Fragments of luxury: Decorated glass from the Palace of Mystras, Greece

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Abstract
An assemblage of luxurious glass findings from the Palace of Mystras, Lakonia, Greece, was analysed via SEM/EDS and Raman spectroscopy, aiming to determine their raw materials, manufacturing technology and likely provenance. Twenty six fragments of everyday use objects of exceptional quality were studied. All fragments are decorated (with white and blue trail or canes, enameled or filigrana glass). Their date cannot be determined with specificity but has to span between the mid 13th and mid 19th c. AD, the period since the construction of the site and throughout its continuous use as the administrative centre of the Despotate of Mystras.
The examination of the glass body resulted in the distinction between two groups: soda and potash glass. The provenance of the soda glass cannot be specifically determined, but production in multiple workshops seems likely, based on the relative heterogeneity of the composition. The presence of manganese as a decolourant suggests that they probably date before the 17th c.
All potash glasses are enameled and likely originating from Bohemia. The examination of the enamels further corroborates the likely provenance from Bohemia, since the enamels are produced using different manufacturing processes than the Islamic and Venetian. Overall, it seems that this group of glasses was selectively imported to Mystras to cover specific stylistic preferences of the higher ranking members of the Palace, as suggested by the recovery of the samples from Building E, the administrative centre of the Palace Complex.
The present study contributes to the acquisition of new knowledge about the post-Byzantine glass production and trade in southern Greece. Further analyses will provide with significant insights into the commercial, artistic and technological interactions of Mystras with the Ottoman Empire and Europe.

Keywords: Post-Byzantine glass; Palace of Mystras; Enamel; Chemical groups; Provenance

Introduction
During the Middle Ages, groundbreaking innovations took place that completely changed the production of glass in Europe. During the 9th c. the main source of alkalis changed from natron to plant ash (Verità 2013). Additionally, the production model gradually changed, with the introduction of local workshops which carried out both the activities of glassmaking and glassworking. These two changes marked the beginning of a period of experimentation, with an emphasis on the use of new raw materials, local and easily accessible, as well as the introduction of new production methods. At the same time, the production of older types of high quality glass continued, using traditional raw materials. As a result, the glass produced between the 14th and the 19th c. in Europe and the Middle East is characterized by exceptional chemical diversity, with Na-rich (Byzantine, Islamic, Venetian and 'façon de Venise' glass), K-rich (forest glass, High Alumina Low Alkali, crystal, Bohemian and English lead glass) and Mixed Alkali glass produced to cover the need for different qualities of glass objects. An overview of the main chemical groups and manufacturing centers is given elsewhere (Schalm et al. 2007; Palamara et al. 2017).
Although the post-medieval glass of central and northern Europe has been extensively studied, the production, distribution and use of glass of the same period in the territories of the Ottoman Empire remains almost unknown. It is known that Venice mass produced glass intended for the markets of the East and the Balkans, following local aesthetic standards (Wenzel 1977). In the regions of present-day Turkey and the Middle East, small quantities of glass were produced by local workshops, but only at the end of the 18th c. there was a significant increase in production in the Beykoz district of Istanbul, with strong influences from Venetian and Bohemian glass (Whitehouse 2012).
Chemical studies have been carried out in few collections (i.e. glass from Istanbul - Canav-Özgümüş 2012) and from limited locations in the Balkans (Topić et al. 2016 - and references therein-; Palamara et al. 2017; Šmit et al. 2009). These studies showed extremely high heterogeneity in the chemical

composition of glass, indicating the parallel introduction and use of glass from many different centers in the East and the West.

In Greece, the existence of small glass-working, or even glass-making, workshops cannot be ruled out, although there is no clear historical or archaeological evidence to support this hypothesis. The only workshop that has been found to date, in the area of the Agora in Corinth, dates much earlier to the 11th c. (Davidson 1940). In addition, there is no clear historical information about the main glass import centers and the possible commercial networks. The study of utilitarian glass objects from two public bathhouses in Kyparissia and Methoni, southern Greece, indicated an unexpected chemical heterogeneity, and consequently the import of glass from a large number of glass centers both from the West and the East. Additionally, there were indications of a local production of HLLA glass in Methoni (Palamara et al. 2017; Palamara and Zacharias, forthcoming).

The present study focuses on the chemical study of a large assemblage of luxurious glass objects of the post-Byzantine period, from the Palace of Mystras in Lakonia, Greece. The site of Mystras presents immense significance, being the administrative, commercial and cultural centre of the late Byzantine and post-Byzantine period.

The Palace of Mystras

The city was founded in 1249 by Frank commander William II of Villehardouin, who built a castle fortress on top of Myzithra Hill (Runciman, 1980). It soon became one of the most prosperous cities in Greece and the administrative centre of the broader region and retained significant political and financial ties both to Constantinople and to Italy (and primarily Venice). From the 13th c. onwards, Mystras underwent successive occupation periods by the Byzantines, the Franks and the Ottoman Turks, until it was liberated in 1821. The city declined after the founding of the new city of Sparta in the early 19th c., while the last inhabitants left in 1953 (Chatzidakis, 1992).

The Palace of Mystras has undergone extensive study; the successive construction phases of the various buildings are well-documented and there is a lot of available information on the use of each building. The construction of the Palace is divided in 5 phases (Figure 1): (1) Period of Latin occupancy: 1249 - 1262, (2) Late - Byzantine I: 1262 - 1348, during which it served as the seat of the Byzantine General, (3) Late - Byzantine II: 1348 – 1384 (Reign of Kantakouzenos), (4) Late - Byzantine III: 1384 – 1460 (Reign of Palaeologos), and (5) Post – Byzantine: 1460 – 1821 (Sinos 2021).

The decorated glasses under study in this paper were primarily found in Building D, Building E and in the Bathhouses. Building D, built by Manouel Kantakouzenos, was a 2-storey building which originally functioned as the residence of the Despots. It is interesting to note that in this building there was an overall higher percentage of good quality decorated glass and a lower percentage of low quality glass, compared to the other buildings of the complex. Building E, built by Palaeologi, was a 3-storey building with multiple functions: in the ground level there were stables and storage, in the 1st level the quarters of the palace guard, and in the 2nd level the throne hall (Sinos 2021). The Bathhouse located in the west of the palace is dated in the Ottoman period but probably was built upon a previous Byzantine similar construction (Arvanitopoulos, 2004).

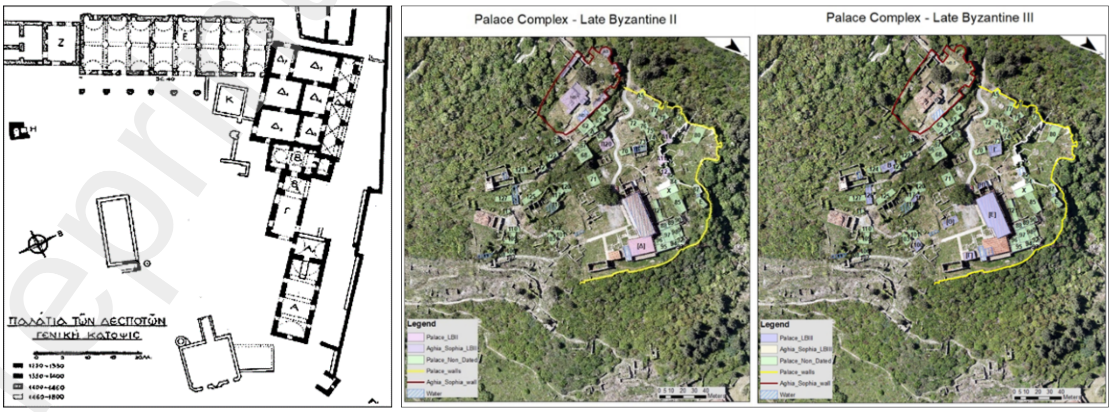

Figure 1. Map of Mystras Palace Complex (after Sinos 2021) and digital reconstructions of the Palace Complex during the Late Byzantine II and Late Byzantine III period (after Panagiotidis and Zacharias 2022)

Samples and methodology

A large assemblage of 140 glass fragments, recovered during the excavation period of 1984-1985 from different buildings of the Palace complex, were analysed in order to identify their raw materials, manufacturing technology and likely provenance. The fragments belong to utilitarian objects (glasses, bottles, lamps, etc.) of exceptional quality. Their date cannot be determined with specificity but has to span between the mid 13th and mid 19th c. AD, the period during which the Palace was active.

The present paper focuses specifically on 26 fragments of decorated glass. The vast majority are colourless, while there is also one purple and one amber fragment. The fragments are small and a typological description is not always possible, but the majority seem to correspond to the base and or the body of small vessels; three funnel-shaped rims, one knop stem and one sample that could be attributed either to the base of a cesendello lamp or a lid, are also identified. Most fragments are decorated with primarily white (and in a few cases blue) trail or canes, 3 fragments can be described as filigrana, with white (and in one sample white and blue) glass threads and 6 fragments are enameled and present floral decorative patterns, formed with white, yellow, red and blue enamel (Figure 2).

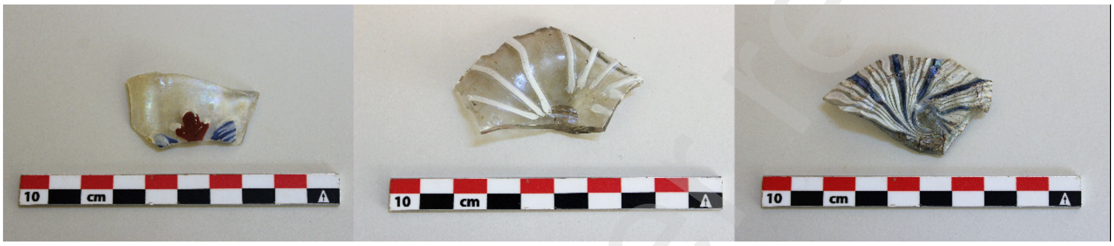

Figure 2. Images of representative samples of enameled glass (sample MY16), glass with white opaque canes (sample MY61), and filigrana glass (sample MY133)

Prior to the chemical analysis, all samples were examined with a portable LED microscope (Moritex I Scope). Areas with strong corrosion were documented and the most suitable areas for micro-sampling were selected. On their outer surface, the samples show common effects of corrosion, such as iridescence, dulling, milky weathering and pitting.

In order to avoid the chemical effects of corrosion, micro-samples were cut off, embedded in resin and polished. For each sample, three to four analyses were performed in different areas, using a Scanning Electron Microscope (JEOL JSM-6510LV), combined with an Oxford Instruments energy dispersive spectrometer. Quantitative data were obtained with INCA software, based on a pre-existing internal calibration of the device. The analyses were performed in high vacuum and at a voltage of 20 kV. The spectra were obtained in areas of 300x400 μm (x100 magnification) and with a collection time of 300 sec. The precision and accuracy of the device used is described in detail by Palamara et al. (2017).

Raman spectra were collected on a Renishaw InVia Reflex confocal micro-Raman spectrometer using the excitation line at 488 nm of an argon ion laser. All Raman spectra were measured at room temperature with 2 cm^{-1} resolution in the frequency range from ca. 120 to 2000 cm^{-1}. More detailed information on the settings are described by Möncke et al. (2013).

Results

Analysis of glass. The average value of concentration of the main oxides for the samples under study are presented in the Appendix. Based on the diagram of Figure 3 and 4, and the categorization described by Schalm et al. (2007), two chemical groups can be distinguished: (1) The majority of the samples, including the two coloured glasses, belong to the category of soda glass; (2) The six enameled samples belong to the K-rich glasses, and more specifically to the category of potash glass. The colour of the amber and the purple glass is attributed to their high iron and manganese content, respectively.

The decorated soda glass samples present an overall heterogeneous composition of their major elements and they do not form a distinguishable group compared to the rest of the soda glasses of the assemblage. Only one filigrana sample (MY22) is easily distinguished from the rest of the assemblage, based on its very high sodium, relatively high alumina and very low calcium content. One other undecorated colourless glass presents similar chemical traits, which do not seem to match any of the published data for the major glassmaking centres of the period. Moreover, in the vast majority of the samples iron was not detected, suggesting a careful selection and/or preparation of the raw materials. Manganese was detected in most samples in relatively high amounts, suggesting its deliberate addition as a decolourant. Manganese was used as a decolourant primarily until the 17th c., whereas later on the use of arsenic (on its own or combined with manganese) was more common (Verità, 2013). Therefore, we can assume that the majority of the glasses date before the 17th c.

It should be noted that all types of soda glass of the period have significant similarities in their composition, as virtually all have arisen from the constant interaction of the respective workshops (the Venetians were influenced by Islamic tradition and consequently influenced the glassmaking of northern/central Europe and Istanbul). In addition, common raw materials were often used, i.e. Venetian workshops importing sodium-rich plant ash from Egypt and Syria (Verità and Zacchin, 2007). A more detailed trace element analysis of the glasses is currently under way and will hopefully assist in identifying the likely provenance of the glasses.

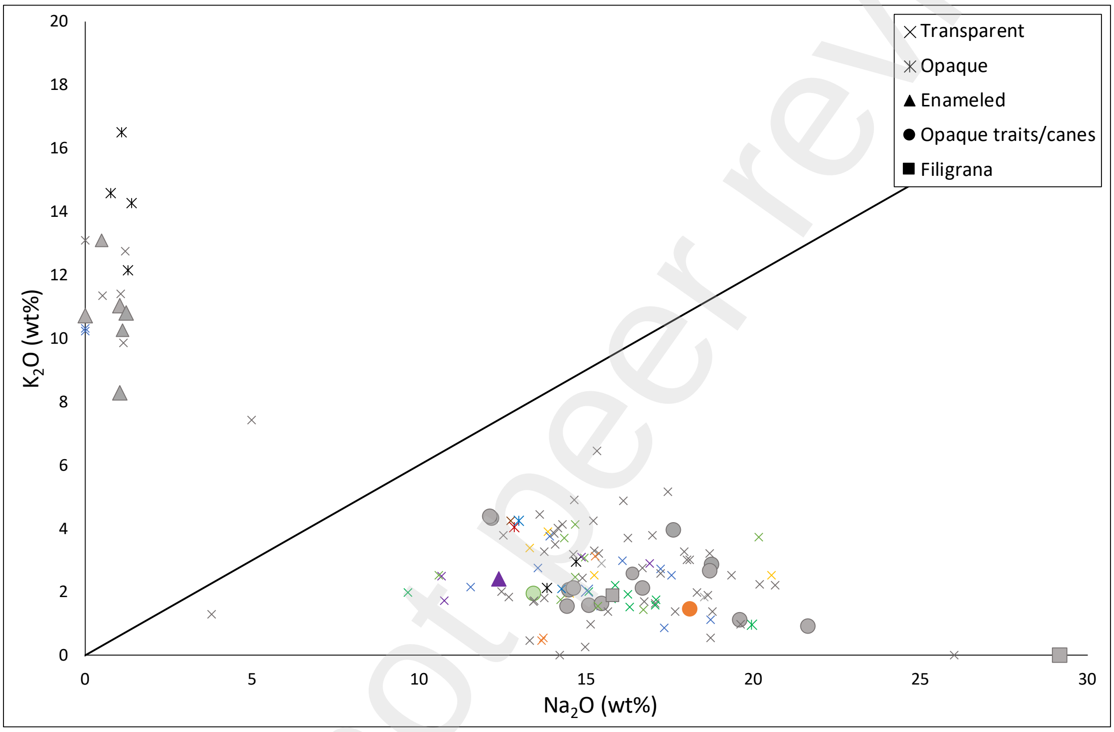

Figure 3. Concentration of Na_2O versus K_2O for the decorated glasses from the Palace of Mystras. The rest of the samples of the assemblage are shown for comparative reasons. The colour of each symbol corresponds to the colour of the sample (colourless glasses represented with the grey colour).

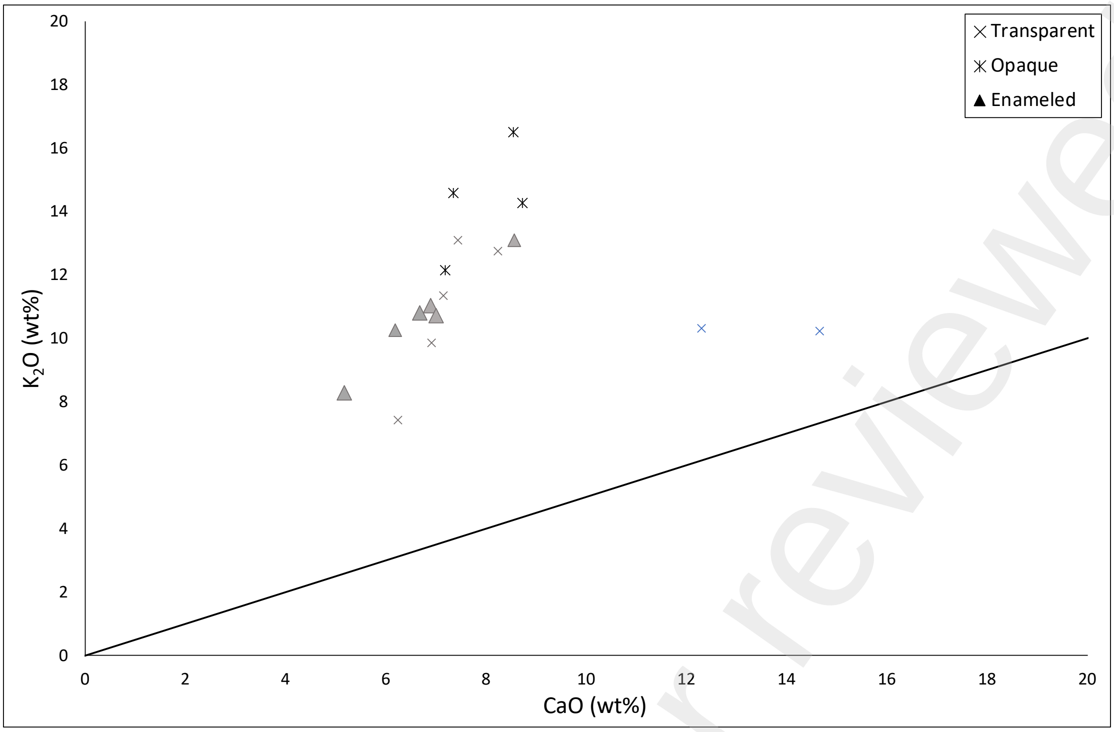

Figure 4. Concentration of CaO versus K_2O for the decorated potash glasses from the Palace of Mystras. The rest of the potash samples of the assemblage are shown for comparative reasons. The colour of each symbol corresponds to the colour of the sample (colourless glasses represented with the grey colour).

For the potash glass, the low concentration of magnesium and especially phosphorus excludes the use of plant ash as a source of alkali and suggests to the use of potassium nitrate (following the production technology of Bohemian glass). The low concentration of iron and the complete absence of manganese is an additional indication that the samples do not belong to the type of forest glass, as this glass contains relatively high concentrations of iron as a mixture in the raw materials, and manganese, which is added as a decolourant.

It should be highlighted that out of the overall 140 samples of the assemblage only 18 belong to the potash group; all of them share the same chemical traits, similar to the Bohemian technological tradition. Among this group, 6 are enameled with floral patterns and 4 are thin-walled white opaque glasses. Overall, it seems that this group of glasses was selectively imported in Mystras to cover specific stylistic preferences.

Figure 5 shows the concentration of the main oxides of the potash decorated samples from Mystras, compared to published data from other potash glasses: (1) Forest glass from various locations in Central and Northern Europe (Kuisma-Kursula et al. 1997; Wedepohl and Simon 2010; Schalm et al. 2007), (2) Bohemian glass and crystal (Mádl and Kunicki-Goldfinger 2006; Kunicki-Goldfinger et al. 2003), (3) Glass from Beykoz, Istanbul (Canav-Özgümüş 2012). It should be noted that the concentration of silicon for most samples of this group is particularly high (silicon concentrations above 75 wt% are not expected in pristine glass), which can be attributed to extensive chemical deterioration due to corrosion. This phenomenon is not surprising, as it is known that K-rich samples are the most vulnerable to chemical corrosion. Along with the increase in silicon, a decrease in potassium is expected during corrosion. Therefore, these two elements cannot be used as safe chemical markers when compared to other glass objects from the literature. Elements such as magnesium, phosphorus, aluminum, calcium, manganese and iron, are expected to show greater chemical stability, while they can also provide important information regarding the raw materials used and the preparation they have undergone.

Overall, the composition of the potash glasses from Mystras presents significant chemical similarities with the glass from Beykoz of Istanbul as well as the glass of Bohemia. Given the fact that the production of glass in Beykoz became significant in the early 19th c., when Mystras had already declined, this suggests that the latter origin is more probable.

Earlier examples of enameled glass found in Bohemia (12th-14th c.) were typically Na-rich and were considered imports primarily from Venice and Syria (Černá et al. 2012). After the invention of Bohemian glass, and especially during the 17th and 18th c., Bohemia became one of the most significant glass centres in Europe and a large exportation centre (Václav 1981). Given the significance of Mystras, it would not be surprising if bohemian glass was exported there, as part of the trade of luxurious objects. It should be noted that apart from one enameled glass that was recovered in the Bathhouses, all other samples were found in Building E, which was the administrative centre of the Palace complex.

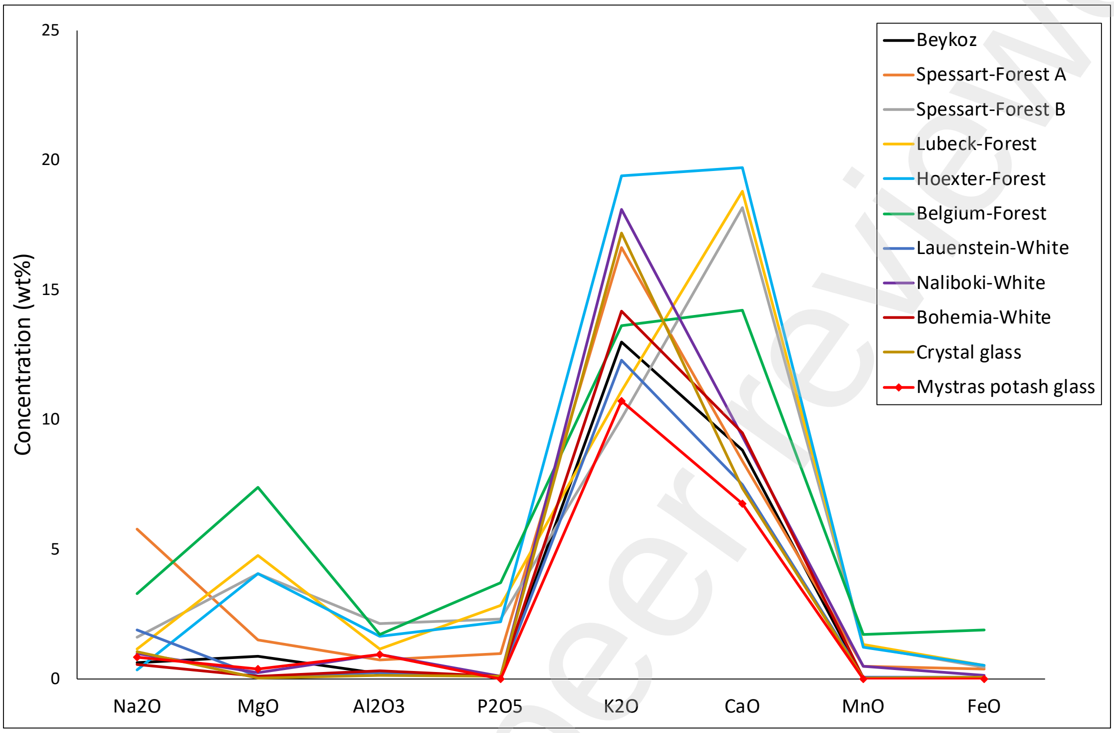

Figure 5. Major oxides concentration for the potash glasses of Mystras compared to bibliographic data.

<u>Analysis of enamels and opaque glass.</u>

In all the examined enamels the alkali used is primarily potassium, in accordance to the bulk glass of these samples. Thus, the enamels are clearly differentiated by both Islamic and Venetian examples of the same period, all of which are made using a soda-lime-silica glass (Freestone and Bimson 1995; Freestone and Stapleton 1998). Based on the SEM microscopical and chemical analysis and the Raman examination of undissolved particles (Table 1, Figure 6 and 7), the following observations can be made for each colour of enamel:

- The white enamel is composed mainly of silica, lead oxide and tin oxide. Its microstructure is characterised by the large number of small cassiterite grains (a few μm wide), which are responsible for its opacity. Compared to Islamic and Venetian enamels, the tin content is here higher and the total alkali content is lower; more importantly, calcium is present in very low amounts (<1 wt%).
- The red enamel presents fewer undissolved particles of larger size, which are usually characterised as haematite grains. In some cases, a more complex Raman spectrum resulted, including bands attributed to haematite, cassiterite and lead compounds (the latter at 980 and 1040 cm^{-1} in the form of a lead-silicate glass - e.g. Palamara et al., 2017, Palamara et al. 2016). Its overall composition is similar to that of low-lead Islamic red enamels (apart from the alkali source) (Freestone and Stapleton 1998), although here the iron content is higher (approx. 8 wt%).
- The blue enamel is characterised by a significantly different composition compared to Islamic/Venetian enamels, with higher amounts of tin and lead, almost no calcium and low total alkali content. The overall composition is very similar to that of the white enamels of the samples under study. Cobalt, which is the most common colourant for blue enamels of the period was not detected, though that could be a result of the relatively high detection limit of the SEM/EDS.

- The yellow enamel presents a unique composition compared to other yellow enamels of the period. The base of the enamel appears to be a lead-silica glass containing tin oxide. However, the colour is attributed to the presence of Naples yellow (undissolved particles can be seen with a size <2 μm). Additionally, the microstructure of the enamel is characterised by the presence of quartz crystals, up to 20 μm wide, which may have been added to increase the opacity.

Table 1 Composition of major elements of the enamels estimated by SEM/EDS. The mean group values are given as wt% (normalized to 100%) (nd: not detected).

Sample	Colour	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	MnO	FeO	SnO_2	Sb_2O_5	PbO
MY4	Yellow	nd	nd	3.03	30.82	2.76	0.88	nd	1.96	7.13	10.53	43.60
MY17	Yellow	0.67	0.62	2.28	31.67	1.85	nd	nd	1.23	12.69	6.52	42.79
MY16	Blue	3.92	nd	1.18	48.50	2.52	0.55	nd	nd	20.21	nd	22.37
MY18	Red	1.37	0.84	0.82	68.72	8.97	6.81	nd	7.98	nd	nd	4.50
MY18	White	2.68	nd	1.31	45.15	2.37	0.84	nd	nd	23.84	nd	23.81

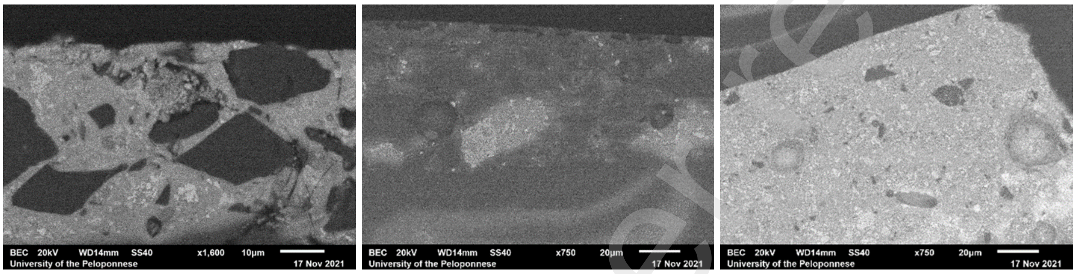

Figure 6. Representative SEM images of a yellow (left), red (middle) and white (right) enamel.

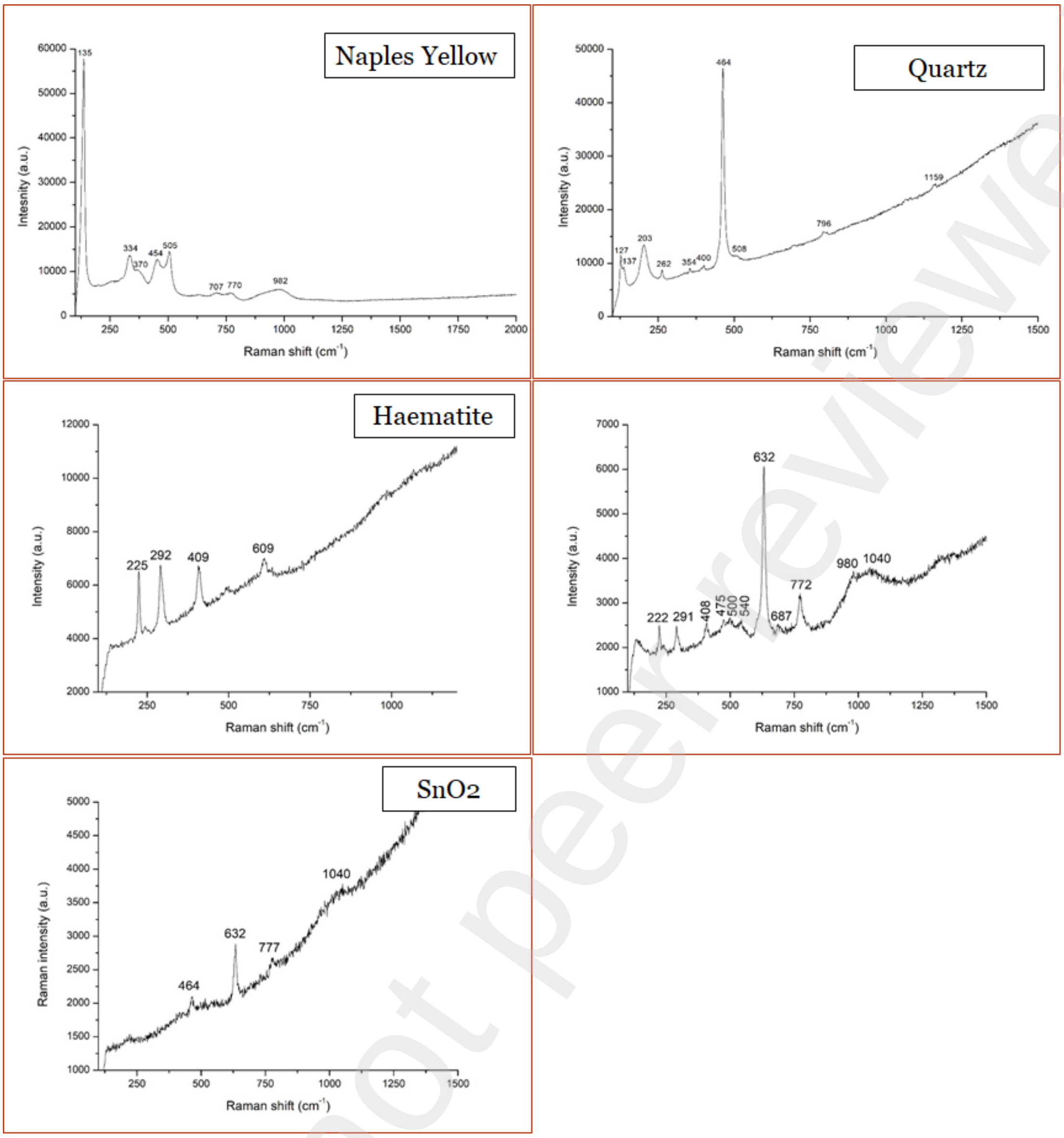

Figure 7. Representative Raman spectra of undissolved particles: Top: a yellow enamel (Naples yellow and quartz); Middle: a red enamel (haematite and composite spectrum with bands attributed to haematite at 221, 291 and 208 cm^{-1}, tin oxide at 475, 632 and 772 cm^{-1} and a lead-silicate glass at 980 and 1040 cm^{-1}); Bottom: a white enamel (tin oxide).

The opaque glass used to form the decorative traits and canes is Na-rich, composed mainly of silica, lead oxide (varying between 7.5 and 21.5 wt%) and tin oxide (varying between 9 and 23.5 wt%) (Table 2). Its microstructure is similar to that of white enamel, with a large number of small cassiterite grains (a few µm wide), which are responsible for its opacity (Figure 8). The white decorative cane of sample MY22, the one sample presenting a singularly high amount of sodium, presents a unique composition, again characterised by the high amount of sodium. In the EDS analyses of the glass matrix tin and lead were absent. However, undissolved particles of tin oxide have been identified, although they are present in significantly smaller amounts compared to the other white samples.

Finally, the blue decorative layers, are composed mainly of silica, lead oxide and tin oxide. As in the case of the blue enamels, no colouring agent was detected, probably due to the relatively high detection limits of the applied technique.

Table 2. Composition of major elements of the glass forming the decorative layers estimated by SEM/EDS. The mean group values are given as wt% (normalized to 100%) (nd: not detected).

Sample	Colour	Na_2O	MgO	Al_2O_3	SiO_2	K2O	CaO	FeO	SnO_2	PbO

MY22	White	31.43	8.33	nd	60.24	nd	nd	nd	nd	nd
MY61	White	14.24	1.88	0.81	46.58	0.80	2.29	nd	12.98	19.35
MY84	White	16.90	1.68	1.14	48.56	1.38	1.38	nd	10.01	18.15
MY88	White	10.85	1.03	0.67	44.69	1.48	2.77	nd	19.74	17.90
MY89	White	14.75	4.00	1.48	54.81	1.76	6.13	nd	9.14	7.45
MY93	White	10.02	1.16	0.24	42.55	0.95	2.57	nd	22.30	19.24
MY133	White	11.28	1.86	0.51	39.91	1.06	3.17	0.82	20.22	20.66
MY133	Blue	10.91	2.17	0.49	37.85	0.71	2.50	nd	23.30	21.24

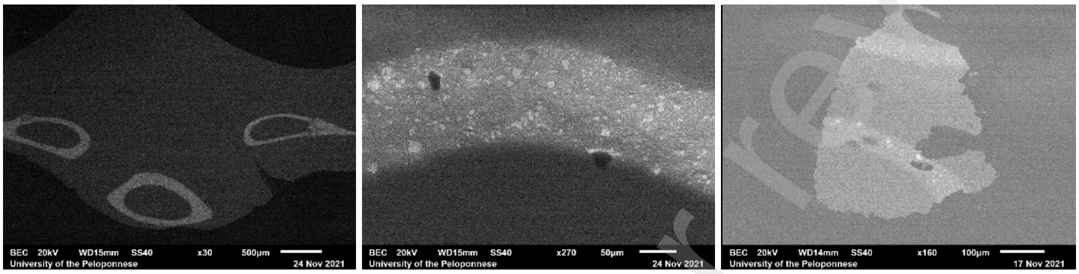

Figure 8. Left and middle: Representative SEM images of the white decorative layers (sample MY133). Right: SEM image of sample MY22 (high-Na sample)

Conclusions

The present study focuses on the microscopic and chemical analysis of 26 samples, enameled or decorated with traits/canes, recovered from the Palace of Mystras. The analysis led to the identification of raw materials, the grouping of samples into broader chemical types and a preliminary evaluation of their likely provenance. Despite the significance of Mystras, as the administrative centre of the broader region and a city with continuous political and commercial ties both to the East and the West, this is the first archaeometric study of glass artefacts from the region.

The examination of the glass body resulted in the distinction between two groups: the majority of the samples belong to the category of soda glass, while the 6 enameled samples belong to the category of potash glass. The provenance of the soda glass cannot be determined based on the composition of the major elements. The samples seem to follow the same manufacturing process but are likely produced in different workshops, based on the relative heterogeneity of their composition. This hypothesis is further supported by the composition of the opaque glass used for the decorative canes. The presence of manganese as a decolourant in the majority of the samples, suggests that they probably date before the 17th c.

The composition of the enameled glass suggests that they likely originate from Bohemia. The examination of the enamels further corroborates the likely provenance from Bohemia, since the enamels are produced using K-rich glass and with distinct manufacturing process than the Islamic and Venetian enamels (as shown with the use of Naples yellow for the yellow enamels). Overall, it seems that this group of glasses was selectively imported to Mystras to cover specific stylistic preferences. Additionally, the recovery of the samples from Building E, the administrative centre of the Palace Complex, suggests that the artefacts were traded to high ranking members of the Palace as valuable objects.

The present study contributes to the acquisition of new knowledge about the glass and the glass centres of southern Greece. Further analyses will enhance our understanding the glass manufacture and trade and will provide with significant insights into the commercial, artistic and technological interactions of Mystras with the Ottoman Empire and Europe.

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Appendix

Composition of major elements of the glass samples estimated by SEM/EDS. The mean group values are given as wt% (normalized to 100%) (nd: not detected).

Sample	Colour	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	MnO	FeO
MY1	Colourless	12.17	3.41	1.16	70.09	nd	0.81	4.34	7.23	nd	0.81	nd
MY4	Colourless	1.13	0.75	nd	81.69	nd	nd	10.25	6.19	nd	nd	nd
MY7	Light green	13.41	5.16	1.97	64.50	1.05	0.86	1.96	9.53	nd	0.97	nd
MY16	Colourless	1.05	nd	0.94	84.55	nd	nd	8.29	5.18	nd	0.00	nd
MY17	Colourless	1.03	0.89	nd	80.15	nd	nd	11.02	6.89	nd	0.00	nd
MY18	Colourless	0.00	nd	nd	82.28	nd	nd	10.71	7.00	nd	0.00	nd
MY22	Colourless	29.18	6.56	nd	64.27	nd	nd	nd	nd	nd	0.00	nd
MY26	Colourless	12.12	2.98	2.03	68.95	nd	0.66	4.41	8.10	nd	0.74	nd
MY37	Colourless	1.23	nd	nd	80.92	nd	0.36	10.81	6.68	nd	0.00	nd
MY47	Colourless	14.47	3.67	1.59	67.99	nd	0.71	2.08	8.47	nd	1.03	nd
MY52	Colourless	17.62	3.46	0.67	65.67	nd	1.08	3.97	7.53	nd	nd	nd
MY55	Colourless	18.75	2.24	1.54	66.97	1.18	1.04	2.87	5.41	nd	nd	nd
MY61	Colourless	19.58	1.67	1.49	71.47	nd	1.03	1.12	3.08	nd	0.55	nd
MY63	Amber	18.10	4.35	1.34	68.14	nd	0.94	1.47	5.67	nd	nd	nd
MY84	Colourless	18.71	4.78	0.95	66.98	nd	0.78	2.69	5.11	nd	nd	nd
MY88	Colourless	14.42	1.93	0.93	72.83	nd	1.25	1.57	5.79	nd	0.82	0.46
MY89	Colourless	14.62	4.39	1.32	66.43	nd	0.80	2.13	8.75	nd	0.91	1.00

MY93	Colourless	15.08	2.82	1.02	70.52	nd	1.10	1.60	5.96	nd	1.40	0.50
MY97	Colourless	16.67	3.13	1.03	67.79	nd	0.56	2.13	7.54	nd	1.16	nd
MY107	Colourless	21.64	2.67	1.39	69.60	nd	0.67	0.92	3.11	nd	nd	nd
MY108	Colourless	15.47	2.00	1.33	72.52	nd	0.78	1.64	5.85	nd	0.41	nd
MY110	Purple	12.37	3.92	1.60	69.40	nd	0.95	2.43	6.87	nd	2.47	nd
MY122	Colourless	16.42	3.74	1.18	66.75	nd	1.29	2.57	8.06	nd	nd	nd
MY133	Colourless	15.82	2.11	0.98	70.50	nd	0.80	1.89	6.56	nd	0.63	nd
MY138	Colourless	0.50	0.74	nd	77.10	nd	nd	13.09	8.56	nd	nd	nd