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Izumi Nakai, Mayumi Matsuzaki, Daichi Sawamura, Yoshinari Abe, Andreas Schachner

Chemical Characterization of Roman and Early Byzantine Glass from Boğazköy/Hattuša and its Vicinity

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Chemical Characterization of Roman and Early Byzantine Glass from Boğazköy/Hattuša and its Vicinity

Keywords: Late Antiquity, Boğazköy, Glass, Trade
Schlüsselwörter: Spätantike, Boğazköy, Glas, Handel
Anahtar sözcükler: Geç Antik, Boğazköy, Cam, Ticaret

INTRODUCTION

Although the understanding of the Hittite levels of the ancient site of Boğazköy was always the main focus of the excavations going on there for more than a century, the periods earlier and later than the second millennium B.C. were not neglected. In fact, a reconstruction of the cultural development of the region covering all periods of human activity was among the aims of the different researchers¹.

During the excavations and surveys, remains of the Roman and Byzantine periods were encountered in various areas of the ancient Bronze Age city. From the relevant records, a comparatively detailed picture of a remote village-type settlement can be reconstructed². Beside accidental finds in different areas, the settlement remains have mostly been discovered through geophysical research and small-scale excavations. They attest the existence of a medium-sized village lying on slightly elevated terraces at the southern edge of the Budaközü plain, which stretches to the north of the site. Surface surveys in the plain indicate a loose settlement structure scattered in the plain. The Roman road from Tavium (mod. Büyüknefesköy) passes a few kilometers west of the Bronze Age city and traverses the plain to its north to continue eastward until Amasia (mod. Amasya).

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Sources of Illustrations: Fig. 1–3 = I. Nakai. – Fig. 4–8 = Boğazköy-Archive.

¹ Schachner 2011, 21–32.

² For a general overview: Schachner 2011, 331–342; Kühn 2014.

At Boğazköy, the most important finds of the Antiquity come from a necropolis, which was used extensively and was perpetually used from the Hellenistic to the Late Imperial Times. It mainly stretches out across the areas west and south of the Great Temple of the Hittite period³. The dating of the individual graves is possible thanks to the different burial customs and grave types – ranging from the small tumuli of the Hellenistic period to clay sarcophagi, terracotta coffins, stone cists, graves covered with roof tiles, and simple inhumations of the Roman and Late Roman period – typologically different finds, and coins⁴. A number of glass vessels and fragments of the vessels analyzed in the work presented here have been excavated from this Roman cemetery. A second group of material incorporated into the present study comes from a looted cemetery of the same imperial Roman age in the vicinity of Sungurlu, which lies about 30 km northwest of Boğazköy. Typologically, the studied materials represent a typical repertoire of the Roman period.

In contrast, their chemical composition has not yet been researched. As these finds we are dealing with have been found in a region that must be considered very remote in terms of the geography of the Roman Empire, an analysis of their chemistry seemed promising for various reasons: it is very interesting to know the origin of these glasses, whether they were produced using local raw materials or produced using raw materials such as glass chunks imported from central production centers, namely, secondary production. In the summer of 2013, we were able to analyze these glass finds housed at the Boğazköy museum using nondestructive methods and so chemically characterized the glass.

Chemical Composition of Glass: General Remarks

The chemical composition of the glass reflects the source material and where it was produced, hence the origin of the glass. Therefore, chemical analysis plays a very important role in studying historical glass of unknown origins.

In western Asia, the basic chemical type of the glass is soda-lime-silica glass, whose typical composition is as follows: $\text{SiO}_2 \sim 70\%$, $\text{Na}_2\text{O} + \text{K}_2\text{O} \sim 20\%$, and $\text{CaO} + \text{SrO} + \text{Al}_2\text{O}_3, \sim 10\%$. Namely, the major component of the glass is SiO_2 (silica), whose source materials are desert sands or coastal sands. These sands tend to contain iron oxide, titanium oxide, manganese oxide, etc. as impurities, reflecting the local geology where the sands are collected. Therefore, Fe, Ti, and Mn are important target elements for the analysis of the glass.

Alkali such as soda (sodium carbonate) was added to silica as flux to lower the melting temperatures of the glass. It is well known that two different sources of alkali were used as flux to produce glass in ancient times: plant ash and mineral soda⁵. The former is literally ash of certain types of plants. The latter is well known as natron, which is a natural mineral with the chemical formula $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and is collected from Wadi Natrun, a salt lake located between Cairo and Alexandria in Egypt. The word natron is often more widely used as a general term referring to the minerals of sodium carbonates used in ancient West Asia. It is generally recognized that soda-lime glass with both K_2O and MgO levels being greater than approximately 1.5% were made with plant ashes⁶, because Mg and K are essential to plant life and are abundant in plants.

³ Schachner 2011, 328 Abb. 152.

⁴ Kühne 1969; Kühn 2014.

⁵ Sayre – Smith 1961.

⁶ Sayre – Smith 1961.

Typical plant ash glasses include Sassanian glass and Islamic glass after the eighth century A.D.⁷ On the other hand, it is known that Roman and Hellenistic glasses were produced using natron as an alkali source⁸, which does not contain K or Mg as essential components. Therefore, the concentrations of K₂O and MgO are lower than in plant ash glasses.

The chemical compositions of the natron glass of Roman and Hellenistic glass produced around the Mediterranean regions can be further subdivided into the following three types based on the impurities of the source materials such as silica sands: i) (Roman) blue-green type, ii) Levantine type, and iii) HIMT (high iron manganese titanium) type. These groups were introduced by Freestone and his colleagues⁹. Each type has following characteristics¹⁰.

Blue-green glass is typical of Roman glass of the late first to third centuries A.D. The chemical composition is soda-lime-silica glass. The term blue-green is due to the color of this type of glass, which is due to the presence of the Fe²⁺ ion. The origin of the sand was examined by isotope analysis¹¹, and there is a possibility that the sands used to produce the glass come from several coasts around the Mediterranean seashore. It should be noted that there is a possibility of the recycling of old glass in this type of glass. It is characteristic of this glass that antimony was used as a decolorizer, especially in early Roman glass. After the second century A.D., manganese began to be used as a decolorizer, resulting in a reduction in the use of antimony, and finally the use of antimony as a decolorizer disappeared¹².

HIMT glass was first recognized by Freestone and also identified as a widely traded variety in the western Mediterranean regions. This type of glass exhibits high levels of the oxides of iron (> 0.7%), magnesium, manganese (~ 1–2%), and titanium (> 0.1%). They exhibit a strong positive intercorrelation and also exhibit a correlation with alumina. It is reported that this glass was traded in Italy, Britain, Germany, and Sinai. In the Romano-British assemblage, there are two groups of HIMT glasses, i. e., HIMT 1 and HIMT 2. HIMT 1 glasses contain, on average, double the proportion of iron, manganese, and titanium oxides than HIMT 2 samples. They became widespread sometime in the fourth century. HIMT 2 glass became common during the early fourth century A.D., while HIMT 1 became common after the mid fourth century A.D.

The Levantine coast has been the location of large-scale glass making in Antiquity¹³. Glass making factories were discovered at archaeological sites such as Bet Shearim, Bet Eli'ezer (Hadera), and Apollonia (Arsuf), all in present-day Israel¹⁴. Levantine 1 glass was first reported by Freestone and his group¹⁵ and corresponds to Group 3 as defined by Foy and his colleagues¹⁶. The glass was made using coastal sand of the Syro-Palestinian region around the mouth of the River Belus, in the Bay of Haifa. It appears to have been the typical glass of the Levant between the fourth and seventh centuries A.D.

⁷ Brill 1999.

⁸ Whitehouse 2002; Shortland *et al.* 2006.

⁹ Freestone 2006.

¹⁰ Jackson *et al.* 1991; Freestone 2006; Foster – Jackson 2009; Geilmann 1955.

¹¹ Ganio *et al.* 2012.

¹² Sayre 1963; Jackson 2005.

¹³ Freestone 2002.

¹⁴ Gorin-Rosen 2000.

¹⁵ Freestone *et al.* 2000.

¹⁶ Foy *et al.* 2003.

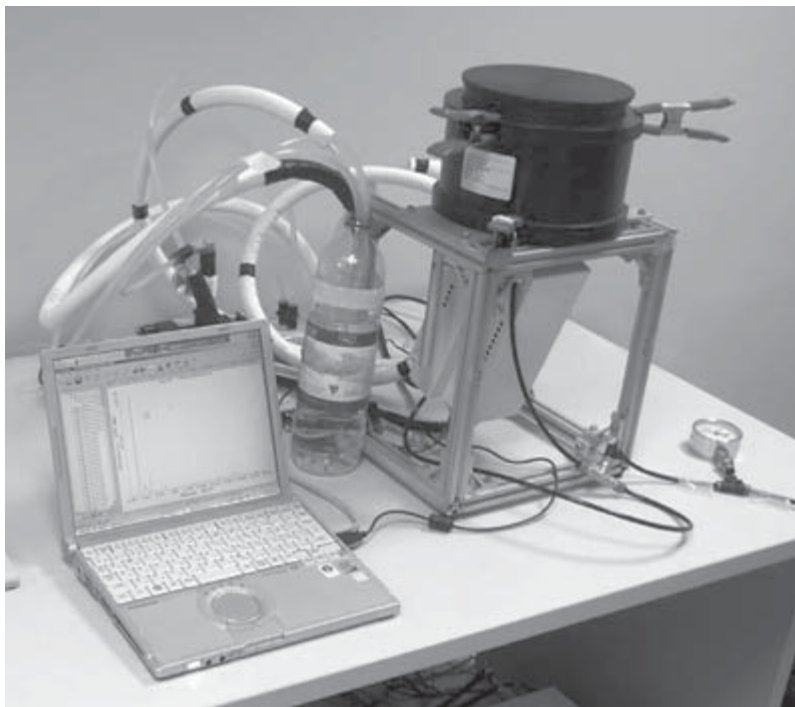


Fig. 1 General photograph of the XRF spectrometer developed by the authors' group

Levantine 1 and the HIMT glasses can be distinguished from the typical Roman blue-green glass of the first through third centuries based on their chemical composition¹⁷. The reason is hidden in the fact that large-scale political and economic changes occurred in the fourth and fifth centuries A.D., causing the fragmentation of the Roman Empire into two parts. Such political instability must also effect glass production, including changes in the silica sources, which results in changes in the chemical compositions of the glasses. We will discuss the analytical data of the Roman and possibly early Byzantine glasses excavated from a Late Roman cemetery at Boğazköy and purchased by the local Museum from the immediate vicinity based on the above criteria. The purpose of the present study is to estimate the possible origin of the Boğazköy glass.

THE CHEMICAL ANALYSIS OF THE BOĞAZKÖY GLASS SAMPLES

Samples

Table 1 lists the samples quantitatively analyzed in this study. A total of 29 samples of vessels or fragments of vessels were excavated from a Late Roman cemetery at Boğazköy, Turkey or were purchased by the local Museum from the immediate vicinity. Photographs of these samples are shown in *Figs. 4–7*. The samples are naturally colored glasses or artificially decolorized glasses. The samples were subjected to nondestructive XRF analysis as detailed below.

¹⁷ Foy *et al.* 2003; Freestone *et al.* 2002.

Fig. 2 General photograph of the XRF spectrometer developed by the authors' group with a glass sample inside the vacuum chamber



XRF Analysis

A portable XRF spectrometer, OURSTEX 100FA IV (OURSTEX Co.), was brought to an experimental room in the museum at Boğazköy. The XRF instrument used (*Fig. 1–2*) was specially designed for glass analysis. The analytical procedures and conditions are described elsewhere¹⁸. The measurement time was 200 s (live time), and the tube current (mA) was adjusted so that the dead time did not exceed 30 %. Standard glass samples (NIST SRM610, 612, 621, 1412, 1830, 1831) and 15 synthetic glasses that had been analyzed using inductively coupled plasma-atomic emission spectrometry (ICP-AES) by the authors' group were used to generate calibration curves for quantitative analysis. For elements from Co to Zr, monochromatic X-ray mode operated at 40 kV and 1.0 mA was used. For the analysis of light elements, from Na to Fe, and heavier elements than Sn, white X-ray mode at 40 kV and 0.25 mA was applied. The measured net XRF intensity was normalized to the Compton scattering peak of the Pd $K\alpha$ line (21.125 keV).

A multivariate statistical analysis using StatPartner ver. 2.0 was carried out to characterize the chemical composition of the glass samples and to distinguish between glasses with similar compositions. For cluster analyses of the samples, Ward's method was applied, and the following elements were used as index elements: Mg, Ca, Ti, Fe, Sr, Zr, and Sb. The dendrogram was obtained using Ward's method.

¹⁸ Kato *et al.* 2009; Kato *et al.* 2010; Tantrakarn *et al.* 2009; Abe *et al.* 2012.

Assigned group	Reg. No.	Sample ID	Concentration/wt%			
			Na ₂ O	MgO	Al ₂ O ₃	SiO ₂
1a	"exhibits No.5 left 1-219-72"	T-004	12.89	0.55	1.78	76.80
1a	"1-145-88 Bo 88/146"	T-066	17.69	0.72	2.02	70.19
1a	1-223-72	T-084	15.42	1.09	2.40	71.17
1a	1-224-72	T-085	22.97	0.71	2.30	63.61
1a	1-66-69	T-101	23.14	0.71	2.11	64.23
1b	1-215-72	T-077	12.02	1.31	5.92	70.71
1b	1-213-72	T-088	18.17	0.91	3.28	65.98
1b	1-210-72	T-092	13.08	1.22	6.24	68.28
1	1-68-68	T-100	11.61	0.87	1.96	77.44
1	"Bo 1983 e-83-24 (b)"	T-127	16.52	0.65	2.38	71.65
1	e-83-119	T-139	16.74	0.62	1.92	72.14
1	e-83-121	T-140	14.37	0.57	1.92	74.28
1	–	T-159	12.22	0.62	2.49	76.61
1c*	"exhibits No.4 1-221-72"	T-002	15.79	1.47	7.41	59.06
1c	"exhibits No.3 1-227-72"	T-003	11.11	0.85	5.75	69.49
1c*	1-225-72	T-086	25.45	0.86	4.53	53.41
2	"1-181-77 Bo 77/181"	T-031	20.12	0.97	2.47	65.74
2	1-218-72	T-081	17.16	1.45	2.97	67.41
2*	302-57	T-141	22.94	1.23	2.63	60.74
2	Bo 10/75	T-144	18.53	0.78	2.56	68.31
2*	BO 09/234	T-148	23.86	1.33	2.68	60.65
2	BO 09/1030	T-149	18.80	0.96	2.24	66.87
3	"exhibits No.2 right 1-230-72"	T-005	10.85	0.89	5.66	70.00
3	1-211-72	T-076	21.05	0.65	3.30	64.37
3	1-214-72	T-079	13.38	0.98	5.54	71.23
3	1-208-72	T-094	11.11	1.08	6.56	71.53
3	1-71-69	T-096	18.76	1.07	3.24	65.40
3	1-70-69	T-097	21.90	0.88	3.10	63.98
3	1-64-69	T-102	13.29	0.72	3.33	71.84

Table 1a XRF analysis of the glass samples from Boğazköy, listed according to their assigned compositional groups. Interpretation of the possible decolorizer is shown; major elements (%); cont. p.242

Concentration/wt%								"Possible decolorizer"
K ₂ O	CaO	MnO	Fe ₂ O ₃	CuO	PbO	SnO ₂	Sb ₂ O ₃	
0.55	6.01	0.716	0.351	tr.	0.005	tr.	0.348	Sb/Mn
0.74	7.37	0.588	0.505	tr.	0.007	n.d.	0.162	Sb/Mn
0.62	7.39	0.823	0.745	tr.	0.023	n.d.	0.319	Sb/Mn
0.76	8.07	0.843	0.442	tr.	0.009	tr.	0.272	Sb/Mn
0.70	7.58	0.615	0.585	0.003	0.016	n.d.	0.316	Sb/Mn
0.84	7.92	0.381	0.631	tr.	0.005	n.d.	0.272	Sb/Mn
1.11	9.32	0.509	0.634	0.006	0.010	n.d.	0.058	Sb/Mn
0.94	8.66	0.638	0.752	0.008	0.014	tr.	0.139	Sb/Mn
0.57	6.28	0.121	0.571	0.107	0.011	tr.	0.544	Sb/Mn
0.61	7.14	0.353	0.419	0.027	0.014	tr.	0.273	Sb/Mn
0.59	6.89	0.361	0.425	tr.	0.005	tr.	0.304	Sb/Mn
0.65	7.09	0.370	0.464	tr.	0.006	n.d.	0.277	Sb/Mn
0.53	6.38	0.209	0.489	tr.	0.007	tr.	0.438	Sb/Mn
1.48	12.36	1.873	0.548	tr.	tr.	tr.	n.d.	Mn
0.83	9.04	2.394	0.528	tr.	tr.	tr.	n.d.	Mn
0.94	12.06	2.110	0.629	tr.	tr.	tr.	n.d.	Mn
0.62	7.92	1.056	1.059	0.007	0.008	tr.	tr.	–
0.67	8.16	1.247	0.902	tr.	tr.	tr.	n.d.	–
1.02	8.78	1.155	1.460	0.012	0.010	0.007	tr.	–
0.48	6.97	1.159	1.179	0.007	0.012	0.010	tr.	–
0.68	8.44	1.214	1.122	0.006	0.006	n.d.	n.d.	–
0.66	8.34	1.243	0.860	0.007	0.008	n.d.	tr.	–
1.18	10.08	0.879	0.445	0.005	tr.	tr.	n.d.	Mn
0.68	9.22	0.336	0.381	tr.	tr.	tr.	n.d.	Mn
0.73	7.06	0.587	0.471	tr.	tr.	tr.	tr.	Mn
0.84	8.10	0.392	0.361	0.084	tr.	tr.	tr.	Mn
0.86	9.31	0.799	0.519	0.106	0.004	tr.	tr.	Mn
0.58	8.01	1.197	0.347	0.093	tr.	0.008	n.d.	Mn
0.73	8.22	1.434	0.403	0.094	tr.	0.009	n.d.	Mn

* Poor quality data (SiO₂ content < 60%) tr.: trace amount n.d.: not detected

Assigned group	Reg. No.	Sample ID	Concentration/ppm				
			TiO ₂	CoO	NiO	ZnO	As ₂ O ₃
1a	"exhibits No.5 left 1-219-72"	T-004	482	n.d.	n.d.	32	n.d.
1a	"1-145-88 Bo 88/146"	T-066	682	n.d.	tr.	28	n.d.
1a	1-223-72	T-084	889	n.d.	n.d.	33	n.d.
1a	1-224-72	T-085	614	n.d.	n.d.	32	tr.
1a	1-66-69	T-101	732	tr.	tr.	40	tr.
1b	1-215-72	T-077	790	tr.	n.d.	35	tr.
1b	1-213-72	T-088	742	tr.	tr.	41	n.d.
1b	1-210-72	T-092	1022	tr.	tr.	44	n.d.
1	1-68-68	T-100	945	tr.	tr.	tr.	n.d.
1	"Bo 1983 e-83-24 (b)"	T-127	673	n.d.	n.d.	tr.	n.d.
1	e-83-119	T-139	581	tr.	tr.	29	tr.
1	e-83-121	T-140	573	tr.	n.d.	27	tr.
1	–	T-159	679	n.d.	n.d.	tr.	n.d.
1c*	"exhibits No.4 1-221-72"	T-002	727	n.d.	tr.	30	tr.
1c	"exhibits No.3 1-227-72"	T-003	696	n.d.	tr.	28	tr.
1c*	1-225-72	T-086	614	n.d.	n.d.	32	tr.
2	"1-181-77 Bo 77/181"	T-031	1937	tr.	n.d.	65	n.d.
2	1-218-72	T-081	1042	tr.	tr.	26	n.d.
2*	302-57	T-141	1599	tr.	tr.	55	n.d.
2	Bo 10/75	T-144	2130	tr.	n.d.	40	n.d.
2*	BO 09/234	T-148	1951	tr.	tr.	37	n.d.
2	BO 09/1030	T-149	1256	tr.	n.d.	27	n.d.
3	"exhibits No.2 right 1-230-72"	T-005	689	tr.	n.d.	tr.	n.d.
3	1-211-72	T-076	498	n.d.	n.d.	tr.	n.d.
3	1-214-72	T-079	648	tr.	n.d.	tr.	n.d.
3	1-208-72	T-094	547	tr.	n.d.	tr.	n.d.
3	1-71-69	T-096	732	n.d.	n.d.	29	n.d.
3	1-70-69	T-097	557	n.d.	n.d.	tr.	n.d.
3	1-64-69	T-102	575	n.d.	tr.	tr.	tr.

Table 1b XRF analysis of the glass samples from Boğazköy, listed according to their assigned compositional groups. Interpretation of the possible decolorizer is shown; trace elements (ppm).

Concentration/ppm				Compositional type	Archaeological dating
Rb ₂ O	SrO	Y ₂ O ₃	ZrO ₂		
9	479	tr.	56	Roman blue-green	2 nd –4 th cent A.D.
tr.	502	tr.	64	Roman blue-green	2 nd –4 th cent A.D.
tr.	490	tr.	57	Roman blue-green	2 nd –4 th cent A.D.
tr.	499	tr.	51	Roman blue-green	2 nd –4 th cent A.D.
10	544	tr.	72	Roman blue-green	2 nd –4 th cent A.D.
9	527	tr.	70	Roman blue-green	2 nd –4 th cent A.D.
13	567	tr.	59	Roman blue-green	2 nd –4 th cent A.D.
9	531	tr.	67	Roman blue-green	2 nd –4 th cent A.D.
9	446	tr.	89	Roman blue-green	2 nd –4 th cent A.D.
tr.	434	tr.	56	Roman blue-green	2 nd –4 th cent A.D.
tr.	466	tr.	59	Roman blue-green	2 nd –4 th cent A.D.
7	453	tr.	56	Roman blue-green	2 nd –4 th cent A.D.
tr.	447	tr.	63	Roman blue-green	2 nd –4 th cent A.D.
15	691	tr.	42	"Roman blue-green or Levantine"	2 nd –4 th cent A.D.
10	711	tr.	49	"Roman blue-green or Levantine"	2 nd –4 th cent A.D.
tr.	499	tr.	51	"Roman blue-green or Levantine"	2 nd –4 th cent A.D.
tr.	489	tr.	123	HIMT	2 nd –3 rd cent A.D.
7	442	tr.	48	HIMT	2 nd –3 rd cent A.D.
7	632	tr.	104	HIMT	"2 nd –1 st cent B.C.E. ? But also 2 nd –3 rd cent A.D. possible."
tr.	502	tr.	151	HIMT	2 nd –3 rd cent A.D.
7	563	tr.	99	HIMT	2 nd –3 rd cent A.D.
tr.	673	tr.	97	HIMT	2 nd –3 rd cent A.D.
10	579	7	47	Levantine	2 nd –3 rd cent A.D.
8	506	tr.	50	Levantine	2 nd –3 rd cent A.D.
7	415	tr.	49	Levantine	2 nd –3 rd cent A.D.
9	481	tr.	50	Levantine	2 nd –3 rd cent A.D.
7	547	tr.	56	Levantine	2 nd –3 rd cent A.D.
tr.	406	tr.	43	Levantine	2 nd –4 th cent A.D.
8	533	tr.	51	Levantine	2 nd –4 th cent A.D.

* Poor quality data (SiO₂ content < 60%) tr.: trace amount n.d.: not detected

Limitations and Opportunities of Non-destructive Analysis

The present XRF analyses were carried out nondestructively. Before analysis, a small part of the sample surface was polished with fine emery papers (> #1000) if possible. It is known that the Na₂O contents of typical soda-lime glass range from 15 to 20 wt%. If the Na₂O content of the sample is significantly less than 15 wt% (Table 1a), this may be due to heavy weathering of the glass surface, caused by long-term underground burial conditions. The analytical data for SiO₂ were obtained by subtracting the total analytical data other than silica from 100%. The silica content of typical soda-lime glass should not exceed 80% nor be below 60%; some of the data are seriously affected by surface weathering. Such data are marked with an asterisk in Table 1 and are considered for reference and not used for calculating average values. This is a limitation of the nondestructive analysis of archaeological glass by XRF. However, our experience suggests that though the contents of Na and Si are seriously affected by weathering, it is not such a concern for the heavier elements as long as the glass has a shiny surface¹⁹.

Results of the Analysis

The results of the XRF analysis are given in Table 1a and b. The major and minor element compositions (wt%) of the glass samples are listed in Table 1a, while the trace element compositions (ppm) are given in Table 1b. Characterization of the samples was carried out using a two-components plot, and the analytical data were compared with the literature data. The literature analytical data of the following four compositional glass types were used for comparison: Roman blue-green type (118), Levantine type (24), HIMT type 1 (123), and HIMT type 2 (221), where the number of data is given in parentheses²⁰. These literature data are obtained by destructive analysis of colorless or naturally colored glass with well-documented archaeological contexts.

The Base Glass Composition, Source of Alkali, and Decolorizer

It was found that all the glasses are soda-lime-silica glass. To reveal the alkali source, the concentrations of K₂O and MgO of the analyzed glasses are plotted in Fig. 3 (a), where the analytical data of Roman to post-Roman glass are shown for comparison. All the samples exhibited K₂O and MgO levels lower than 1.5 wt%, indicating that natron was used as an alkali source. The analytical data are comparable to the reference data shown in Fig. 3 (a). These characteristic chemical compositions are typical of Roman and early Byzantine glass.

The colors of the analyzed glass range from pale green and pale greenish blue to colorless. The greenish color is due to iron impurities. Therefore, these glasses do not contain any colorants and are naturally colored or decolorized. In order to reduce the greenish color, antimony or manganese was added as a decolorizer. Antimony oxides act as an oxidizing agent of Fe²⁺, which is responsible for the green color. Manganese ion compensates for the color of iron; the blue-green coloring can be corrected by the addition of the purple color of Mn³⁺.

It has been suggested that the beginning of the use of antimony as a decolorizer dates back to the eighth century B. C. in Nimrud (Northern Mesopotamia). On the other hand, the beginning of the use of manganese as a decolorizer is not clear because many glasses more or less contain

¹⁹ Kato *et al.* 2009; Kato *et al.* 2010.

²⁰ Freestone *et al.* 2005; Jackson 2005; Silvestri *et al.* 2005; Foster – Jackson 2009.

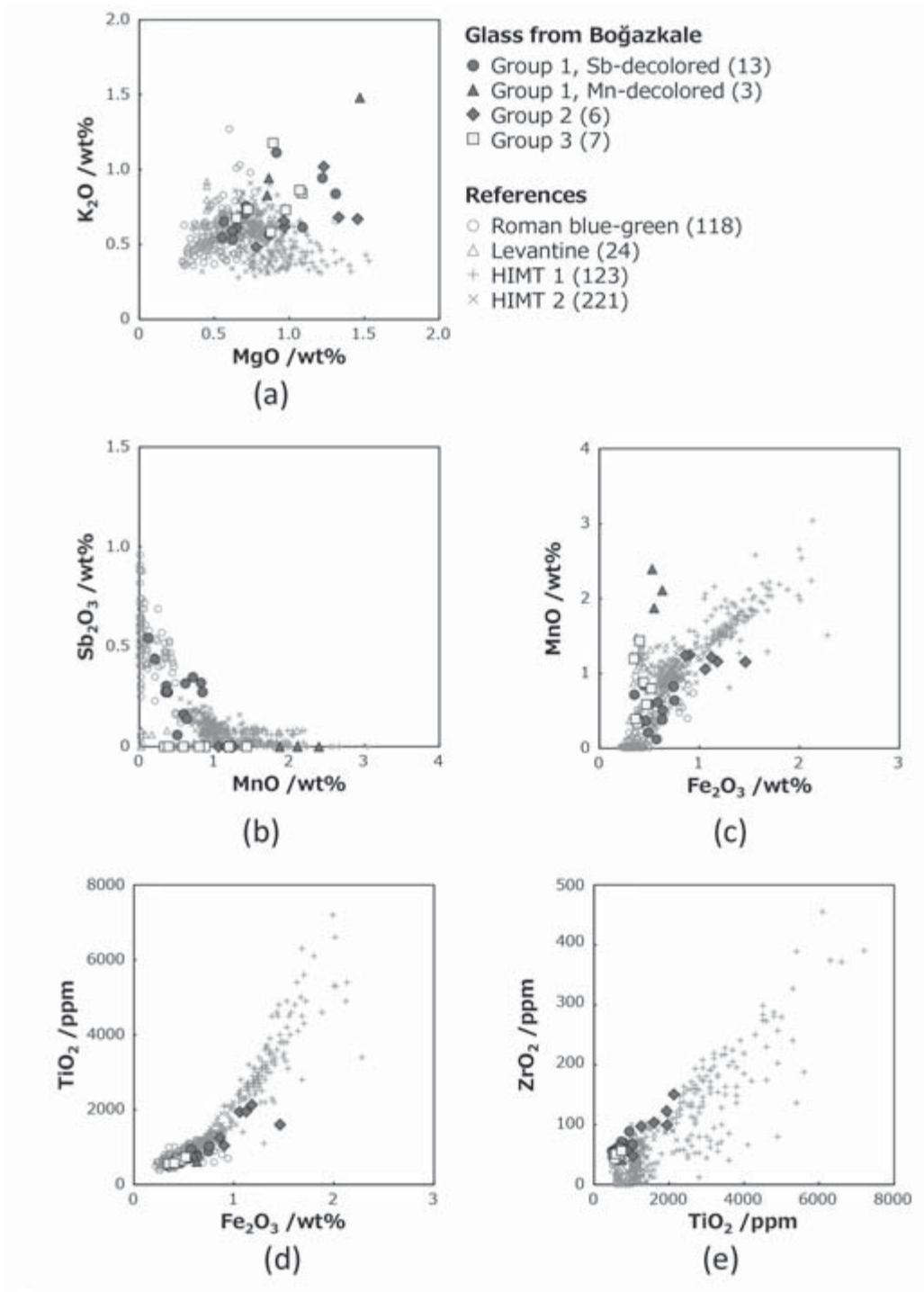


Fig. 3 Characterization of the glass samples from Boğazköy using two component plots, which also show reference analytical data for four compositional types (Blue-green, Levantine, HIMT 1, and HIMT 2): (a) K_2O vs. MgO , (b) Sb_2O_3 vs. MnO , (c) MnO vs. Fe_2O_3 , (d) TiO_2 vs. Fe_2O_3 and (e) ZrO_2 vs. TiO_2 plots.

manganese as an impurity in sand. Therefore, it is often difficult to judge whether manganese is intentionally added or not. It is known that the Roman blue-green type used antimony for decoloring until the second century A.D. However, in later periods, antimony was not used for Levantine nor HIMT glass. *Fig. 3 (b)* shows an Sb_2O_3 vs. MnO plot for the analytical and reference data. The reference Roman green samples (open circles) are located in the Sb-rich region from 0.1 to 1.0 % Sb_2O_3 . It is found from *Fig. 3 (b)* that the literature data for the Sb_2O_3 contents of the HIMT glass (\times , + marks) and Levantine glass (open triangle marks) are 0.2 % or less. In contrast, the MnO content of the HIMT glass is higher than 0.5 %. It is found that our Group 1 samples from Boğazköy (\bullet marks) are located in the region of the Roman blue-green type. The black triangle, diamond, and square marks represent glass containing a certain amount of manganese and an absence of antimony. Thus, the former samples are examples of Roman blue-green type, and the latter samples are not. The color of the former glass is slightly bluish in tint compared with the latter.

The highest Mn content for Boğazköy glass was 2.4 %. There are two possibilities of the origin of manganese, i. e., intentionally added decolorizer or an impurity associated with iron. *Fig. 3 (c)* shows the MnO vs. Fe_2O_3 plot for the Boğazköy glass and the literature data. If manganese is an impurity in the iron source, there will be a linear correlation between iron and manganese. This tendency was observed for the literature data of the HIMT glass in *Fig. 3. (c)*. The Boğazköy glass, represented by the diamond marks, is located in the region of the literature data of the HIMT glass (\times and + marks). On the other hand, the Boğazköy glass shown with the black triangle marks are significantly more-rich in Mn compared with Fe_2O_3 .

CLASSIFICATION OF THE BOĞAZKÖY GLASS SAMPLES

Roman Blue-green Glass

It is known that Sb_2O_3 decolorizer was only used for Roman blue-green glass, while Levantine glass and HIMT glass do not use Sb_2O_3 . The accidental inclusion of antimony into the glass as a contaminant of the raw materials is unlikely because antimony is a unique element, and neither silica nor natron contain antimony as an impurity. Therefore, the Sb_2O_3 content from 580 ppm to 5440 ppm in the 13 Boğazköy glass samples is assumed to be intentionally added to the glass as a decolorizer (*Fig. 4*). These glasses are assigned as Group 1, Roman blue-green type, in Table 1.

The concentration of manganese oxide for these glasses ranged from 0.12 to 0.84 %. Sayre and Smith found that colorless glasses from the Syrian coast are characterized by the increasing use of manganese oxide (MnO) rather than antimony oxide, with concentrations on the order of 1 %²¹. In Italy and northern Europe, glass was generally decolorized with either antimony or antimony/manganese until the end of the third century A. D., when an increase in manganese is observed. This assumes that the intentional addition of decolorizers is above 0.2 % in each case²². In Boğazköy glass, the Group 1a glass samples contain MnO above 0.5 %, and we assume that manganese was intentionally added together with antimony as decolorizer. On the other hand, the Group 1b glasses contain high levels of alumina (> 3 %), implying a different origin of the

²¹ Sayre – Smith 1961.

²² Jackson 2005; Sayre 1963; Sayre – Smith 1967.

source sand. The rest of the Group 1 samples are decolorized using antimony oxides ($> 0.25\%$), predominantly with a relatively moderate level of iron oxide ($< 0.6\%$). The archaeological dating of these glasses is from the second to fourth century A. D., which is in good agreement with the chronology of the blue-green glass of the Roman empire and also that of the antimony/manganese decolorizer.

Group 1c is Roman blue-green glass with manganese decolorizer ($\text{MnO } 2.39\%$) (Fig. 5). The samples do not contain any antimony. The characteristic of this group is that the alumina contents are high (Al_2O_3 5.75%). There is a possibility that the Group 1c glasses belong to the Levantine 1 type. Aluminum is a light element, and analytical data obtained by XRF analysis tend to be affected by the surface conditions. Accordingly, information of the chemical composition is not enough to clearly characterize Levantine 1 glass. Further information will be obtained through the Sr, Nd, and Pb isotope analysis of the samples²³.

HIMT Glass

HIMT glass can be characterized by high levels of iron ($> 0.7\%$), manganese (usually $\sim 1\text{--}2\%$), and titanium ($> 0.1\%$). HIMT glass can be further classified into two types: HIMT 1 and HIMT 2, depending on the concentrations of the transition elements. HIMT 1 glasses contain, on average, higher amounts of iron (1.36%), manganese (1.71%), and titanium (0.33%) than HIMT 2 glasses (0.72%, 0.98%, and 0.12%, respectively). The glasses in Group 2 ($n = 4$) have medium levels of Fe_2O_3 (0.86–1.18%, av. 1.0%), MnO (1.06–1.25%, av. 1.18%), and TiO_2 (0.104–0.213%, av. 0.159%), which are closer to the criteria of HIMT 2 glass.

Fig. 3 (d) shows the TiO_2 vs. Fe_2O_3 plot. From the literature, HIMT glass (+ and × marks) shows a strong positive correlation between iron and titanium. The Boğazköy glasses in Group 2 are indicated with a diamond mark and are located in the region of the HIMT glass in Fig. 3 (d). Fig. 3 (e) shows the ZrO_2 vs. TiO_2 plot; there is a positive correlation of the two elements for HIMT glass. Group 2 samples are distributed among the data of the reference HIMT glasses. From these observations, the Group 2 glasses from Boğazköy are considered to be HIMT 2 glass (Fig. 6). The presence of higher soda (18–19%) and magnesia (usually $> 0.8\%$) and lower lime ($\sim 6\%$) contents are other characteristics of HIMT glass. The Group 2 glass ($n = 4$; Table 2) contained Na_2O (17.16–20.12%, av. 18.65%) and MgO (0.78–1.45%, av. 1.04%) concentrations that are consistent with the criteria, though the CaO contents are slightly higher (6.97–8.34%, av. 7.85%).

HIMT glass was produced using relatively impure sand sources, and a recent isotope analysis suggested that the sand came from the Near East, probably Egypt²⁴. Elemental and lead isotope data show that HIMT glass was traded as far afield as the south of Britain, Italy, Germany, and the Sinai. It is assumed that HIMT glass became widespread sometime in the fourth century A. D.²⁵. The archaeological dating of the Group 2 samples from Boğazköy are the third and probably fourth centuries A. D., which seems to roughly fit with the general occurrence of HIMT glass.

²³ Degryse *et al.* 2005; Degryse – Schneider 2008.

²⁴ Freestone *et al.* 2005; Leslie *et al.* 2006.

²⁵ Freestone *et al.* 2002.



Fig. 4 Photographs of the glass samples from Boğazköy Museum classified as group 1 (Roman blue-green type, Sb decolorizer) based on the chemical composition. Scale 1:2



Fig. 5 Photographs of the glass samples from Boğazköy Museum classified as group 1c (Roman blue-green type, Mn decolorizer) based on the chemical composition. Scale 1:2



Fig. 6 Photographs of the glass samples from Boğazköy Museum classified as group 2 (Roman HIMT type) based on the chemical composition. Scale 1:2

Assigned group		Concentration/wt%										Concentration/ppm		
		Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	MnO	Fe ₂ O ₃	Sb ₂ O ₃	TiO ₂	SrO	ZrO ₂	
1a (n = 4)	min	12.89	0.55	1.78	63.61	0.55	6.01	0.59	0.35	0.16	482	479	51	
	max	23.14	1.09	2.40	76.80	0.76	8.07	0.84	0.75	0.35	889	544	72	
	mean	18.42	0.75	2.12	69.20	0.67	7.28	0.72	0.53	0.28	680	503	60	
	st.dev.	4.56	0.20	0.24	5.44	0.09	0.77	0.12	0.15	0.07	150	25	8	
1b (n = 3)	min	12.02	0.91	3.28	65.98	0.84	7.92	0.38	0.63	0.06	742	527	59	
	max	18.17	1.31	6.24	70.71	1.11	9.32	0.64	0.75	0.27	1022	567	70	
	mean	14.42	1.15	5.15	68.32	0.96	8.63	0.51	0.67	0.16	851	542	65	
	st.dev.	3.29	0.21	1.63	2.36	0.14	0.70	0.13	0.07	0.11	150	22	6	
1c (n = 1)		11.11	0.85	5.75	69.49	0.83	9.04	2.39	0.53	–	696	711	49	
	min	17.16	0.78	2.24	65.74	0.48	6.97	1.06	0.86	–	1042	442	48	
	max	20.12	1.45	2.97	68.31	0.67	8.34	1.25	1.18	–	2130	673	151	
	mean	18.65	1.04	2.56	67.08	0.61	7.85	1.18	1.00	–	1591	526	105	
3 (n = 7)	st.dev.	1.21	0.29	0.31	1.07	0.09	0.61	0.09	0.15	–	524	101	44	
	min	10.85	0.65	3.10	63.98	0.58	7.06	0.34	0.35	–	498	406	43	
	max	21.90	1.08	6.56	71.84	1.18	10.08	1.43	0.52	–	732	579	56	
	mean	15.76	0.90	4.39	68.34	0.80	8.57	0.80	0.42	–	606	495	50	
3 (n = 7)	st.dev.	4.69	0.16	1.47	3.58	0.19	1.01	0.41	0.06	–	85	65	4	

Table 2 Average chemical composition of the glass samples from Boğazköy for each of the five compositional groups

Glass at Sagalassos		Concentration / wt%									
		Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	MnO	Fe ₂ O ₃	TiO ₂	
Blue (n = 8) 1–150 AD	min	16.03	0.57	1.85	67.52	0.60	6.69	0.29	0.76	0.09	
	max	18.15	1.26	2.21	69.86	1.11	8.36	0.92	1.65	0.15	
	mean	17.14	0.78	2.00	68.82	0.81	7.45	0.43	1.14	0.12	
	st.dev.	0.71	0.23	0.15	0.75	0.16	0.49	0.21	0.28	0.02	
Green (n = 20) 1–150 AD	min	15.28	0.34	1.65	69.11	0.42	5.94	0.03	0.51	0.09	
	max	17.30	0.57	2.59	73.48	0.64	8.03	1.52	0.76	0.10	
	mean	16.38	0.50	1.97	70.92	0.54	6.83	0.51	0.66	0.10	
	st.dev.	1.03	0.11	0.43	1.94	0.09	0.92	0.69	0.11	0.01	
Colorless (n = 2) 1–150 AD	min	17.60	0.42	1.55	71.35	0.35	5.01	0.02	0.52	0.09	
	max	18.38	0.42	1.70	71.77	0.47	6.06	0.02	0.60	0.10	
	mean	18.12	0.42	1.60	71.63	0.39	5.36	0.02	0.55	0.09	
Colorless (n = 7) 300–450 AD	min	17.04	0.48	1.54	66.09	0.29	5.94	0.02	0.60	0.09	
	max	19.36	0.90	2.25	71.28	0.57	7.84	1.04	1.60	0.14	
	mean	18.30	0.68	1.81	69.35	0.41	6.71	0.27	0.87	0.11	
	st.dev.	0.94	0.14	0.24	2.33	0.10	0.74	0.38	0.33	0.02	

Table 3 Average chemical compositions of contemporary glass from Sagalassos, southwest Turkey (Degryse *et al.* 2005)



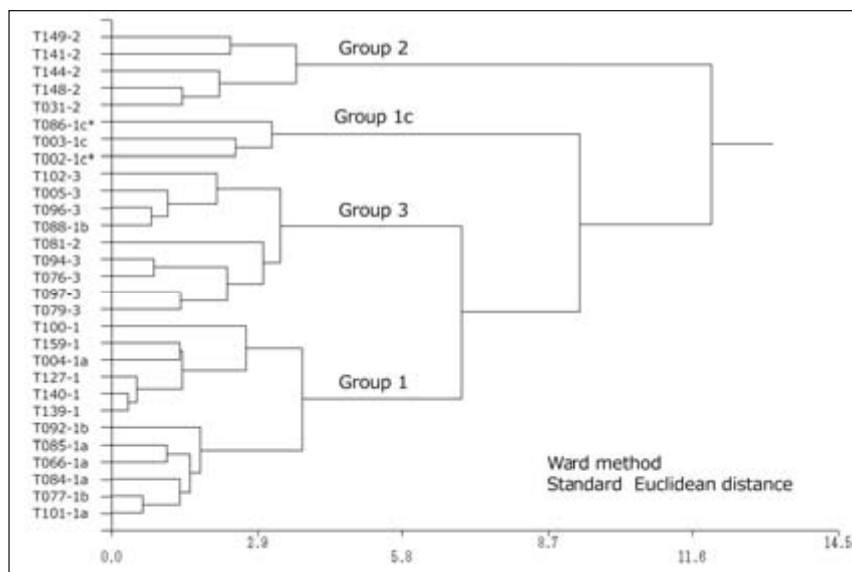
Fig. 7 Photographs of the glass samples from Boğazköy Museum classified as group 3 (Levantine type, Mn-decolorizer) based on the chemical composition. Scale 1:2

Levantine 1 Glass

Levantine 1 glasses are characterized by lower soda (~ 15 %) and higher lime (~ 9 %) concentrations, and they often contain low levels of iron oxide (0.4 %) and a relatively high alumina content (~ 3 %). The glasses in Group 3 ($n = 7$; Table 2) contain soda (10.85–21.90 %, av. 15.76 %), high level of CaO (7.06–10.08 %, av. 8.57 %), low levels of Fe_2O_3 (0.35–0.52 %, av. 0.42 %), and high Al_2O_3 (3.10–6.56 %, av. 4.39 %), which satisfy the criteria of Levantine 1 glass mentioned above. The Group 3 glasses contain high levels of MnO (0.34–1.43 %, av. 0.80 %), and manganese was used as a decolorizer (Fig. 7).

The archaeological dating of the samples T-97 and T-102, which belong to Group 3, is second to fourth century A. D. The rest of the Group 3 samples are archaeologically identified as second to third century, which conforms to the early time period for Levantine 1 glass (Fig. 7).

Fig. 8
Dendrogram
showing the four
compositional
groups of the glass
samples from
Boğazköy ob-
tained by a cluster
analysis



Classification Based on Statistical Analysis

Fig. 8 shows the results of the cluster analysis as a dendrogram of the glass samples based on the CaO , TiO_2 , MnO , Fe_2O_3 , SrO , ZrO_2 , and Sb_2O_3 contents of the analyzed glass. The glass samples can clearly be separated into four compositional types: Group 2 (HIMT glass), Group 1c (Roman blue-green type with Mn decolorizer or Levantine 1), Group 3 (Levantine type), and Group 1 including 1a, 1b, and 1 (Roman blue-green type with Sb decolorizer). These results are in good agreement with the classifications assigned in the preceding sections based on the comparisons with the reference data, showing the consistency of the grouping. However, there were two exceptions; sample T-081 and T-088 were classified into Group 3 (Levantine 1). Sample T-081 contains high level of TiO_2 (0.1 %), MnO (> 1 %), and Fe_2O_3 (> 0.7 %), which is in accordance with the criteria of Group 2 (HIMT glass) more so than the Levantine 1 type. On the other hand, sample T-088 contains a significant amount of Sb_2O_3 ; even though the level is low (0.058 %), this fact is distinct from the Levantine type (Group 3) and should be classified into Group 1b.

Comparison with Roman Glass from Sagalassos

It is reported that local secondary production of glass was carried out at Sagalassos in southwest Turkey from Imperial Roman to early Byzantine times. It is interesting to compare this glass with the Boğazköy glass of the same period. Table 3 shows the average chemical composition of the blue ($n = 8$), green ($n = 20$), and colorless ($n = 2$) glasses of 1–150 A. D. and the colorless glass of 300–450 A. D. excavated from Sagalassos. The comparable data for the Boğazköy glass are listed in Table 2. The analytical data of the glasses from Sagalassos show quite uniform chemical compositions with a low std. deviation: Al_2O_3 (av. 1.70–2.00 %), CaO (av. 6.06–7.45 %), MnO (0.02–0.43 %), TiO_2 (0.10–0.12 %), and Fe_2O_3 (0.60–1.14 %). The trace element composition is only given for a blue glass ($n = 1$, 1–150 A. D.): Zr 51 ppm, Sb 894 ppm, Sr 384 ppm, and Pb 204

ppm. These analytical data are comparable with those of Group 1a for Boğazköy glass; the average composition ($n = 4$) is Al_2O_3 2.12 %, CaO 7.28 %, MnO 0.72 %, TiO_2 0.068 %, Fe_2O_3 0.53 %, Zr 60 ppm, Sb 2750 ppm, Sr 503 ppm, and Pb 110 ppm. This observation suggests the similarity of the source sands used in the two glasses. It can be assumed that both glasses used sands of East Mediterranean coasts. However, it is found that a larger amount of antimony/manganese decolorizer was used for the Boğazköy glass, suggesting different places of secondary production.

Origin of the Boğazköy Glass

Our analytical data suggest that the chemical composition of the Boğazköy glass can be understood based on the existing glass types. It is remarkable that the wide variety of glass excavated from Boğazköy and its vicinity can be classified into only a few compositional glass types: blue-green, HIMT, and Levantine. Namely, glass vessel assemblages are governed by a small number of compositional groups. This suggests that there were just a few production centers of making glass from raw materials. Recent excavations revealed the existence of large primary glass-making installations of Greco-Roman date in Egypt²⁶ and Byzantine to early Islamic date in Israel²⁷. The Levantine coasts were indeed the location of large-scale glass making in Antiquity. This might explain the uniformity of glass compositions throughout wide areas of the Mediterranean world. The glasses are distributed in the form of rough chunks to fabrication workshops around the Mediterranean and surrounding countries. Sagalassos may be one such place. In fact, glass chunks, fuel ash slag, and kiln fragments related to glass processing have been excavated at Sagalassos.

In the Upper Town of Boğazköy, J. Seeher unearthed a two-roomed building, which he identifies as a possible workshop for glass making during the imperial period based on finds of greenish glass slags²⁸. Moreover, R. Czichon suggested several sites in the close vicinity of Boğazköy to be glass workshops²⁹ because he found glass slags there. It can be presumed that there was local but secondary glass production at least during the imperial Roman period.

The results also seem to indicate that the compositional grouping of the glass may be restricted by chronological differences rather than geographical differences between excavated place and production site because of the active trading during the Roman and Byzantine times.

Abstract: During investigations in the Hittite capital Hattuşa the remains of a small, probably village settlement from late antiquity were discovered at various points in the urban area. A necropolis from this epoch is of particular importance. Finds from it as well as finds purchased in the vicinity by the local museum provided the material for archaeometric analyses of the chemical composition of glass specimens from late antiquity. Several groups can be chemically distinguished and they fit well into the known spectrum of eastern Mediterranean glass production. Given these findings, we may assume that the then relatively remote region of Boğazköy was nonetheless integrated in the transregional network supplying raw materials for glass production.

²⁶ Nenna *et al.* 1997; Nenna *et al.* 2000.

²⁷ Gorin-Rosen 1995; Gorin-Rosen 2000.

²⁸ Seeher 1997, 338; S. Kühn dates this building to the second century A.D. based on an analysis of the archaeological materials found in it (Kühn 2014, 71).

²⁹ Czichon 2003, Sites No. 15, 17–19; Kühn 2014, 72.

CHEMISCHE CHARAKTERISIERUNG VON RÖMISCHEM
UND FRÜHBYZANTINISCHEM GLAS AUS BOĞAZKÖY/ḪATTUŠA UND SEINER UMGEBUNG

Zusammenfassung: Im Zuge der Erforschung der hethitischen Hauptstadt Ḫattuša wurden an verschiedenen Stellen des Stadtgebietes Reste einer kleinen, wahrscheinlich dörflichen Siedlung der Spätantike freigelegt. Eine besondere Bedeutung kommt einer Nekropole dieser Epoche zu. Funde aus dieser sowie solche, die durch das lokale Museum aus der unmittelbaren Umgebung angekauft wurden, bilden die Grundlage für naturwissenschaftliche Analysen der chemischen Zusammensetzung spätantiker Gläser. Es können mehrere Gruppen chemisch unterschieden werden, die sich gut in das bekannte Spektrum der ostmediterranen Glasproduktion einfügen. Anhand der Ergebnisse kann vermutet werden, daß die in dieser Zeit relativ abgelegene Region Boğazköy dennoch in die überregionale Versorgung mit Rohmaterialien zur Glasproduktion eingebunden war.

BOĞAZKÖY/ḪATTUŠA VE ÇEVRESİNDEN ROMA
VE BİZANS CAMLARININ KİMYASAL KARAKTERİZASYONU

Özet: Hitit başkenti Ḫattuşaş'ın farklı yerlerinde sürdürülen araştırmalar sırasında Geç Antik döneme ait, olasılıkla bir köy yerleşiminin kalıntıları ortaya çıkmıştır. Bu döneme ait bir nekropol özellikle anlam kazanmıştır. Gerek sözü edilen kazılar sırasında ortaya çıkan gerekse yerel müze tarafından civardan satın alınan buluntular, Geç Antik dönem camlarının kimyasal analizine temel oluşturmaktadır. Buluntuların, Doğu Akdeniz bölgesinin bilinen cam ürünleri çeşitliliğine uygunluk gösteren, kimyasal açıdan birkaç gruba ayrıldığı görülmüştür. Sonuçlardan yola çıkılarak, o zamanlar merkezi konumda olmayan Boğazköy yöresinin, yine de, cam üretiminde kullanılan hammadde bakımından bölgeler arası bir öneme sahip olduğu düşünülebilir.

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