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Lead-glazing technology from Medieval Central Asia: A case study from Aktobe, Kazakhstan

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ABSTRACT

A representative group of lead-glazed ceramics excavated from the Medieval city of Aktobe, in what is today southern Kazakhstan, was analyzed to reconstruct the production technology. Fifteen sherds, which date from the 9th–12th c. CE, were previously identified by neutron activation analysis as locally produced (Klesner et al., 2019). The ceramics, which represent four common Early Islamic wares (monochrome, underglaze painted, underglaze slip-painted, and opaque) were examined by scanning electron microscopy and electron microprobe analysis to establish the variability of local artisans' use of raw materials, glazing methods, and decorative techniques. Early Islamic ceramics are the first glazed wares produced in southern Kazakhstan, and through their technological reconstruction, we determined how this new ceramic technology was produced. We show evidence that the ceramics were introduced by skilled craftspeople who knew the production technology that was being used in Islamic centers in southwest and Central Asia. The ceramic technology differs, however, in the use of antimony as an opacifier in opaque glazes containing high-lead oxide. This research adds to the growing body of knowledge about glazing technologies in Central Asia and helps to define the technological and cultural ties present in the Early Islamic Period.

1. Introduction

Glazed ceramic production is an important and dynamic technology that can be easily traced due to the durability of the product. During the Early Islamic Period in southwest Asian and the eastern Mediterranean we find a period of increased innovation occurring in glazed ceramic technologies, as it was a significant element for economic competition in the long-distance trade networks (Mason, 1995). These ceramic technological innovations, especially tin-opacified opaque wares (Matin, 2019, Matin et al., 2018, Ting and Taxel, 2020, Tite et al., 2015, Watson, 2014) and lustre wares (Mason and Tite, 1997, Pradell et al., 2008a; Pradell et al., 2008b) have been examined to reconstruct their development and spread in the Early Islamic Period. In 8th, 9th, and 10th c., we see the evidence for widespread production and trade of lead-glazed ceramics across southwest Asia that extends into Central Asia. Central Asia has been the connecting bridge facilitating the long-distance trade of goods across Eurasia through the so-called Silk Road. While Central Asian communities have were trading centers, their craftspeople also produced specialty goods for elite consumption.

It has been assumed that the glazing technology in Central Asia was strictly derivative of western Islamic traditions, and potentially even directly introduced by immigrant craftsmen patronized by the emerging governing elites in eastern cities such as Nishapur, Merv, and Samarkand (Henshaw, 2010). An increase in research focused on the lead-glazed ceramics produced in the eastern Islamic world (Central Asia and Iran) from the 8th-12th c. CE has allowed a more thorough investigation of this assumption. This includes the recent projects on ceramics from the sites of Termez, Nishapur, Afrasiyab, Akhsiket, Tashkent, and Sirjan (Fig. 1) (Gradmann et al., 2015, Henshaw et al., 2006, Henshaw, 2010, Holakooei et al., 2019, Martínez Ferreras et al., 2019, Molera et al., 2020, Morgan and Leatherby, 1987). In southern Kazakhstan, where no local glazing tradition existed before the Early Islamic period (Heinsch et al., 2018, Henshaw, 2010), we sought to determine how this region began producing their own glazed ceramics after the introduction of nonlocal glazed ceramics imported through Silk Road trade. The primary aim of this paper is to reconstruct the production technology of Early Islamic period ceramics made at Aktobe, Kazakhstan, and to examine how the ceramics produced in this region compare to Islamic

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glazed ceramics from other sites in Central Asia and the southwestern Asia.

1.1. Aktobe

Aktobe is a large, fortified city that lies in the Chu River valley between the Serhau, Aksu, Kara-Balta, and Toktas rivers just northeast of the modern Kazakhstan-Kyrgyzstan border (Figs. 1 and 2). The city is located along the northern edge of the Tien Shen mountain range in the oasis region of Central Asia, which stretches along the foot of the mountain ranges and encompasses the river valleys which bring much needed water from the mountains into the deserts and steppe. A major artery of the Silk Road passed by the city during the Early Islamic Period. Aktobe was occupied from the 6th c. and through the early 13th c. CE (Akymbek and Baibugunov, 2014) when Turkic states were centred in the Chu-Talas region. The site, which has been excavated by archaeologists from the Al-Farabi Kazakh National University and the A.Kh. Margulan Institute of Archaeology has been proposed to be the location of the Medieval city of Balasagun (Akymbek and Baibugunov, 2014, Shalekenov, 2009, p. 52).

>40 years of excavations in four main areas of the city have been investigated. These consist of the citadel, two shahristans (lower cities), and the rabad (suburbs and agricultural area) which lie within the larger defensive walls. The citadel occupies the highest location of the site and has an area of 1 ha. It sits inside of Shahristan I, which is rectangular in shape and is roughly 380 × 250 m in extent. There are two entryways into this Shahristan, and the area is surrounded by defined walls. Shahristan II is located to the southeast of Shahristan I, and is slightly smaller, only 300 × 250 m. The agricultural area in the rabad is extensive and is located within a radius of two to seven km of the citadel. This rabad is surrounded by two defensive walls; the external wall is 20.5 km in circumference while the inner wall is 14.7 km. Within the site of Aktobe the remains of a palace, bath, minaret, urban domestic quarters, winemaking workshops, pottery production area, agricultural homesteads, sewage systems, potable water supply system, and irrigation ditches have been excavated. The archaeological research suggest that the city was one of the largest political and economic centers in the region during the Turkic Khaganate (552–603 CE), the Western Turkic Khaganate (603–704 CE), Turgesh Khaganate (704–756 CE), Karluk Khaganate (756–940 CE), and the Karakhanid Khaganate (942–1212 CE) (Shalekenov, 2006). The city appears to have been abandoned in the first half of the 13th c., although no fire or other destructive debris is present to suggest it was the result of a violent destruction.

2. Materials and methods

2.1. Materials

Compositional study of 9-15th c. CE ceramics from seven Silk Road sites in southern Kazakhstan identified a group of lead-glazed ceramics as likely produced at Aktobe based on their paste composition (Klesner et al., 2019). The locally produced glazed ceramics were dated from the 9th-12th c. CE and have a high-lead oxide glaze in a variety of wares including monochrome and underglaze wares. In this group are ceramics dated to two periods based on the ceramic styles and excavation contexts. Ceramics dated from the 9th – 10th c. CE were excavated from a residential building east of the citadel in Shahristan I. Ceramics dating from the 11th – 12th c. CE were excavated in 2009 from the foundations of the minaret located within the citadel. ^{14}C dating of charcoal remains found in context with the ceramics from the minaret provided a date for the construction of 1015 – 1030 CE (68% probability, Table 1).

In 2017–2018 two kilns were uncovered approximately 500 m west of the citadel, one of which has evidence of glazed ceramic production, confirming the presence of locally produced glazed ceramics in the Early Islamic Period at Aktobe (Fig. 2). Ceramic sherds excavated from around the glaze kiln, concentrated in a pit 50 cm to the west, were included in this compositional and technological analysis. Further compositional analysis on an additional 94 ceramics from southern Kazakhstan

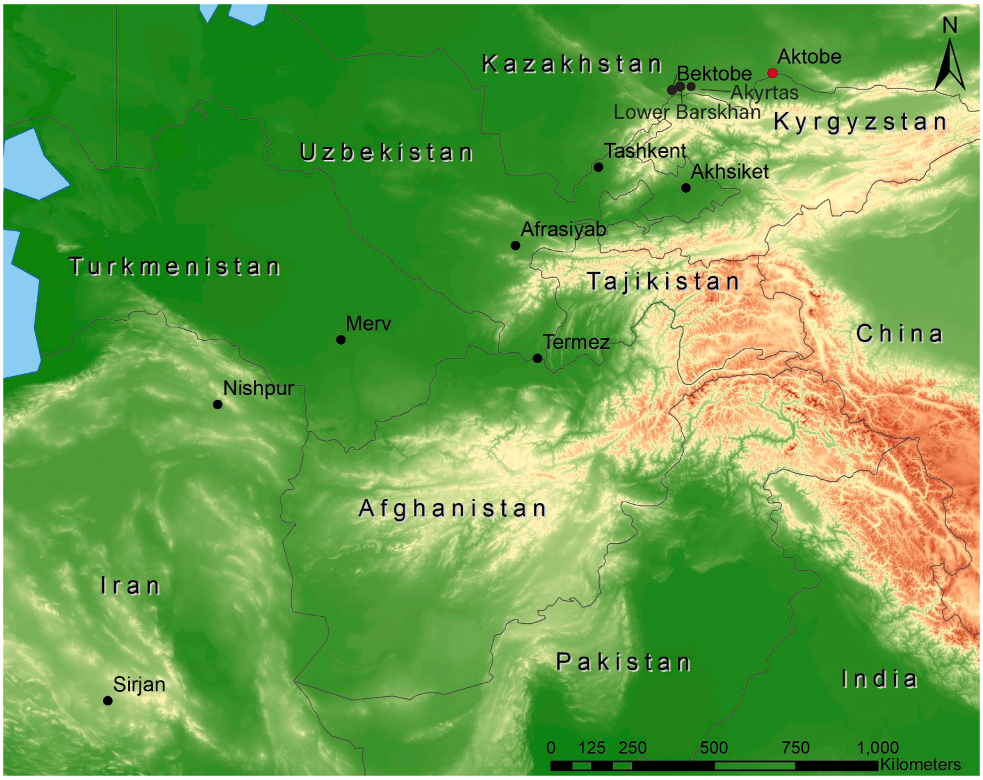

Fig. 1. Map showing the location of Aktobe, Kazakhstan (indicated in red) and the other sites in Central Asia that are mentioned in the text. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

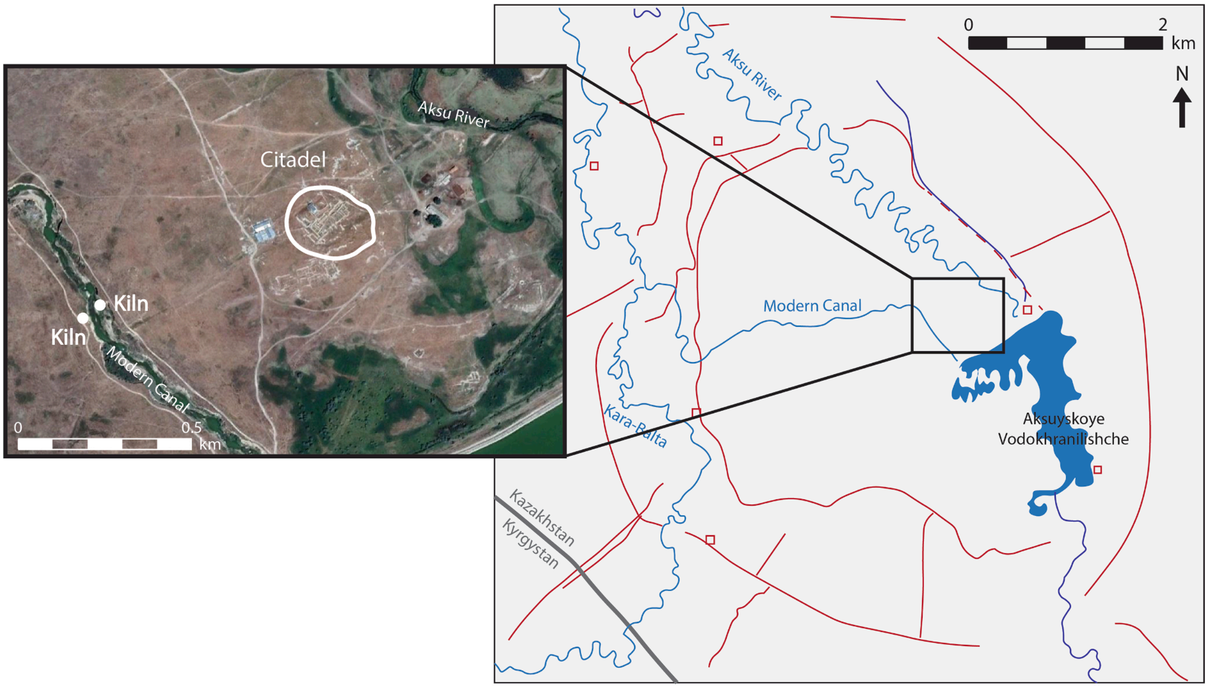

Fig. 2. Plan of Aktobe and the location of the ceramic production center. Blue lines indicate waterways while red lines indicate fortification walls (after Akymbek 2015). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 1
Radiocarbon date from the minaret at Aktobe, Kazakhstan. Accelerator Mass Spectrometry (AMS) analysis was conducted at the Beta Analytic Radiocarbon Dating Laboratory.

ANID	Material	Lab ID	δ^{13}C (°/$_{oo}$)	^{14}C Age (year B.P.)	Calibrated age	Probability
Aktobe 2 (minaret)	Charcoal	Beta-433905	−25.1	1000 ± 30	CE 1015–1030	68%

identified four sherds excavated from other cities in the region, Bektobe, Lower Barskhan, and Akyrtas, that fall into the locally produced Aktobe compositional group, indicating the occurrence of regional trade of ceramics produced at Aktobe.

Fifteen of the local glazed ceramics (K047, K048, K050, K069, K070, K071, K077, K099, K193, K198, K199, K200, K201, K203, and K212) were chosen for this technological study (see Fig. 3). They come from well contextualized excavation contexts and are representative of the larger ceramic assemblage. The earthenware ceramics have a reddish yellow (5YR 6/6) to red (2.5YR 5/6) body, with the exception of K077, which has a grayish brown (10YR 5/2) body. Four of the five samples that were excavated from the ceramic production area are visibly overfired and have a reduced ceramic fabric ranging from reddish brown (2.5YR 5/4) to dark reddish gray (10R 4/1) in color. All the ceramics have a transparent high-lead glaze, with the exception of K212, which has an opacified lead glaze. The ceramics in this study are fragmentary, which allows in most cases for only general assignments of ware. The sherds are too small to assign a specific shape, although most are from open vessels, bowls or plates, and are glazed solely on the interiors. Based on the decoration and application we assigned six of the samples as monochrome glazed ware (K047, K050, K077, K193, K198, and K200), six as underglaze painted ware (K048, K071, K099, K199, K201, and K203), and two as underglaze slip-painted ware (K069 and K070). Incising of linear decoration was employed on some of the ceramics (K070, K201). These common ceramic styles were present throughout the Islamic world during this period. K212 is an opacified glazed ware, and the glaze is pale yellow in color (5Y 8/4). Yellow opaque ceramics are less common in this period and in Central Asia. We do not have examples of other common Islamic wares in the locally produced groups, specifically white tin-opacified wares and lustre-ware. While these two wares were common throughout the western Islamic world, they occur less frequently in Central Asia, and their absence is not unexpected for the local ceramic production group (Henshaw, 2010).

2.2. *Analytical methods*

To reconstruct the production technology of the Central Asian Early Islamic lead-glazed ceramics, we characterized the composition and microstructure of the ceramic slips, glazes, and colorants for a representative collection of wares present in the archaeological assemblage. Sherds were first observed under low magnification (8-35x) by binocular optical microscopy (OM) using a Leica EZ4 HD microscope with integral digital camera. To examine the composition and microstructure of the samples, polished thin sections and embedded and polished samples were prepared and further examined by OM, scanning electron microscopy (SEM) and electron microprobe analysis (EPMA).

Backscatter electron (BSE) images focused on the glaze, glaze-slip, and slip-body interaction zones were recorded at magnifications from 40× to 10,000× to investigate the structural variation of the ceramics. BSE images on polished sections highlight several features of glazed ceramics including the homogeneity of the glaze, the size and distribution of particles and inclusions in the glaze, the nature of the glaze-slip interaction zone, the presence of bubbles and crazing in the glaze, and the extent of weathering. These analyses were conducted on a TM4000 Plus benchtop SEM (University of Arizona) operated at 15 kV accelerated voltage.

Compositional information for the glaze and slips was obtained from polished thin-sections and embedded and polished samples using a CAMECA SX100 Ultra electron probe microanalyzer (EPMA) with 5 spectrometers. Wavelength-dispersive Spectroscopy (WDS) data was

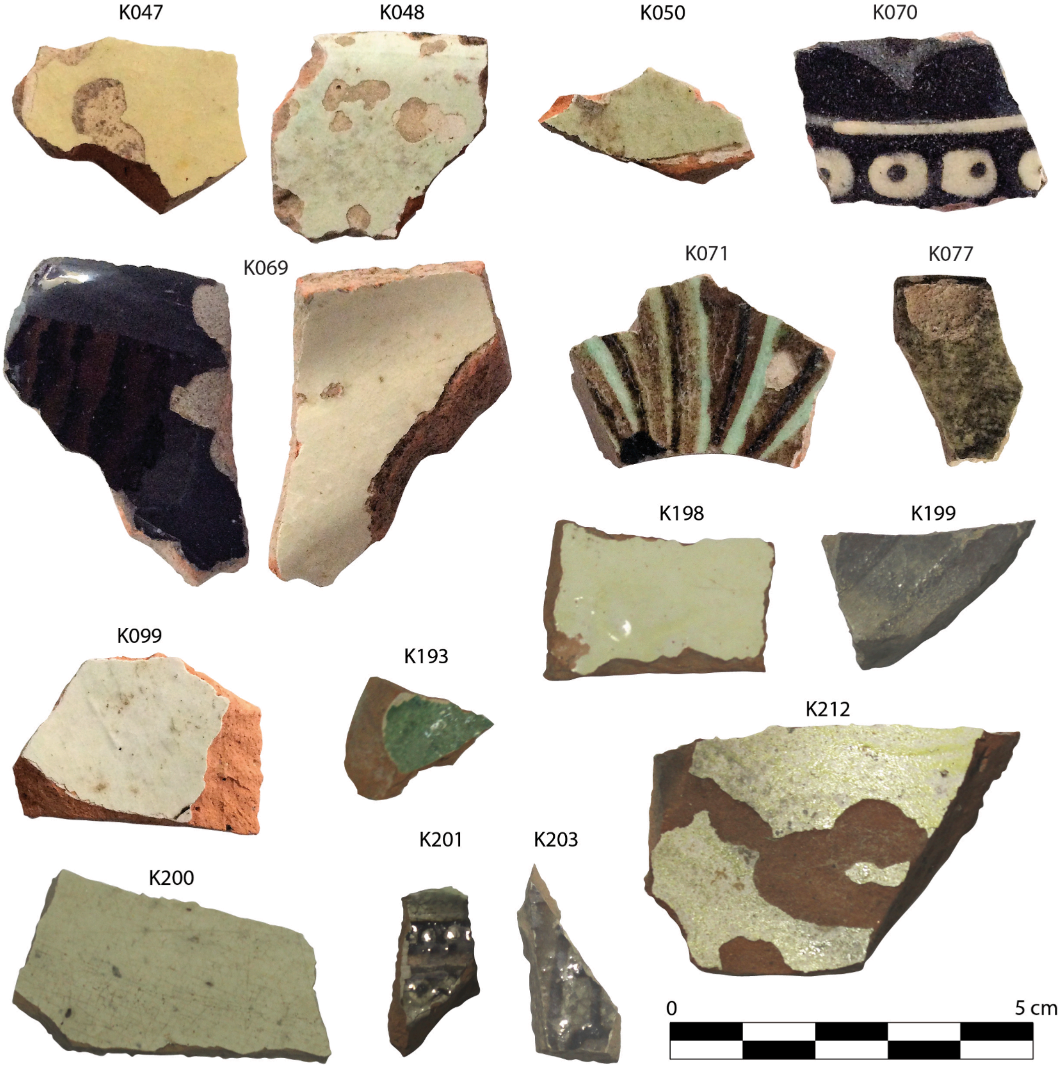

Fig. 3. Glazed ceramics analyzed from Shahristan I and the minaret at Aktobe (K047, K048, K050, K069, K070, K071, and K077), the ceramic production center at Aktobe (K198, K199, K200, K201, and K203), and ceramics recovered at neighboring sites identified as Aktobe products (K099, K193, and K212).

obtained for 16 elements: Si, Na, Mg, Al, P, K, Ca, Sb, Sn, Mn, Fe, Cu, Pb, Co, Mo, Ti, and Cr. The elements were calibrated with the following standards chosen and maintained by K. Dominik in the Michael J. Drake Electron Microprobe Laboratory at the University of Arizona: Crete albite from University of Washington, orthoclase OR1 from Penn State, San Carlos Olivine, Hakone anorthite, NIST SRM K0229 Lead Silicate glass, Rockport fayalite, rhodonite 104,791 from University of Washington, natural cuprite, Spex Inc. 99.999% Co metal, natural cassiterite, natural stibnite, Smithsonian Chromite USNM 117075, rutile, and synthetic apatite. Instrumental parameters included an accelerating voltage of 15 keV, 8nA for alkalis (Na and K), which were counted first, and 40nA for all other elements, and a spot size of 1 or 10 μm. The 10 μm beam was the default setting, but for compositions on small particles or near the exterior of the glazed region a 1 μm beam was employed. Wherever possible at least five WDS collections were obtained for each region of interest on each sample, which is consistent with the methods employed by other researchers on Islamic glazes (Matin et al., 2018, Ting and Taxel, 2020, Tite et al., 2015). Additionally, WDS maps of the distribution of Si, Al, Fe, Mn, and Pb were collected for cross-sections of the black glaze on samples K069 and K070, and Si, Al, Pb, Sn, and Sb were collected for cross-sections of the opaque yellow glazed on sample K212.

Theoretical firing temperatures were determined for eight of the samples using the Fulcher-Tammann equation as described by Lakatos et al. (1972) based on the composition of the glaze as determined by laser-ablation inductively coupled plasma mass-spectrometry (LA-ICP-MS) and reported in Klesner et al. (2019). Compositions determined by LA-ICP-MS where chosen for this analysis because the LA-ICP-MS technique produces concentrations of a larger number of elements important in determining firing temperature (SiO_2, Na_2O, MgO, Al_2O_3, K_2O, CaO, PbO, BaO, Li_2O, and ZnO) than does data from EPMA WDS. The calculations assume a processing viscosity of 10^4P, which is characteristic for the firing of a glaze as reported by Hamer and Hamer (1997) and assumed by Tite et al. (1998) in their study of methods of lead glaze production.

3. Results

3.1. Slip

The general structure of the ceramics consists of a ceramic body, on which a slip is applied and then covered with a glaze high in lead oxide, typically containing over 35% PbO by weight (Tite, 2011). This can be seen in backscatter images of all fourteen of the transparent glazed ceramics in Fig. 4. A white slip, also termed engobe, was applied over the ceramic on all of the samples, while a colored slip was additionally used in regions of two of the samples that were underglaze slip-painted (K069, K070). The slip has a thickness ranging from 10 to 190 μm

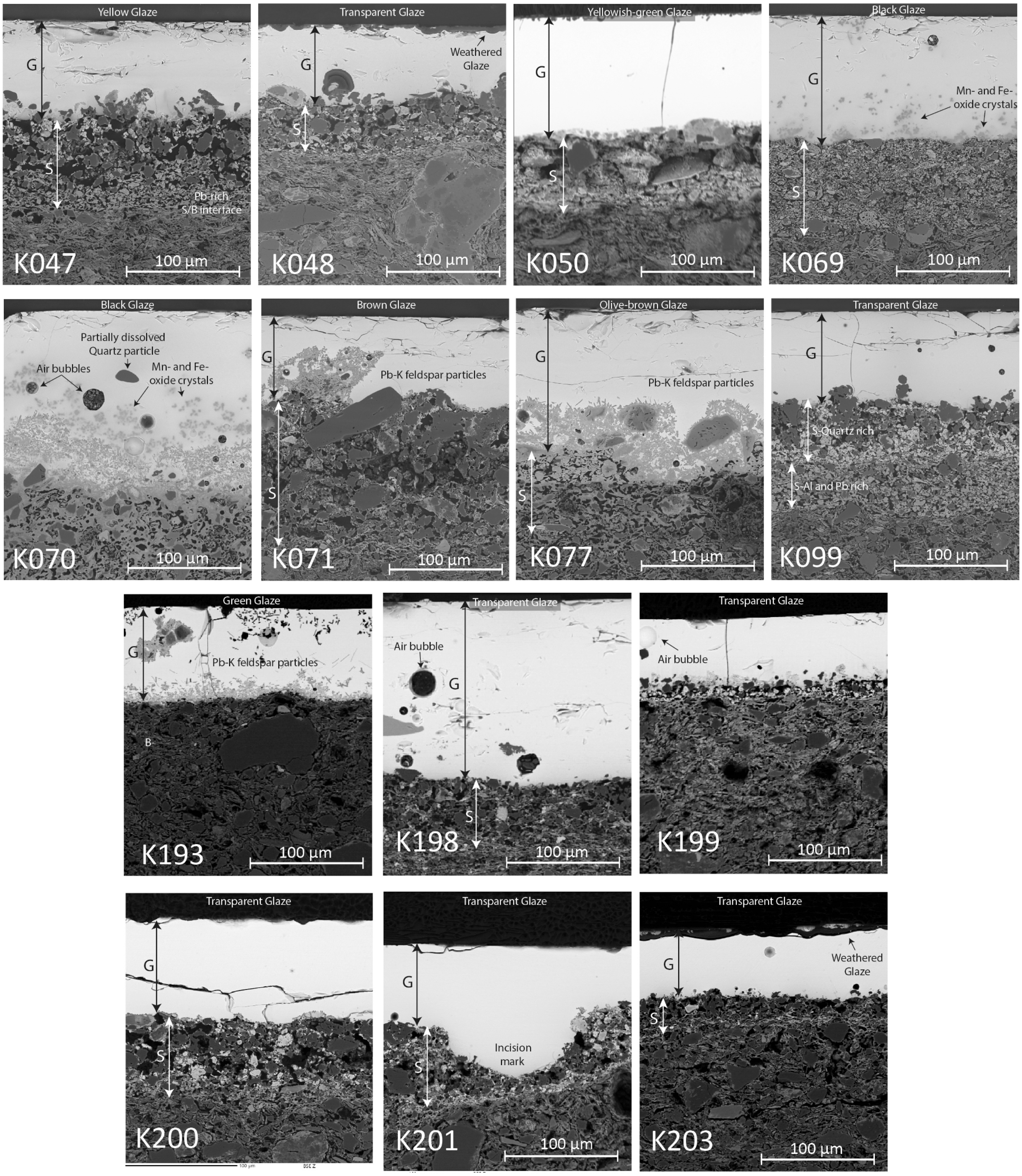

Fig. 4. Backscattered electron (BSE) images of the glaze-slip-body cross sections of the transparent lead-glazed samples in this study scaled for direct comparison. The sample number is indicated in the lower left corner of each image. G = Glaze, S = Slip, B = Body.

(Table 2), although a thickness of 100 µm or less is recorded for the slips on twelve of the fifteen samples. Two of the samples (K193 and K199) have a very thin and fragmentary slip. In all but four examples (K071, K099, K201, and K212) the slip is thinner than the overlaying glaze.

The white slip is generally distinct from the underlying body in both microstructure and composition. The slip has a range of particle sizes, including large particles (>30 µm) (Fig. 4). The ceramic body was made of a low-calcareous iron rich clay, which has an average composition of 61.9 wt% SiO_2, 17.3 wt% Al_2O_3, 6.3 wt% Fe_2O_3, 5.2 wt% CaO, 3.8 wt% K_2O, 2.9 wt% MgO, 1.2 wt% Na_2O, and less than 1 wt% P_2O_5, MnO, TiO_2, and Cl. Based on the bulk compositions determined by EPMA WDS (Table 3), the slips are primarily a mixture of an alumina-rich clay with added quartz. The clay used for the slip has very little iron present, averaging 1.03 wt% Fe_2O_3 compared to the 6.3 wt% in the underlying ceramic body. One sample, K047, has a higher concentration of calcium in the slip, 8.56 wt%, which suggests the use of a calcareous clay. A significant amount of PbO is present in the slips, ranging from an average of 10.90 to 24.60 wt%. The PbO is distributed throughout the entire depth of the slip, and there is also observed an accumulation of lead rich particles on the slip-body interface (Fig. 4). We see minimal particle growth on the slip-glaze interface for the white slips covered in a transparent glaze. This suggests that the slip was applied and fired onto

Table 2

Properties of fifteen lead-glazed ceramics from Kazakhstan. Glaze composition it reported in oxide weight %.

ID	Site	Date (c. CE)	Ware	EFT (°C)	Glaze color	Slip (µm)	Glaze (µm)	Color-ant	Na_2O	K_2O	MgO	Al_2O_3	SiO_2	PbO	CaO	Fe_2O_3	MnO	CuO	SnO_2	Sb_2O_5	P_2O_5	TiO_2	Total
K047	Aktobe	9-10th	M	935	Y	60	80	–	0.21	0.73	0.36	4.35	42.41	50.42	0.84	0.60	0.00	0.05	b.d.	0.24	0.14	–	100.30
K048	Aktobe	9-10th	UG	920	T	60	70	–	0.18	0.59	0.21	2.77	37.90	55.19	2.33	0.39	0.01	0.16	b.d.	–	0.08	–	99.83
K050	Aktobe	9-10th	M	880	Y/G	60	115	–	0.06	0.07	0.12	2.11	27.66	64.81	1.09	0.27	0.01	0.11	b.d.	0.29	–	–	96.61
K069	Aktobe	11-12th	SP	945	Bl	80	120	Fe, Mn	0.29	0.81	0.62	3.63	36.75	47.19	1.99	4.42*	2.58	0.02	b.d.	b.d.	0.14	–	98.46
			UG		T	110	80	–	0.30	0.70	0.62	3.34	40.78	51.21	1.83	0.45	0.03	0.05	b.d.	b.d.	0.17	–	99.46
K070	Aktobe	11-12th	SP	985	Bl	Up to 150	90–230	Fe, Mn	0.27	1.16	0.50	4.78	41.97	44.96	1.07	3.32*	1.70	0.03	b.d.	b.d.	0.14	–	99.91
			UGI		T			–	0.20	0.83	0.48	4.10	45.14	48.10	0.87	0.48	0.09	0.01	b.d.	b.d.	0.08	–	100.37
K071	Aktobe	11-12th	UG	930	Br	190	80	Fe	0.24	0.84	0.38	5.08	35.21	54.89	0.72	1.32	0.06	0.48	b.d.	–	0.09	–	99.36
					T			–	0.24	0.79	0.38	4.72	35.92	54.59	0.79	1.12	0.07	0.48	b.d.	–	0.09	0.19	99.39
K077	Aktobe	11-12th	M	835	Ol/Br	50–80	100–130	Clay	0.55	1.11	0.46	7.63	35.48	49.88	1.89	0.72	0.03	0.07	b.d.	–	0.18	0.30	98.31
K099	Bektobe	11-12th	UG	1020	T	100	85	–	0.17	0.58	0.50	4.53	43.68	47.98	0.96	0.34	0.02	0.05	b.d.	b.d.	0.07	0.11	99.03
K193	Akyrtas	11-12th	M	–	G	0–10	85	Cu	0.24	0.38	0.31	5.65	27.84	62.70	0.38	0.54	0.00	0.78	b.d.	b.d.	0.12	0.13	99.13
K198	Aktobe-Kiln	10-12th	M	–	T	30–50	80	–	0.25	0.49	0.61	2.95	35.94	55.82	2.38	0.41	0.01	0.08	b.d.	–	0.11	0.12	99.20
K199	Aktobe-Kiln	10-12th	UG	–	Br	0–20	120	Fe	0.32	0.60	0.60	4.72	32.11	54.52	1.25	3.85	0.55	0.05	b.d.	0.16	0.07	0.21	99.03
					T	20	50	–	0.29	0.50	0.39	4.11	37.34	54.91	0.80	0.51	0.01	0.03	b.d.	0.18	0.05	0.12	99.25
K200	Aktobe-Kiln	10-12th	M	–	T	70	90	–	0.26	0.66	0.19	3.50	33.34	60.46	0.50	0.25	0.00	0.12	b.d.	b.d.	0.05	0.13	99.49
K201	Aktobe-Kiln	10-12th	UGI	–	T	120	90	–	0.17	0.47	0.24	3.51	34.65	59.24	0.82	0.30	0.02	0.09	b.d.	–	0.06	0.14	99.72
K203	Aktobe-Kiln	10-12th	UG	–	T	30–50	60–75	–	0.33	0.98	0.35	3.41	37.37	55.66	0.74	0.46	0.02	0.10	b.d.	b.d.	0.09	0.20	99.79
K212	Lower Barskhan	11-12th	O	–	Y	80	10–50	Sn, Sb	0.22	0.85	0.69	4.70	43.02	44.79	1.87	0.49	0.01	0.19	0.85	0.39	0.11	0.25	98.30

EFT = Estimated firing temperature, M = Monochrome, UG = underglaze, SP = Slip-painted, UGI = underglaze with incision, O = opaque, Y = Yellow, Y/G = Yellowish-green, G = Green, Bl = Black, Br = Brown, Ol/Br = Olive-brown, T = transparent glaze, b.d. = below detection. A dash (-) indicates the element was not analyzed.

Note: *FeO is reported for the Black Glazes.

Table 3
Average (m), standard deviation (σ), and normalized average compositions of the white slips (n = 10) and black slip (n = 1) on the locally produced ceramics as determined by WDS. The low totals are indicative of porous slips. *FeO is reported for the black slip.

Ox. Wt %	White slip			Black slip		
	m	σ	Normalized m	m	σ	Normalized m
Na_2O	0.57	0.22	0.68	0.56	0.38	0.61
K_2O	2.45	0.75	2.95	1.95	1.75	2.16
MgO	0.79	0.85	0.95	0.55	0.15	0.61
Al_2O_3	14.02	3.26	16.88	7.00	4.40	7.75
SiO_2	45.23	8.39	54.46	25.67	23.79	28.43
PbO	16.63	4.43	20.03	6.64	5.70	7.35
CaO	1.68	2.09	2.02	0.55	0.24	0.61
Fe_2O_3	1.03	0.86	1.24	33.02*	22.57*	36.58*
MnO	0.02	0.02	0.02	13.91	12.35	15.41
CuO	0.02	0.02	0.02	0.15	0.18	0.17
P_2O_5	0.25	0.31	0.30	0.01	0.01	0.01
TiO_2	0.36	0.44	0.43	0.25	0.21	0.28
Total	82.96	6.33	100.00	90.27	6.66	100.00

the ceramic before the application of the overlying glaze.

The high amount of PbO in the slip has been suggested by Molera et al. (2020) to have been intentionally added into the slip as a component, since it is too high of a concentration to solely be the result of glaze-slip interaction during the forming and firing process. Ting and Taxel (2020) similarly found high concentrations of PbO (14.2–23.0 wt %) in the white slips on 19 of the 20 samples of Early Islamic Coptic Glazed Ware, while Henshaw et al. (2006) found up to 13 wt% PbO in the white slips from Akhsiket. Molera et al. (2020) suggests the addition of PbO in the slip may be to better adhere the slip to the body. The added lead in the slip additionally serves as an intermediary helping the glaze fit better with the underlying ceramic body. For a good glaze fit, the glaze needs to have a smaller thermal expansion coefficient than the ceramic body (Nordyke, 1984), typically 5–15% less than the body (Tite et al., 1998). By adding lead oxide into the slip layer, the composition of the slip will have a thermal expansion coefficient between that of the high lead glaze and the underlying ceramic body and thus helps the glaze adhere to the body.

Black slips are seen in samples K069 and K070. In both cases the black slip is not applied over the white slip, but it is applied directly to the ceramic body in areas where there is intended black decoration. The black slip can be seen clearly in the mapping of the distribution of Pb, Si, Mn, Al, and Fe for both K069 and K070 (Fig. 5). The black slip has a significantly smaller average particle size than the white slips, as can be seen in Fig. 6. The composition of the black slip for K070 is shown in Table 2. The amount of FeO in the slip (33.02 wt%) suggests that iron was added to the slip as a separate metal oxide pigment not an iron-rich clay. MnO (13.91 wt%) is also added to the slip as a pigment to create the deep black color. We do see an increase in the interaction between the black slip and the glaze compared to the white slip, with micron sized particles of FeO and MnO rising up from the slip into the more fluid glaze (Fig. 6). Identification of the mineral phases of the Fe- and Mn-oxides will require the use of X-ray diffraction and is an area of future study.

3.2. *Glaze*

The glaze layer ranges from 50 to 230 μm in thickness, although most samples have thicknesses between 80 and 110 μm. All the transparent glazes are homogeneous with few undissolved quartz grains or other inclusions present. For transparent glazes on a white slip, we found no appreciable particle growth at the slip-glaze interface. As discussed above, for the black glazes we did find some particles of Mn- and Fe-oxide extending into the glaze from the underlying black slip layers. For samples with an underlying painted decoration (discussed below) there is particle growth extending 10–40 μm from the painted layer into the glaze (Fig. 4). The composition of the particles indicates they are a lead-potassium feldspar with an average composition of 3.63 wt% K_2O, 22.86 wt% Al_2O_3, 40.79 wt% SiO_2, and 29.67 wt% PbO.

When we compare the composition of the glaze after the colorants are removed and renormalized, we see that it is almost exclusively composed of PbO and SiO_2 (Table 4), with only minor amounts of Al_2O_3 (4.36 wt%), CaO (1.19 wt%) and K_2O (0.71 wt%). The glaze is made of a mixture of PbO and SiO_2 and not as a straight application of PbO. This is determined by comparing the ratio of SiO_2 in the slip and in the glaze (Fig. 7). The significantly higher ration of silica in the glaze compared to the underlying slip, and the depleted aluminum content indicates that the potters were mixing SiO_2 with the lead oxide to create the clear glaze. The glazes themselves do not show extensive weathering and corrosion, with the exception of K048 and K203 which do display weathering on the surface and along crazing lines, averaging 3–5 μm in depth and extending up to 10 μm. Other glazes with more PbO do not show surface weathering, this weathering of K048 and K203 is probably due to wetter local burial conditions.

3.3. *Decorative techniques*

Colors were achieved in the glazed ceramics by two main techniques that are common in Early Islamic Glazed ceramics. The first method of introducing color was used on the underglaze ceramics and is achieved is by applying the color decoration as a paint or slip-paint on an unglazed ceramic and then coating it with a transparent lead glaze. Both the paints and the slip-paints were colored using metal oxides. Black color was achieved by a mixture of MnO and FeO oxide, 3.84 wt% and 2.48 wt% respectively, for the paint on K071, and 33.02 wt% and 13.91 wt% respectively in the slip-paint on K070. Brown was achieved by an iron-rich paint, 3.92 wt% Fe_2O_3 for K048, while olive-brown was colored by both Fe_2O_3 and CuO, 1.32 and 0.48 wt% respectively for K071. The slip-painted ceramics (K069 and K070), as discussed above, have the colorant mixed with clay and applied as a thick (~100 μm) slip directly on the ceramic body. The underglaze painted ceramics have a colored paint applied over the white slip in a thin layer. The paints appear to be applied either solely as the metal oxide pigment suspended in water, or with a small amount of clay. The other primary method of achieving colored decoration is seen in the green (K193) monochrome ceramic, which has the colorant in solution. The most common monochrome glaze color in the Early Islamic Period in Central Asia is green, which is achieved by having Cu^{2+} in solution. K193 has 0.78 wt% CuO in the glaze which gives it the distinct green color.

Four of the samples analyzed had measurable amounts of antimony in the glaze when analyzed by EPMA. When antimony is present in glass it serves either as a yellow opacifier (>1 wt% Sb) in the form of lead-antimonate ($Pb_2Sb_2O_7$) particles, or in low concentrations, antimony (~0.5 wt% Sb) functions as a decoloriser (Dillis et al., 2019). Two of the ceramics, K047 and K050, have very similar concentrations of antimony, 0.24 wt% Sb_2O_5 for K047 and 0.29 wt% Sb_2O_5 for K050. The antimony in the transparent glazes is in solution. No visible lead-antimonate particles are present either throughout the glaze or concentrated at the glaze-slip interface. The low amount of antimony in these glazes is likely the result of contamination in the original lead source. A third transparent glaze, K199, has both the brown and transparent glaze sections contain minor amounts of Sb_2O_5, 0.18 wt% and 0.16 wt% respectively. The brown glaze is colored by iron (3.85 wt% Fe_2O_3) and manganese oxides (0.55 wt% MnO) as seen in the other locally produced brown and black glazes. However, in the brown glaze a small number of particles are suspended in the glaze layer (Fig. 8) which have an average composition of 54.89 wt% Fe_2O_3, 24.80 wt% PbO, 5.68 wt% SiO_2, 5.05 wt% MnO, and 3.30 wt% Sb_2O_5. The particles are thin rods ranging from sub-micron to 10 μm in length. This glaze is transparent despite the particles throughout the glaze, and this is likely due to an insufficient number of particles present to effectively scatter or absorb the light

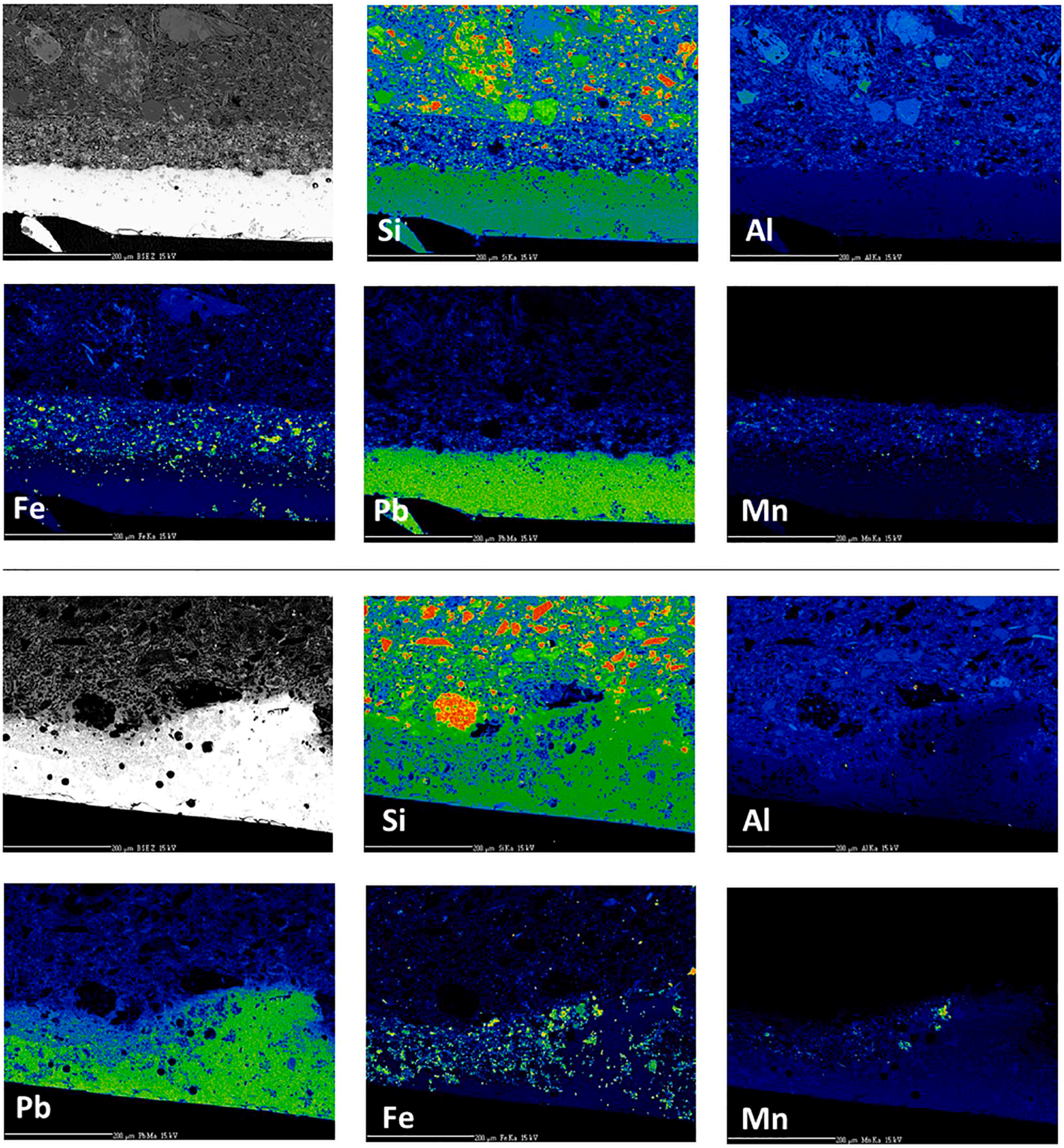

Fig. 5. Electron microprobe elemental mapping of the black glaze on K069 (*above*) and K070 (*below*) showing the distribution of Si, Al, Fe, Pb, and Mn across the glaze-slip-body cross section of each sample.

creating an opaque glaze. The presence of antimony in this sample is thus likely an impurity in the brown colorant.

The opaque yellow glazed ceramic, K212, also has an antimony-based opacifier. As shown in Fig. 8, the glaze is thin and varies in thickness from 10 to 50 μm. Distributed throughout the glaze are micron and sub-micron sized particles that are composed of SnO_2 and Sb_2O_3 (Fig. 9), with an average ratio of 2:1, respectively. In K212 the number of Sn and Sb particles suspended throughout the glaze both act as opacifiers.

3.4. Firing

These earthenware ceramics have a fairly soft body, and minimal vitrification in the ceramic paste as observed by electron microscopy. The theoretical firing temperature for eight of the ceramics was determined to be 935 ± 55 °C using the Fulcher-Tammann equation. We see no appreciable change in firing temperature across the two periods of study, with the average firing temperature for the 9th–10th c. CE sherds being 915 ± 35 °C, and for the later period 945 ± 65 °C. These temperatures fall in the expected range for a high lead-glazed earthenware, typically 900–1050 °C (Pradell and Molera, 2020) similar to what has been reported for other Islamic ceramics. Tite et al. (1998) calculated that the temperature range for high lead glaze (45–60 wt% PbO) was 820–1030 °C. Martínez Ferreras et al. (2019) estimated that the firing temperature for locally produced glazed wares from Termez from the same period was between 800 and 1100 °C based on XRD identification of mineral phases in the ceramic body.

The glazes have only minor amounts of bubbles trapped in the glaze, indicating that the ceramics were fired for long enough at peak temperature for the bubbles to escape. Minimal crazing of the Aktobe glazes

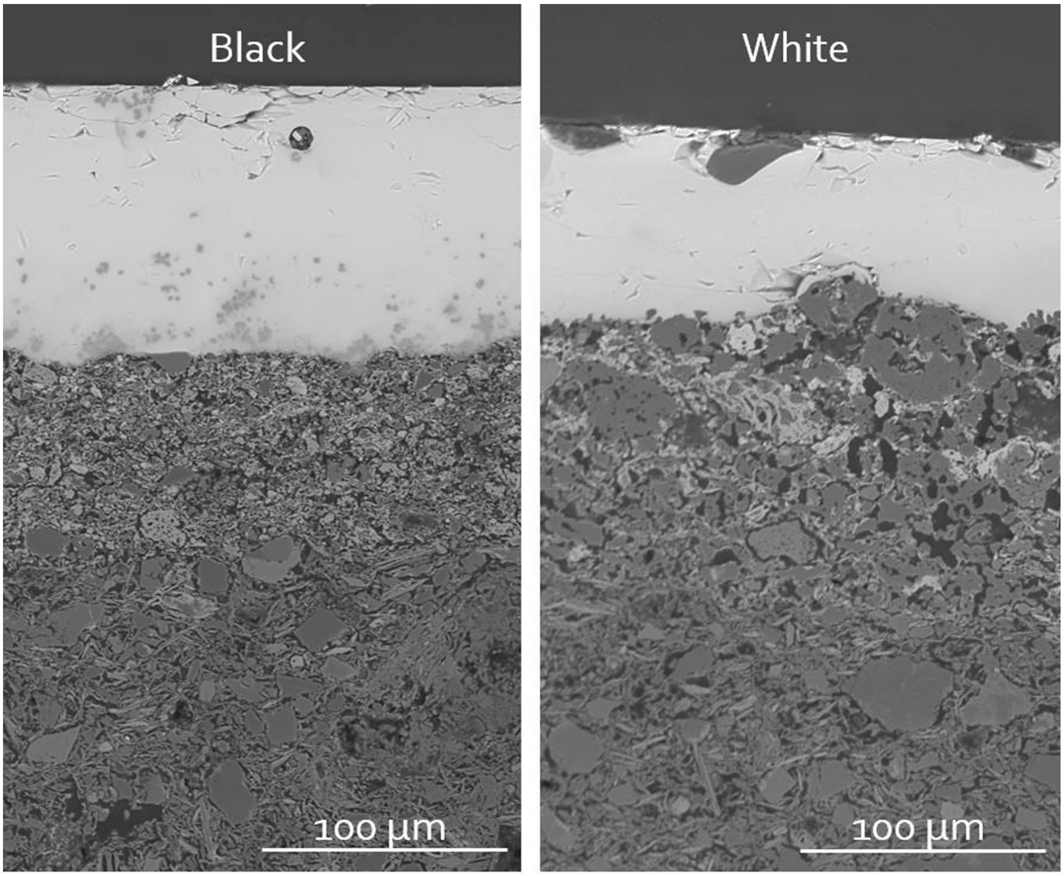

Fig. 6. SEM backscatter images of the glaze-slip-body interface for the black (*left*) and white (*right*) areas on sample K069. The images are scaled for direct comparison of the slip microstructures.

Table 4
Mean (m) and standard deviation (σ) of lead glaze compositions in this study as determined by EPMA compared to published studies. All data is normalized to 100% after removing colorants.

Oxide	Lead glaze composition from Early Islamic ceramics in southern Kazakhstan (n=14)		9-11th c. CE lead glazes from ancient Termez (southern Uzbekistan) reported by Molera et al. (2020)		Colorless glaze (n=5) on Early Islamic ceramics from Akhsiket, Uzbekistan reported by Henshaw et al. (2006)	Al-Raqqa, Syria, 8th-9th c. CE (n=2) reported by Shen (2017)		Kish-shaal Ghazna, Iraq, 10th-13th c. CE (n=23) reported by Shen (2017)		Kish or Hira, Iraq, 8th-14th c. CE, maybe later than 10th c. CE (n=10) reported by Shen (2017)	
	m	σ	m	σ	m	m	σ	m	σ	m	σ
SiO_2	37.44	4.88	39.8	3.9	42.2	39.28	0.49	33.74	4.60	32.73	4.72
Na_2O	0.26	0.10	0.9	0.3	0.6	0.58	0.26	0.75	0.41	0.98	0.77
MgO	0.41	0.15	0.5	0.2	-	0.64	0.15	0.61	0.46	0.53	0.41
Al_2O_3	4.36	1.35	2.7	1.0	2.7	1.60	0.62	2.80	1.34	2.06	1.11
P_2O_5	0.11	0.04	-	-	-	-	-	-	-	-	-
K_2O	0.71	0.26	1.0	0.7	2.0	0.56	0.28	1.10	0.74	0.80	0.37
CaO	1.19	0.61	1.7	0.6	0.8	2.54	0.19	1.81	0.61	1.75	0.76
PbO	55.53	5.34	53.7	5.1	51.7	54.79	1.46	59.19	6.64	61.13	6.44

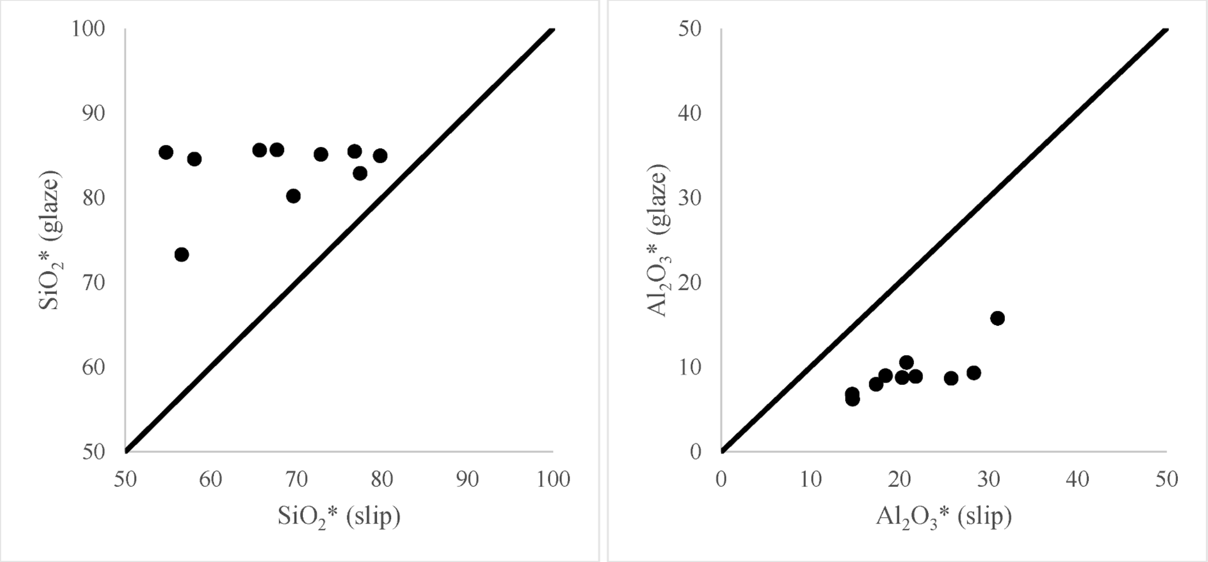

Fig. 7. Comparison between the normalized concentration (reported in ox. wt%) of SiO_2*(*left*) and Al_2O_3* (*right*) in the glaze and the slip of the samples after subtracting PbO.

indicates a reasonable glaze fit to the ceramic body.

4. Discussion

4.1. Reconstructing the ceramic production technology

Synthesizing the results, we were able to reconstruct the production processes of the Aktobe ceramics based on the composition and microstructural variation of the glazes and slips and the choice and application methods of colorants. Comparing analyses of the Aktobe sherds to the larger assemblage of excavated sherds we were able to assess manufacturing sequences and gain insights into firing practices. Based on previous analyses of the samples (Klesner et al., 2019), Aktobe ceramics were made using a local clay source. Once a vessel was formed, a thin slip was applied on the interior of the vessel. For the monochrome and underglaze painted wares, a white slip composed of white clay, quartz, and added of lead flux (10.90–24.60 wt% PbO), was applied over the entire decorated area. The use of a white slip over a red body is a characteristic feature of Islamic potters, attested as a novel invention in the Early Islamic period (Mason, 2004). By covering the red body with white slip, the craftspeople created a white background on which colored decoration was applied, that often mimics the fashionable imported porcelain from China (Henshaw, 2010). When we compare the slip composition (Table 3) to other Central Asian Early Islamic ceramics, we see some differences. At Akhsiket, Henshaw (2010) observed a higher concentration of silica (60–75% range) and less clay used in the slip. In Termez, the slip is alumina rich, but calcia poor clay (Molera et al., 2020) compared to the ceramic body. While most of the ceramics have a low concentration of CaO and MgO indicating an alumina rich non-calcareous clay was used, one sample (K047) has a relatively high concentration of CaO (8.56 wt%) and MgO (3.94 wt%) which could indicate that the artisans were also using a calcareous clay source for the white clay.

The white slip appears to have been applied and bisque fired on the vessel before the application of any pigment or the lead-glaze, as attested by the small interaction zone between the white slip and overlying glaze. Many factors contribute to the formation and thickness of the slip/glaze interaction zone, including the type of clay used in the slip, the cooling rate, and whether the slip has been bisque fired prior to the firing of the glaze (Tite et al., 1998, Molera et al., 2001). In general, the formation of lead-rich feldspars on the interface between the slip and glaze occurs to a greater extent when applied to an unfired ceramic body compared to a bisque fired ceramic (Molera et al., 2001). The type of clay also has a large effect, with illitic clays forming much thicker interfaces than kaolinitic clays, and calcareous clays forming Ca-rich pyroxene crystallites that float up into the glaze. The ceramic slips, in this case, are composed of an alumina-rich, non-calcareous clay that has an average of 2.95 wt% K_2O. The small interaction zone observed in our sample could be explained by the slip being composed of kaolinitic clay what was cooled at a fast rate, however other indications suggests that the small interaction zone is the result of the glaze being applied after the slipped ceramic has been first bisque fired.

We do not observe any artifacts in the microstructure that would indicate the application of a glaze slurry on an unfired ceramic body, which is more difficult than applying glaze onto a bisque fired ceramic. The application of a wet glaze slurry on a dry, unfired ceramic body would lead to expansion and contraction of the slip and ceramic body a result of wetting and subsequent drying of the ceramic. This would result in the flaking of the slip and glaze or displacement of sections of the slip layer into the glaze. The application of the glaze on a bisque fired ceramic also is usually more uniform in thickness than on an unfired body (Tite et al., 1998). We observed that the samples have consistent glaze thicknesses, except when more than one slip is applied (K069 and K070) or when the glaze extends around the rim (K077). Finally, the ceramic bodies are fully oxidized, which suggests the ceramic was bisque fired. The formation glassy phase during firing for a lead glaze occurs at a low temperature and would prohibit the full oxidation of the ceramic body if it was not first bisque fired.

The bisque firing of slipped ceramics was noted by Molera et al. (2020) in the ceramics from Termez, and on the slip-painted wares from Sirjan (Morgan and Leatherby, 1987). At Sirjan, sherds were uncovered that had only undergone the initial bisque firing and revealed that the initial firing was low enough not to completely harden the fabric (Morgan and Leatherby, 1987, p. 57). While the general technique of applying a thin white slip under the transparent lead-glaze and bisque firing the vessel is the same as that used in other Early Islamic glazed ceramics made in Central Asia, the potters at Aktobe do appear to be using different raw materials. The use of a local raw materials for the slip is to be expected, as the long-distance transport of a widely available and inexpensive raw material like white clay is usually not economically viable.

For the ceramics with extensive areas of black designs, black slip is

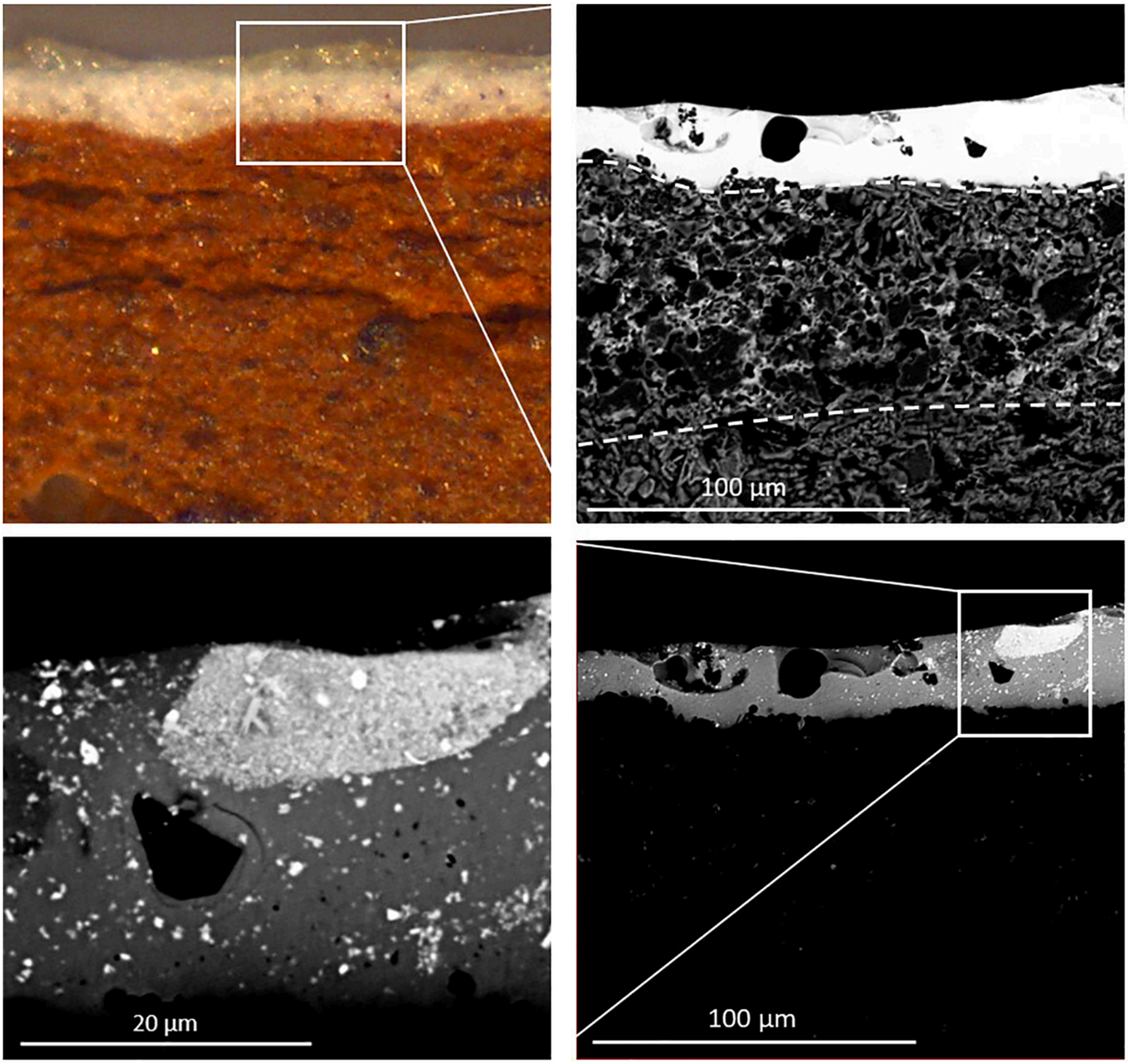

Fig. 8. Cross section of the opaque yellow glaze on K212 showing body, white slip, and glaze layer in the optical microscopy image (*top right*) as well as the BSE image of the cross section (*top left*). BSE image of the sample at higher contrast shows the heterogeneity of the lead glaze (*bottom right*) and at higher magnification the distribution of micron and sub-micron particles containing Sb and Sn oxides that appear white (*bottom left*). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

used in place of the white slip. This slip appears to be made by mixing clay with a relatively large proportion of Mn- and Fe-oxides. The Mn- and Fe-oxides are crushed much smaller than the quartz used in the white slip, resulting in an overall smaller particle size range for the black slip. The use of black slips has been observed on ceramics from Akhsiket and Tashkent (Henshaw, 2010). Molera et al. (2020) also observed the application of a black slip in two samples from Termez. The black slip on those samples appears to be composed of large grains of iron oxides, magnesium oxides, and some grains of a spinel with chromium. Black slips were observed on the ceramics from Nishapur and Afrasiyab by Holakooei et al. (2019). They observed the use of Fe- and Mn-oxides in the Afrasiyab slips, and the use of chromite ($(Mg,Fe)Cr_2O_4$) for the black glazed sherds from Nishapur. Similarly, Bouquillon et al. (2012) identified the use of magnesiochromite in the black slips from Nishapur. The use of chromite colorant for black slips in the ceramics from Nishapur is reflective of the fact that the Sabzevar-Torbat-e-Heydrireh ophiolite belt in the region provided easily accessible chromite minerals as a raw material for potters at Nishapur. While the use of chromium to produce a black color is found on Early Islamic Ceramics excavated from southern Kazakhstan sites, those samples have been identified by NAA to have been imported into the region, likely from Iran (Klesner et al., 2019). The choice of Fe- and Mn- oxides instead of magnesiochromites at Aktobe indicates that the potters are exploiting local raw materials and processing them in similar methods as in other Central Asian cities to create black decoration on their lead-glazed ceramics.

The slipped ceramics had painted decoration applied as a pigment with water or with a small amount of added clay. The underglaze painted ceramic style is characteristic of the ceramics produced in the eastern portion of the Islamic world. Aktobe decoration includes black, brown, and green painted designs. An increased interaction zone between the paint and glaze was observed in the decorated sections, which ranges from 20 to 70 µm. It is unclear whether this is the result of paints being applied after the ceramic was slipped and bisque fired, but before the glaze was applied, or whether the increased interactions is the result of nature of the paints used in decoration. The application of painted decoration on slipped ceramics which were then bisque fired prior to the second glaze firing is noted at Termez (Molera et al., 2020) and on green painted ware from Sirjan (Morgan and Leatherby, 1987).

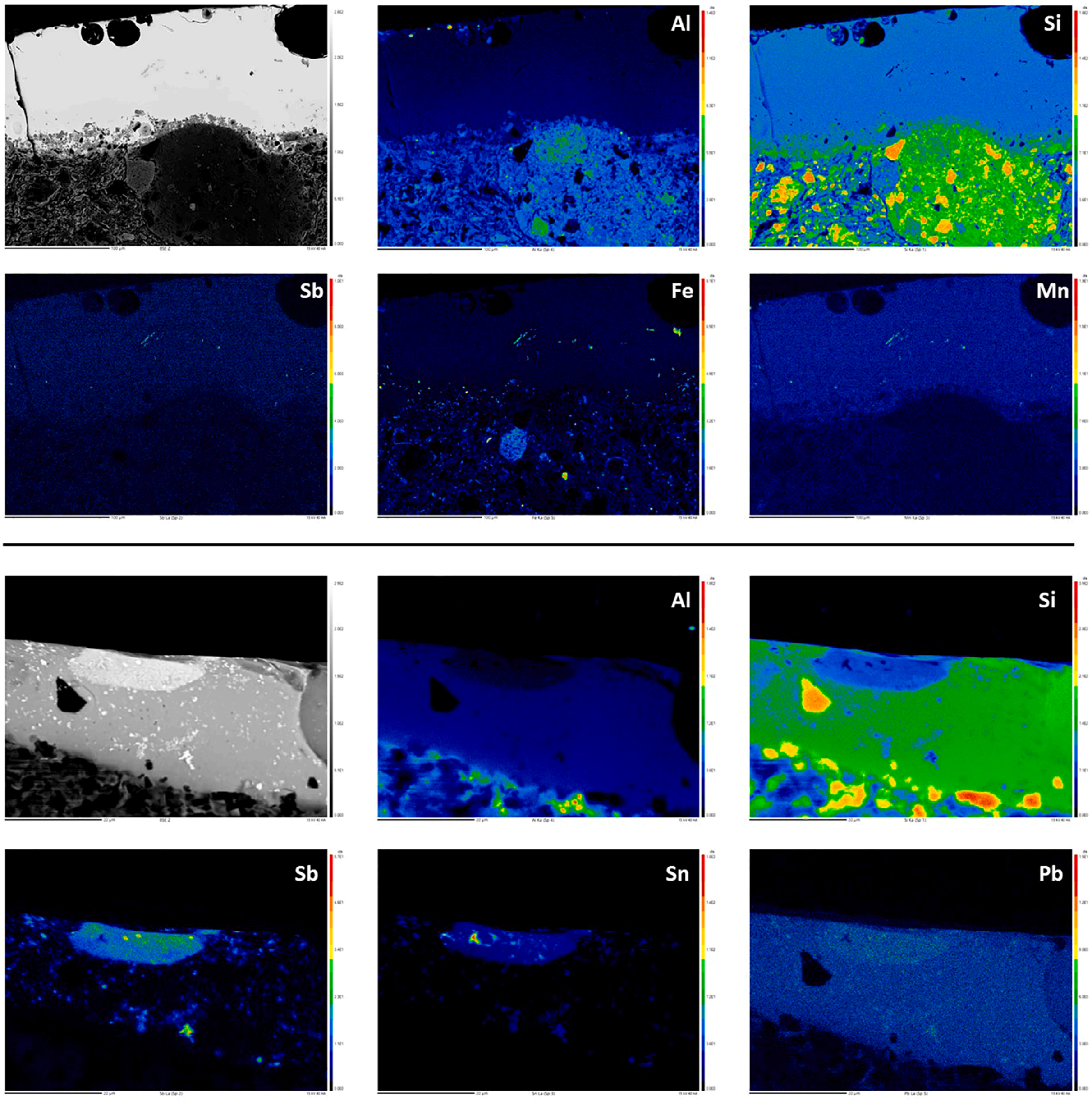

Fig. 9. Electron microprobe elemental mapping of the brown glaze on K199 (*above*) showing the distribution of Al, Si, Sb, Fe, and Mn, and of the opaque yellow glaze on K212 (*below*) showing the distribution of Al, Si, Sb, Sn, and Pb in the glaze and slip of the samples. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

The lead glaze was made by prefritting lead oxide and silica, and not by the direct application of lead oxide onto the slip. Unlike the later Roman period lead glazes (Walton and Tite, 2010) and the Early Byzantine lead glazes (Waksman et al., 2007) which were produced by only PbO, the Early Islamic lead glazes appear to be a mixture of PbO and SiO_2 (Molera et al., 2020, Ting and Taxel, 2020, Tite et al., 2015, Walton and Tite, 2010). Given the homogeneity of the glaze and the general lack of any undissolved quartz particles, the results suggest that the lead oxide and quartz were fitted before being applied as a glaze. Allen (1973) in his translation of Abu'l-Qasim's treatise on pottery production from 1301 CE describes the lead-glaze as being applied as a glaze frit which has been ground, finely sifted, and mixed with water. The fritting of PbO and SiO_2 was also noted by Henshaw (2010) for the ceramics from Akhsiket. The relatively high amount of Al_2O_3 (4.36 wt%) in the glaze, and the presence of Aluminum throughout the glaze depth at relatively consistent composition, also suggests that some clay particles were added to the frit when it was mixed with water (Tite et al., 2015). The added clay in the glaze would have not only increased the plasticity of the glaze, but also helped bind the glaze to the body of the ceramic (Tite et al., 1998) and lowers the expansion coefficient of the glaze (Nordyke, 1984). Table 4 shows the glaze recipe is very similar to those reported for the 9-11th c. CE lead glazes from ancient Termez (Molera et al., 2020) and the colorless glaze on Early Islamic ceramics from Akhsiket (Henshaw et al., 2006). Shen (2017) also reported similar compositions of 8th-9th c. CE ceramics from Syria, but higher proportions of lead in the glazed on Early Islamic glazes from Iraq (Table 4).

The ceramics were then fired in a kiln in oxidizing conditions with temperatures ranging from 800 – 1000 °C. The partial kilns uncovered at Aktobe are round and made of fired bricks. Two kilns were recovered, one of which had associated glazed ceramics. While the full extent of the kiln is not preserved due to the cutting of a modern irrigation canal through the ceramic production area, the projected maximum diameter of the kiln with associated glazed wares is 3.2 m and the interior firing chamber diameter is 2 m. Similar circular kilns dating to the 11th

-beginning of the 12th c. CE were uncovered at Termez (Martínez Ferreras et al., 2019) and Afrasiyab (Henshaw, 2010). These results indicate that not only were the production steps similar for the forming and decorating of lead-glazed ceramics at other Central Asian ceramic production centers, but they were also firing the ceramics in the same way with similar kiln structures.

The largest technical departure from the other Central Asian ceramics is in the presence of antimony in the opaque glaze, which is unusual for the Early Islamic period in the eastern Islamic World. The first opaque lead glazes in the Islamic world, produced in Egypt between the 7th and early 9th c. CE, where made with lead stannate as the opacifying agent (Watson, 2014, Tite et al., 2015, Matin et al., 2018, Ting and Taxel, 2020). The tin-opacified glazes could be either yellow (opacified by $Pb(Sn,Si)O_3$ crystals) or white (opacified by SnO_2 crystals), depending on the composition and firing temperature of the glaze. The yellow glazes were the first to be produced, followed by the white glazes, and both technologies spread throughout the Islamic world in the 9th and 10th c. CE. Yellow tin-opacified ceramics were present in Central Asia and ceramics from Merv and Nishapur have been recorded to contain 2.8–4.9 wt% SnO_2 in the glaze (Matin et al., 2018).

In the second half of the 9th c. CE, workshops in Fustat (Egypt) began using yellow lead-antimonate as a glaze opacifier, rediscovering the technology used to opacify glass and alkaline glazes prior to the Islamic period (Salinas et al., 2019). The opaque yellow glaze, as well as opaque amber and green glazes were opacified also with lead antimonate particles, and these were used on multi-colored glazed finewares (Salinas et al., 2019). The reintroduction of lead-antimonate as an opacifier is attributed to local innovation of the Egyptian potters experimenting with stibnite (Sb_2S_3) and galena (PbS) minerals to produce new glaze colors (Salinas et al., 2019). This technology appears to have spread in the Western edges of the Islamic world to Tunisia, the Mediterranean basin (Salinas et al., 2019), and Yemen (Hallett et al., 1988), but not into the eastern Islamic world. Henshaw (2010) notes that the use of antimony yellow was not widely used in Central Asia, and not found in the Ferghana valley at all (Henshaw, 2010, p. 258). Holakooei et al. (2019) also notes that while two treatises on the production of glazes, Nayshaburi (12th c. CE) and Abu'l Qasim (early 14th c. CE), mention a substance named *ithmid* or *athmad* (traditionally translated as antimony sulfide) was used to produce a yellow glaze, no antimony yellow glaze has been recorded in Iran (Allan, 1973; Holakooei et al., 2019). The use of antimony in an opaque yellow glaze at Aktobe, therefore, is a unique technology in the Eastern Islamic World. It is probable that the Aktobe technology is not linked to the production of antimony yellow in Egypt, but instead is likely the result of experimentation with antimony bearing minerals. The fact that both tin and antimony are used in the glaze suggests that the local potters were not aware of the opacifying nature of the Sb-oxide particles, and instead were using the antimony as a colorant and tin as an opacifier. Tite (2011) observed that tin-opacification is rare to completely absent for colorants that act as opacifiers themselves, including for lead antimonate yellow colorants in glazed Islamic ceramics.

The use of antimony to produce a yellow opaque glaze by Aktobe craftsmen represents a distinct technological choice that departs from the technology of the rest of the eastern Islamic world. This raw material choice could be due to availability of antimony in the region. In Central Asia, the main silver ore sources all contain the unusual Ag-Sb mineralization type (Pavlova and Borisenko, 2009), which contain high-Sb sulfosalts, Sb sulfides, and native antimony. Two of these sources, the Talas silver ore district and the Pamir silver ore district were known silver producers in antiquity (Pavlova and Borisenko, 2009). The Talas silver ore province, located in modern Kyrgyzstan, is only 200 km from Aktobe and has native Sb, Ag, as well as galena (Pavlova and Borisenko, 2009). If local craftsmen were sourcing their silver or lead ore from this mining region, it is possible that they were also obtaining antimony to use as a glass and glaze opacifier and colorant. The presence of antimony in the transparent glazes as an impurity from the lead source suggests that they are exploiting antimony-rich lead ore sources in their glaze production.

4.2. Technological conservatism

The ceramics in this study come from two different periods, 9–10th c. and 11–12th c. CE, but we see no significant stylistic or technological change occurring across the span of about 400 years. The glazing technology that is first introduced in the 9th c. CE to the region remains consistent, which represents a long period of technological conservatism. The period under study saw continual political change. From the 8th c. CE, Aktobe was under that control of the Turgesh Khaganate (704–756 CE), Karluk Khaganate (756–940 CE), and Karakhanid Khaganate (942–1212 CE) (Shalekenov, 2006). Despite this political turmoil, the economic growth of Silk Road cities in southern Kazakhstan appears to have been strong during the 8th-12th c. CE (Dawkes and Jorayev, 2015). The ceramic technology, similarly, appears to not have been affected by the political instability of the region.

This period also saw the introduction and gradual conversion to Islam of the people in the area around Aktobe. Beginning in the early 8th c. CE, Arabs came to the territory of modern-day Kazakhstan. In 751, in the Talas region approximately 200 km west of Aktobe, a decisive battle between the Arabs and the Chinese army occurred which resulted in the local Karluks aligning with the Arabs and defeating the Chinese army. From that time, Islam spread to the region. While it is difficult to say that the population of Aktobe converted to Islam immediately after the arrival of the Arabs, we do see a gradual increase in material culture associated with the Islamic religion and the rest of the Islamic world. In the 9th-10th c. CE at Aktobe we see the transition from Sogdian inscriptions, widely used through the 9th c. CE at Aktobe (Akymbek and Baibugunov, 2014), to Arabic writing, which became widespread in the 10th and 11th c. CE. In 960 CE, the state religion of the Karakhanids was proclaimed to be Islam after the death of the former ruler, Satuq Bughra Khan (History of Kazakhstan, 1996: 399). It can be assumed from the remains of the 11th c. CE minaret at Aktobe, which was built in the Muslim style, that the Islamic religion was present in medieval Aktobe (Akymbek and Baibugunov, 2013). The discovery of a clade with 3057 copper coins from the Shahristan excavations in 1984 also indicates the significant spread of Islamic culture in the region (Shalekenov, 1985: 22-36).

Decorated glazed ceramics are one of the most defining features of the "Islamic" archaeological record (Watson, 2014). The increase in the sheer number of glazed ceramics, and the variety of glazed wares, found across the Islamic world in the Early Islamic period points to the importance that they held as cultural markers. The prevalence of lead-glazed ceramics in the major Early Islamic styles at Aktobe beginning in the 9th c. CE suggests that the local people saw ownership of these wares as a cultural marker associating them with the rest of the Islamic world. Not only were the people of Aktobe importing these products, they were also producing them, indicating further cultural ties with the Islamic world. The results indicate that the glazing technology in place at Aktobe in the 9-12th c. CE is remarkably similar to the production technology of the lead-glazed ceramics produced in other Central Asian Islamic cities in this period. As there are no technological precursors for glazed ceramics in the region (Heinsch et al., 2018), the glazed wares that were produced in the 9th c. CE are the first glazed wares made by local craftsmen at Aktobe, and their style and production method indicate a direct cultural and technological link to potters in the eastern Islamic world. While there are differences in the choice of raw materials, specifically in the use of antimony opacifiers, between the Aktobe ceramics and the other Central Asian ceramics, the technology otherwise falls into the standardized techniques used by potters throughout the eastern Islamic world. The degree of standardization and specialization in the multiple-step production required to produce these lead-glazed ceramics indicates that skilled craftsmen with direct knowledge of how the glazed and decorated ceramics were produced in other regions was introduced at Aktobe.

This is not the first time we see technological transfer coincide with stylistic transfer in the Islamic world. Recent research into early tin-opacified Islamic ceramics has identified the technological transfer of yellow tin-opacified ceramics from Egypt and Syria produced in the 8th c. CE to the cities of Samarra, Kish, and Susa in Mesopotamia in the 9th c. CE (Matin et al., 2018, Tite et al., 2015). Similarly, once opaque white glazing technology was developed in the 9th c. CE in Iraq, it then was transferred throughout the Islamic world, both east to Iran, Central, and East Asia, and west to the Mediterranean (Matin et al., 2018). This direct transfer of ceramic production technology across large regions in the Early Islamic period demonstrates that strong knowledge networks were in place across the Islamic world, which facilitated the introduction of glazed ceramics into southern Kazakhstan in the 9th c. CE.

5. Conclusions

By characterizing the variability of the local artisans' use of raw materials, glazing methods, and decorative techniques for a group of representative Early Islamic ceramics from Aktobe, Kazakhstan, we reconstructed the production technology. The local craftspeople produced fine decorated ceramics with a high lead glaze applied over a white slip. The white slips were composed of white clay, quartz, and lead oxide, which was applied before the first firing. Their use of slips with lead oxide indicated that they had learned to overcome the problem of adhering the white slip to the ceramic in a two-step firing, and also how to prevent lead glazes from chipping and peeling, or crazing and weathering as a result of the problem of glaze fit. Putting lead in the slip layer allows the slip to have a thermal expansion coefficient intermediate between the body and glaze. Decoration was applied on top of the white slip a paint or slip colored with metal oxides, iron and manganese oxides for black decoration and iron oxide for brown decorations, or as the metal oxide in solution with the lead glaze as is seen in the green (copper) glazes. A unique opaque yellow glaze was also characterized, which had a high lead glaze and micron and sub-micron sized particles that are composed of tin and antimony oxides. The glazed ceramics were fired in a circular kiln similar to those used throughout Central Asia in the Early Islamic period to temperature typical for high-lead glazes, 800–1000 °C.

This study of southern Kazakhstan documents the direct adoption of the lead-glazing technology from the Islamic world. As Islam spread across Central Asia during the 8th and 9th c. CE, the cultural material across this region and into what is today southern Kazakhstan began to shift. At Aktobe we see the local production of the traditional Early Islamic style glazed ceramics using the same methods as those employed by potters throughout the eastern Islamic world. These glazed ceramics, which were not produced at all in the region before the 9th c. CE, are a cultural marker associating the people in southern Kazakhstan with the rest of the Islamic world. Given the similarity of production technology, it is likely that the craftspeople in Aktobe learned how to make the glazed ceramics directly from Islamic potters from the West. While the technology is similar in most aspects, we do see local innovation in the use of antimony in the opaque glazed ceramics at Aktobe. The availability of antimony as a raw material in the region suggests that local craftspeople were sourcing their own opacifiers from local materials and experimenting with different pigments to produce opaque lead glazes. This introduction of antimony-based opacifiers is likely not linked to the use of antimony in opaque polychrome ceramics in Egypt during the same Early Islamic period.

CRediT authorship contribution statement

C.E. Klesner: Conceptualization, Investigation, Writing - original draft, Writing - review & editing. **Y. Akymbek:** Resources. **P.B. Vandiver:** Supervision, Methodology, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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References

Akymbek, Y., 2015. Ceramics of medieval Aktobe city (7th-early 13th century). (Ph.D. Dissertation) Almaty: Department of Ethnology and Archaeology, Al-Farabi Kazakh National University (in Kazakh).

Akymbek, Y., Baibugunov, B. 2013. Minaret of medieval city Aktobe. World Academy of Science, Engineering and Technology, 78, 2093–2096.

Akymbek, Y., Baibugunov, B., 2014. Inscriptions on ceramics of medieval city Aktobe. Proc. Soc. Behav. Sci. 122, 77–81. https://doi.org/10.1016/j.sbspro.2014.01.1306.

Allan, J.W., 1973. Abu'l-Qasim's Treatise on Ceramics. Iran 11, 111–120.

Bouquillon, A., Coquinot, Y., Doublet, C., 2012. Pottery Study and Analyses. In: Rante, R., Collinet, A. (Eds.), Nishapur Revisited: Stratigraphy and Ceramics of the Qohandez. Oxbow Books, Oxford, pp. 56–135.

Dawkes, G., Jorayev, G., 2015. A case study of an early Islamic city in Transoxiana: Excavations at the medieval citadel in Taraz, Kazakhstan. Archaeol. Res. Asia 4, 17–24. https://doi.org/10.1016/j.ara.2015.09.001.

Dillis, S., Van Ham-Meert, A., Leeming, P., Shortland, A., Gobejishvili, G., Abramishvili, M., Degryse, P., 2019. Antimony as a raw material in ancient metal and glass making: provenancing Georgian LBA metallic Sb by isotope analysis. STAR; Sci. Technol. Archaeol. Res. 1–15 https://doi.org/10.1080/2054923.2019.1681138.

Gradmann, R., Berthold, C., Schussler, U., 2015. Composition and colouring agents of historical Islamic glazes measured with EPMA and μ-XRD. Eur. J. Mineral. 27, 325–335.

Hallett, J.R., Thompson, M., Keall, E.J., Mason, R.B., 1988. Archaeometry of medieval Islamic glazed ceramics from North Yemen. Can. J. Chem. 66 (2), 266–272. https://doi.org/10.1139/v88-045.

Hamer, F., Hamer, J., 1997. The Potter's Dictionary of Materials and Techniques, fourth ed. A & C Black; University of Pennsylvania Press, London: Philadelphia.

Heinsch, M., Vandiver, P.B., Lyublyanovics, K., Choyke, A.M., Reedy, C., Tourtellotte, P., and Chang, C. 2018. "Ceramics at the Emergence of the Silk Road: A Case from Southeastern Kazakhstan" Materials Issues in Art and Archaeology X, MRS Symposium Proceedings, 1656:251–282.

Henshaw, C.M. 2010. Early Islamic Ceramics and Glazes of Akhsiket, Uzbekistan. (PhD Dissertation) London: Department of Archaeology, University of College London.

Henshaw, C.M., Rehren, Th., Papachristou, O., and Anarbaev, A.A. 2006. "The Early Islamic Glazed Ceramics of Akhsiket, Uzbekistan" from J. Perez-Arantegui, Ed. 34th International Symposium on Archaeometry.

History of Kazakhstan: from ancient times to the present day. (1996) Volume I. – Almaty: Atamura, 544 (in Kazakh).

Holakooei, P., de Lapérouse, J.-F., Carò, F., Röhrs, S., Franke, U., Müller-Wiener, M., Reiche, I., 2019. Non-invasive scientific studies on the provenance and technology of early Islamic ceramics from Afrasiyab and Nishapur. J. Archaeolog. Sci.: Rep. 24, 759–772. https://doi.org/10.1016/j.jasrep.2019.02.029.

Klesner, C.E., MacDonald, B.L., Dussubieux, L., Akymbek, Y., Vandiver, P.B., 2019. Local production and long-distance trade of Islamic glazed ceramics in Central Asia: A compositional analyses of ceramics from Southern Kazakhstan by NAA and LA-ICP-MS. J. Archaeolog. Sci. Rep. 26, 101905. https://doi.org/10.1016/j.jasrep.2019.101905.

Lakatos, T., Johansson, L.G., Simmingskold, B., 1972. Viscosity temperature relations in the glass system SiO2-Al2O3-Na2O-K2O-CaO-MgO in the composition range of technical glasses. Glass Technol. 13, 88–95.

Martínez Ferreras, V., Fusaro, A., Gurt Esparraguera, J.M., Ariño Gil, E., Pidaev, S.R., Angourakis, A., 2019. The islamic ancient termez through the lens of ceramics: a new archaeological and archaeometric study. Iran 58 (2), 250–278. https://doi.org/10.1080/05786967.2019.1572430.

Mason, R.B., 1995. New looks at old pots. Muqarnas 12, 1–10.

Mason, R.B. 2004. Shine Like the Sun (Costa Mesa and Toronto). Mazda Publishers & Royal Ontario Museum.

Mason, R.B., Tite, M.S., 1997. The beginnings of Tin-opacification of pottery glazes. Archaeometry 39, 41–58.

Matin, M., 2019. Tin-based opacifiers in archaeological glass and ceramics glazes: a review and new perspectives. Archaeol. Anthropol. Sci. 11, 1155–1167. https://doi.org/10.1007/s12520-018-0735-2.

Matin, M., Tite, M.S., Watson, O., 2018. On the origins of tin-opacified ceramic glazes: New evidence from early Islamic Egypt, the Levant, Mesopotamia, Iran, and Central Asia. J. Archaeol. Sci. 97, 42–66.

Molera, J., Vendrell-Saz, M., Pérez-Arantegui, J., 2001. Chemical and textural characterization of tin glazes in Islamic ceramics from Eastern Spain. J. Archaeol. Sci. 28, 331–340. https://doi.org/10.1006/jasc.2000.0606.

Molera, J., Martinez Ferreras, V., Fusaro, A., Gurt Esparraguera, J.M., Gaudenzi, M., Pidaev, S.R., Pradell, T., 2020. Islamic glazed wares from ancient Termez (southern Uzbekistan). Raw materials and techniques. J. Archaeolog. Sci.: Rep. 29, 102169 https://doi.org/10.1016/j.jasrep.2019.102169.

Morgan, P., Leatherby, J., 1987. "Excavated ceramics from Sirjan. In: Allan, J.W., Roberts, C. (Eds.), Syria and Iran: Three Studies in Medieval Ceramics. Oxford University Press, Oxford, pp. 51–110.

Nordyke, J.S. (Ed.), 1984. Lead in the world of ceramics. The American Ceramic Society Inc, Columbus, OH.

Pavlova, G.G., Borisenko, A.S., 2009. The age of Ag-Sb deposits of Central Asia and their correlation with other types of ore systems and magmatism. Ore Geol. Rev. 35, 164–185. https://doi.org/10.1016/j.oregeorev.2008.11.006.

Pradell, T., Molera, J., 2020. Ceramic technology. How to characterise ceramic glazes. Archaeol. Anthropol. Sci. 12, 189. https://doi.org/10.1007/s12520-020-01136-9.

Pradell, T., Molera, J., Smith, A.D., Tite, M.S., 2008a. The invention of lustre: Iraq 9th and 19th centuries AD. J. Archaeol. Sci. 35, 1201–1215.

Pradell, T., Molera, J., Smith, A.D., Tite, M.S., 2008b. Early Islamic lustre from Egypt, Syria and Iran (10th to 13th century AD). J. Archaeol. Sci. 35, 2649–2662.

Salinas, E., Pradell, T., Matin, M., Tite, M.S., 2019. From tin- to antimony-based yellow opacifiers in the eary Islamic Egyptian glazes: Regional influences and ruling dynasties. J. Archaeol. Sci. Rep. 26, 101923 https://doi.org/10.1016/j.jasrep.2019.101923.

Shalekenov, U.Kh. (1985) "Archaeological and Ethnographic Study of the Chui Valley" Report in the Archive of the Institute of Archaeology named A.Kh.Margulan – Almaty, Kazakhstan (in Russian).

Shalekenov, U.Kh. 2006. Balasagun city in the 5-13 centuries. Almaty. (in Kazakh).

Shalekenov, U. Kh. 2009. Balasagun city in the 5-13 centuries. Almaty. (in Russian).

Shen, J. 2017. Chemical and isotopic analysis in the investigation of glazed from Northern China and the Middle East, 7th-14th centuries AD. (Ph.D. thesis) Nottingham, University of Nottingham.

Ting, C., Taxel, I., 2020. Indigeneity and innovation of early Islamic glaze technology: the case of the Coptic Glazed Ware. Archaeol. Anthropol. Sci. 12 (27) https://doi.org/10.1007/s12520-019-01007-y.

Tite, M.S., 2011. The technology of glazed islamic ceramics using data collected by the Late Alexander Kaczmarczyk. Archaeometry 53 (2), 329–339. https://doi.org/10.1111/j.1475-4754.2010.00546.x.

Tite, M.S., Freestone, I., Mason, R.B., Molera, J., Vendrell-Saz, M., Wood, N., 1998. Lead glazes in antiquity – methods of production and reasons for use. Archaeometry 40 (2), 241–260.

Tite, M.S., Watson, O., Pradell, T., Matin, M., Molina, G., Domoney, K., Bouquillon, A., 2015. Revisiting the beginnings of tin-opacified islamic glazes. J. Archaeol. Sci. 57, 80–91.

Waksman, S.Y., Bouquillon, A., Cantin, N., and Katona, I. (2007) "The first Byzantine 'Glazed White Wares' in the early medieval technological context" in Archaeometric and archaeological approaches to ceramics (papers presented at EMAC'05) (ed. S. Y. Waksman) Oxford: BAR International Series 1691, 129–135.

Walton, M.S., Tite, M.S., 2010. Production technology of roman lead-glazed pottery and its continuance into late antiquity. Archaeometry 52 (5), 733–759. https://doi.org/10.1111/j.1475-4754.2009.00506.x.

Watson, O., 2014. Revisiting Samarra: the rise of Islamic glazed pottery. Beitrage zur Islamischen Kunst und Archaologie 4, 125–144.