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THE FRAGMENT OF ENAMELED GLASS VESSEL FROM THE BOLGAR EXCAVATION (RUSSIA)

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This paper is devoted to the interdisciplinary study of an enameled glass fragment found in the excavation of the Bolgar fortified settlement (Russia). The artifact comes from excavation site CLXXII of the so-called aristocratic district of the city. A comparison to a collection of Islamic drinking glasses from the Nasser Khalili collection shows the identity of the enamel pattern decor. The artifact was investigated by a series of analytical methods: scanning optical and electron microscopy (OSEM) and optical emission spectral analysis (OES). The results of the OES studies revealed that the basis is soda-lime glass. OSEM determined that different enamel colors were obtained from lapis lazuli, nepheline, diopside, bone ash, hematite, and lead-tin additive. Comparison of element's concentrations with data of the Brill catalog of archaeological glass made it possible to identify the Bulgarian fragment as Egyptian glass produced in the late 13th – early 14th centuries.

Keywords: archaeological glass, enamel, 13–14th centuries, scanning electron microscopy, colorants, Egyptian import.

Introduction

Enameled glassware is one of the great achievements of Islamic culture and technology. Richly enamel and gold decorated vessels, as well as luxury objects such as fabrics or weapons, were popular import objects in Europe (Piotrovsky, 2008). Enameled glass accounts for a substantial part of the glass finds in excavations in the European part of Russia, especially in fortified settlements, such as in Novgorod, Moscow, Yaroslavl, Vladimir or Suzdal (Schapova, 1963; Stoliarova, 2004; Stoliarova & Engovatova, 2013; Plokhov, 2007; Zelentsova & Kuzina, 2008; Kuzina, 2007). Research has focused on the manner by which these expensive Islamic imports came into these cities (Busiatskaya, 1972; Darkevich, 1974; Rodina, 2003; Limonov, 1961). The city of Bolgar, located in the center of Eastern Europe along the Volga River (Zakharov & Kuzina, 2010; Koval', 2010), is the most probable agent for this trade. The archaeological excavations of the fortified settlement (inhabited between the 7th and the 15th c. ACE) have revealed a large number of fragments of enameled glassware.

This paper is devoted to the study of an enameled glass fragment with tricolor ornaments and gilding, found in 2012, dating by stratigraphic association to the end of the 13th – beginning of the 14th century ACE, during the heyday of enameled glass (Carboni, 2001). By that time, the masters of the "Golden Age" of Islamic glass (Israeli, 2003) from Egypt and Syria have brought their wares to perfection, and the artifacts of this time can be defined as works of high art. Thanks to the study of rich museum and private collections, a large database on the history, morphology (Lamm, 1929–1930, 1941; Gudenrath, 2006; Ward, 1998), manufacturing techniques and chemical composition of enameled artifacts (Carboni, 2001; Freestone & Stapleton, 1998; Brill, 2001; Henderson, 2003; Colomban, 2013; Verita, 1995; Greiff & Hartman, 2008) is known for comparative purposes.

The use of two independent analytical methods – scanning optical and electron microscopy (OSEM) and optical emission spectral analysis (OES) – has shown high

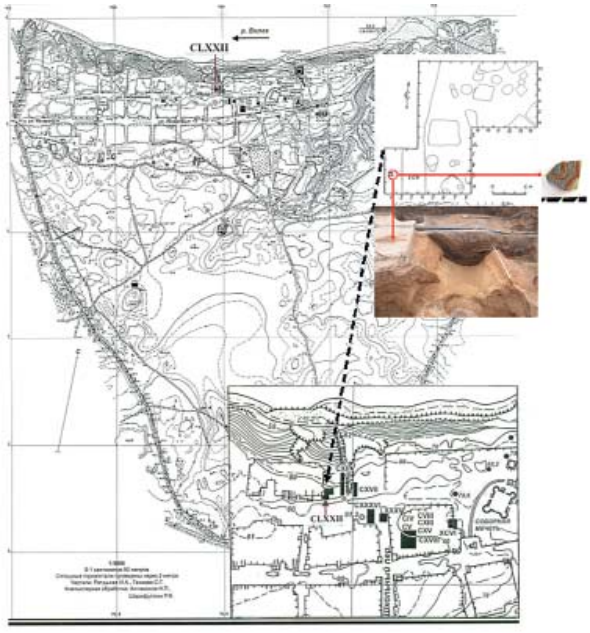

Fig. 1. Excavation CLXXII of the Bolgarian fortified settlement with the finding point of glass fragment.

Рис. 1. Раскоп CLXXII Болгарского городища с местом обнаружения стеклянного фрагмента.

research efficiency. As the experience of the authors has shown, emission analysis provides important insights into the macro and micro components of the glass base (Khramchenkova et al., 2016; Khramchenkova et al., 2017; Khramchenkova et al., 2017b).

Stratigraphy

The northern part of the Bolgar fortified settlement belonged to the so-called "aristocratic" territory of the city. The most significant and monumental architectural structures are concentrated there. In this area, archeologists have also identified a number of residential buildings, belonging to the rich households of aristocrats, craftsmen-jewelers, merchants, etc. An example is the "Craftsman's House", in the remains of which fragments of glass vessels were found, almost undamaged items of expensive imported glazed earthenware and celadon ware, and many other things (Gubaidullin, 2013).

Excavation CLXXII with a total area of 264 sq.m. was performed in 2012. A fragment of enameled glass (no. 12 / CLXXII-23) was found at a depth of 188 cm in B/1 square (fig. 1). It originates from layer IV, the early Golden Horde layer. Four copper Golden Horde coins were also found (fig. 2: a) in the same layer, minted in the name of Nasir Lid Dina (minted ~1240 ACE, Bolgar), Mengu Kaana (minted the end of the 13th century ACE, Bolgar), Saray al-Jadid (minted –in the first half of the 14th century ACE) or identified as a possible Bolgar coin (likely from the first half of the 14th century ACE). (Khlebnikova, 1987). Additionally, a significant number of fragments of glazed red clay wares and crockery, as well as fragments of celadon, dating to the second half of the 13th to the 14th centuries ACE, were found in the same layer (Figure 2b), next to rare fragments of imported amphorae, most likely produced in the Syro-Palestinian region (Volkov & Gubajdullin, 2012).

Material

The investigated find is a transparent colorless glass with an ornament applied in the enamel technique (fig. 3: a). A

Fig. 2. Finds from the excavation CLXXII of the Bolgarian ancient settlement: a – copper coins, b – glazed ceramics.

Рис. 2. Находки из раскопа CLXXII Болгарского городища: a – медные монеты, b – глазурованная керамика.

fragment of 24×20 mm^2 in size and 17 mm in thickness has a convex shape, the outer part of which is patterned. The pattern has three colors – red, blue and white, with a gold bordering. The ornament is formed by arched interleaved lines of red and white colour, the red color on both sides contoured with gold. One red line adjoins a gold line. There is a blue field with a floral pattern above the arches, marked down with red and gold lines. The central red-gold part of the lines is applied to white enamel and is contoured with a red strip applied to the blue enamel. The pattern has a thin structure of alternation of a dark red outside, blue, white and gold inside, and a bright red in the center of the line. The thickness of each colored line is not more than 0.4 mm and the total width of the lines does not exceed 3 mm.

Microscopic examination of the artifact revealed an application sequence of the enamel in four stages:

– application of the primary colors – white, red and blue, after which the vessel was subjected to high-temperature heat treatment in the furnace;

– application of a red border;

– application of gold paint over the gold border;

– application of thin red lines.

Examples from the collection of Professor Nasser Khalili (Khalili collections 2016) (shown in fig. 3: a) are very close analogue to the Bolgar find. They form a unique set of three equally designed undamaged glasses, measuring 18 by 12.7 cm (rim diameter), 15.8 by 11.4 cm (rim diameter) and 12.5 by 8.4 cm (rim diameter), dating to the 13th century ACE. It is assumed that similar glasses were used as liturgical vessels in Europe (Khalili collections, 2016).

A glass with similar decoration is located in the National Museum of Kuwait (Gibson, 2005). It has been postulated that this vessel was originally part of a full set, together with the three glasses of the Khalili collection. The reconstruction of the Bolgar archaeological fragment`s diameter using the curvature radius, has shown that the vessel had a diameter of 6 cm at the point to which the fragment belongs. The glasses of the Khalili collection have the following diameters in those corresponding areas (decoration ring): 8 cm, 7 cm and 5 cm. The size reconstruction of Bolgar glass give the value near of 6 cm (fig. 3: b) and finding likely corresponded in size to the artifact from the museum of Kuwait.

Fig. 3. a – the fragment of enameled glass from the Bolgar excavation CLXXII with glasses from the collection of N. Khalili, b – the reconstruction of enamel decoration.

Рис. 3. a – фрагмент эмалевого стекла из болгарского раскопа CLXXII со стаканами из коллекции Н. Халили, b – реконструкция эмалевого рисунка.

Chemical composition

Studies of the chemical composition (base and trace elements) (tab. 1) of the Bolgar sample were made using optical emission spectral analysis (OES) (Khramchenkova & Sitdikov, 2014). Micro-samples of the base glass, with inclusions of red and blue colors, were selected for this analysis. The study of the enamel pattern composition was carried out by scanning electron microscopy (SEM), using an EVO LS 10 SEM with an INCA X-MAX energy- dispersive spectrometer. The sample was fixed on a current-conducting carbon tape, on an aluminum holder, placed in the electron microscope. VPSE mode (secondary electron detection mode, in low vacuum) was used during the electron microscopic analysis to visualize the total morphology of the coating in a panoramic survey, as well as to perform microprobe quantitative and qualitative analysis (at 20 kV and 800 pA). The microprobe X-ray spectral analysis of the samples based on the energy dispersive spectrometer INCA X-Max (sensitivity at 127 eV) included the determination of the sample's composition and mapping of element distributions. Photographs were taken using an Axio Observer Z1 optical microscope. A snapshot of the analyzed area is given in fig. 3: c. Results are given in Table 1. The analytical results of the optical emission spectral (OES) and energy dispersive (ED SEM) analyses are very close for the base glass composition.

Colorless transparent base glass

The investigated sample is a soda-lime glass. The average Na_2O and CaO content is around 14% and 8% respectively. Significant concentrations of potash and magnesia, around 2% and 3.5% respectively, prove the use of plant ash as a flux. The presence of 2% alumina and 0.8% iron oxide is likely representative of the silica sand source used (Freestone 1998), though this can also be part of the ash used in the manufacture of this glass. Manganese contents between 1.0 to 1.8% are standard for Islamic glass.

The tables with our analysis also show data for other (Islamic) enameled glass. Most samples of enameled glass analyzed by Freestone (Freestone & Stapleton, 1998) have a composition close to the Bolgar sample, with the exception of the aluminuma content. The authors conducted a comparative analysis of the chemical composition of the Bolgar base glass fragment with data provided in the R. Brill catalogue, in order to identify possible analogies (Brill, 1999). Two samples (# 965 and # 963) were found to be almost identical in chemical composition to the Bolgar material for all elements measured. These glass fragments were found in the north-western tower of St. Catherine's Cathedral, located on Mount Sinai. It is

Table 1.

The chemical composition of the colorless base glass, the results of emission (OES) and spectral (SEM) analysis respectively. Comparative data by Brill 1999, Freestone 1998 – A, Eremine 2003 - B, Catalog mineralov, 2005 - C.

	colourless	**CuO**	**MnO**	**PbO**	**SnO**	**TiO_2**	**P_2O_5**	**CaO**	**Fe_2O_3**	**Al_2O_3**	**K2O**	**MgO**	**Na_2O**	**SiO_2**	**SO_3**			
SEM	base		1,88			0,26		6,76	0,77	1,28	2,18	2,58	12,73	70,78				
SEM	base		0,94			0,22		6,08	0,6	3,92	2,05	3,03	14,38	66,63				
SEM	base		1,17			0,3		8,15	1,17	2,55	1,88	4,69	12,34	66,38				
ESA	base with blue incl.	0,003	1,81	0,006	0	0,22	0,3	8,82	0,84	2,84	1,94	3,15	15,21	65,12				
BRILL/965	Sinai	0,01	1,23	0,005		0,13	0,4	8,52	0,53	2,17	2,32	3,09	13,2	68,25				
BRILL/963	Sinai	0,005	1,38	0,005		0,17	0,42	8,68	0,69	2,58	2,6	3,05	14,2	66,09				
A	1900.6-21.20		1,1			0,2	0,3	9,2	0,6	1,5	2,1	3,1	13,2	67,7				
A	1900.6-21.54		1,6			0,3	0,3	8,4	0,6	1,3	2,6	3,2	13,7	67,1				
B	153 colourless		0,81			0,05	0,38	7,9	0,38	1,27	3,39	3,37	13,14	68,71	0,18			
Eremin	154 colourless	0,05	0,76			0,06	0,32	7,73	0,37	1,24	2,42	3,14	14,83	67,26	0,23			
Eremin	349 colourless		0,78			0,08	0,31	7,39	0,42	1,24	2,73	3,11	14,64	67,42				
	blue	**CuO**	**MnO**	**PbO**	**SnO**	**TiO_2**	**P_2O_5**	**CaO**	**Fe_2O_3**	**Al_2O_3**	**K2O**	**MgO**	**Na_2O**	**SiO_2**	**SO_3**			
Chirvinskij	nefelin							0,43		**32,89**	**5,45**		15,73	43,97				
SEM	blue, inclusion 1							0,59		**31,79**	**5,52**	0,18	16,88	44,95				
C	diopsid							**25,9**				**18,5**		55,6				
SEM	blue, inclusion 2							**20,73**	0,34	2,94	0,08	**16,95**	1,93	57,04				
A	lazurite							6,3		**29**	0,5		16,9	34,4	**13,7**			
SEM	blue, inclusion 3		0,68					5,71	0,36	**11,36**	1,81	1,92	15,71	56,57	**4,74**			
SEM	blue, inclusion 4		0,94					6,07	0,44	**7**	2	2,27	14,63	62,9	**2,93**			
SEM	blue		0,87					6,42	0,89	**4,47**	1,85	3,04	13,91	65,65	**n.d.**			
A	1900.6-21.48		1,5			0,2	0,4	9,2	0,5	**4,4**	2,4	3,7	8,7	66,7	**1,3**			
A	1900.6-21.30		0,9	0,5		0,2	0,2	11	0,6	**4,2**	2,1	3,9	11,2	62,4	**1,2**			
B	153 blue	0,06	0,78	0,1			0,3	7,82	0,41	**1,7**	3,07	3,4	14,05	66,81	**0,51**			
B	154 blue	0,06	0,84	0,09			0,36	8,74	0,42	**2,03**	2,92	3,27	13,99	65,29	**0,53**			
B	349 blue	0,16	0,39	**5,8**	0,03			3,33	0,3	**2,05**	0,78	0,28	17,28	64,65	**0,22**			
B	153 blue lazurite							3,16	0,04	**25,52**	1,83	0,34	14,33	36,4	**13,07**			
B	154 blue lazurite							3,15		**28,97**	0,61		18,22	35,39	**15,52**			
	red	**CuO**	**MnO**	**PbO**	**SnO**	**TiO_2**	**P_2O_5**	**CaO**	**Fe_2O_3**	**Al_2O_3**	**K2O**	**MgO**	**Na_2O**	**SiO_2**	**SO_3**	**Au**	**As**	**Zn**
SEM	red enamel		1,21				1,02	5,01	1,2	4,34	2,18	2,78	12,62	65,17	0,24			
SEM	red enamel		0,88			0,31	0,77	6,13	0,87	6,42	2,35	2,71	12,28	62,31	1,07			
A	1900.6-21.32		1,1	1,7		0,3	0,2	7,4	3,9	1,3	2,2	2,4	11,2	66,8	0,4			
B	349 red	0,04	0,02	0,4				1,78	0,22	0,41	7,63	0,04	12,9	61,39	0,29			**10,46**
SEM	red, inclusion		0,43					1,37	50,54	1,6	0,58	1,29	11,88	28,4			2,12	
SEM	red, inclusion		0,44			0,15		2,16	37,56	1,27	0,88	1,85	12,45	40,83	0,94		0,78	
ESA	c/less with red incl.	0,007	1,42	**0,78**	**0,77**	0,32	0,48	7,56	0,52	2,28	2,18	3,62	15,76	64,5		0,048	0,09	0,001
SEM	red line			**10,81**		0,2	**12,39**	8,27	**37,12**	4,97	1,85	0,93	**0,94**	17,19		4,4		
A	1900.6-21.30			**34,5**		na	bd	1,7	**19,8**	0,9	1	0,9	**1,7**	36,7	bd			
A	1900.6-21.32			**45,6**		na	bd	1,6	**12,9**	0,8	0,7	1	**1,6**	32,3	bd			
B	154 red, hematite	0,29	0,53	**11,92**	6,55			4,42	**23,3**	0,65	1,31	2,32	6,41	39,32	0,31			
B	349 red hematite	0,25	0,02	0,35				0,39	**34,38**	0,19	1,49	0,02	3,18	52,4	0,1			
	white	**CuO**	**MnO**	**PbO**	**SnO**	**TiO_2**	**P_2O_5**	**CaO**	**Fe_2O_3**	**Al_2O_3**	**K2O**	**MgO**	**Na_2O**	**SiO_2**	**SO_3**	**Au**	**Ag**	**As**
SEM			0,68	9,45	4,36			4,3	0,51	2,03	1,65	2,94	14,13	59,96				
A	1900.6-21.20		0,5	7,7	9,9	0,1	0,2	8,1	0,4	1,2	2,3	3,8	11,8	53,2				
A	1900.6-21.62		0,3	13,8	20		1,1	7,9	0,3	0,7	1,6	2,7	11,7	48,5	0,5			
B	154 white		0,69	17,61	10,3			6,04	0,31	0,79	1,41	2,73	9,34	52,51	0,23			
SEM	**gold**	**CuO**	**MnO**	**PbO**	**SnO**	**TiO_2**	**P_2O_5**	**CaO**	**Fe_2O_3**	**Al_2O_3**	**K2O**	**MgO**	**Na_2O**	**SiO_2**	**SO_3**	**Au**	**Ag**	**As**
				0,84			2,27	1,58	8,81	2,07	0,91			8,94		73,19	1,09	0,11

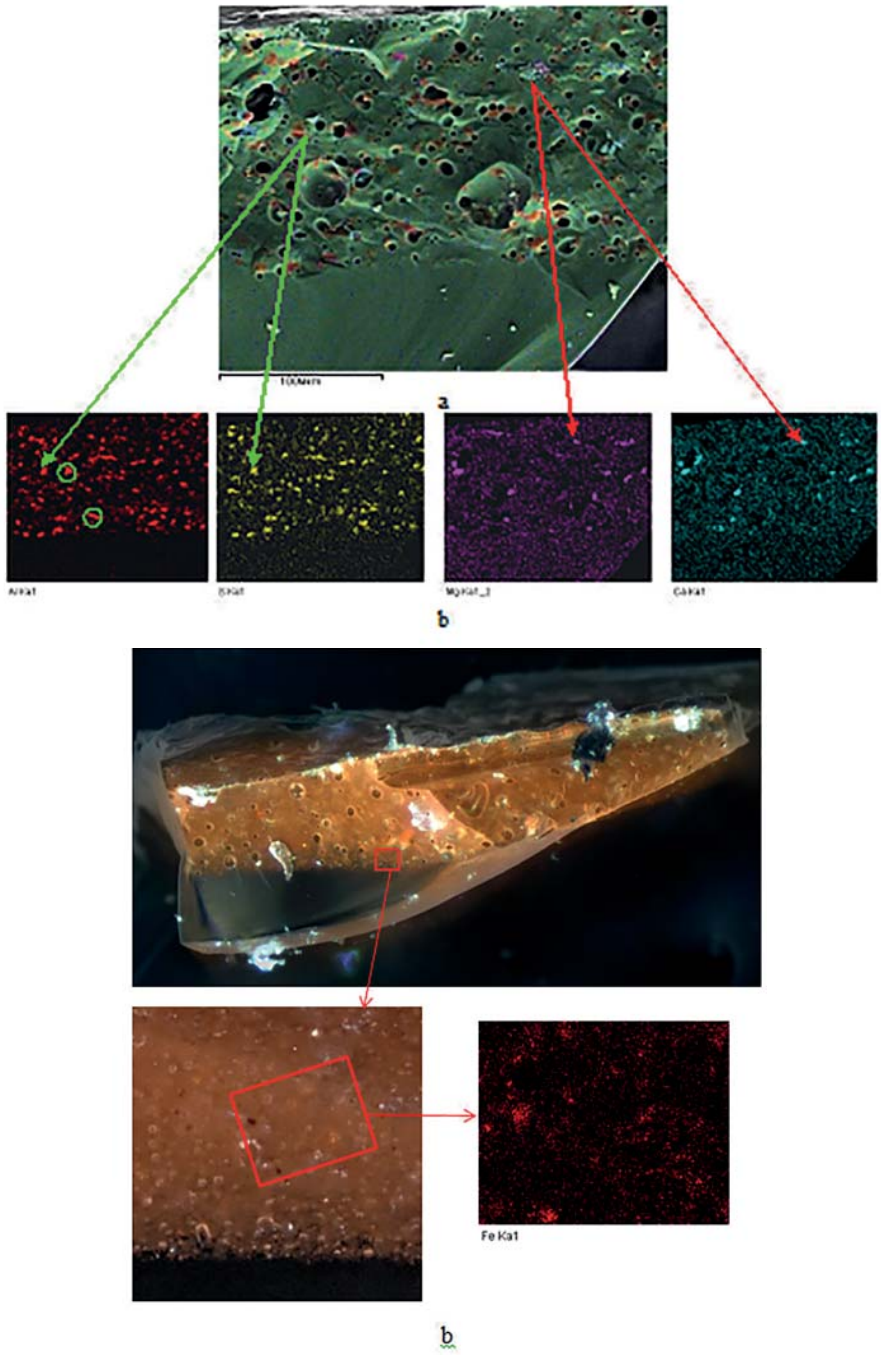

Fig. 4. a – enamel with the blue area: OM-SEM image, SEM image with color differentiation of sections of different compositions (green area without color inclusions – base glass) and map of sulfur, aluminum, calcium and magnesium; b – picture of OM-SEM image of red area and a cartogram of iron distribution in enamel.

Рис. 4. a – эмаль с синей областью: изображение OM-SEM, изображение SEM с цветовой дифференциацией участков разного состава (зеленая область без цветных включений – базовое стекло) и карта серы, алюминия, кальция и магния; b – снимок ОМ-СЭМ изображения красной области и картограмма распределения железа в эмали.

indicated in the catalogue that the tower was built in the 6th century, but the glass is identified as being from a window. It is noted in the catalogue that these windows have a younger origin, but the exact date of manufacture is unknown. Data by K. Eremin (Eremin & Al-Khamis, 2003) for two mosque lamps, dating to the middle of the 14th century (# 153 and # 154) and a perfume bottle from Syria of the same date (# 349), were used as further comparative materials. These Mamluk vessels with enamel painting are in the National Museum of Scotland. This Islamic Mamluk glass can be distinguished from the Bolgar sample through its lower manganese, titanium and alumina contents.

Blue enamel

The blue enamel, compared to the base glass, has a clearly increased alumina and phosphorus content of 15.2% and 1.64% respectively. This blue enamel composition was compared to other enameled glass (Freestone & Stapleton, 1998; Eremin & Al-Khamis, 2003). The use of lazurite $Na_6Ca_2[AlSiO_4]_6(SO)_4]$, known as "lapis lazuli", as a blue coloring agent is suggested by the high alumina content. However, the sulfur content in our glass is quite low, not exceeding 1.4%, and must have been reduced by the heating process (Tauson & Sapozhnikov, 2003). Fig. 4: a shows an SEM image with color differentiation of areas of different compositions and elemental maps. These elemental maps show that the blue color is obtained by adding compounds including Al-S and Mg-Ca complexes to the base glass (fig. 4: a, the lower portion without inclusions). In addition, there are points on the aluminuma map that do not contain sulfur. These were characterized by spot analysis (Tab.1, blue inclusion). The chemical composition of inclusion 1 (Table 1, blue inclusion 1) shows nepheline – a kind of feldspar. The second inclusion with the Si-Ca-Mg complex is a diopside (http://www.catalogmineralov.ru/mineral/diopside.html, 2005). The third inclusion, which includes alumina and sulfur, c orresponds to lapis lazuli mixed with the base glass.

Red enamel

Like with the data by Freestone (Freestone & Stapleton, 1998), there are two types of red pigment used on the enameled glass from the Bolgar excavation. Thin lines are drawn with hematite red enamel (Fe_2O_3 -37.12%), mixed in with a Si-Pb-Ca-P basis (17.19%-10.81%-8.27%-12.41%). This composition differs from data of (Freestone & Stapleton, 1998; Eremin & Al-Khamis, 2001). The same compound is moreover applied as a base surface for the gold dye. The main red ornament is painted with a different type of enamel. Red and orange particles are visible in the red glass body (fig. 4: b) and are similar to enamel as described by Freestone (Freestone & Stapleton, 1998). The map of iron distribution over the glass chip surface shows the correspondence of the red inclusions to Fe-rich points, the composition of which is given in Table 1 (red inclusion). It can be concluded that the "basic" red enamel is made by adding the ground pigment, by which thin red lines were applied, to the base glass.

White enamel and gold plating

The composition of the white enamel (Table 1) does not differ from other, well-known examples (Freestone & Stapleton, 1998; Eremin & Al-Khamis, 2003). A lead-tin compound causes the white color. A presence of 1% silver and 0.1% arsenic can be noted in the composition of the gold paint applied to the red hematite line.

Discussion and Conclusions

Interdisciplinary research involving the historical analysis and study of the structure of the glass artifact makes it possible to answer questions about the origin of the finds (Sitdikov et al., 2016, Khramchenkova et al., 2017a).

Analysis showed that the base glass of the enameled fragment from the Bolgar excavation site CLXXII was made on the basis of the traditional soda-silica-lime Islamic formula (Brill, 2001; Salvant et al., 2016; Henderson & Allan, 1990; Henderson, 2003a). Plant ash was used as a flux (Freestone & Stapleton, 1998; Henderson, 2003a; Whitehous, 2002). Enamel of three colors is close in composition to the main colors used by medieval masters when painting Islamic vessels (Freestone & Stapleton, 1998; Eremin & Al-Khamis, 2003; Colomban et al., 2012; Stapleton, 1998).

The ornament on the Bolgarian archaeological fragment is similar to the one depicted on the drinking glasses from a set in the Nasser Khalili collection. According to the catalogue, this set dates to the 13th century ACE, which is consistent with the stratigraphic dating of the fragment from the Bolgar excavation but does not have exact information on the place of production.

Comparative analysis of the chemical composition of the Bolgar glass fragment revealed two very similar analogies. An enameled glass window found in St. Catherine's Cathedral, located in the center of the Sinai Peninsula (Brill, 1999) (Table 1), and has no exact date. These samples are almost identical in composition to the Bolgar glass. It could be hypothesized that the enamel vessel imported to Bolgar was made in Egypt. The presence of Egyptian imports in Bolgar, silk fabrics found in the graves of young women (Fedotova et al., 2015), can support such hypothesis.

Also the decoration pattern of the glass could indirectly point to an Egyptian origin. With the ascension to power of Sultan An-Nasir Addin Muhammad, who ruled and lived in Egypt in 1293–1341, the tradition of decorating enamelware changed. Genre scenes gave way to ornamental decoration with the use of three colors – red, white and blue with gilding (Nofal', 1992).

The enamel pattern on our investigated fragment is obtained by applying three colors with gilding. Blue enamel is made by adding a mixture of lapis lazuli, nepheline and diopside to the colorless base. Such a composition indicates that the ancient masters used raw lapis lazuli material, consistent with e.g. the Sare-Sang blue pigment sources of northern Afghanistan (Efimov & Suderkin, 1967; Blaise & Cesbron, 1966; Bariand, 1981). The red lines of the pattern are applied with a hematite dye the red enamel of the ornamentations is made of the base glass with the addition of ground hematite. The white enamel has a lead-tin compound.

To summarize, it can be assumed that the enamel vessel brought to Bolgar, as well as the set from the collection of N. Khalili, are of Egyptian origin and were made in the late 13th – early 14th centuries.

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ФРАГМЕНТ ЭМАЛЕВОГО СОСУДА ИЗ РАСКОПА БОЛГАРСКОГО ГОРОДИЩА (РОССИЯ)

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Статья посвящена междисциплинарному исследованию фрагмента эмалевого стекла, найденного при раскопках Болгарского городища (Россия). Артефакт происходит из раскопа CLXXII так называемого аристократического района города. Сравнение декора исламских стаканов из коллекции исламского стекла Насера Халили (Лондон) показывает полную идентичность эмалевого узора болгарской стеклянной находки. Объект был исследован с помощью ряда аналитических методов, таких как сканирующая оптическая и электронная микроскопия (OSEM) и оптико-эмиссионный спектральный анализ (OES). Результаты исследований OES выявили, что основой является натриево-известковое стекло. OSEM определил, что различные цвета эмали получены в результате использования лазурита, нефелина, диопсида, костной золы, гематита, а также свинцово-оловянная добавка. Сопоставление колличественных характеристик элементных концентраций с каталогом археологического стекла Brill позволило идентифицировать болгарский фрагмент как египетское стекло, произведенное в конце XIII – начале XIV веков.

Ключевые слова: археологическое стекло, эмаль, XIII–XIV век, сканирующая электронная микроскопия, красители, египетский импорт.

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