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Sylvie Yona Waksman, Georgia Giannaki, Jacques Burlot. Chemical analyses of bodies and glazes of ceramics from the pottery workshop excavated at the site of Lozenets (Sofia, 6th - early 7th c. AD): preliminary results. ПРИНОСИ КЪМ БЪЛГАРСКАТА АРХЕОЛОГИ [CONTRIBUTIONS TO BULGARIAN ARCHAEOLOGY], 2025, 14, pp.75-84. <10.53250/cba14.75-84>. <hal-05336327>

HAL Id: hal-05336327

<https://hal.science/hal-05336327v1>

Submitted on 15 Jan 2026

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CHEMICAL ANALYSES OF BODIES AND GLAZES OF CERAMICS FROM THE POTTERY WORKSHOP EXCAVATED AT THE SITE OF LOZENETS (SOFIA, 6TH – EARLY 7TH C.): PRELIMINARY RESULTS

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ABSTRACT

A selection of 40 glazed and unglazed pottery sherds from the 6th – early 7th c. pottery workshop at Lozenets, Sofia (ancient Serdica) was studied to investigate their manufacturing technique. Analysis of the sherds' ceramic bodies indicates that the same low-calcareous clay was used for the production of all categories of vessels (glazed and unglazed, for cooking and not). The glazes are high-lead ones, with a noticeable bismuth content in some inclusions which might be indicative of the provenance of the lead compounds. The latter were probably applied directly onto vessels' low-calcareous bodies to form the glaze. As for the firing process, several clues point to a single firing. Based on this data, the production of glazed pottery at Serdica seems to be related to the technical tradition observed in the Balkans and northern Italy between the 5th and 7th c. that continued in the “Byzantine Glazed White Ware I” of the 7th c.

Keywords: high-lead glazed ware, Early Byzantine pottery, Serdica, Bulgaria, manufacturing techniques, body and glaze analysis, chemical analysis, WD-XRF, SEM-EDS

The 6th – early 7th c. pottery workshop in Sofia (the Lozenets residential district) is of particular interest since production sites of glazed wares that have been identified archaeologically are rare for this specific period (for earlier periods see Magrini, Sbarra 2005; Cvjetičanin 2006; Hárshgyi, Ottományi 2013). These glazed wares shortly precede the production of a repertoire of Byzantine glazed wares in the early 7th c., according to John W. Hayes' study at Saraçhane, Istanbul (Hayes 1992). They may therefore be seen as the “missing link” between late antique and Byzantine glazed wares. Archaeometric investigations have been undertaken to characterize the materials used for the bodies and glazes of glazed ceramics produced in Sofia and to determining the technological tradition to which these ceramics are related (Walton 2004; Waksman et

al. 2007; Waksman et al. 2008; Capelli et al. 2009; Capelli et al. 2010a; Capelli et al. 2010b; Walton, Tite 2010).

We considered a sample of 40 ceramics from the excavations of the Lozenets pottery workshop (fig. 1) that were carried out in 2000 by a team from the National Archaeological Institute with Museum of the Bulgarian Academy of Sciences directed by Metodi Daskalov and Snezhana Goryanova (Daskalov, Goryanova 2009; Даскалов, Горянова 2009; Daskalov 2024, in this volume). This sample comes from a large assemblage found in close proximity to the pottery kilns that has been interpreted as their dump (Todorova 2024, in this volume). Although this assemblage seemed fairly homogeneous, we included in the sample overfired or distorted wasters in order to secure local references. The sample also contains tablewares as well as common and cooking wares that are both glazed and unglazed. The glazed and unglazed wares sometimes have the same form, thus raising the question of double firing (fig. 1: top left). Most of the sherds in the sample belong to the most common fabrics and forms of glazed wares, mainly jugs and mortars. Less common fabrics



Fig. 1. Examples of sherds sampled from the Lozenets workshop: fragments of glazed jugs and unglazed counterparts (top left), glazed mortar (top right), jug with lumps on the surface (bottom left), unglazed rim showing drops of glaze with a central hole (bottom right). Analyzed samples are indicated by Lyon laboratory id (Photos: S. Y. Waksman)

Обр. 1. Примери за фрагменти, взети от работилницата в кв. „Лозенец“: фрагменти от глазирани кани и техните неглазирани аналози (горе вляво), глазиран мортарий (горе вдясно), кана с бучки по повърхността (долу вляво), неглазирано устие с капки глазура, в центъра на които има „кратерчета“ (долу вдясно). Анализираните проби са обозначени с идентификатор на лаборатория в Лион (Снимки: С. Й. Ваксман)

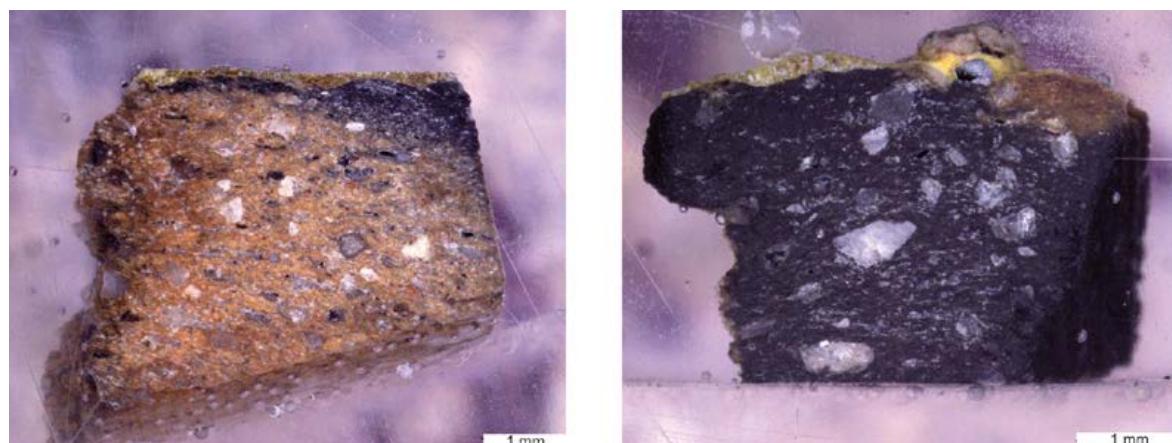


Fig. 2. Photos of polished cross-sections of samples under the binocular microscope, showing total reduction (right) or partial reduction (left) of the clay body on the glazed side. A large bubble at the surface of the glaze covered by crusting is also visible at right (samples BZN469 on the left and BZN473 on the right; Photos: J. Burlot)

Обр. 2. Снимки на полирани напречни сечения на проби, погледнати под бинокулярен микроскоп, показващи пълна редукция (вдясно) или частична редукция (вляво) на глинено то тяло от страната на глазурата. Вдясно се вижда и голямо мехурче на повърхността на глазурата, покрито с коричка (вляво – проба BZN469, вдясно – проба BZN473; Снимки: Ж. Бурло)

and forms were sampled as well to include representatives of most of the glazed repertoire (e.g., jars and cooking wares). A restricted number of unglazed wares was included as well to compare their chemical characteristics.

A smaller sample of six sherds was selected for glaze analysis. Four were jugs (BZN467, BZN475) and mortars (BZN469, BZN478). Two special cases were also considered: one jug bearing a large number of round lumps on the surface of its glaze (fig. 1: BZN473) and an otherwise unglazed sherd showing a large drop of glaze with a hole in the center (fig. 1: BZN487), a phenomenon known as pitting or pin-holing depending on the depth of the “crater”.¹ These two samples were expected to provide further information on the glazing process. Unglazed sherds with either drops or trails of glaze are frequent, suggesting that glazed and unglazed wares were fired together in the same load in the kilns. The glaze applied to the former would have dripped onto the latter in a (semi-)liquid state.

Optical microscopy

Observations of cross-sections of the sherds under a binocular microscope show their bodies to be fairly coarse with inclusions reaching millimetric size (fig. 2). Most glazed sherds were only partly reoxidized during cooling in the kiln; a few remained completely reduced (fig. 2: left and right, respectively). In the former case, the reduced part of the sherd always corresponds to the glazed side, which supports a single firing process where reoxidation during cooling could only occur through the unglazed side.² Round lumps at the surface of the glaze, occasional on many sherds and particularly abundant at the surface of sample BZN473, are bubbles trapped directly below the surface rather than remains of unmelted glaze. Crusting may also be seen at the surface of a large lump on BZN473 (fig. 2: top right). The pinhole in sample BZN487 would be another

¹ We thank Dr. Rumyana Koleva from Sofia University “St. Kliment Ohridski” for information about similar defects on later glazed wares found at Silistra (Koleva 2009).

² See Picon 2002 for the successive steps of the firing cycle and the effects of an oxidizing or reducing atmosphere in the kiln on the color of the pottery.

id.	CaO	Fe ₂ O ₃	TiO ₂	K ₂ O	SiO ₂	Al ₂ O ₃	MgO	MnO	(Na ₂ O)	(P ₂ O ₅)	Zr	(Sr)	Rb	Zn	Cr	Ni	(Ba)	V	Ce
BZN459	1.49	5.77	0.707	2.59	64.63	21.36	1.59	0.0464	1.56	0.08	181	188	148	92	78	25	552	119	75
BZN460	1.46	5.45	0.671	2.70	65.95	20.27	1.55	0.0463	1.65	0.06	164	198	142	85	74	25	571	115	77
BZN461	1.48	5.58	0.715	2.57	65.32	20.81	1.64	0.0447	1.60	0.08	179	187	143	90	78	27	550	124	75
BZN462	1.43	5.81	0.721	2.48	65.35	20.89	1.61	0.0454	1.39	0.10	177	183	143	89	78	24	543	126	76
BZN463	2.00	5.38	0.638	3.51	65.67	19.15	1.55	0.0533	1.71	0.17	178	213	153	83	74	24	578	96	73
BZN464	1.49	5.44	0.680	2.71	65.98	20.25	1.56	0.0450	1.58	0.09	166	192	148	85	76	25	544	117	81
BZN465	1.48	5.36	0.630	2.64	66.91	19.69	1.44	0.0452	1.59	0.08	153	187	140	82	71	25	557	104	70
BZN466	1.45	5.83	0.718	2.50	65.22	20.94	1.60	0.0478	1.38	0.14	170	184	143	89	78	25	556	127	67
BZN467	1.42	5.56	0.680	2.59	66.59	19.95	1.46	0.0502	1.41	0.12	166	181	143	84	73	22	565	123	72
BZN468	1.73	5.23	0.651	3.43	66.14	19.50	1.37	0.0466	1.58	0.14	166	248	137	79	69	22	779	113	59
BZN469	1.49	5.70	0.680	2.65	66.11	20.00	1.51	0.0522	1.53	0.11	170	199	143	84	74	23	593	120	73
BZN470	1.54	5.66	0.669	3.12	65.09	20.07	1.68	0.0446	1.75	0.15	158	186	143	89	80	25	567	115	77
BZN471	1.41	5.31	0.655	2.72	66.73	19.84	1.46	0.0419	1.58	0.08	164	187	142	90	71	23	547	111	76
BZN472	1.44	5.46	0.691	2.66	66.15	20.18	1.54	0.0482	1.54	0.10	183	183	145	88	75	25	543	116	88
BZN473	1.99	5.64	0.696	2.52	65.07	20.42	1.51	0.0437	1.51	0.42	175	266	141	89	78	25	794	122	80
BZN474	1.51	5.45	0.692	2.64	65.95	20.27	1.56	0.0476	1.59	0.12	192	190	149	89	77	23	576	115	87
BZN475	1.65	5.55	0.672	2.63	66.21	19.87	1.47	0.0526	1.53	0.20	173	209	138	83	74	24	652	111	74
BZN476	2.05	5.75	0.707	2.56	65.03	20.29	1.48	0.0450	1.45	0.45	175	261	135	89	78	25	811	119	66
BZN477	1.36	5.71	0.683	2.57	65.79	20.52	1.56	0.0436	1.54	0.07	164	183	140	89	77	26	545	119	77
BZN478	1.44	5.31	0.681	2.64	66.29	20.22	1.54	0.0460	1.57	0.10	170	190	145	85	81	24	551	116	81
BZN479	1.48	5.49	0.683	2.62	66.40	19.90	1.51	0.0444	1.53	0.14	196	191	143	83	72	24	562	122	75
BZN480	1.43	5.67	0.670	2.71	66.02	20.13	1.50	0.0479	1.58	0.07	163	187	151	87	77	25	558	112	88
BZN481	1.48	5.42	0.670	2.68	66.20	20.17	1.50	0.0427	1.56	0.11	163	187	144	87	74	24	556	111	75
BZN482	1.37	5.70	0.646	2.69	66.55	19.86	1.41	0.0459	1.50	0.08	173	178	150	83	74	23	532	106	71
BZN483	1.45	5.45	0.679	2.60	66.54	19.89	1.49	0.0458	1.59	0.11	177	189	143	86	74	23	572	116	83
BZN484	2.05	5.76	0.702	2.62	64.96	20.29	1.50	0.0463	1.42	0.44	166	254	134	88	83	28	899	126	75
BZN485	2.14	5.76	0.663	2.82	65.09	20.00	1.38	0.0523	1.48	0.41	166	245	136	89	79	24	980	106	66
BZN486	1.41	5.56	0.682	2.60	65.98	20.40	1.53	0.0434	1.54	0.08	174	184	141	88	74	24	569	115	71
BZN487	1.45	5.51	0.676	2.62	66.10	20.25	1.52	0.0455	1.51	0.11	167	187	141	84	74	25	564	118	77
BZN488	2.12	5.35	0.618	2.62	66.27	19.41	1.28	0.0444	1.56	0.52	147	280	132	83	71	23	777	104	72
BZN489	1.45	5.57	0.680	2.97	65.74	20.25	1.53	0.0431	1.50	0.10	169	186	147	89	77	24	550	119	84
BZN490	1.41	5.96	0.748	2.44	64.51	21.50	1.71	0.0458	1.40	0.08	173	181	141	93	84	27	542	129	81
BZN491	1.43	5.83	0.732	2.45	65.16	21.03	1.64	0.0460	1.44	0.09	178	186	144	90	77	26	548	128	84
BZN492	1.39	5.74	0.717	2.52	65.50	20.82	1.59	0.0466	1.42	0.08	172	175	144	91	83	25	543	128	81
BZN493	1.41	5.47	0.679	2.64	65.96	20.45	1.51	0.0430	1.55	0.11	163	184	142	87	74	24	550	118	77
BZN494	1.63	5.57	0.665	2.76	66.00	20.01	1.46	0.0429	1.57	0.13	154	202	136	87	76	25	703	116	75
BZN495	1.47	5.44	0.648	2.71	66.60	19.82	1.45	0.0418	1.56	0.10	168	189	142	84	75	25	594	112	75
BZN496	2.03	5.51	0.656	2.86	65.93	19.52	1.33	0.0585	1.51	0.39	167	305	136	84	70	24	825	109	74
BZN497	1.44	5.64	0.676	2.71	66.12	20.09	1.51	0.0457	1.50	0.09	180	195	139	86	72	23	584	114	74
BZN498	1.43	5.38	0.655	2.63	66.73	19.84	1.49	0.0428	1.56	0.08	159	191	145	82	80	24	564	110	83
m	1.57	5.57	0.680	2.69	65.86	20.20	1.51	0.0463	1.53	0.15	170	202	142	87	76	24	614	116	76
σ	0.24	0.17	0.028	0.22	0.60	0.50	0.09	0.0035	0.08	0.13	10	32	5	3	4	1	113	7	6
min	1.36	5.23	0.618	2.44	64.51	19.15	1.28	0.0418	1.38	0.06	147	175	132	79	69	22	532	96	59
max	2.14	5.96	0.748	3.51	66.91	21.50	1.71	0.0585	1.75	0.52	196	305	153	93	84	28	980	129	88

Table 1. Chemical composition of the ceramic body (WD-XRF; major and minor elements in oxide weight %, trace elements in ppm; m: mean, σ: standard deviation, min: minimum value, max: maximum value; elements between brackets were not taken into account in the histogram presented on fig. 3)

Таблица 1. Химичен състав на керамичното тяло (WD-XRF; основни и второстепенни елементи в тегловни % на оксида, микроелементи в ppm; m: средно, σ: стандартно отклонение, min: минимална стойност, max: максимална стойност; елементите в скоби не са взети предвид в представената хистограма на обр. 3)

aspect of the same phenomenon; bubbles of gas were either trapped in the glaze (the surface lumps) or exploded leaving a cavity in it (the pinholes). Both would be favored by a single firing, as a first biscuit firing prior to glazing and a second firing would have liberated gases caused by organic material contained in the clay body and so would have avoided such glazing defects.

Chemical analysis of the ceramic bodies

Chemical analysis of the ceramic bodies was carried out through wavelength-dispersive X-ray fluorescence (WD-XRF) at the Lyon laboratory (CNRS UMR 5138) using a Bruker S8 Tiger spectrometer. Samples were cut out with a diamond-coated saw, and any coating as well as the external layer, chemical composition of which is more liable to be altered by the burial environment, were removed. After heating at 950°C (for the removal of water, volatile elements, organics), cooling,

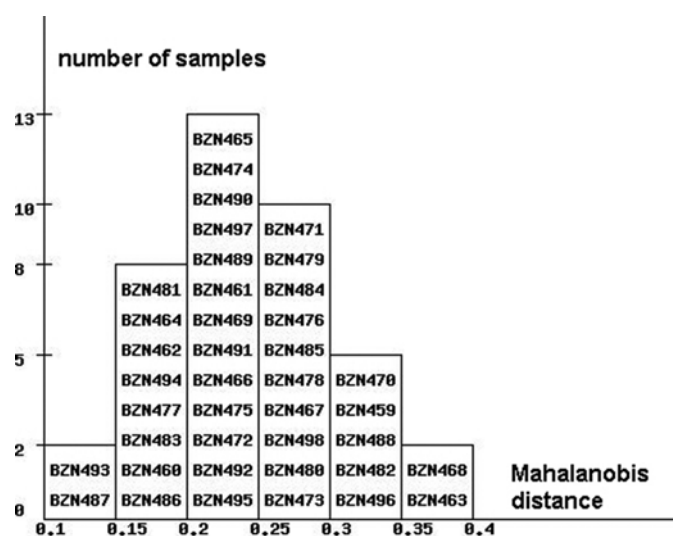


Fig. 3. Histogram of the Mahalanobis distances between each individual sample and the distribution of the group

Обр. 3. Хистограма на разстоянията по Махаланобис между всяка отделна проба и груповото разпределение

and grinding, 800 mg of ceramic powder was mixed with 3200 mg of flux (lithium metaborate and tetraborate). The mix was heated to a liquid state in a gold and platinum crucible then cast into a bead. Analysis was carried out on these homogeneous beads of fixed size to determine a mean chemical composition representative of the initial clay material. The calibration of the set-up was based on the analysis of 40 geological standard reference materials (including CRPG, USGS, and NIST certified international standards).³

The chemical compositions of the samples are very homogeneous, and the histogram of their individual Mahalanobis distances to the distribution of the group shows its statistical consistency (Table 1, fig. 3). They correspond to low calcareous bodies (mean CaO ~ 1.6 %) that are not very ferruginous (Fe_2O_3 ~ 5.6 %) and with concentrations in alkali and alkali-earths too high for us to consider them kaolinitic (mean K_2O ~ 2.7 %, mean MgO ~ 1.5 %). Elements such as Sr, Ba, and P have more variable concentrations, with distributions skewed towards high values that were interpreted as pollution during burial (e.g. Picon 1991) and were not taken into account in the histogram (fig. 3). Two samples differ because of a slightly higher potassium content (BZN463, BZN468). The same clays were used in Serdica to manufacture the whole repertoire, whether glazed or unglazed and designed for cooking or not. These low-calcareous clays are suitable for cooking wares that have to withstand thermal stress, so it was not necessary to look for a different more appropriate material.⁴ It may be noted that the sherds that looked a bit different visually, with a clearer surface reminding us of kaolinitic wares, were not distinguished from the others through chemical analysis.

Chemical analysis of the glazes

To analyze the ceramic glazes, samples were embedded in a polyester resin, cross-sectioned to expose all the layers, polished up to a 1 μm diamond paste, and then carbon-coated for SEM-EDS⁵ analysis. Prior to carbon-coating, the cross-sections were observed under a binocular microscope with magnifications ranging from 10x to 115x.

Glazes analyses were carried out with a SEM (FEI Quanta FEG 250) at the Technological Center for Microstructures (CT μ) in Lyon. The study of the microstructures was mainly performed

³ A detailed description of the analytical setup and its performances may be found in Thirion-Merle 2024.

⁴ This is not the general case, and it is commonly observed that different clays were used in workshops manufacturing both cooking and tablewares; see, for example, Marchesi et al. 1997.

⁵ Scanning Electron Microscopy/Energy Dispersive Spectroscopy.

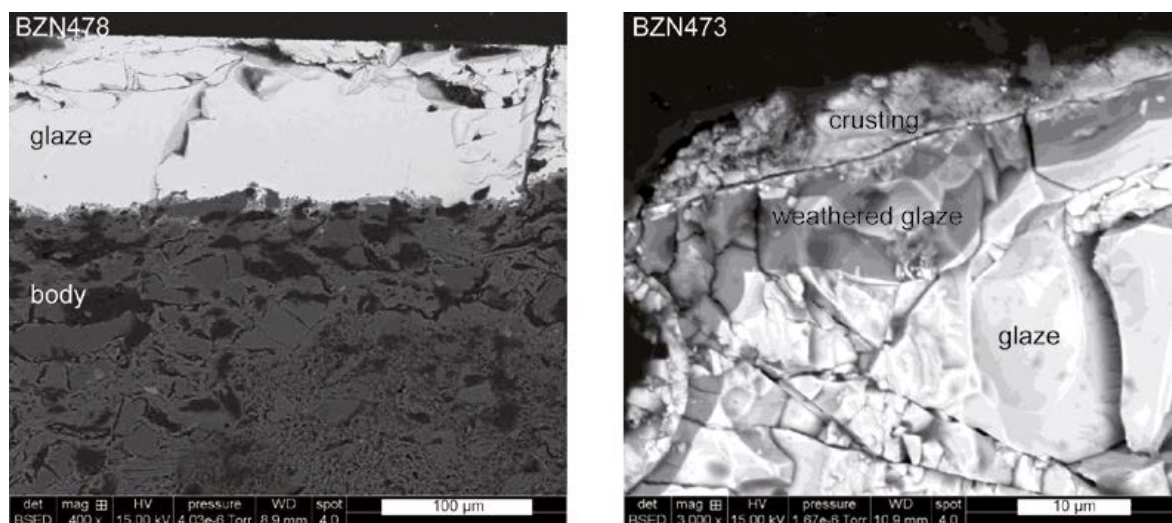


Fig. 4. Backscattered electron (BSE) SEM images of cross-sections of glazed ceramics, showing the chemical contrast between the high-lead glaze and the underlying body (left), and zooming in on the surface structures of a crusting and of altered glaze on top of the initial glaze (samples BZN478 on the left and BZN473 on the right; Photos: J. Burlot)

Обр. 4. Изображения на напречни сечения на глазирана керамика, получени чрез обратно разсеяни електрони (BSE) и сканираща електронна микроскопия (SEM), показващи химическия контраст между глазурата с високо съдържание на олово и намиращото се под нея тяло (вяляво) и увеличение на повърхностните структури на кора и на изменена глазура върху първоначалната глазура (вяляво – проба BZN478, вдясно – проба BZN473; Снимки: Ж. Бурло)

using backscattered electron (BSE) images that differentiate various phases according to the atomic numbers of their elementary content (fig. 3). Elemental compositions were determined by energy-dispersive X-ray spectrometry (EDS) using an Oxford Instruments Ultim[®] Max 65 mm2 silicon drift detector. All measurements were operated at 15 kV acceleration voltage in 60 s by setting the working distance at about 10 mm. Standardless quantification was performed using a PAP correction method of the intensities. AZtecLive software was used for data acquisition and evaluation, and the reliability of the results was tested by measuring reference glasses and geological standards (Corning Brill B, C and D, DR-N). The compositions of the glazes are given as an average of three to five measurements on different zones that avoided weathered areas and glaze-body interfaces. In addition, spot analyses were carried out on selected inclusions.

The analysis of the glazes shows homogeneous composition. Lead is the only flux, with concentrations reaching 70 % PbO and the contents in alkali being below 1.4 % Na₂O+K₂O. The iron concentrations are between 1.7 and 2.4 % FeO, which may account for the greenish to brownish color of the glazes (when applied to a reduced or oxidized body surface, respectively; Table 2). The respective concentrations of silicon in the bodies and glazes support that lead was applied directly to the body surface and reacted with the silicon of the body to form the glaze (see, for example, Waksman et al. 2008). Weathered layers may be observed as well as superficial layers rich in lead, calcium, and phosphorus that correspond to crustings on some of the round lumps at the surface of the glaze (fig. 4: right).

An interesting feature is the localized concentration of bismuth (up to 4 % Bi₂O₃) in inclusions rich in lead at the body-glaze interface (sample BZN467, Table 2, fig. 5).⁶ Such high concentrations in Bi are rare in lead objects (L'Héritier et al. 2015) but were found in a wooden-lead anchor sunk in the Black Sea off the coast of Bulgaria (Kuleff et al. 1995). Although the attribution of this

⁶ Further investigations by LA-ICP-MS analysis are ongoing.

id.	Al ₂ O ₃	SiO ₂	PbO	Na ₂ O	MgO	K ₂ O	CaO	FeO	TiO ₂	SrO	Bi ₂ O ₃
glazes											
BZN467	5.8	18.1	71.3	0.4	0.5	0.6	0.7	1.8	0.2	0.3	
BZN469	5.8	16.2	73.4	0.2	0.5	0.4	0.9	1.8	0.2	0.3	
BZN473	5.1	15.6	74.2	0.3	0.4	0.3	1.3	1.7	0.2	0.3	
BZN475	5.3	17.7	71.3	0.2	0.5	0.6	0.7	2.8	0.2	0.2	
BZN478	6.0	19.6	69.6	0.3	0.5	0.5	0.5	1.8	0.2	0.6	
BZN487	7.4	22.2	64.2	0.6	0.7	0.7	0.9	2.4	0.3	0.4	
inclusions at the body-glaze interface, BZN467											
spectrum 1	7.9	23.1	57.4	0.2	0.2	2.6	0.8	2.5	0.4	0.1	3.9
spectrum 2	12.9	29.6	50.1	0.3	0.2	3.2	0.8	1.3	0.2	0.2	1.0

Table 2. Semi-quantitative chemical analysis of the glazes and of the selected inclusions shown on fig. 5, scanning electron microscopy with energy-dispersive spectroscopy (SEM-EDS; oxide weight %)

Таблица 2. Полуколичествен химичен анализ на глазуриите и на избраните включения, показани на обр. 5, сканираща електронна микроскопия с енергийно-дисперсионна спектроскопия (SEM-EDS; % тегло на оксида)

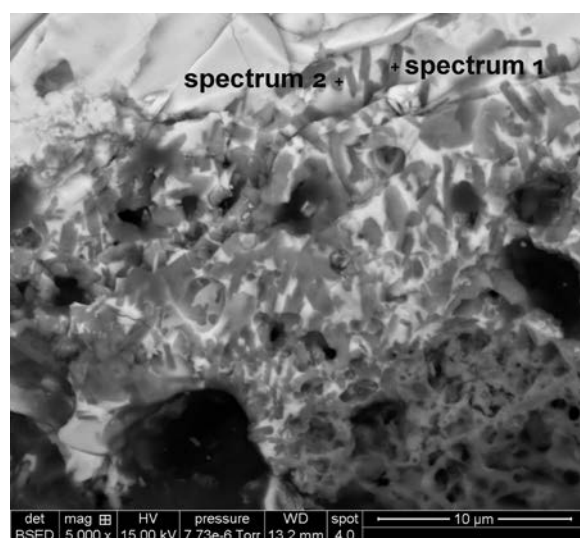


Fig. 5. Inclusions at the body-glaze interface, backscattered electron (BSE) image showing the location of SEM-EDS analysis reported in Table 2 (sample BZN467; Photo: J. Burlot)

Обр. 5. Включения на границата на взаимодействие между керамичното тяло и глазурата, изображение чрез обратно разпръснати електрони (BSE), показващо местоположението на SEM-EDS анализите, отчетени в Таблица 2 (проба BZN467; Снимка: Ж. Бурло)

anchor to a metal source in the Rhodope mountains was not clear, it is probably not by chance that high-Bi lead is found in the glazes of a Bulgarian pottery production as well.

Concluding remarks

Chemical investigations of a sample of ceramics produced in the 6th – early 7th c. Lozenets pottery workshop in Sofia indicate that all the categories of wares, whether glazed or unglazed and designed for cooking or not, were manufactured with the same clay material. Several clues point to a single firing.

Chemical analyses of the bodies and glazes show them to be low-calcareous bodies associated with high-lead glazes. Such features belong to a technical tradition observed in the Balkans and northern Italy in a period that is usually not very well-defined between the 5th and the 7th c. (Capelli et al. 2009; Capelli et al. 2010a; Capelli et al. 2010b). The Lozenets material enables us to specify more precisely that these characteristics are found in a 6th – early 7th c. production that just precedes that of Glazed White Ware I, considered to be the first Byzantine glazed wares *stricto sensu*. The latter continues this tradition, with the nuance that the low-calcareous clays are kaolinic (Waksman et

al. 2007; Waksman et al. 2008).

Our analysis of wares produced in Sofia also shows that the glazes contain phases with high concentrations of bismuth, a potential marker of the lead's provenance.

Acknowledgments

This research was supported by a French-Bulgarian PHC RILA program (project number: 48049UB) co-directed by Evelina Todorova and Sylvie Yona Waksman and funded by the French Ministry for Europe and Foreign Affairs, the French Ministry for Higher Education and Research, and the Bulgarian National Science Fund according to contract No. КП-06-Рила-6/15.12.2021. This work was also made possible by grants to Georgia Giannaki from the Hellenic Foundation for Research and Innovation (HFRI) under the 3rd call for HFRI PhD Fellowship grant (Fellowship Number: 6804) and from the French Academy (Jean Walter Zellidja grant). Our thanks also goes to the staff of the "Céramologie" (CNRS UMR 5138) and the СТμ (Lyon 1 University) platforms in Lyon and to the MONARIS laboratory (UMR 8233) in Paris.

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ХИМИЧНИ АНАЛИЗИ НА КЕРАМИЧНИ ТЕЛА И ГЛАЗУРИ ОТ ГРЪНЧАРСКАТА РАБОТИЛНИЦА, РАЗКОПАНА В КВ. „ЛОЗЕНЕЦ“ (СОФИЯ, VI – НАЧАЛОТО НА VII В.): ПРЕДВАРИТЕЛНИ РЕЗУЛТАТИ

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РЕЗЮМЕ

Подбрани са 40 глазирани и неглазирани керамични фрагмента от VI – началото на VII в., произлизаци от грънчарската работилница в кв. „Лозенец“, София (антична Сердика), за да се изследва техниката им на производство. Химичните анализи на керамичните тела показват, че един и същ нисковаровит глинени материал е използван за производството на всички категории съдове (глазирани и неглазирани, предназначени или не за готвене). Глазурите са с високо съдържание на олово, със забележимо съдържание на бисмут в някои включения, което може да е показателно

за произхода на оловните съединения. Последните вероятно са нанасяни директно върху нисковаровитите тела за образуване на глазурата. По отношение на процеса на опалване на съдовете, съществуващите податки сочат, че то се е осъществявало само веднъж. Въз основа на тези данни се предполага, че производството на глазирана керамика в Сердика е свързано с техническата традиция, наблюдавана на Балканите и в Северна Италия между V и VII в., която продължава да съществува и във „Византийска глазирана белоглинена керамика I“ (GWWI) от VII в.

Ключови думи: съдове с покритие от високооловна глазура, ранновизантийска керамика, Сердика, България, производствени техники, анализ на керамично тяло и глазура, химичен анализ, WD-XRF, SEM-EDS