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Received: October 16<sup>th</sup> 2022  
Accepted: November 15<sup>th</sup> 2022  
Original research article  
UDC: 904.666.1"652"(497.11)  
[https://doi.org/10.18485/arhe\\_apn.2022.18.10](https://doi.org/10.18485/arhe_apn.2022.18.10)

## ROMAN AND LATE ANTIQUE GLASS IN THE MEDITERRANEAN AREA AND SERBIA: ITS PRODUCTION, COMPOSITIONAL TYPES AND PROVENANCE

### ABSTRACT

*The paper gives a synthesis of Roman glass production, compositional types and provenance of Roman soda-lime-silica glass (natron glass) during the Roman and Late Antiquity epochs. It briefly discusses a small production of plant-ash glass, which appears among the Serbian glass finds. The paper describes the production process and components used in glass production and the two-phase production model of Roman glass. It presents the main compositional features of the most typical Roman glasses during the first three centuries CE: Roman blue-green glass, naturally colourless glass, and antimony-decolourised glass. It also describes new glass types that appeared during the 4th century: Roman manganese-decolourised glass, HIMT, Foy série 3.2, Jalame, and Roman Sb+Mn. It then gives characteristics of the most-represented glass-type of the 6th century, the Foy série 2.1 and its subtypes with elevated concentrations of iron.*

*The paper discusses the provenances of the mentioned types and the methods used for their determination: circumstantial evidence, major and minor element concentrations, and isotopic ratios and rare earth patterns. There is also a discussion on the types of glass from Serbia, giving a brief sketch of its evolution in time and contextualising it within finds reported from the wider Mediterranean area. It shows that the distribution of particular glass types in Serbia generally reflects the distribution in the wider area. The important exception to this is Foy 3.2, which seems to be more present in Serbian assemblages, started to appear earlier (3rd century) and lasted longer (6th century) than in the Western Mediterranean.*

**KEYWORDS:** GLASS, COMPOSITIONAL TYPE, PROVENANCE, ROMAN PERIOD, NATRON, PLANT ASH.

### INTRODUCTION

Glass in the Roman Empire was not just a material, it was a highly valued product and prestigious commodity whose manufacture needed significant technological knowledge and skill. Glass production during Roman times followed the manufacturing practice used in several other ancient technologies, namely a two-phased production. This means that the product was first partially manufactured in a convenient place (usually close to the resources and energy sources), and then transported to the place of consumption, where it would be completed to a finished product (Foy and Nenna 2001; Foyet

Nenna 2003). Roman glass was also produced in a two-stage process: first, raw glass was produced in furnaces close to the natural sources of good-quality sand and natron flux. After cooling, the glass slab was broken into small pieces of raw glass and transported by ships across the Roman Empire. In the furnaces close to the consumption sites, the glass was remelted and blown or cast into glass objects for everyday use (Freestone et al. 2000; Freestone (2003; 2004); Gorin-Rosen 2000; Foy et al. 2003; Nenna et al. 2000). To differentiate between the two phases of glass production, it is customary to label the sites primary production sites (glass making) and secondary production sites (glass working).

Archaeological studies have established that raw glass was produced in relatively few places in the Eastern Mediterranean and transported across the vast space of the Roman Empire for local glass production (Freestone et al. 2000, Nenna et al. 2000; 2003). To discover the provenance of primary glass, to establish the main compositional types and to map their spread across the Empire through time, means to cast light on the vast Roman commercial routes and trading patterns and, thus, to gain an insight into the important part of the Roman society, its economy and commerce.

The most typical Roman glasses during the first three centuries CE were: Roman blue-green glass, naturally colourless glass, and antimony-decoloured glass. New glass types appeared during the 4<sup>th</sup> century: Roman manganese-decoloured glass, HIMT, Foy série 3.2, Jalame, and Roman Sb+Mn. The most-represented glass-type of the 6<sup>th</sup> century was the Foy série 2.1 and its subtypes with elevated concentrations of iron.

We will give a general picture of what is known about the Roman glass production, glass types and glass commercialisation during the Roman and Late Antiquity epoch, and briefly discuss how data from Serbian contemporary sites fits into the broader picture.

## ROMAN GLASS PRODUCTION

Glass is an amorphous solid produced from silica and lime, with various additives used for glass modification, like colouring, decolouring, and opacifying. Glass in the Roman Empire was mostly natron glass (or soda-lime-silica glass, according to its main constituents) (Nenna et al. 1997; Foy et al. 2003). It was produced by adding natron (mineral soda low in potash) to batches to serve as a metallurgical flux to lower the silica melting temperature. The main source of sodium for Roman natron glass was the mineral trona, found in high quantities in the Wadi El Natrun evaporitic lakes in Egypt, close to Cairo (Freestone et al. 2000). The Wadi El Natrun region is well documented with primary glass production sites (Nenna et al. 1997).

Lime was added to the glass as a stabiliser. Its role can be seen in the case of the glass from ancient Egypt, produced without lime, that was unstable and dissolved with time. The most common

source of CaO in natron glass were sea-shells, washed ashore, ground by waves and mixed naturally with beach sands. On more rare occasions, when silica sand was used, shells might have been deliberately added to batches of natron glass to increase the lime content (Freestone et al. 2000). The best sand used in Roman glass making, which was also mentioned by Pliny the Elder (Gaius Plinius Secundus, *Naturalis Historia*, ed. Karl Friedrich Theodor Mayhoff, Teubner, 1897), was the beach sand around the mouth of ancient River Belus, close to Haifa in what is today Israel. Brill (1988) has demonstrated that Roman glass could have been produced using Belus-type sand and natron from Wadi El Natrun. Natron glass was by far the most represented type of glass during the first seven or eight centuries CE.

Another type of flux used was ash, produced by burning halophytic plants, which grow on saline soils rich in potassium. The main source of CaO in this type of glass is plant-ash flux, so a sand low in lime or crushed quartz could have been utilised in the glass production (Freestone et al. 2000). In the Mediterranean basin, this technology was employed during the Old Era, and from the Late Byzantine/Early Islamic glass making transition that occurred during the late 7<sup>th</sup> to 8<sup>th</sup> century CE (Phelps et al. 2016). The reason for replacing natron with plant-ash as flux is assumed to have been the exhaustion of trona reserves in Wadi El Natrun. However, evidence of a small, perhaps specialised production of plant-ash glass in Egypt during this era of natron has been discovered (Rosenow and Rehren 2014, 2018). Several glasses of this type are reported from two sites outside Egypt, in Crete (Oikonomou et al. 2021) and Serbia (Balvanović et al. 2022).

### *Roman glass technology*

The two-phase model of Roman glass production is based on the scarce evidence of primary glass furnaces, and on the fact that they are all located near the shores of the Eastern Mediterranean. The only Roman primary glass production sites were discovered in Egypt (Nenna et al. 2000; 2003) and Levant (Freestone et al. 2000). Furnaces from Bet Shearim, Israel, show that each firing might have produced several tons of raw glass. Upon cooling, primary glass blocks were crushed

to small chunks and exported throughout the Empire, as shown by several ship-wrecks carrying large amounts of raw glass or crushed glass (cullet) (Freestone et al. 2000). Ship-wreckages such as the 2<sup>nd</sup> century CE Iulia Felix (Silvestri et al. 2008), 3<sup>rd</sup> century Ovest-Embiez (Mardikian and Girard 2010), and 11<sup>th</sup> century Serçe Limanı (Bass 1979), contained raw glass, cullet and glassware. This evidence strengthens the two-phase model assumption and indicates that glass was widely traded over the Roman maritime trading network across the Mediterranean Sea.

The two-phase model of glass production facilitates pinpointing the origins of primary glass production, since the composition of a finished glass object depends on the composition of primary glass used for its production and, thus, on the composition of sand from which it was made. Since the raw glass, being an imported product, was not always available at a local glass-working workshop, and to lower costs of raw materials, broken old glass, cullet, was often added to the batches in the secondary workshops. Iulia Felix had glass cullet sorted according to its colour, indicating that glass-makers and glass-workers were in good control of the glass-manufacturing process (Silvestri et al. 2008). The practice of recycling varied both in time and with compositional types of the glass. Some glass types were not recycled much (Foy 3.2) while some were recycled very much (Foy 2.1). Roman Sb+Mn glass was produced using antimony-decolourised and manganese-decolourised cullets. Recycled glass leaves, as a chemical signature, elevated concentrations of trace elements like copper, antimony, tin, nickel and zinc, well above normal concentration in sands (Foy et al. 2003). The cut-off values for these elements need to be established for every compositional type of glass, starting from its primary glass concentrations.

In order to market glass objects, the glass was fashioned to the contemporary market demands. The glass could be initially produced colourless (by using very clean sand) or decolourised (using antimony or manganese). It could be of more bluish or greenish colour, depending on the furnace redox conditions (reducing conditions producing a bluish colour, oxidising conditions producing a greenish tinge).

It could be naturally coloured, like green HMT or Foy 2.1 glass types. It could deliberately coloured, often light blue (with copper) or dark blue (with cobalt), or black (with iron). The colouring was mostly done in the secondary workshops, and is confirmed by elevated trace elements concentrations, like copper, cobalt, or iron (Balvanović and Šmit 2022).

### ***Glass trading patterns***

The small number of primary production sites (situated mostly in the Eastern Mediterranean) and large number of local workshops across the Empire used for glass working, were interconnected over a vast glass trading network. The geographical distribution of various glass types, as demonstrated by many works, are not equal for particular types. Some types are found more in the entire Mediterranean (Foy groups 1-3, Foy et al. 2003), others only in the East (Foy groups 4-10). They are not equally distributed even within smaller regions, as shown by the high presence of Foy 3.2 in the Balkans, compared to Italy and other regions in the Western Mediterranean. This shows that trading patterns for the glass were quite complex and changed over time. The glass might have travelled over direct links, or by coastal routes, hopping from port to port. It might have been imported to inner regions by land, or by river routes, like the Danube waterway. It is an open question whether it usually travelled with some other commodities, and with which ones, or on ships dedicated to glass transport, or if it was just an add-on commodity. Reconstructing these routes by mapping the entire Mediterranean region, with compositions and provenance analysis of raw glass, is a major effort.

## **COMPOSITIONAL TYPES OF ROMAN GLASS**

Two most represented types of glass during the 1<sup>st</sup> – 3<sup>rd</sup> century CE in the Roman Mediterranean are common Roman blue-green glass and colourless glass. Both are transparent and are either very lightly coloured or colourless.

### **Roman blue-green glass**

Roman glass, common glass with bluish or greenish hues, was the most common glass in the 1<sup>st</sup> to 3<sup>rd</sup> centuries CE. It also had a remarkably stable elemental composition, making it a suitable reference glass with which to compare other glass types. Average compositions, calculated for 227 glasses of this type, are around 16.6% of Na<sub>2</sub>O, 2.6% of Al<sub>2</sub>O<sub>3</sub>, 7.5% of CaO, 0.6% of Fe<sub>2</sub>O<sub>3</sub>, 0.13% of TiO<sub>2</sub> and 0.6% of MgO (Table 1, Nenna et al. 1997). Roman glass was produced using clean sands, with low heavy mineral concentrations in sand, Fe<sub>2</sub>O<sub>3</sub> + MgO + TiO<sub>2</sub> = 1.9% on average (Table 2). Its colour, bluish or greenish, depends on the redox conditions in the furnace, resulting in different amounts of ferrous and ferric ions. Brill has shown that this glass might have been produced using Belus-type sand and natron from Wadi El Natrun. (Brill 1988; Freestone et al. 2000)

### **Roman colourless glass**

Roman naturally colourless glass was produced using very clean sands, with the least amount of the sand impurities, like heavy minerals, that give colour to glass (Table 2). Thus, Group 1b (a single glass, Jackson 2005), contains 0.29% of iron, 0.45% of magnesium, 0.07% of titanium oxides and virtually no manganese oxide. Naturally colourless glass from Kosmaj, group K3, had similar concentrations, on average 0.32% of iron, 0.48% of magnesium, 0.05% of titanium and 0.2% of manganese oxides (Stojanović et al. 2015). Heavy mineral concentrations in sands from which the Kosmaj naturally colourless glass is produced is 1.2%. Objects manufactured from this type of glass were likely considered luxury products.

During the 1<sup>st</sup> to 3<sup>rd</sup> centuries CE, Roman glass was decolourised using antimony. Antimony, the principal decolouriser of this period, is a strong decolouriser. The amount of antimony used to decolourise the colouring effect of iron is roughly 1:1. The ratio of antimony to iron oxides in antimony-decolourised glasses in Mala Kopašnica (Stamenković et al. 2017), is 1.01±0.37, and in Egeta 0.83±0.28 (Balvanović et al. 2022).

### **Fourth century change in glass compositions**

During the 4<sup>th</sup> century, manganese started to replace antimony as the main decolouriser. The reason is hypothesised to have been the exhaustion of antimony bearing ores. Manganese is a less efficient decolouriser than antimony, requiring around double that of antimony to produce the same result. The ratio of manganese to iron oxides in the manganese-decolourised glass of Mala Kopašnica is 2.67±1.13 and in Egeta is 1.72±0.43. Since, in this period, the amount of colourless glass cullet available from earlier times, decolourised with antimony, was sufficient, it was widely used for recycling with new, manganese-decolourised raw glass and cullet. In this period, Roman Sb+Mn decolourised glass was quite represented in the archaeological record. In Kosmaj, the group K2 was decolourised using 0.21±0.05 of antimony and 0.29±0.017 of manganese oxides, while iron was 0.42±0.07. The average ratio of the sum of the two decolourising agents to iron is 1.2:1. As the antimony glass became less and less available, the amount of antimony in recycled glass decreased, but it was still found in smaller quantities. The amount of antimony in 6<sup>th</sup> century window glass of type Foy 2.1 in Jelica is 125 ppm (Balvanović et al. 2018, Balvanović and Šmit 2020).

While during the first three centuries CE, the Roman glass was mostly lightly-coloured blue-green glass or colourless glass, in the 4<sup>th</sup> century it was often darker colours, such as green, olive-green or amber. It is not clear what brought this change; possibly a change of fashion or the exhaustion of supplies of very clean sands. The darker glass began to show in significant percentages in archaeological records of the 4<sup>th</sup> century.

Several naturally coloured glass types were reported from this period. They obtained their darker colour from higher concentrations of heavy minerals, derived from sand impurities (4.3% for Foy série 2.1, 7.5% for Foy group 1, Foy et al. 2003). Authors reporting these glass types gave names that best described their respective chemical compositions, like HIMT (High Iron, Manganese, Titanium, Mirti et al. 1993), HIMT 1 and HIMT 2 (Foster and Jackson 2009), strong HIMT and weak HIMT (Foster and Jackson 2009), HIT (High Iron, Titanium, Freestone 1994), HLIMIT (High Iron, Lime, Magnesium, Titanium, Glioz-

glass group/No	location/c. AD	wt%										ppm					
		Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	MnO	Fe <sub>2</sub> O <sub>3</sub>	Sr	Zr	Sb			
Roman glass	Roman Empire	mean	16.63	0.59	2.59	69.54	0.12	0.75	7.48	0.13	0.73	0.62					
n=227	1 <sup>st</sup> -4 <sup>th</sup>	std	1.50	0.29	0.38	2.53	0.05	0.24	1.18	0.14	0.74	0.48					
group E	Aosta	mean	17.44	1.02	2.55	67.06		0.94	5.67	0.56	2.09	1.51	550				
n=9	3 <sup>rd</sup> -5 <sup>th</sup>	std	1.25	0.16	0.34	3.66		0.38	0.4	0.15	0.22	0.55	40				
HIMT 1	Romano-British	mean	19.11	1.00	2.49		0.05	0.50	6.08	0.33	1.72	1.36	501	117	400		
n=123	4 <sup>th</sup> -5 <sup>th</sup>	std	1.12	0.16	0.29		0.02	0.12	0.66	0.11	0.33	0.26	55	61	300		
HIMT 2	Romano-British	mean	19.65	0.78	2.17		0.05	0.58	6.02	0.12	1.00	0.72	446	31	900		
n=220	4 <sup>th</sup> -5 <sup>th</sup>	std	0.97	0.10	0.18		0.01	0.12	0.67	0.02	0.17	0.10	52	19	500		
HIMTa	Cyprus	mean	18.2	1.07	2.98	66.3	0.06	0.44	5.9	0.48	2.10	1.76	400	219	7700		
n=9	5 <sup>th</sup> -7 <sup>th</sup>	std	1.0	0.17	0.28	1.5	0.02	0.10	0.9	0.15	0.44	0.33	38	55	7700		
HIMTb	Cyprus	mean	17.7	1.15	3.28	65.1	0.14	0.41	5.5	0.58	1.68	3.55	410	250	4900		
n=5	5 <sup>th</sup> -7 <sup>th</sup>	std	0.1	0.12	0.22	0.5	0.03	0.03	0.2	0.08	0.16	0.14	12	33	3100		
group 1	France, Tunisia, Egypt	mean	19.12	1.23	2.88	64.49	0.11	0.41	6.22	0.49	2.02	2.28	498	216	7		
n=43	5 <sup>th</sup>	std	1.34	0.24	0.26	1.36	0.04	0.08	0.85	0.12	0.4	0.86	87	61	15		
série 2.1	France, Tunisia, Egypt	mean	18.49	1.23	2.54	64.43	0.18	0.79	7.80	0.16	1.60	1.35	669	85	132		
n=51	6 <sup>th</sup> -7 <sup>th</sup>	std	1.24	0.15	0.15	1.06	0.04	0.14	0.69	0.02	0.37	0.66	86	10	202		
série 3.2	France	mean	18.79	0.65	1.92	68.07	0.08	0.44	6.99	0.09	0.95	0.70	536	57	18		
n=17	turn 5 <sup>th</sup> /6 <sup>th</sup>	std	0.85	0.16	0.15	1.49	0.03	0.08	0.74	0.02	0.34	0.15	106	11	34		
Levantine I	Apollonia, Israel	aver	15.17	0.63	3.05	70.64	0.15	0.63	8.07	0.12	<0.10	0.48	391	52			
n=9	6 <sup>th</sup> -7 <sup>th</sup> c.	std	0.91	0.09	0.17	1.98	0.05	0.20	1.48	0.00		0.13	80	9			
Levantine II	Bet El'ezzer, Israel	mean	12.20	0.63	3.32	74.89	0.12	0.46	7.16	0.13	0.10	0.58	369*	71*			
n=27	7 <sup>th</sup> -8 <sup>th</sup> c.	std	1.32	0.09	0.28	1.48	0.03	0.08	0.59	0.03	0.00	0.22	50*	11*			
Egypt I		mean	17.07	0.84	4.46	70.94	0.08	0.43	2.72	0.55	0.04	1.79	185	189	1		
n=2	Ahihus, Tiberias, Israel, 7 <sup>th</sup> -8 <sup>th</sup> century	Std	1.41	0.04	0.11	1.68	0.04	0.01	0.06	0.01	0.00	0.04	6	10	1		
Egypt II	several sites, Israel	mean	14.17	0.58	2.53	70.32	0.10	0.30	9.53	0.26	0.09	0.95	185	175	4		
n=55	8 <sup>th</sup> -9 <sup>th</sup> century	Std	1.11	0.19	0.24	1.09	0.03	0.13	0.82	0.04	0.23	0.15	41	33	12		

\*n=5.

Table 1. Mean values and standard deviations of glass groups mentioned in the text. (Sources: Roman glass - Nena et al. 1997; Group E - Mirti et al. 1993; HIMT 1, HIMT 2 - Foster and Jackson 2009; HIMTa, HIMTb - Ceglia et al. 2015; Ceglia et al. 2017; group 1, série 2.1 and série 3.2 - Foy et al. 2003; Apollonia and Bet'elzezer - Freestone et al. 2000. Egypt I, II - Phelps et al. 2016; Blank entries - data not published).

group	cent. AD	(Fe <sub>2</sub> O <sub>3</sub> + TiO <sub>2</sub> +MgO)/SiO <sub>2</sub>	(Al <sub>2</sub> O <sub>3</sub> +K <sub>2</sub> O+ CaO)/SiO <sub>2</sub>	sum	TiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>
Roman glass <i>n</i> =227	1 <sup>st</sup> – 4 <sup>th</sup>	0.019	0.156	0.175	0.05
naturally colourless 1b <i>n</i> =1	Late 2 <sup>nd</sup> – 4 <sup>th</sup>	0.012	0.147	0.159	0.02
naturally colourless Kosmaj K3 <i>n</i> =4	1 <sup>st</sup> – 3 <sup>rd</sup>	0.012±0.003	0.147±0.009	0.159±0.01	0.02±0.001
AD/N1 (Mn) <i>n</i> =45	1 <sup>st</sup> – 4 <sup>th</sup>	0.017±0.004	0.158±0.014	0.174±0.016	0.024±0.009
AD/N2 (Sb) <i>n</i> =4	2 <sup>nd</sup> – 3 <sup>rd</sup>	0.014±0.003	0.112±0.007	0.127±0.010	0.047±0.015
série 3.2 (non t.) <i>n</i> =2	1 <sup>st</sup> – 2 <sup>nd</sup>	0.023±0.003	0.124±0.007	0.147±0.009	0.045±0.011
groupe 1 (HIMT) <i>n</i> =43	5 <sup>th</sup>	0.075±0.017	0.148±0.014	0.222±0.024	0.169±0.032
série 3.2 <i>n</i> =17	5 <sup>th</sup> / 6 <sup>th</sup>	0.021±0.004	0.138±0.016	0.159±0.020	0.049±0.009
série 2.1 <i>n</i> =51	6 <sup>th</sup> – 7 <sup>th</sup>	0.043±0.011	0.173±0.012	0.216±0.016	0.062±0.007
Levantine I (Apol- lonia)	6 <sup>th</sup> – 7 <sup>th</sup>	0.017±0.003	0.168±0.028	0.185±0.030	0.021±0.010
Levantine II (Bet'Eliezer)	7 <sup>th</sup> – 8 <sup>th</sup>	0.018±0.003	0.147±0.008	0.164±0.010	0.029±0.013
Egypt I	7 <sup>th</sup> – 8 <sup>th</sup>	0.045±0.001	0.107±0.005	0.152±0.006	0.122±0.005
Egypt II	8 <sup>th</sup> – 9 <sup>th</sup>	0.026±0.004	0.176±0.015	0.201±0.016	0.103±0.015

Table 2. Amounts of heavy and light minerals in the glass making sands for several Roman glass types, and TiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio differentiation between Levantine and Egyptian sands. Note that the naturally colourless glass from such distant places as England (1b) and Serbia (K3 from Kosmaj) have the same concentrations of sand impurities. This hints at the use of the same sand. Note also that Levantine types have a lower TiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> (< 0.25) ratio than Egyptian groups (> 0.45). (Sources: Foy et al. 2003; Gallo et al. 2013; Nenna et al. 1997; Foster and Jackson 2010; Foster and Jackson 2009; Phelps et al. 2016; Freestone et al. 2000).

zo et al. 2016a), Ca-rich HIMT, Gliozzo et al. 2016b). However, new data demonstrated that some of these names cover very similar compositions. HIMT 1, strong HIMT and Foy group 1 are now considered HIMT. HIMT is now divided into HIMTa and HIMTb, according to the iron content (Ceglia et al. 2015). HIMT 2 is now considered to be very similar to Foy 3.2, while Weak HIMT, HLIMT and Ca-rich HIMT are considered akin to Foy 2.1 (Freestone et al. 2018).

Egypt, perhaps Sinai, is considered to be the source of sand used to produce HIMT glass. Sand from Egyptian beaches has high concentrations of heavy minerals, derived from the Nile and washed ashore by sea currents and winds (Nenna 2000, 2003; Freestone et al. 2005; Gliozzo et al. 2015).

Roman antimony-decoloured glass and série 3.2 are also considered to be of Egyptian provenance (Schibille et al. 2016), but different to that of HIMT, since the sand used in its production is much cleaner.

However, not all the glass of the 4<sup>th</sup> century had darker colours. Roman manganese-added glass was colourless. Its supposed provenance is the Levantine coast (Brill 1988). The Jalame-type glass, with or without added manganese, shows a wide colour pallet (colourless, aqua, green or olive), and appeared in the mid-4<sup>th</sup> century in Palestine. The type with no added manganese has higher alumina (2.7%) and lime (8.77%) and lower sodium (15.74%) than the Roman glass. Higher alumina and lime are explained by feldspar-rich sand,

while lower sodium by the geographical distance from the Wadi El Natrun natron deposits.

Série 3.2 was almost colourless or slightly coloured (Foy et al. 2003), and had a manganese-decoloured composition, manufactured with more pure sands. Its composition is broadly similar to that of antimony-decoloured Roman glass and is considered of Egyptian provenance.

### ***Sixth and seventh century glass types***

The 6<sup>th</sup> – 7<sup>th</sup> century glass from Apollonia in Israel, termed Levantine I (Freestone et al. 2000), is high in feldspar derived alumina (3.05%) and lime (8.07%), and low in heavy mineral derived iron (0.47%) and titanium (0.07%), and low sodium (15.17%), very similar to Jalame glass (also classified as Levantine I).

Glass from the 7<sup>th</sup> – 8<sup>th</sup> century furnaces in Bet Eliezer is termed Levantine II (ibid.). It has even lower sodium (12.3%) and lime (7.36%) and higher alumina (3.26%) than Levantine I. Even lower sodium in later Levantine II is interpreted as the exhaustion of the trona deposits of Wadi El Natrun over time.

Late Antique natron glass from Egypt is termed Egypt I (Gratuze and Barrandon, 1990). It is richer in alumina (4.46%) and iron (1.79%) compared to Levantine glass. It was produced from the late 7<sup>th</sup>/early 8<sup>th</sup> to the late 8<sup>th</sup> century CE. Around 780 CE, this type was replaced by the type Egypt II, the last natron glass produced in Egypt (Schibille et al. 2019). Egypt II is lower in alumina (2.53%) and higher in lime (9.53%) than Levantine glass (Foy et al. 2003, Phelps et al. 2016).

Independently, Foy et al. (2000, 2003) describe ten glass groups from the Mediterranean, dated to Late Antiquity. Four of these also appear in the Western Mediterranean (groups 1-4). Compositionally, Foy group 1 is similar to HIMT 1, and group 2 is similar to HIMT 2. Foy group 3.1 corresponds to Levantine I, while group 4 is similar to the Roman antimony decoloured glass. Regarding Eastern Mediterranean types, Foy group 7 is akin to Egypt II, and groups 8 and 9 to Egypt I. The compositional differences between these groups are depicted in the Primary Component Analysis diagram (Fig. 1).

### ***Plant ash glass***

During the period between from the 1<sup>st</sup> to the 7<sup>th</sup> century CE, the flux used in the production of glass in the Mediterranean and continental Europe was the mineral natron. During the same period, the Mesopotamian region, east of the Euphrates river, continued its centuries-old tradition of halophytic plant-ash glass production. Plant-ash glass would not make its comeback to the Mediterranean before the Islamic transition during the 8<sup>th</sup> – 9<sup>th</sup> centuries CE (Phelps et al. 2016). No plant-ash glass production is recorded in the Mediterranean and Western Europe during this epoch, with the exception of a very limited, presumably specialised, production of halophytic plant-ash glass, evidenced first in Egypt (Rosenow and Rehren 2014, 2018) and most recently reported from Crete (Oikonomou et al. 2021) and Serbia (Balvanović et al. 2022).

### **PROVENANCE**

“In Syria there is a region known as Phoenicia... In this region is the source of the river called Belus that, after five miles, disembogues into the sea close to the Ptolemy colony... The sand from the river Belus is ground and purified by the power of sea waves and it becomes very clean... The coast upon which this sand is deposited is no more than five miles long, but nevertheless this was for many centuries the only place which provided materials for glassmaking” (Gaius Plinius Secundus, *Naturalis Historia*, ed. Karl Friedrich Theodor Mayhoff, Teubner, 1897). This testimony of Pliny the Elder was the map for early archaeological efforts to find primary glass production sites.

### ***Primary production sites and supposed provenances***

As already mentioned, there are relatively few reported Roman primary glass production sites, and all are located in the coastal areas of the Eastern Mediterranean, like Apollonia, Jalame, Bet Eliezer (Hadera), today Israel, and in Wadi El Natrun in Egypt, near the Nile delta. Seventeen furnaces were discovered in Bet Eliezer, each capable of producing several tons of raw glass in a single firing, as evidenced by the nine ton glass

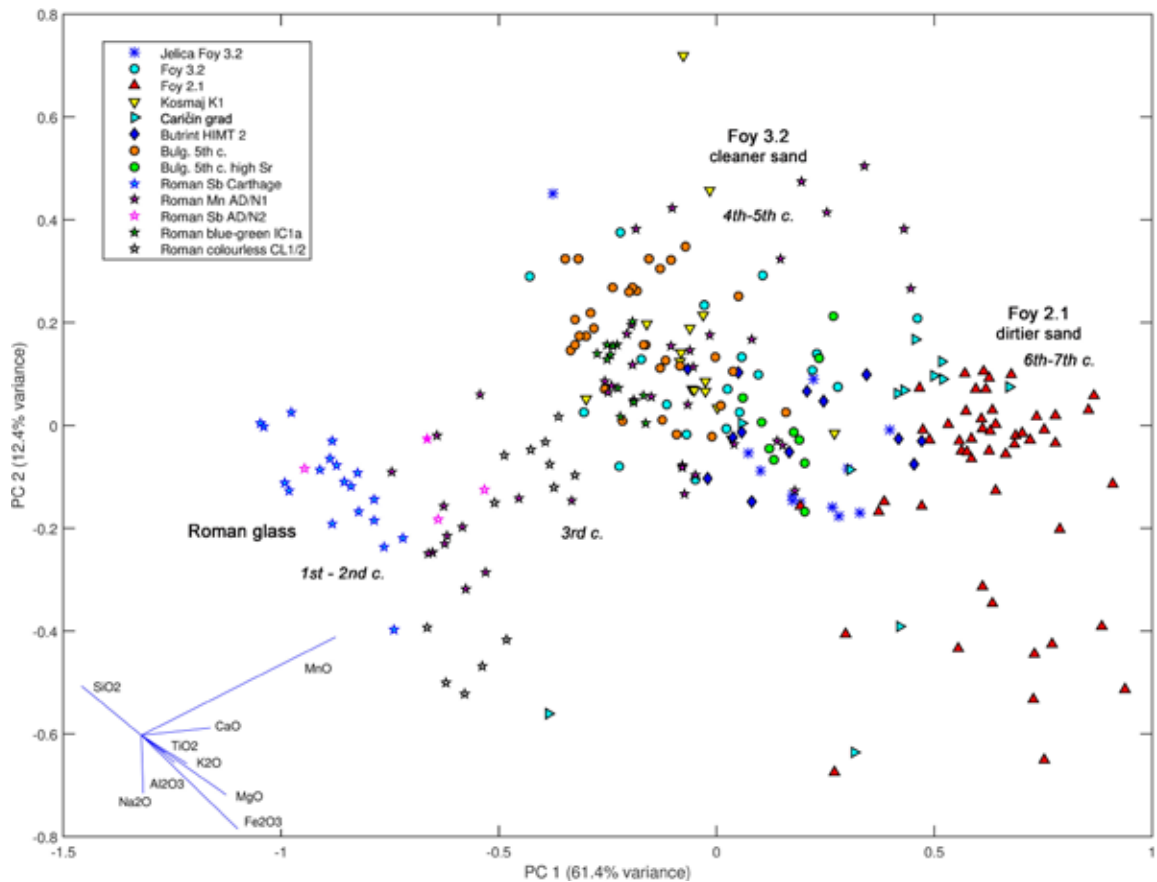


Fig 1. Principal component analysis of 256 glasses from 14 Roman and Late Antiquity natron glass groups. The diagram compares compositions of six glass groups from the Balkans, five Roman glass groups (with added manganese and with no added manganese, naturally coloured and decolourised) and Foy série 3.2 and 2.1. Vectors of oxides:  $\text{Na}_2\text{O}$ ,  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{K}_2\text{O}$ ,  $\text{CaO}$ ,  $\text{TiO}_2$ ,  $\text{MnO}$ ,  $\text{Fe}_2\text{O}_3$  lower left (Source: Balvanović and Šmit, 2022).

slab discovered in nearby Bet She'arim (Freestone et al. 2000). Such a topography of primary glass production sites, together with circumstantial evidence of glass finds in the region, and the writings of Pliny the Elder, formed the basis for the two-phase production model of the Roman glass.

The two-phased glass production model, in general, facilitates the possibility of the determination of the primary production sites. The elemental composition of a glass object reflects the composition of the primary glass used in its production and this, in turn, reflects the composition of the sand used in the primary furnace. Several relationships between concentrations of particular elements in glass were used as indicators of the compositional characteristics of particular sands and, thus, their possible origins.  $\text{TiO}_2/\text{Al}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3/\text{SiO}_2$  ratios differentiate high-titanium-low-alumina Egyptian sands from high-alumina-low-titanium Levantine sands (Schibille et al. 2016). A  $\text{SrO}/\text{CaO}$  ver-

sus  $\text{MnO}$  biplot is used to evaluate the amount of strontium that derives from lime (because manganese ore can contain some strontium), which can be useful in provenance determination in some cases. A value of  $\text{SrO}/\text{CaO}$  around 60 is characteristic of Eastern Mediterranean coastal sands (Freestone et al. 2018). Higher values of  $\text{Na}_2\text{O}$  to  $\text{SiO}_2$  possibly indicate an Egyptian primary glass manufacture, implying the abundance and availability of the mineral trona in Egypt (Freestone et al. 2000). How these minor elements ratios can differentiate between the types of sands used for glass production, is depicted in Fig. 2.

Taking such relationships and the circumstantial evidence into account, several hypotheses for the provenance of particular glass groups were suggested. It is considered that Levantine glass was manufactured with sands from around the mouth of the ancient Belus river in today's Israel (Brill 1988). This sand has high concentrations of feldspars and lime



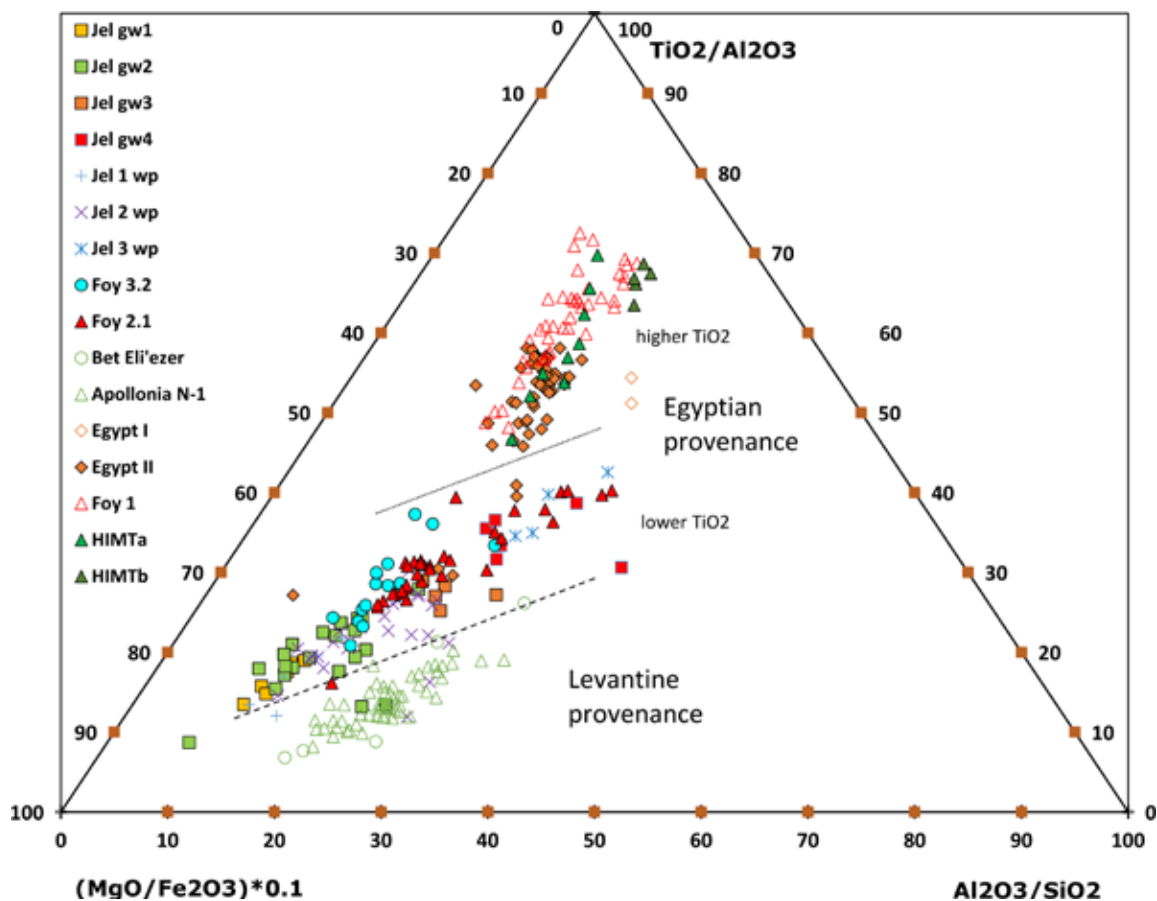


Fig. 2 Triangular diagram of  $\text{TiO}_2/\text{Al}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3/\text{SiO}_2$  and  $\text{MgO}/\text{Fe}_2\text{O}_3$  for Jelica glassware (gw1-gw3) and Jelica windowpane glasses (Jel wp 1-3), plotted against selected Late Antiquity glass groups. The diagram is indicative of mineralogical composition of glass-making sands, thus of sand provenance. Ratio  $\text{MgO}/\text{Fe}_2\text{O}_3$  is scaled for easier differentiation of groups. Note that there are two groups of glasses of Egyptian provenance, differentiated mostly by the  $\text{TiO}_2/\text{Al}_2\text{O}_3$  ratio.

and low concentrations of heavy minerals. Roman manganese-decoloured glass is also thought to be of Levantine origin (Brill 1988, Schibille et al. 2016).

It is considered that HIMT glass, very rich in heavy minerals, was manufactured using sands from Egypt, possibly Sinai. This is supported by isotopic analyses (Nenna 2000, 2003; Freestone et al. 2005; Gliozzo et al. 2015). Roman antimony-decoloured glass and Foy 3.2, are thought to be of Egyptian provenance (Schibille et al. 2016, Maltoni et al. 2016, Paynter and Jackson 2018). A very different type, Foy 2.1, with high concentrations of impurities, is also considered to be of Egyptian provenance (Foy et al. 2003). A unique Late Antiquity glass with exceptionally high alumina has been discovered in Turkey (Rehren et al. 2015).

Other provenances of glass-producing sands were also suggested. These include Western Mediterranean (Brems et al. 2012; 2013) and Greece (Silvestri et al. 2017). Wadi El Natrun in Egypt is considered the main

source of mineral soda used for fluxing. Other possible locations, like the lakes al-Barnuj in Egypt (Shortland et al. 2006), and Pikrolimni in Greece (Dotsika et al. 2009), are also suggested.

#### *Determination of provenance by isotopes and rare earths*

Some other tracers are indicative of geochemical processes underlying the sand formation and can serve as “fingerprints” of particular sands. The most common ones used in provenance determination of Roman natron glass are isotopic ratios of strontium, neodymium and hafnium, and rare earth patterns.

**Isotopic Ratios.** The ratio of strontium isotopes  $^{87}\text{Sr}/^{86}\text{Sr}$  can differentiate between beach sands and inland sands. This stems from the fact that beach sands derive most of their CaO from seashells, while inland sands derive it mostly from lime. Seashells have a  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio close

to contemporary seawater (0.7092), while these ratios in lime reflect the ratios at the time of its geologic formation. Thus, the  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio can provide a differentiation between coastal and inland glass-making sand. However, this ratio does not differentiate between Levantine and Egyptian sands (Barfod et al. 2020), suggesting the use of beach sands in both cases.

Barfod et al. (2020) showed that hafnium isotope composition is very good discriminator between Egyptian and Levantine sands and, by implication, glasses. This stems from the fact that hafnium on Egyptian and Levantine beaches derives from the mineral zircon, brought in by the Nile and longshore sand transport by sea currents and winds. It is known that zircon drops out from the transport along the route, yielding lower concentrations of hafnium and, significantly, changing its isotopic composition along the way. This difference in Hafnium isotopic ratios indicates that Roman Sb glass, Foy 2.1 and Egypt I were produced in Egypt, while Roman Mn, Levantine I glass from Apollonia and Jalame glass have a Levantine provenance.

**REE Patterns.** Another set of provenance indicators are rare earths patterns. The geochemistry of rocks participating in sand formation is reflected in rare earth patterns. REE patterns are concentrations of rare earths normalised to the upper continental crust (Kamber al. 2005). REE patterns of glass assemblages of Foy 2.1 type of glass from Visigothic Spain (Balvanović and Šmit 2020) are shown in Fig. 3. The Foy 2.1 type has a variant with higher concentrations of iron oxide, called Fe-rich Foy 2.1. The assemblage is divided into three groups according to the iron oxide concentrations, with low iron (average of 0.94%), high iron (1.77%) and very high iron (2.66%). Their respective REE patterns are depicted.

With the increase of iron from the low iron to high iron group, concentrations of REEs also increase, while the shapes of the REE pattern remain the same, i.e., the differences in their respective heights are due to iron concentrations (0.94% versus 1.77%). This indicates the same origin of iron in both groups. The same is true for the patterns of high iron and very high iron, but with the exception of cerium and hafnium, which decrease with increases of iron concentrations. This might indicate different type of mineral iron and, thus, likely point to a different provenance of the sand.

## GLASS IN SERBIA DURING THE ROMAN EMPIRE AND LATE ANTIQUITY

How do Roman and Late Antiquity glass finds from Serbia fit into the wider picture of glass type distribution over the entire Mediterranean, and through time? The changes in glass compositions that occurred during the 4<sup>th</sup> century and again during the 6<sup>th</sup> century CE, are also reported from Serbia (Table 3, Fig. 4). After the domination of Roman glass (mostly naturally colourless and decolourised with antimony), manganese became the main decolourant in the 4<sup>th</sup> century, just as in the other areas of the Mediterranean. The newer, manganese-decoloured glass, was recycled with older, antimony-decoloured glass, yielding Roman Sb+Mn glass. New types of glass appeared, like Foy 3.2 and HIMT. The specificity of Serbian Foy 3.2 glass is that it seems to be among the oldest yet reported in the literature. The Kosmaj K1 group (Foy 3.2 type of glass) is dated to the 3<sup>rd</sup> to 4<sup>th</sup> century and some glasses to the 2<sup>nd</sup> to 3<sup>rd</sup> century. The lightly coloured Foy 3.2 type, with small concentrations of sand impurities, was also sometimes recycled with antimony - decolourised glass, although not often. During the period between the 2<sup>nd</sup> and 4<sup>th</sup> century, glass was imported to Serbia from two sources of primary glass production: Egypt (around three quarters of analysed glasses) and the Levantine coast (around one quarter, Fig. 4, Tab. 3).

From the 6<sup>th</sup> century, earlier compositional types vanish from the archaeological record, and a new type, Foy 2.1, with a darker colour, reflecting high concentrations of impurities in the sand, and heavily recycled, dominate the Serbian landscape. The exception to the disappearance of earlier types is Foy 3.2, which continued well into the 6<sup>th</sup> century. Foy 3.2 type among Serbian finds spans the period from the late 2<sup>nd</sup> to early 3<sup>rd</sup> century (Kosmaj K1) to the late 6<sup>th</sup> century (Jelica). It is noteworthy that Foy 3.2 appears among the 6<sup>th</sup>/early 7<sup>th</sup> century Serbian assemblages of windowpanes and glassware from Jelica and among raw glass from Caričin Grad (Iustiniana Prima) (Drauschke and Greiff, 2010) with not insignificant percentages (on average 12%). Thus, Foy 3.2 glass from Kosmaj and Caričin Grad significantly widen the 4<sup>th</sup> to 5<sup>th</sup> century timespan of Foy 3.2 glass, as earlier considered. A comparison with the published data shows that this type of glass was much more common in the Balkans than in contemporary Italy. In addition, the percentages of Foy 3.2 type

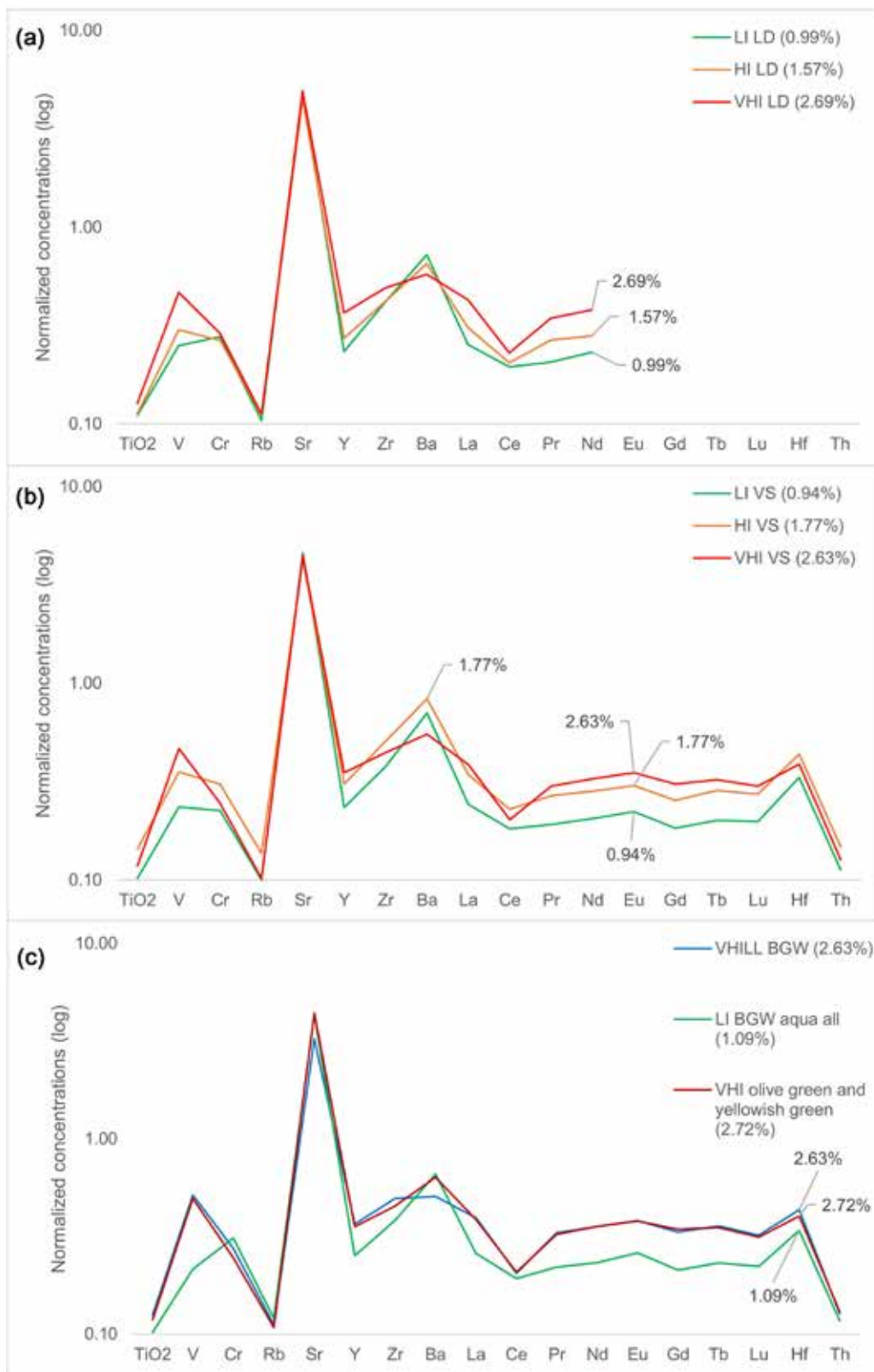


Fig 3. Trace element patterns of Fe-rich Foy 2.1 glasses, grouped by iron concentrations. Values normalised to the upper continental crust [38]. Groups from the Lower Danube (a), Visigothic Spain (b) and Byzantine glass weights (c). Note that for the Lower Danube REE dataset only La-Nd measurements are reported (Source: Balvanović and Šmit, 2022).

**Table 3.**

compositional type	Mala Kopašnica 2 <sup>nd</sup> – 4 <sup>th</sup>	Kosmaj late 2 <sup>nd</sup> /early 3 <sup>rd</sup> – 4 <sup>th</sup>	Viminacium 3 <sup>rd</sup> – 4 <sup>th</sup>	Mediana late 3 <sup>rd</sup> – 5 <sup>th</sup>	Egeta 4 <sup>th</sup>	Jelica 6 <sup>th</sup> /early 7 <sup>th</sup>	Caričin grad 6 <sup>th</sup> /early 7 <sup>th</sup>	Sum
Roman blue-green								0
Roman naturally colourless		4						4
Roman Sb decolourized	7		9		7			23
Roman Mn decolourized	8		6	11	3			28
Roman Sb+Mn decolourized		14			1	1		16
Foy série 3.2		15	5	19		12	2	53
HIMTa			1	8				9
HIMTb			1					1
Jalame Mn			1					1
plant-ash coloured			1				2	3
Foy série 2.1 LI	2		2			5	4	13
Foy série 2.1 HI						48	14	62
Foy série 2.1 VHI						5	2	7
outlier						11	1	12
TOTAL	17	33	27	38	11	83	26	235

Table 3. Glass from 2<sup>nd</sup> – 6<sup>th</sup>/early 7<sup>th</sup> century sites in Serbia, divided into compositional groups (Sources: Stamenković et al. 2015; Stojanović et al. 2015; Balvanović et al. 2022; Drauschke and Greiff 2010).

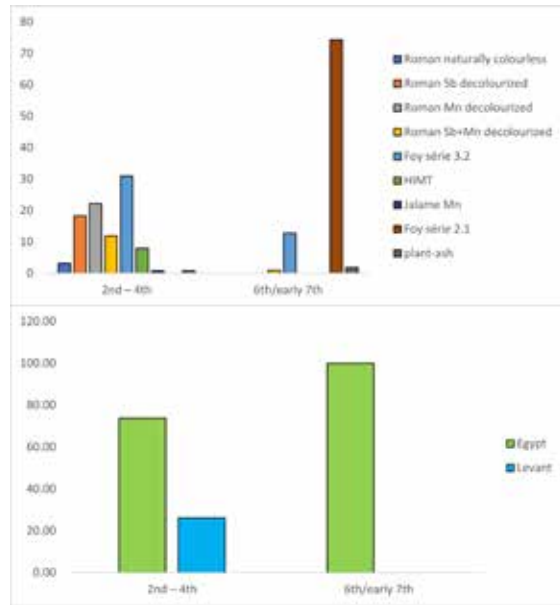


Fig 4. Up: Percentages of compositional types among 126 glasses from 2nd – 4th century and 109 glasses from 6th century, that are reported from Serbia. Down: supposed provenance of these glasses (Sources: Stamenković et al. 2015; Stojanović et al. 2015; Balvanović et al. 2022; Drauschke and Greiff 2010).

among the glass assemblages published from the Balkans seem to increase eastwards, possibly indicating that the import of Foy 3.2 raw glass came from the East rather than from the West. The possible candidate is the Danube waterway (Fig. 5). However, this hypothesis will have to be re-examined when more data on Balkan glass finds becomes available.

Another important change in the Balkans, regarding raw glass trading routes, seems to have happened during the 6<sup>th</sup> century. Data shows that raw glass in this period was imported exclusively from Egypt. This is also in accordance with the wider picture of the Mediterranean region, notably Italy.

## CONCLUSIONS

The Roman soda-lime-silica (natron) glass evolved, both compositionally and in its distribution, with time. The compositional change reflected the changing compositions of sands used in the production of primary (raw) glass. This was the consequence of a change of sand mines, a routine that had to be practiced whenever good quality sand was exhaust-

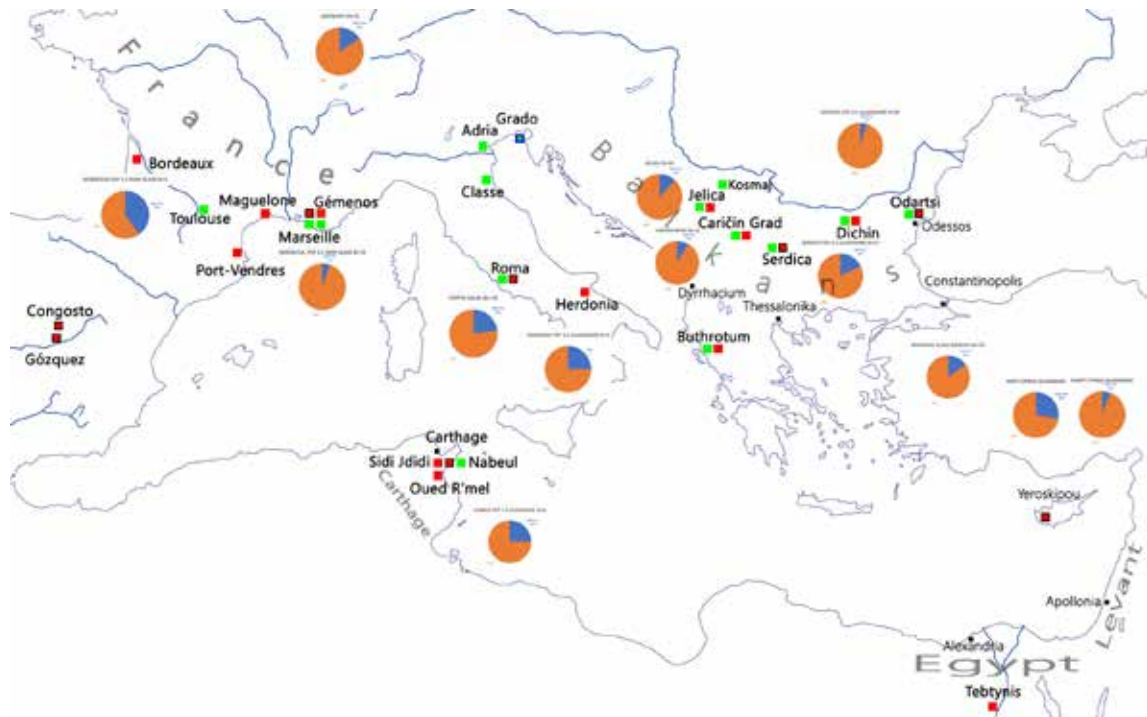


Fig. 5. Distribution of Late Antique glass types in the Mediterranean. Pie charts show percentages of glass types within reported assemblages. Yellow and blue pie charts show percentages of high iron glasses among Foy 2.1 types in particular regions. Note that distributions are different in different regions. In the Balkans, Foy 3.2 is more represented than in Italy (Sources: Foy et al. 2003; Schibille et al. 2016; Stamenković et al. 2015; Stojanović et al. 2015; Balvanović et al. 2022; Drauschke and Greiff 2010; Cholakova et al. 2016; Conte et al. 2016; Schibille 2011; Ceglia et al. 2015; Mirti et al. 1993; Gliozzo et al. 2015a).

ed at a particular quarry. Despite this, the “classical” Roman blue-green glass was of remarkable compositional stability, and it lasted for several centuries. Considerable change started during the 4<sup>th</sup> century, when manganese started to replace antimony as a decolouriser, and when several new types of glass, often with darker colours, appeared, such as the HIMT class of glasses. Besides this darker glass, a variety of other colours also appeared, like Jalame glass, with or without added manganese, and very lightly coloured Foy série 3.2. Later, yet newer glass compositions appeared, in the 6<sup>th</sup> century Foy série 2.1, in the 7<sup>th</sup> century Levantine II and Egypt I, and in the 8<sup>th</sup> century Egypt II. The era of natron glass ended with the replacement of natron with plant-ash flux, a change that happened with the Islamic transition, during the 8<sup>th</sup> and 9<sup>th</sup> centuries. However, a small, possibly specialised, production of plant ash glass production has been reported from Egypt, and glass of this types has also been reported from Crete and Serbia.

The glass compositions and period of their distribution followed this general picture of the Mediterranean, with an important exception. It seems that glass of the type série 3.2 appeared in the archaeological record earlier and lasted longer than in other parts of the Mediterranean, notably Italy. It also seems that the ratios of this type of glass in the reported assemblages in Serbia (and the eastern and Central Balkans as a matter of fact) are higher compared to other parts of the Roman Empire. This suggests that the commercial routes importing the raw glass of this type were different. The data suggests that série 3.2 was imported to the eastern and Central Balkans via the Danube.

## APPENDIX – GLASS COLOURS

Natural colours of the Roman glass range from colourless and nearly colourless to coloured, depending mostly on the amount of iron in glass-making sand and redox conditions in furnace. Roman glass could also be intentionally coloured (with iron, cobalt, copper, manganese) or decolourised (with antimony and manganese). Below are the photographs of glasses from several sites in Serbia, showing various types of glasses and their typical colours (Figs. A-C).

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Fig. A. Left: colourless glass C-123e from Egeta (4<sup>th</sup> century). Middle: naturally coloured glass, sample 17b (Gradina on Jelica, 6<sup>th</sup> c. AD), of type Foy 2.1, with  $\text{Fe}_2\text{O}_3 = 0.91\%$ . Right: naturally coloured glass, sample 28 from Jelica, of type Foy 2.1 high-iron, with  $\text{Fe}_2\text{O}_3 = 1.96\%$  (Sources: Balvanović and Šmit 2020, C-123e is not published).



Fig. B. Left: glass 16 from Gradina on Jelica (6<sup>th</sup> century) coloured with 580 ppm of cobalt; right: glass 24 from Jelica (6<sup>th</sup> century) coloured with 1830 ppm of copper (Source: Balvanović and Šmit 2020).

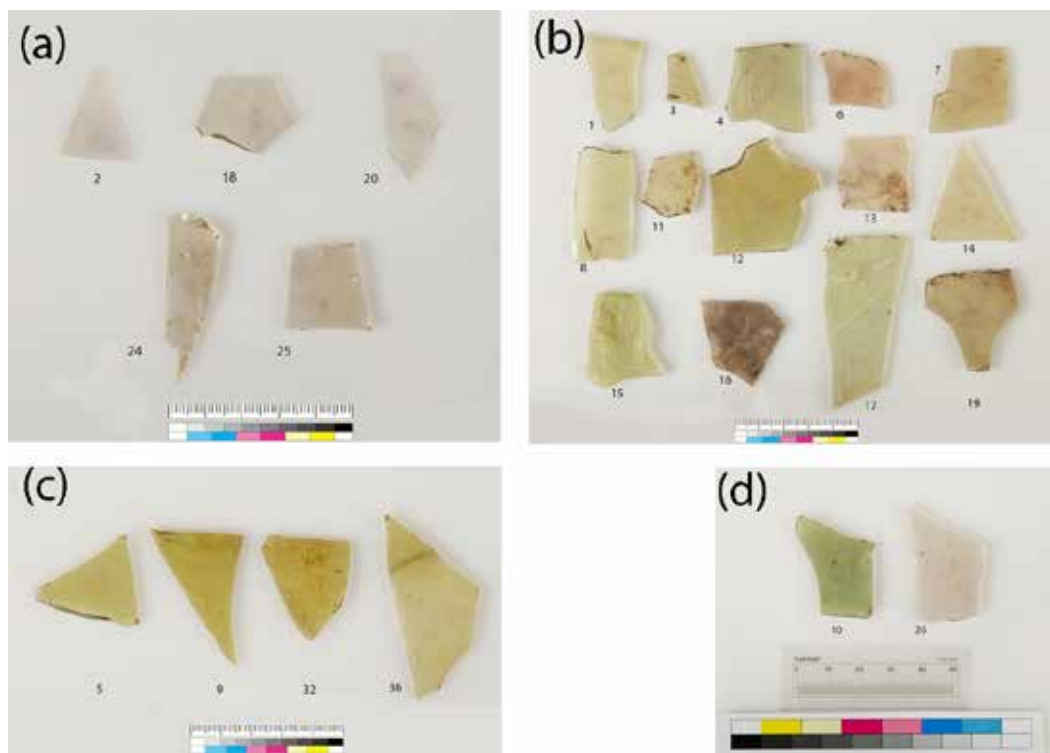


Fig. C. Photographs of windowpane glasses from Jelica: a. Jelica 1 - almost colourless glass of type Foy série 3.2 (average  $\text{Fe}_2\text{O}_3 = 0.70\%$ ); b. Jelica 2 - light olive-green and amber glass of type Foy série 2.1 (average  $\text{Fe}_2\text{O}_3 = 1.00\%$ ); c. Jelica 3 - deeper olive-green glass of type Foy série 2.1 high-iron (average  $\text{Fe}_2\text{O}_3 = 2.71\%$ ); d. Jelica 4 - glass of type Foy 2.1 with 2.26 % of iron oxide (left) and glass of type Foy 3.2 with 0.46% of iron oxide (right) (Source: Balvanović et al. 2018).

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**REZIME**

**RIMSKO I KASNOANTIČKO STAKLO U OBLASTI MEDITERANA I SRBIJI: PROIZVODNJA, KOMPOZICIONI TIPOVI I POREKLO**

**KLJUČNE REČI: STAKLO, KOMPOZICIONI TIPOVI, POREKLO, RIMSKI PERIOD,**

**NATRON, BILJNI PEPEO.**

Rad daje sintezu proizvodnje rimskog stakla, kompozicionih tipova i porekla primarnog stakla tipa soda-kreč-kvarcni pesak (natronsko staklo) za vreme rimske epohe i epohe kasne antike. Takođe, kratko opisuje malu proizvodnju stakla od biljnog pepela, koje se pojavljuje i među staklima nađenim u Srbiji. Rad opisuje proizvodnju primarnog stakla i komponente koje se u proizvodnji koriste, i dvofazni model proizvodnje rimskog stakla. Rad daje karakteristike sastava tipičnih tipova rimskog stakla: plavo-zeleno rimsko staklo, prirodno bezbojno staklo, bezbojno staklo obezbojeno antimonom; potom tipove stakla koji se pojavljuju tokom IV veka: rimsko staklo obezbojeno manganom, HIMT, Foj 3.2, Džalame i bezbojno staklo obezbojeno antimonom i manganom. Rad opisuje karakteristike najčešćeg tipa tokom VI veka, odnosno Foj 2.1, i njegove podtipove sa povišenom koncentracijom gvožđa.

Daje se prikaz porekla navedenih tipova i opis metoda upotrebljenih za određivanje porekla: nalazi stakla sa okolnostima nalaza, koncentracije glavnih i sporednih elemenata, izotopski odnosi stroncijuma i hafnijuma, obrasci retkih zemalja. Rad opisuje rasprostranjenost kompozicionih tipova stakla pronađenih u Srbiji, daje kratak prikaz promene rasprostriranja tipova sa protokom vremena, i stavlja ove nalaze u širi mediteranski kontekst.

Rad pokazuje da rasprostriranje pojedinih tipova u Srbiji uopšteno prati njihovo rasprostriranje u širem okruženju. Važan izuzetak od ovoga predstavlja tip Foj 3.2, koji je, kako se čini, češće zastupljen među analizovanim srpskim kolekcijama stakla, a koji se pojavio ranije (III vek) i trajao duže (VI vek) nego u širem prostoru Mediterana.

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