Study on Western Asiatic cast ribbed rectangular beads from Kaman-Kalehöyük, Turkey, by using portable X-ray fluorescence

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Abstract

In this article we report a comparative study on excavated objects and artifact from museums to reveal an aspect of ancient trade. The target artifact is Western Asiatic cast ribbed rectangular beads excavated from an architectural remain at Kaman-Kalehöyük, Turkey. Blue glass beads of this type have been excavated in Western Asia from north Iran and Iraq to the Syro-Palestinian coast from second half of 16th to 14th century BC. The analysis of the samples was carried out by using a portable X-ray fluorescence (XRF) spectrometer developed by us. The instrument was brought to the excavation site in Turkey as well as to the museums to analyse typologically similar glass beads from the collection of Okayama Orient Museum and MIHO MUSEUM in Japan, for comparison. Our XRF analyses suggested that all analysed glass artifacts are plant ash soda-lime silica glass with 2-4 wt% magnesium and potassium. The three glass beads exhibited similar compositional characteristic, *i.e.* they contain Sb, Pb, Fe, Cu and Sr in similar quantities. A typological and principal component analysis comparison of the glass beads unearthed from Kaman- Kalehöyük site with those of the museums and literature data support that they should have a similar origin. In addition, archaeological context of the glass from Kaman-Kalehöyük also supports that the artifact belongs to the Middle-Late Bronze Age (16th to 15th centuries BC). This is the first scientific material evidence that shows the possibility of a cultural flow from Mesopotamia region to Kaman-Kalehöyük during Middle-Late Bronze Age.

Introduction

The investigation on archaeological glass is not just important for technological but also in terms of archaeological study. The chemical composition of glass artifact can provide an indication of primary raw materials used to make the glass and the means by which colors were achieved. The trace elements as impurities introduced with the raw materials can be an evidence indicating the manufacturing place.

Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) (Gratuze *et al*., 2001; Gratuze, 1999) is a powerful technique for quantitative analysis of glass. However, for some archaeological objects analysis destroying the artifacts is often not allowed. In this case, X-ray fluorescence (XRF) spectrometry becomes an alternative tool for the elemental analysis of archaeological objects especially for glass material (Van Grieken and Markowicz, 2002; Pillay *et al*., 2000; Mantler and Schreiner, 2000; Milazzo and Cicardi, 1997) since it has the capability of performing non-destructive analysis and no need for complicated sample preparations. Yet, XRF analysis of glass tends to be affected by surface corrosion, which results in loss of Na and increase in Si and Al contents, However, taking the artifacts out of an excavation site or from a museum for analysis is a great restriction. To solve this problem portable XRF is still only choice. We have been developing a portable XRF spectrometer suitable for glass analysis and brought to many museums and archeological sites in Egypt, Turkey, Syria, *etc*. to characterise the archaeological objects (Nakai *et al*., 2005). Fortunately, our experiences suggest that if we could choose sample with good shining condition, the analytical results are fair especially for heavier elements though it may be safe not to use the data of Na and Si. In this article, we report a comparative study on excavated glass objects and artifacts from museums to reveal an aspect of ancient trade. The target artifact is Western Asiatic cast ribbed rectangular beads (Figure 1) which were unearthed from an architectural remain at Kaman-Kalehöyük site. The site is in Çağırkan village, Kaman, Kırşehir province, located in the central Anatolia approximately one hundred kilometers southeast of the Ankara city of the Republic of Turkey (Figure 2a). The site situated at the crossroads of the caravan routes that ran from China via Mesopotamia to Greece and the Mediterranean. The rectangular glass beads had been found in a round structure number 1 (Figure 2b), constructed of stones slanted toward its center with stone paved floor, along with many interesting excavated objects such as bullae with hieroglyphic signs, Old Hittile Period pottery shreds, a bronze pin, a bronze awl, round and cylinder glass beads. Most of them dated to the second half of the second millennium BC or know as the period of Old Hittite Empire in Central Anatolia.

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The rectangular blue glass beads are molded with the characteristic four ribs on the upper surface and doubly perforated longitudinal along the perforations in turquoise blue color. Most of the beads are 1 to 3.5 cm long. They are sometime reported to be found as plain flat disk pendants and figural female pendants with the same color. The rectangular blue glass beads of this type have been excavated in West Asia from north Iran and Iraq to the Syro-Palestinian coast from second half of 16th to 14th century BC. More than 11,000 pieces of the beads and pendants excavated at Nuzi (Yorgan Tepe), near present day Kirkuk in northern Iraq which was a part of Hurrian kingdom of Arrapha, along the south-eastern edge of the Mitanni's area (Stern and Schkucj-Nolte, 1994; Starr, 1939; Lankton, 2003).

Materials and Methods

The analytical instrument used in this study was an energy dispersive portable XRF spectrometer (OURSTEX 100FA-IIL) developed by us in cooperation with Ourstex Co (Tantrakarn *et al*., 2009). The instrument has a distinguished optics system with monochromatic X-ray source as well as white X-ray source excited at 40 kV with Pd target x-ray tube current available for 0.1 to 1.0 mA. The X-ray source has a dual optics system that can be used alternatively, equipped with a bent toroidal monochromator of graphite (0002) to obtain a Pd-K line monochromatic X-ray source for the analysis of medium to heavy elements (K-Y) and a capillary to obtain a white X-ray source for light (Na-Cl) and heavier elements (Ag-). This yields an irradiated beam spot approximately 2.5 mm in diameter at the position of the sample. The samples were placed on the X-ray tube and detector unit through a window covered with a thin film of ULTRALENE No. 3525 (thickness 4 μm) at the bottom of a cylindrical vacuum sampling chamber. The vacuum chamber was composed of a cylinder column 25 cm in diameter and 15 cm in height that could be extended to 45 cm in height by connecting 3 cylinder columns together, thus allowing the analysis of a large artifact with 25 cm diameter and 40 cm height or more. The sampling chamber and X-ray beam path were evacuated down to 500 Pa by a compact vacuum pump to reduce the drastic absorption by air of the low-energy X-rays emitted from the low Z elements such as Na. The introduction of the Moxtek AP3.3 polymer window of the silicon drift detector (SDD), together with an introduction of a vacuum sample chamber allowed us to quantify the light elements such as Na and Mg with energy resolution with a full width at half maximum (FWHM) of Mn Kα at 170 eV. Most recent improvement includes a reduction of the total weight from 25 to 16 kg and an introduction of a C-MOS camera for observation of samples. To analyse the glass samples, a flat surface without or less corrosion has been chosen for each glass sample and cleaned with ethanol before placing inside the sampling chamber on top of the X-ray beam path. X-ray fluorescence spectrum was measured by X-ray beam generated by the Pd target excited at 40 kV with tube current of 0.25 mA for white X-ray source and 1.0 mA for monochromatic X-ray source. The X-ray spectra were measured for 300 seconds in vacuum condition for each analysis mode in the energy range of 0-40 keV. The quantitative analysis of lead has been carried out by Pb Lβ line because Pb Lα line overlap with As Kα line while other elements have been carried out by their Ka line. The integrated XRF intensity of each element was normalised by the scattered X-ray of the Pd Kα line (Compton scattering). A calibration curve method was applied here utilizing various reference standard samples.

The portable XRF instrument was brought to the excavation site in the Republic of Turkey in 2007. Four pieces of the Western Asiatic cast ribbed rectangular beads unearthed during 1994 excavation season of Kaman-Kalehöyük were chosen and the analysis was performed at the site facility.

For comparison with the excavated sample, we have analysed similar glass beads (Figure 3) from the collection of the Okayama Orient Museum and MIHO MUSEUM in Japan which were identified as artifacts from Syro-Palestinian or North Mesopotamian regions. The portable XRF instrument was brought to the museums. The analyses at the museums were made under joint projects with R.

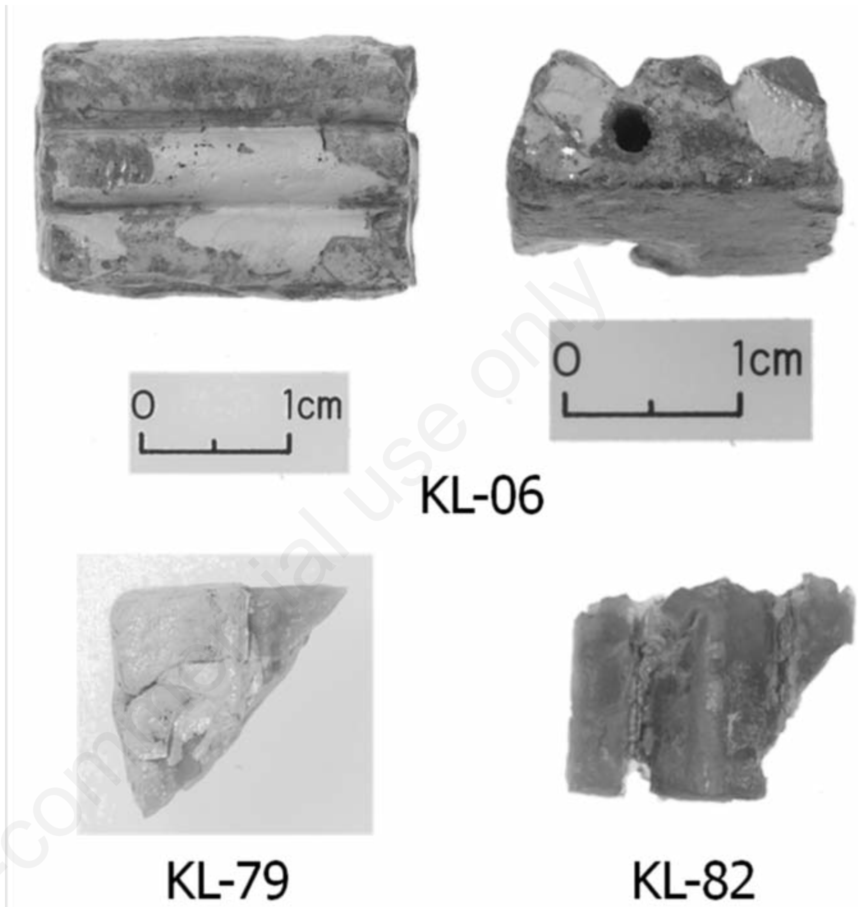

Figure 1. Cast ribbed rectangular glass beads excavated from Kaman-Kalehöyük.

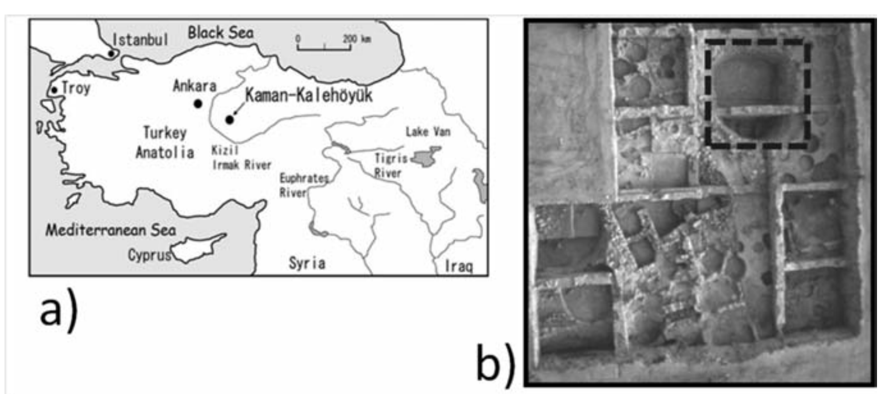

Figure 2. a) Location of Kaman-Kalehöyük site; b) a photo of the round structure number 1 indicated by square where the samples were excavated.

Shikaku (Okajama Orient Museum) and Y. Azuma (MIHO MUSEUM). To determine the geographic origin of the glass, principal component analysis (PCA) of the glasses from Kaman-Kalehöyük and opaque blue glass bead reported in several literatures were performed using the statistics software package Minitab (Minitab Inc., State College, PA, USA). Contents of SrO, MnO, CaO, ZnO Sb_2O_3 Fe_2O_3, MgO, K_2O, and PbO were used as calculating variation by correlation matrix method.

Results

In Table 1, chemical compositions of major and trace elements of the cast ribbed rectangular semi-transparent blue glass beads excavated from Kaman-Kalehöyük, determined by the XRF are shown. The ribbed rectangular glass beads contain 2-4 wt% magnesium oxide and potassium oxide with relatively high in contents of sodium for 6-10 wt% except for the sample KL-71 which was affected from its apparent heavy surface corrosion: *i.e.*, the Si and Al contents are high while the Ca content is low. X-ray fluorescence spectra of the blue ribbed rectangular glass bead (KL-06) are shown in Figure 4. Trace amount of Pb and Rb and a significant amount of antimony was detected in the KL-06 sample. The three glass beads (KL-06, KL-79 and KL-82) exhibited similar compositional characteristics; i.e. their Sb content is fairly high in the range of 1-2 wt%, and Pb, Fe and Cu contents are in a similar level. Copper is responsible for the blue colours.

Chemical compositions of the glass samples from Okayama Orient Museum (OK-1,2) and MIHO MUSEUM (MH-01-05) are shown in Table 2. All of the ribbed rectangular glass beads belonging to the museums are in very bad surface condition. The analysis results affected from the corrosion in the content of silica on the surface. Yet, existence of high level of antimony (1-2 wt%) and trace Pb (10-100 ppm) as well as relatively high level of magnesium and potassium contents are also characteristic to the museum samples resembling to those of the excavated samples from Kaman-Kalehöyük.

Discussion

It should be noted that the excavated samples from Kaman-Kalehöyük were overall lacking in sodium contents which could be due to the depletion of alkali as a result of weathering. Relatively high levels of magnesium and potassium (1.5 wt%<) suggest the use of a plant ash as the alkali source. These glass samples are also characterised by their low alumina and iron oxide levels, reflecting a relatively pure silica source with the mean value of 0.9 and 0.37 wt%, respectively. Low content of titanium oxide indicates that the source of silica does not seem to be come from Egyptian region. The ribbed rectangular glass beads are all blue opaque colored with the use of copper

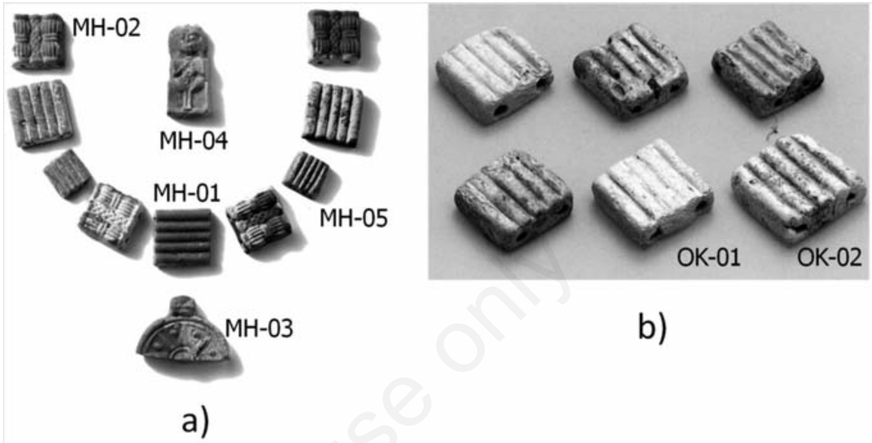

Figure 3. Cast ribbed rectangular glass beads of MIHO MUSEUM (a) and Okayama Oriented Museum (b).

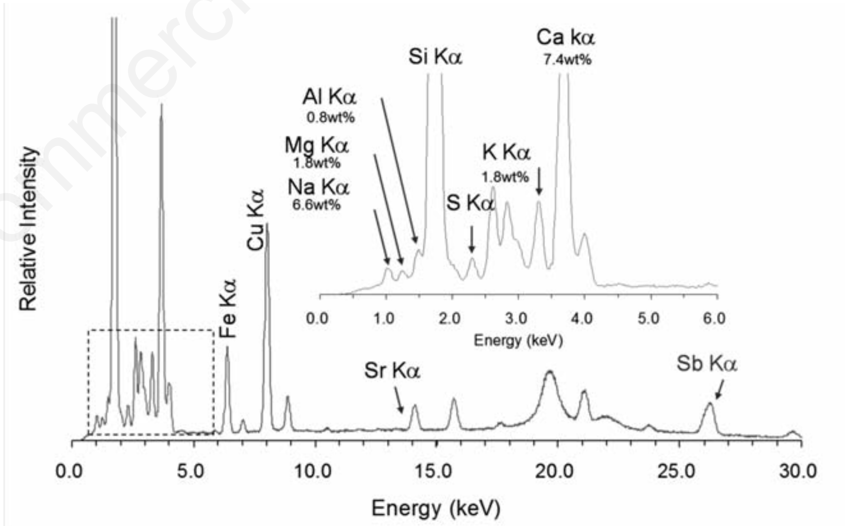

Figure 4. X-ray fluorescence spectra of a blue ribbed rectangular glass bead (KL-06).

Table 1. Analysed spacer bead samples excavated from Kaman-Kalehöyük and their chemical compositions.

No.	Excavated	Na_2O (wt%)	MgO (wt%)	Al_2O_3 (wt%)	SiO_2 (wt%)	K_2O (wt%)	CaO (wt%)	TiO_2 (wt%)	MnO (wt%)	Fe_2O_3 (wt%)	CuO (wt%)	Zn (ppm)	Rb (ppm)	Pb (ppm)	Sr (ppm)	Sb (wt%)
KL-06	Kaman-Kalehöyük	6.6	1.8	0.8	65.3	1.8	7.4	0.1	tr	0.66	0.56	nd	tr	80	360	1.9
KL-71	Kaman-Kalehöyük	nd	nd	1.1	76.2	1.4	1.7	0.17	tr	0.12	4.27	nd	tr	60	80	nd
KL-79	Kaman-Kalehöyük	4.6	2.1	0.8	69.4	2.7	4.2	0.05	tr	0.47	0.71	nd	tr	30	520	1.5
KL-82	Kaman-Kalehöyük	9.7	3.4	0.6	62.2	3	5	0.06	tr	0.44	0.64	nd	tr	30	500	1.3

nd, not detected; tr, trace.

possibly from bronze rust. The typologically same glass beads have been found from the excavation sites at Nuzi and Tell Brak in Mesopotamia and were analysed using an inductively coupled plasma atomic emission spectrometry technique (ICP-AES) by Brill (1999) and electron microprobe technique by Degryse *et al*. (2010), respectively. The analytical data reported by Brill (1999) (Table 3) showed much higher level of sodium but the characteristic elements such as Sb and Pb are in the same levels as those of Kaman-Kalehöyük and the museum samples analysed by us. Both have low aluminum and iron contents. The silica contents of the previous studies are lower and do not show the characteristic of corroded glass, is due to the difference in the analytical methods that ICP-AES and electron microprobe can analyse the bulk composition but the present XRF technique is nondestructive surface analysis.

In terms of the glass opacifier, it is well known that in the ancient glass inducing crystallisation or by direct addition of high density crystalline material, typically antimony or tin based opacifiers such as lead stannate yellow (Pb_2SnO_4 and $PbSnO_3$), tin dioxide (SnO_2), lead antimonate yellow ($Pb_2Sb_2O_7$) or calcium antimonate white ($Ca_2Sb_2O_7$ and $CaSb_2O_6$) would add an opaque property to the glass (Shortland, 2002; Tite and Pradell, 2008). It is the characteristic of the blue ribbed rectangular glass beads that all of the glass contain elevated high calcium contents (7 wt% or higher) much higher than usual plant ash soda-silicate glass. Since lead counterpart in the glass is low, the analytical data may indicate the use of calcium antimonate white ($Ca_2Sb_2O_7$ or $CaSb_2O_6$) as opacifier in the ribbed rectangular glass beads. It is known that calcium antimonate white opacifier was introduced in glass technology in the Middle Bronze Age (Turner and Rooksby, 1961; Nicholson and Shaw, 2009).

Origin of the glass beads based on the principal component analysis

Two possible regions as glass source for the origin of the beads are considered as Mesopotamia and East Mediterranean region.

We have conducted the PCA analysis using the present analytical data and those from the literatures (Brill, 1999). PC1 and PC2 together explained 43% of the total variance of the data. Figure 5 and 6 show the score and loading plots of the first two PCs. Results from cluster analysis indicate 3 groups of light blue glass during the 13th to 16th century BC. Opaque light blue glass beads from Kaman-Kalehöyük (KL-06, KL-69 and KL-72) and MIHO MUSEUM (MH-02, MH-03 and MH-04) are plotted as group A,

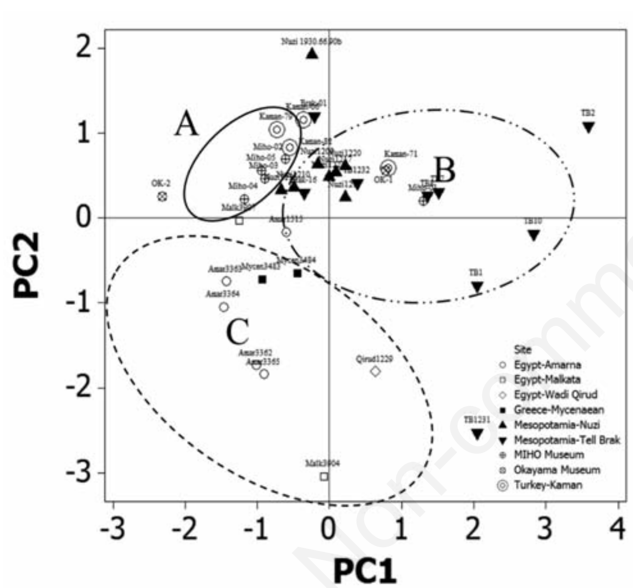

Figure 5. Principal component scores plot of principal component 1 *vs* principal component 2.

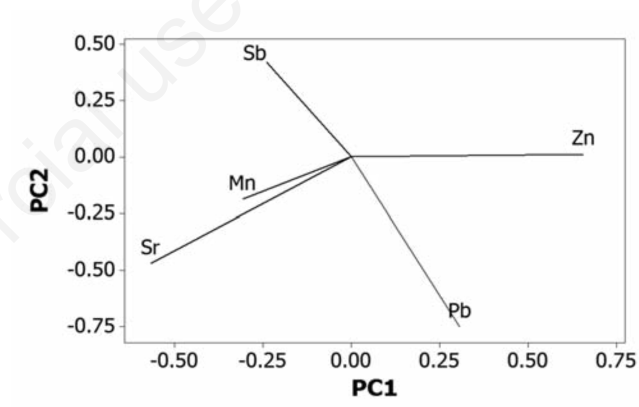

Figure 6. Principal component loading plot of principal component 1 *vs* 2.

Table 2. Analysed spacer bead and pendant glass samples in the collection of MIHO MUSEUM, Okayama Orient Museum and their chemical compositions.

No.	Excavated	Na_2O (wt%)	MgO (wt%)	Al_2O_3 (wt%)	SiO_2 (wt%)	K_2O (wt%)	CaO (wt%)	TiO_2 (wt%)	MnO (wt%)	Fe_2O_3 (wt%)	CuO (wt%)	Zn (ppm)	Rb (ppm)	Pb (ppm)	Sr (ppm)	Sb (wt%)
MH-01	North Mesopotamia	10.6	1.7	0.8	78.1	1.7	4.6	0.05	0.05	0.37	0.54	473	50	100	300	1.3
MH-02	Northern Mesopotamia	10.3	0.9	1.0	74	3.5	7.8	0.09	0.05	0.27	0.58	nd	50	10	330	1.4
MH-03	Northern Mesopotamia	4.5	1.2	0.9	78.7	4.4	7.3	0.08	0.05	0.22	0.9	nd	50	30	460	1.7
MH-04	Northern Mesopotamia	8.8	1.5	0.9	75.5	3.1	7.5	0.07	0.06	0.45	0.49	nd	50	20	560	1.5
MH-05	Northern Mesopotamia	19.4	0.9	1.3	59.2	0.3	16	0.05	0.05	0.36	0.57	nd	50	20	460	1.8
OK-1	Mesopotamia	nd	2.2	0.8	67.9	1.5	4.5	0.1	nd	0.37	2.75	nd	nd	60	100	nd
OK-2	Mesopotamia	3.5	0.5	0.5	65.6	1.9	5	0.08	0.07	0.29	0.76	nd	nd	80	420	1.5

nd, not detected; tr, trace.

Table 3. Chemical composition of light blue glass artifacts from Mesopotamia and Egypt during 16th-14th century BC reported by Brill (1999).

Sample name	Sample	Origin	SiO_2	Na_2O (wt%)	CaO (wt%)	K_2O (wt%)	MgO (wt%)	Al_2O_3 (wt%)	Fe_2O_3 (wt%)	TiO_2 (wt%)	Sb_2O_3 (wt%)	MnO (wt%)	CuO (wt%)	Pb (wt%)	Sr (ppm)	Zn (ppm)
Nuzi 1209	Disk Lt blue opq	Mesopotamia/Nuzi	64.68	14.9	9.31	2.4	3.47	0.49	0.97	0.03	2.32	0.04	1	100	300	78
Nuzi 1210	Medallion Lt blue opq	Mesopotamia/Nuzi	67.09	13	9.29	2.33	3.06	0.41	0.66	0.02	2.35	0.03	1.34	100	500	75
Nuzi 1930	Bead Lt blue opq	Mesopotamia/Nuzi	65.73	12.6	8.93	2.58	3.57	0.28	0.11	nd	3.11	nd	1.69	nd	200	nd
Nuzi 1216	Lt blue opq	Mesopotamia/Nuzi	63.64	17	7.19	3.7	4.81	0.7	0.28	0.03	1.5	0.03	1.03	50	500	nd
Nuzi 1217	Cored vessel Lt blue transp	Mesopotamia/Nuzi	64.68	18.1	6.58	2.38	4.81	1.4	1.02	0.03	nd	0.03	0.91	nd	200	nd
1219	Cored vessel Lt blue transp	Mesopotamia/Nuzi	69.66	15.5	5.11	2.28	4.59	0.42	0.23	0.01	nd	0.03	2.11	50	200	nd
Nuzi 1220	Cored vessel Lt blue transp	Mesopotamia/Nuzi	73.76	12	6.37	2.01	3.91	0.41	0.23	0.01	nd	0.02	1.22	nd	200	nd
Nuzi 1221	Cored vessel Lt blue transp	Mesopotamia/Nuzi	64.74	17	7.93	2.9	4.81	0.94	0.49	0.03	nd	0.04	1.05	nd	200	nd
Brak-01	Ingot Lt blue opq	Mesopotamia/Tell Brak	62.91	16.5	8.35	1.7	2.8	0.18	0.14	nd	2.53	nd	1.25	nd	500	150
Brak-16	Ingot Lt blue opq	Mesopotamia/Tell Brak	69.06	14.2	6.04	1.42	2.44	0.2	0.11	nd	2.34	nd	1.7	140	500	nd
TB8	Ingot Lt blue transp	Mesopotamia/Tell Brak	62.2	19.3	6.5	2.6	6.7	0.4	0.3	0.02	nd	0.04	0.72	100	nd	200
TB1	Ingot Lt blue opq	Mesopotamia/Tell Brak	64.6	17.4	8.5	1.8	3	0.2	0.1	0.03	nd	0.02	1.22	300	nd	200
TB2	Ingot Lt blue opq	Mesopotamia/Tell Brak	70.5	14.6	6.1	1.5	2.6	0.2	0.1	0.03	1.74	0.02	1.63	100	nd	900
TB7	Ingot Lt blue opq	Mesopotamia/Tell Brak	68.6	13.3	8.9	2.1	3	0.5	0.2	0.03	1.36	0.02	0.76	200	nd	200
TB10	Ingot Lt blue opq	Mesopotamia/Tell Brak	66.2	18.6	4.7	2.5	6.3	0.1	0.1	0.02	nd	0.02	0.04	200	nd	500
TB1230	Nugget Lt blue opq	Mesopotamia/Tell Brak	67.5	16.3	8.37	1.67	3.01	0.24	0.1	0.01	1.25	0.02	1.15	50	500	300
TB1231	Pendant Lt blue opq	Mesopotamia/Tell Brak	64.18	15.3	10.9	2.01	4.23	0.86	0.37	0.02	0.79	0.02	0.69	500	500	420
TB1232	Pendant Lt blue opq	Mesopotamia/Tell Brak	70.99	14.4	6.68	1.64	2.6	0.39	0.11	0.01	1.43	0.01	1.63	50	500	260
Mycen 3483	Plaque Lt blue transp	Greece/Mycenaean	65.66	19.8	7.72	1.29	3.1	0.75	0.59	0.08	nd	0.03	0.68	50	800	nd
Mycen 3484	Pendant Lt blue transp	Greece/Mycenaean	63.94	18.9	6.45	2.88	4.91	0.87	0.5	0.05	0.02	0.05	1.19	100	500	nd
Amar 1515	Cored vessel Lt blue transp	Egypt/Amarna	63.88	20.6	7.93	1.71	3.31	1.01	0.61	0.1	nd	0.05	0.73	20	500	nd
Amar 3362	Cored vessel Lt blue transp	Egypt/Amarna	63.65	16.6	4.89	2.47	3.81	0.56	0.32	0.07	0.45	0.02	0.78	200	1000	nd
Amar 3363	Cored vessel Lt blue transp	Egypt/Amarna	57.49	23	10.63	1.23	4.67	1.1	0.62	0.13	nd	0.02	0.98	nd	1000	nd
Amar 3364	Cored vessel Lt blue transp	Egypt/Amarna	68.48	16.2	7.35	1.32	3.58	1.31	0.67	0.13	0.01	0.08	0.7	50	800	nd
Amar 3365	Cored vessel Lt blue opq	Egypt/Amarna	64.3	17.9	8.67	1.68	4.01	0.74	0.45	0.1	1.4	0.05	0.04	300	800	nd
Malk 3904	Cored vessel Lt blue transp	Egypt/Malkata	70.6	14.5	7.6	1	2.7	0.74	0.55	0.12	0.92	0.03	0.95	500	800	nd
Malk 3905	Cored vessel Lt blue opq	Egypt/Malkata	58.41	16.2	11.8	2.33	4.1	0.85	0.61	0.1	3.4	0.03	1.73	20	1500	nd
Malk 3907	Cane Lt blue opq	Egypt/Malkata	60.75	17.9	10.3	2.33	4.11	0.87	0.74	0.08	1.74	0.02	0.96	50	800	nd
Qirud 1229	Vessel Lt blue transp	Egypt/Wadi Qirud	71.4	15.6	3.69	3.39	2.93	0.83	0.51	0.05	nd	0.05	1.18	330	400	80

Opq, opaque; transp, transparent; nd, not detected.

along with the opaque light blue glass beads and pendants from Nuzi and Tell Brak. While partially transparent light blue glass bead KL-71 and corrode MH-01 and OK-01 fallen in to group B along with opaque and transparent light blue glass samples from Nuzi and Tell Brak. These results confirm that the light blue glass bead from Kaman-Kalehöyük are originated from Mesopotamia region and are not from the Egyptian and Mycenaean light blue glasses indentified as group C in the PCA plot.

A typological and compositional comparison of the glass beads unearthed from Kaman-Kalehöyük site with those of the museum collections and literature data clearly indicates that these glass objects can be considered as coming from the same glassmaking tradition.

Conclusions

The cast ribbed rectangular glass beads from Kaman-Kalehöyük site, as described above, are archaeologically well documented materials by the excavation team. However, no previous chemical analysis has been performed on these glasses. This study is the first attempt to characterise this glass type based on the chemical compositions found from Central Anatolia region. The chemical analyses discussed here have demonstrated that the glass beads of Kaman-Kalehöyük site were produced using a plant ash soda-lime silica glass, similar to contemporary material from the Mesopotamia supported by the analysis of the samples from MIHO MUSEUM and Okayama Orient Museum and a comparison with the literature analytical data including PCA. It is found from the present study based on typological and chemical analysis that the glass beads excavated from Kaman-Kalehöyük was produced within the Mesopotamian region. This represents a picture of a certain relationship possibly trading between Kaman-Kalehöyük and the Mesopotamian region. However, due to the limited number of glass analysed in this study, a further investigation of glass from Kaman-Kalehöyük and nearby excavation sites with preferable dating from excavation deposits should be necessary to clarify glass trading in Middle Bronze Age between Central Anatolia and other regions more clearly.

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