumina (23-33 wt% *cf.* 21-23 wt%) contents, and lower lime contents (1-2 wt% *cf.* 10-17 wt%) than those of the bodies. However, the increase in potash content in the slip is more than that in the case of the Nabataean fine pottery, whereas the increase in the iron oxide content is less.

On the basis of the changes, as determined by XRD, in the mineral phases present when samples of local clay or *terra sigillata* are fired or refired, the estimated firing temperature for *terra sigillata* is in the range 1000-1100°C (Sciau et al. 1992, Mirti et al.1999) as compared to about 950°C for the Nabataean fine pottery.

DISCUSSION AND CONCLUSIONS

As contemporary ceramics produced by societies which were definitely in contact with each other, it is interesting to note that the compositions of the Nabataean and Roman *terra sigillata* bodies and slips are in many respects similar, the principle difference being that the lime contents of the Nabataean bodies (5-10 wt% CaO) are less than those of the Roman *terra sigillata* bodies (10-20 wt% CaO)

Therefore, as first defined by Maniatis and Tite (1981) and Tite et al. (1982b), the clays used to produce both ceramic groups can, be classified as calcareous (i.e., >5 wt% CaO), rather than non-calcareous (i.e., < 5 wt% CaO). Maniatis and Tite (1981) further distinguished between fully-calcareous clays containing greater than about 10 wt% CaO and semi-calcareous clays containing some 5-10 wt% CaO. For the former, the open network of vitrified filaments observed in the SEM remains essential unchanged over the firing temperature range from 850-1050ºC, whereas for the latter, as observed by the production of of continuous vitrification when the Nabataean bodies, MS12 and MS16, were refired to 1050ºC, the extent of vitrification can start to increase well before 1050ºC.

Therefore, the Nabataean bodies can be classified as semi-calcareous and the Roman *terra sigillata* bodies as fully-calcareous. However, the open network of vitrified filaments observed in the SEM and the detection of calcium silicate phases by XRD for the Nabataean bodies suggest that there has been sufficient formation of crystalline phases to achieve some of the gains in rigidity and compressive strength resulting from the use of fully- calcareous clays, as for Roman *terra sigillata* bodies (Peters and Iberg 1978).

The greater thickness of the Roman *terra sigillata* bodies (3-4 mm *cf.* as little as 1.5 mm), together with their higher lime content, suggests that Roman *terra sigillata* is both stronger and tougher than Nabataean fine pottery. Furthermore, the high gloss, vitrified slip applied to *terra sigillata* is clearly more robust than the unvitrified, red paint decoration applied to Nabataean pottery.

In summary, therefore, Roman *terra sigillata* is more durably suited for tableware for serving and consuming food than would be the case for Nabataean fine pottery, as well as more successfully suitable for long-distance transport.

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FIGURE CAPTIONS

Figure 1 Photos of Nabataean fine pottery sherds examined by OM, SEM-EDS and XRD: (a) MS10 from SRBR24/VI/02, (b) MS12 from SRBR29/VI/02, (c) MS16 from SRBR29/VI/02, (d) MS19from CSTRQ.

Figure 2 SEM photomicrographs of cross-sections through bodies of Nabataean sherds showing open network of fine, partially vitrified clay filaments with (a) fairly sparse and small inclusions (sherd MS11), and (b) more abundant and larger inclusions (sherd MS18).

Figure 3 SEM photomicrographs of cross-sections through slips and bodies of Nabataean sherds showing (a) very thin, fragmented and unvitrified layer containing a scatter of iron oxide particles (white) associated with the red painted decoration on sherd MS11, and (b) compacted but unvitrified layer associated with white slip applied to sherd MS19.

Figure 4 Observed surface XRF spectrum for Nabataean sherd MS15 after subtraction of the spectrum for the red painted decoration from that for the unpainted body, showing lower calcium, and higher potassium and iron in the red painted decoration.

Figure 5 XRD spectra for (a) painted and unpainted areas of the surface of Nabataean sherd MS16, and (b) unpainted areas of the surface of Nabataean sherd MS16 after refiring to 950°C and 1050°C.

Figure 6 SEM photomicrographs of cross-sections through bodies of Nabataean sherds MS12 and MS16 showing in both cases little change in the open network of fine, partially vitrified clay filaments visible in the as-received (ASR) microstructure and after refiring to 950°C, but a significant change to a continuously vitrified microstructure with bloating pores after refiring to 1050°C.

Figure 7 SEM photomicrograph of cross-section through slip and body of Roman *terra sigillata* sherd BRH1 showing vitrified, impermeable slip layer approximately 20 μm in thickness and containing iron oxide particles (white) (LHS), and open network of fine, partially vitrified clay filaments in the body (RHS).

Table 1

Table 1 Body and slip compositions for Nabataean fine pottery as determined by EDS (wt% normalised to 100wt%)

Sherds	Location[1]	SEM[2]	Na2O	MgO	Al2O3	SiO2	K2O	CaO	TiO2	FeO
Bodies										
MS11	SRBR29/VI/02	C	0.3	2.2	21.8	55.6	2.9	8.6	0.9	7.7
MS12	SRBR29/VI/02	O	0.4	2.2	22.7	55.2	3.2	8.3	0.9	7.1
MS12	SRBR29/VI/02	C	0.4	2.2	22.3	57.0	2.9	8.0	1.0	6.3
MS13	SRBR29/VI/02	C	0.4	2.4	22.0	55.1	2.8	9.7	0.8	6.8
MS15	SRBR29/VI/02	C	0.3	4.1	19.2	56.7	3.1	7.9	1.1	7.7
MS16	SRBR29/VI/02	O	0.4	2.1	22.7	56.3	3.1	6.8	1.0	7.5
Period III[3]			0.1	2.2	22.2	54.6	3.1	7.9	1.0	7.8
MS18	CSTRQ	C	0.4	2.9	21.5	59.5	2.5	6.6	0.8	5.8
MS19	CSTRQ	C	0.5	2.5	22.1	60.3	2.6	6.4	0.7	4.9
Slips										
MS11 - Red	SRBR29/VI/02	C	0.2	2.5	20.9	49.2	4.5	2.2	2.4	17.9
MS19 - White	CSTRQ	C	bd	3.1	3.7	8.1	bd	85.1	bd	bd

[1] SRBR - Shrine Room and Baroque Room, CSTRQ - Corridor between Settling Tank and Residential Quarter

[2] C - Cranfield University, O - Oxford University

[3] Data from Daszkiewicz et al. 2013 - average composition for three sherds from Petra analysed by wavelength dispersive XRF

Table 2 Mineral phases identified in as-received and refired sherds of Nabataean fine pottery as determined by surface XRD measurements

	MS12 - Body			MS16 - Body			MS16 - Slip
	ASR	950°C	1050°C	ASR	950°C	1050°C	ASR
Quartz	X	X	X	X	X	X	X
Ca-Na feldspar	X	X	X	X	X	X	
Diopside	X	X		X	X		
Akermanite	X	X		X	X		
K-Na feldspar	X	X		X	X		X
Haematite	X	X	X	X	X	X	X
Mullite						X	

Table 3 Body and slip compositions for Roman *terra sigillata* (wt% normalised to 100wt%)

Sherds	Na2O	MgO	Al2O3	SiO2	K2O	CaO	TiO2	FeO
Bodies								
BRH1[1]	0.8	2.4	21.0	51.0	1.4	17.2	0.4	5.9
GL03[2]	0.4	1.2	22.9	52.9	3.6	13.4	0.4	5.3
GL12A[2]	0.3	1.2	20.5	58.7	3.0	10.8	0.5	5.0
GL14B[2]	0.5	1.9	23.2	53.5	4.4	9.9	0.7	5.9
La Graufesenque[3]	na	2.3	22.5	52.7	3.7	10.7	1.1	6.0
Slips								
BRH1[1]	0.3	0.8	32.9	43.5	6.9	2.3	0.9	12.4
GL03[2]	0.3	1.3	27.1	47.0	9.6	1.9	0.8	12.0
GL12A[2]	0.4	1.8	23.3	52.8	7.8	1.5	0.4	12.0
GL14B[2]	0.3	1.0	23.3	55.7	8.0	1.1	0.5	10.1

[1] Data from Tite et al. 1982a - analysis by electron microprobe

[2] Data from Mirti et al. 1999 - analysis by EDS

[3] Data from Picon et al. 1975 - average composition for more than 100 sherds analysed by XRF spectrometry

Figure 1

Figure 1a

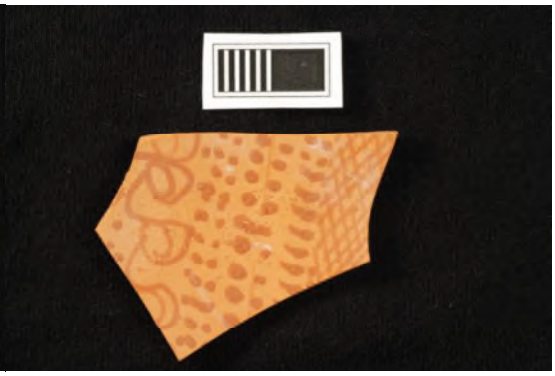


Figure 1b

Figure 1c

Figure 1d

Figure 2-7

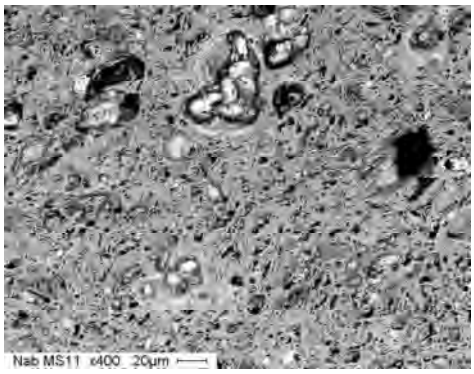


Figure 2a

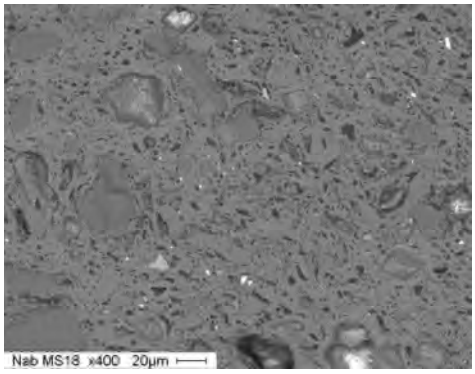


Figure 2b

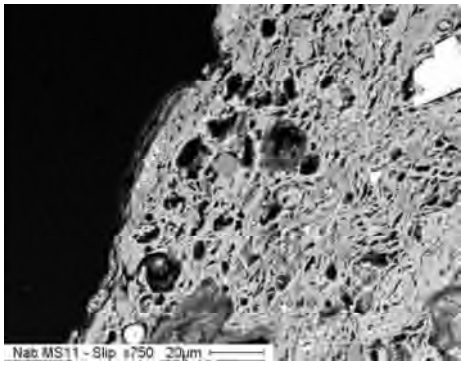


Figure 3a

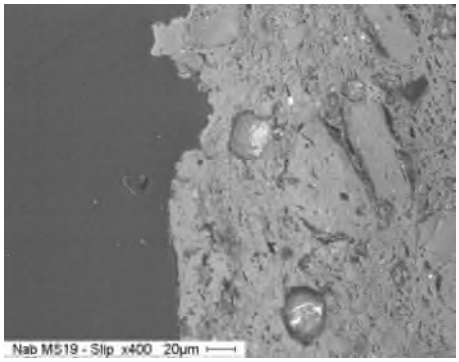


Figure 3b

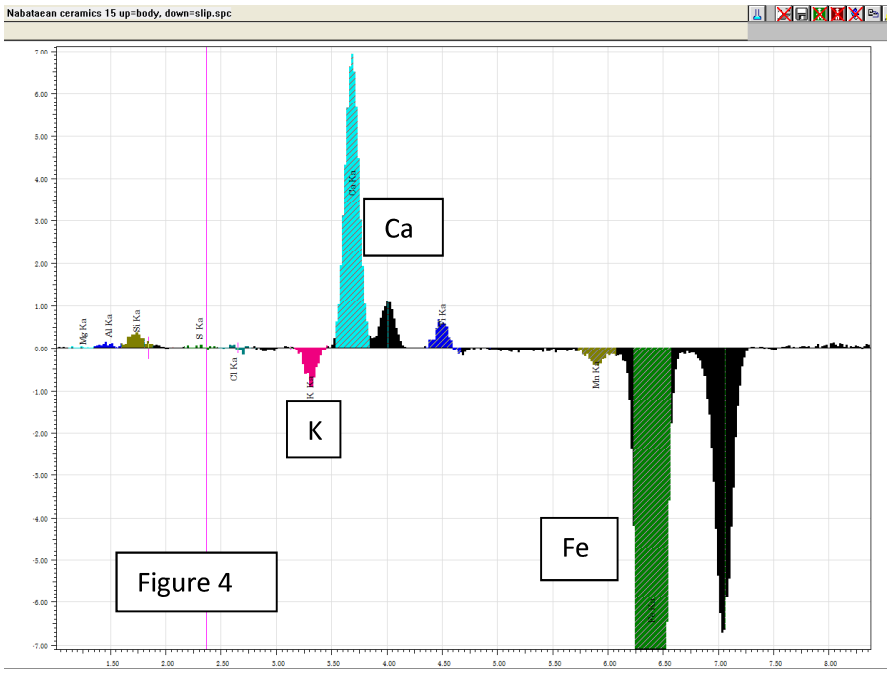


Figure 4

Figure 5a

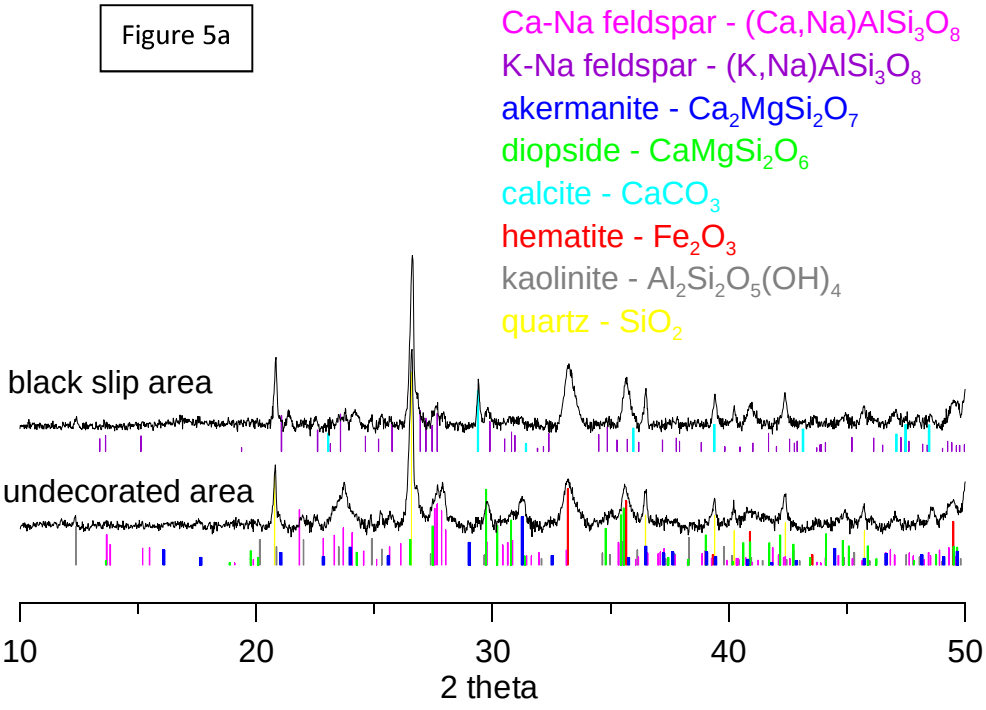
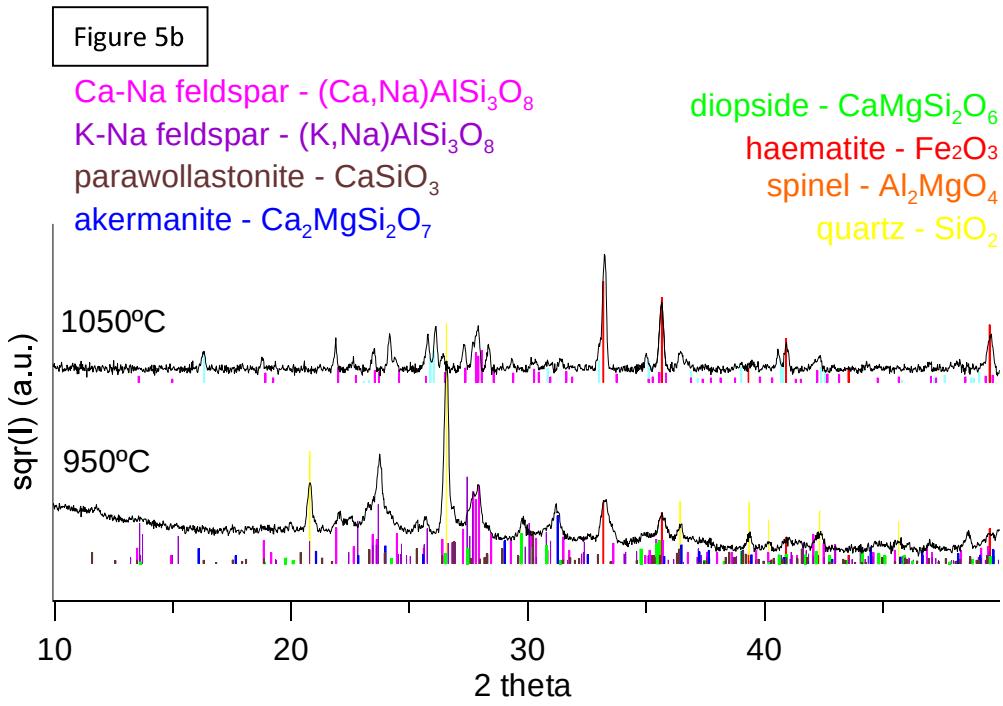


Figure 5b



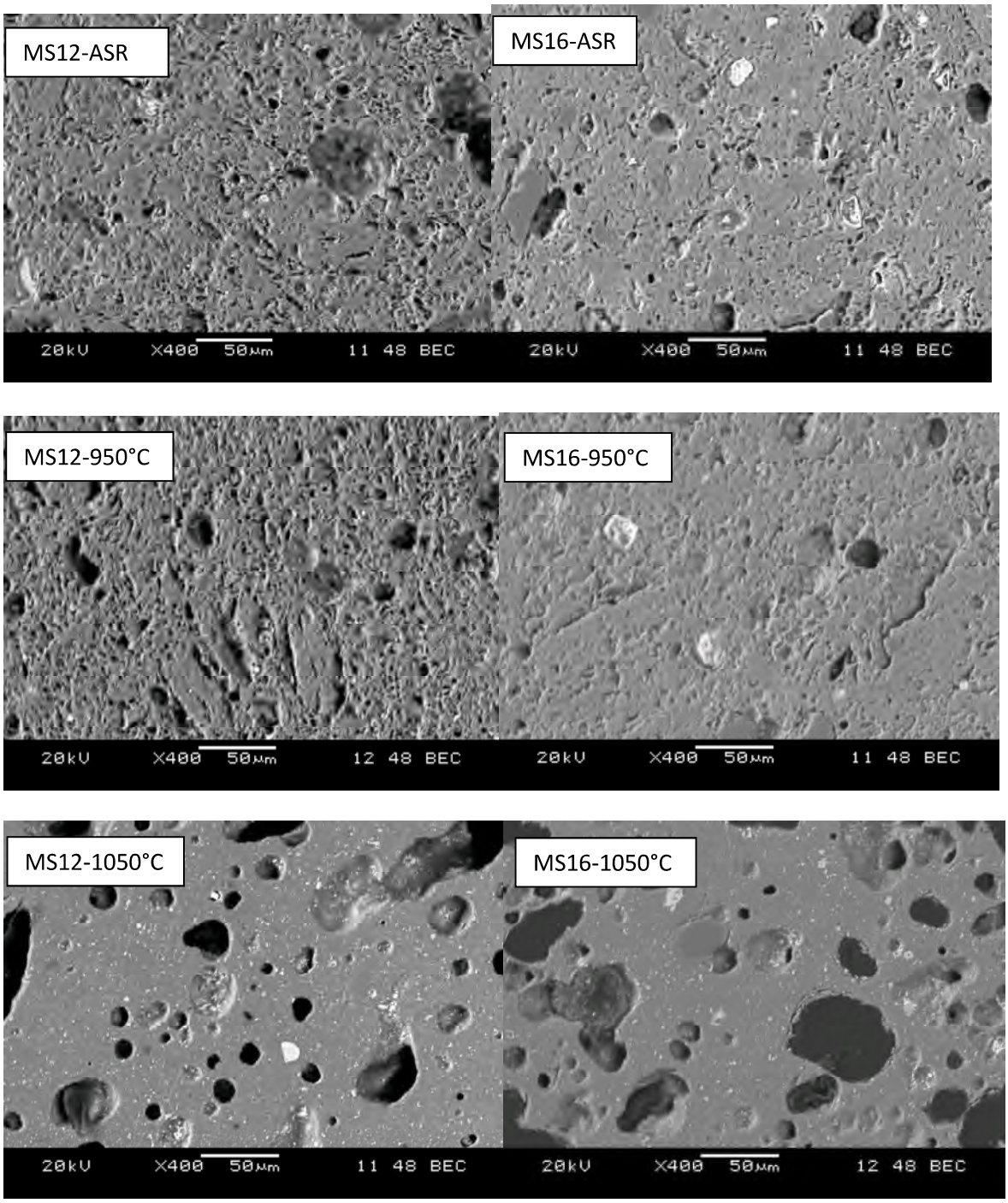

MS12-ASR
MS16-ASR
MS12-950°C
MS16-950°C
MS12-1050°C
MS16-1050°C
20kV X400 50µm 11 48 BEC
20kV X400 50µm 11 48 BEC
20kV X400 50µm 12 48 BEC
20kV X400 50µm 11 48 BEC
20kV X400 50µm 11 48 BEC
20kV X400 50µm 12 48 BEC

Figure 6

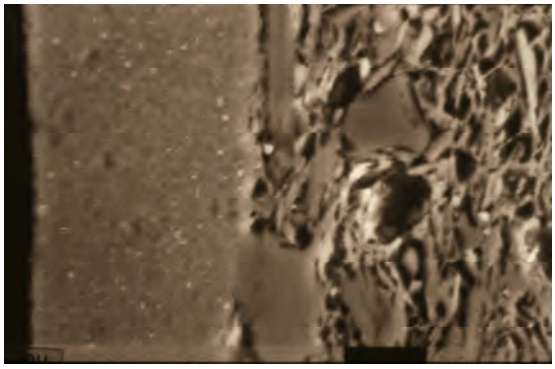


Figure 7

NABATAEAN POTTERY – HIGHLIGHTS

- Nabataean fine pottery produced from semi-calcareous clay and fired to 950°C
- Crystalline phases in Nabataean pottery increase rigidity and compressive strength
- Roman *terra sigillata* produced from fully-calcareous clay and fired to 1000-1100°C
- Roman *terra sigillata* stronger than Nabataean pottery, and better utilitarian ware

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Production technology of Nabataean painted pottery compared with that of Roman terra sigillata

Tite, M.; Herringer, S. N.; Shortland, Andrew J.; Matin, M.; Pradell, T. ...

2016-09-30
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https://doi.org/10.1016/j.jasrep.2016.09.010
Tite M, Herringer SN, Shortland A, et al., (2018) Production technology of Nabataean painted pottery compared with that of Roman terra sigillata. Journal of Archaeological Science: Reports, Volume 21, October 2018, Pages 1073-1078

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