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Composition of Late Hellenistic to Early Roman glass vessels from the Souk Excavations, Beirut

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Abstract
Sixty-six glass vessels from excavations in Beirut and dated first century B.C.E. to the first century C.E. have been analysed by electron microprobe. The majority are relatively high in Al_2O_3, CaO and P_2O_5, are weakly coloured, manganese-colourless, or yellow–brown-amber and on compositional grounds the glass material is considered to have originated in the Levant. Manganese oxide was added as a decolouriser and MnO contents are continuous between 0.02 and 2.0%. Limpid, weakly-coloured glass occurs over the whole MnO range, while most colourless glass has MnO above 0.7%, and all amber and olive glasses have MnO below 0.3%. There is a strong correlation between sulphur and soda concentrations in all the Levantine glass, but total sulphur is lowest in amber and olive, reflecting the reducing conditions required to form the ferri-sulphide chromophore and the lower solubility of the S^{2-} ion as opposed to SO_4^{2-}. Iron is also low in the amber glass relative to other colours, as some Fe was added with the manganese that they contain. Hence, amber glasses were produced at the primary stage from mixtures of natron and sand with no other additives apart from any organic reducing agents. In the second half of the first century C.E. slumped bowls in antimony-decolourised Egyptian glass become apparent, along with colourless cast vessels with mixed antimony-manganese compositions. Antimony is known to have been used as a decolouriser in earlier Hellenistic glass, but it does not appear in the present assemblage until this later introduction of Egyptian glass. The introduction of glass-blowing technology does not seem to have coincided with any significant change in composition.

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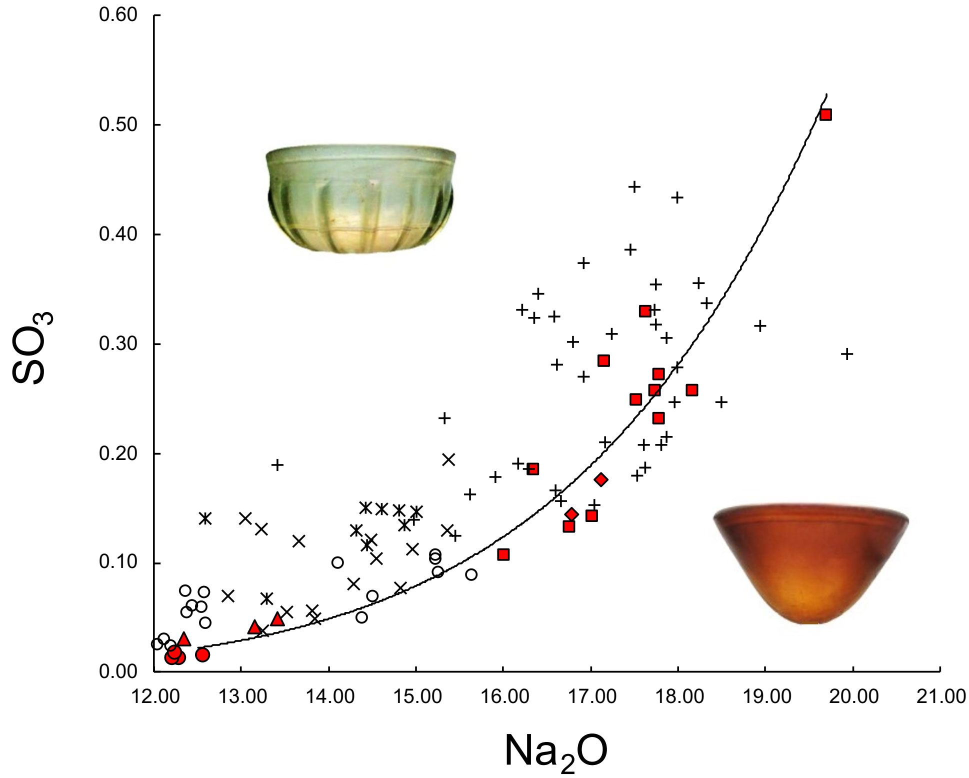
Graphical abstract



Keywords Levantine glass · Glass production · Roman · Hellenistic · Levant · Manganese · Sulphur · Amber glass

1 Introduction

By the middle of the first millennium B.C.E., raw glass was being manufactured from sand and natron in large primary tank furnaces located in the littoral regions of the eastern Mediterranean. The resulting raw glass slabs weighed in the order of 10 tonnes and were broken up and distributed as chunks to be remelted and fabricated into objects across the Mediterranean and Europe (Foy 2017; Ganio et al. 2012a, b). The locations of the primary production centres moved from time to time within the region, but it appears that in any one period raw glass was being produced in both the Levant and Egypt (Freestone 2021). Compositional groupings of the glass reflect the sands exploited at the various production locations.

It has been suggested that later in the first millennium C.E., primary glassmakers in Egypt and the Levant were in competition (Freestone et al. 2015; 2018), but the early development of these glass industries is not fully understood. While analyses of glass from the late Hellenistic and Early Roman periods have been published, they typically present a monotonous compositional assemblage with limited variation (Thirion-Merle 2005; Reade and Privat 2016) and it is, therefore, challenging to unravel patterns of production and distribution. Translucent glass at this time falls into a range of colours, largely controlled either by the addition of transition metals such as copper and cobalt, or the oxidation state of the iron naturally present in the glassmaking sand (Arletti et al. 2013; Möncke et al. 2014; Schreurs and Brill 1984). However, how the glassmakers exerted control over the iron-based colours is not well understood.

Excavations in the Souks area of Beirut, Lebanon (sectors BEY 006, 007 and 045), revealed large amounts of glass artefacts from the Hellenistic to the Islamic period (Jennings 2006). In this study, we investigate the major elemental composition of 66 mid-second century B.C.E–early second century C.E. glass vessels from Beirut and to evaluate the origins of the glass, to provide information on colouration and decolourisation technologies and to investigate any compositional changes in the transition from casting to blowing techniques.

2 Materials and methods

Samples were selected and provided by the late Dr. Sarah Jennings, as part of her programme of research on the glass from the site. They were dated typologically and stratigraphically, and assigned to typo-chronological groups as given in Table 1. The great majority date to the first centuries B.C.E. and C.E. The vessels analysed are all monochrome; they fall into several colour categories: colourless, weakly coloured (including pale greens, blues and weak yellows), yellow-amber, olive green and purple. As pointed out by Jennings (2006), "colourless" is a subjective definition and some of the "colourless" vessels have a very weak tint. However, as will be seen, Jennings's observations are reasonably consistent with the new analyses. Table 1 provides chronological details from the notes provided by Jennings with the samples. It should be noted that the region was annexed by the Roman general Pompey in 64 B.C.E.; prior to this, the glass was considered late Hellenistic. Attention is drawn in particular to the fact that the great majority of the vessels analysed are hemispherical bowls made by the "slumping" method, which involved the production of a glass disc which was heated over a hemispherical former and slowly slumped to form a bowl. The traditional term "cast", although used in Table 1 to remain true to the descriptions provided by Sarah Jennings, is considered misleading. Only a small number of blown vessels were analysed and these were recovered from the so-called "Cistern Group", recovered from a closed context dated 60–70 CE. More details with the unique DNRO identifier for each fragment (Jennings 2006) are provided in the Supplementary material.

Samples were mounted in epoxy resin, polished flat to 1 µm diamond and vacuum coated with a thin layer of carbon. They were analysed by electron probe microanalysis (EPMA) in the Wolfson Archaeological Science Laboratories, UCL. A JEOL JXA 8100 microprobe with three wavelength-dispersive spectrometers was operated at 15 kV accelerating potential, beam current 50 nA, working distance of 10 mm and rastered at a magnification of x800 to minimise sodium loss. X-rays were collected for 30 s on peak and 10 s on each background. Standards were pure elements, oxides and minerals of known composition. Seven areas were analysed on each sample and the means are presented here. Corning Museum Ancient Glass Standards A, B and C (Brill 1999; Vicenzi et al. 2002) were measured during the same analytical run, and the results compare well with the given values (Supplementary Table 1).

3 Results

Full results are given in Supplementary Table 2, means of selected categories are presented in Table 2. All glasses are soda-lime-silica in composition, with less than one percent each of K_2O and MgO, indicating the use of natron as a flux. The samples fall into two main compositional groups. The main group, comprising the great majority of the samples, contains all colours including yellow-amber, strong blue, purple and colourless glass. It has relatively high Al_2O_3, CaO and P_2O_5, but low Na_2O. Samples from this group frequently contain manganese above 0.02–0.03%, which is the background level found in the glassmaking sand (Brems and Degryse 2014; Schibille et al. 2017; Brems et al. 2018), while antimony is consistently below measurement limits of about 0.02%. This group corresponds closely to much of the late Hellenistic and early Roman glass from elsewhere in the eastern Mediterranean and Levant, for example, late second-early first century B.C.E. Jebel Khalid, Jordan (Reade and Privat 2016), and also to manganese-decolourised glass widely distributed in the Roman period (e.g. Silvestri 2008), as well as other late Hellenistic and early Roman glass from Beirut (Thirion-Merle 2005; Paynter 2006). Its origin is generally considered to be on the Levantine coast (Ganio et al. 2012a, b; Schibille et al. op. cit.; Thirion-Merle op. cit.,) and it differs only in minor respects (slightly higher Na_2O, lower Al_2O_3) from the glass found in later primary production centres at Jalame and Apollonia (Freestone et al. 2023; Tal et al. 2004). No evidence for the use of antimony as a decolouriser was found in the Levantine glass analysed here,

Table 1 Summary of numeration, chronology and typology of the analysed vessels as provided by S. Jennings

Group/sample no.	Dating	Vessel type
1/1–1/5	Late Hellenistic	Conical cast bowls
1/10–1/19	Late Hellenistic	Hemispherical cast bowls
1/20–1/29	Late 1st Cent B.C.E-100 C.E.	Cast ribbed bowls
2/1–2/7	Early Roman	Late plain cast bowls
2/10–2/19	Early Roman	Linear-cut cast bowls
2/20–2/27	2nd half of 1st Cent C.E.	Late colourless cast vessels
3/1–3/6	Cistern Group 60–70 C.E.	Cast ribbed bowls and late plain bowls
3/7–3/16	Cistern Group 60–70 C.E.	Blown vessels

Table 2 Mean compositions of selected colours: antimony-colourless, mixed antimony-manganese colourless, manganese colourless, weak colours, amber-yellow and cobalt blue. Also analysed: Zn and Sn, below detection in all cases

	Group 2	Group 3	Group 1			
	Sb-cllss	Sb-Mn	Mn-cllss	Weak cols	Amber	Co-Blue
n	*4*	*4*	*10*	*30*	*12*	*3*
SiO_2	70.07	69.59	68.44	67.55	68.06	67.65
Na_2O	18.55	17.10	16.46	17.27	17.47	16.64
CaO	6.00	7.36	8.31	8.17	8.17	8.48
K_2O	0.43	0.62	0.66	0.62	0.68	0.67
MgO	0.52	0.59	0.55	0.54	0.56	0.51
Al_2O_3	1.93	2.28	2.40	2.45	2.43	2.55
Fe_2O_3	0.41	0.36	0.35	0.36	0.32	1.34
TiO_2	0.07	0.05	0.06	0.05	0.05	0.04
Sb_2O_5	0.64	0.26	< 0.02	< 0.02	< 0.02	< 0.02
MnO	0.02	0.66	0.90	0.72	0.06	0.40
CuO	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	0.07
CoO	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	0.04
PbO	0.07	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
P_2O_5	0.00	0.01	0.04	0.06	0.05	0.06
Cl	1.19	0.97	0.87	0.99	1.00	0.84
SO_3	0.25	0.21	0.28	0.26	0.25	0.29
Total	100.17	100.10	99.37	99.09	99.15	99.63

although the technique is known from the Hellenistic period (Oikonomou et al. 2020).

A second group comprises four colourless cast vessels dated to the second half of the first century C.E. They are decolourised with antimony, with lower CaO, Al_2O_3 and P_2O_5 but higher Na_2O (Fig. 1). This group corresponds to the antimony-decolorised glass of the Roman period, considered to have originated in Egypt (Barfod et al. 2020; Gliozzo 2017). They typically have higher TiO_2/Al_2O_3 ratios than the manganese-decolorised group (Fig. 2), and high TiO_2 is widely accepted as an indicator of Egyptian origin for glass of this period (Foy et al. 2003). The high soda levels of c.

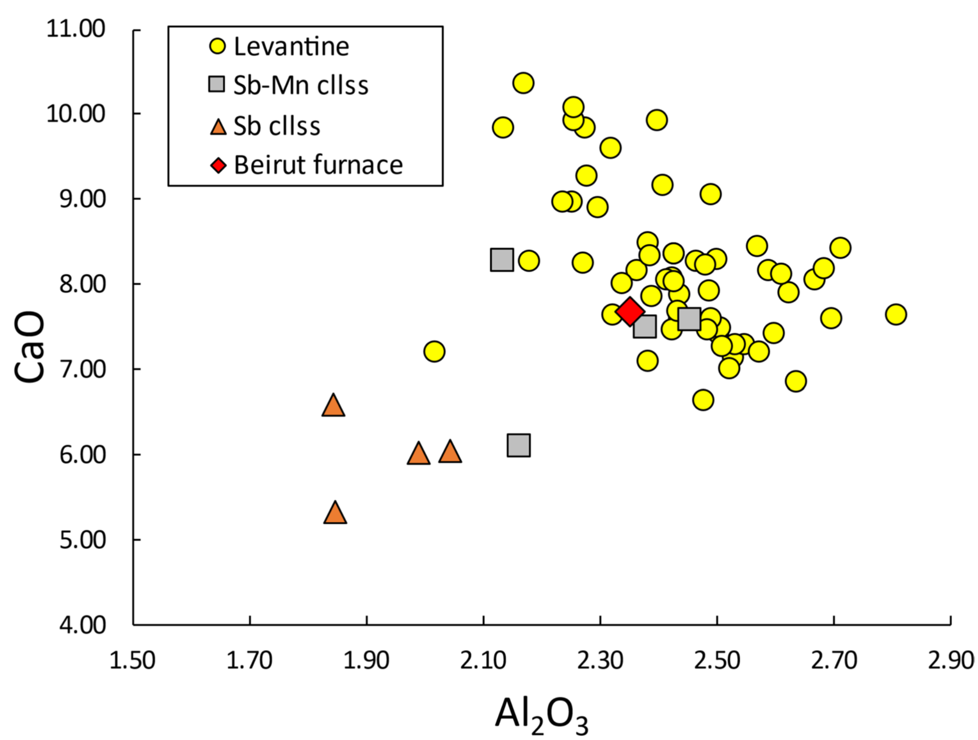


Fig. 1 Weight percent lime and alumina concentrations in the analysed glasses. The diamond indicates the composition of glass from a furnace in Beirut (Henderson 2013: 93)

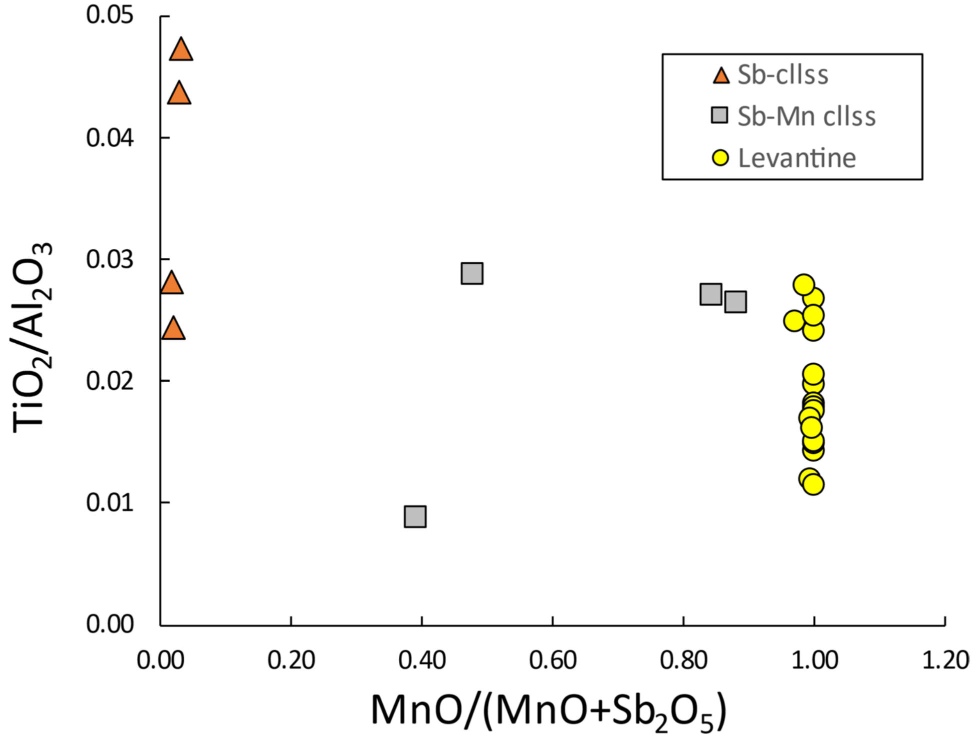


Fig. 2 Compositions of early Roman glasses in terms of their titanium/aluminium oxide ratios versus manganese to antimony, showing the higher TiO_2/Al_2O_3 ratios in the Sb-decolourised glass

18.5% relative to the lower levels in the Levantine group (mean c. 17%) also support the view that the glass material was made closer to the natron deposits of Egypt (Freestone 2021).

A third group of four colourless samples contains both antimony and manganese and is compositionally intermediate to the others (Figs. 1, 2; Table 2). These are generally considered to be a mixture of antimony- and manganese-decolourised glasses generated during the recycling of waste vessel glass (Freestone 2015; Jackson and Paynter 2016; Silvestri 2008).

4 Discussion

4.1 Local production

Evidence for tank furnaces, believed to have been engaged in the production of raw glass and dating to the early first century C.E. are known from elsewhere in Beirut (BEY 015: Kouwatli et al. 2008; Henderson 2013). They produced translucent glass of a range of colours. An analysis of the glass from the Beirut primary furnace complex provided by Henderson (2013:93) corresponds to the Levantine group, as might be expected (Fig. 1).

Glass vessels from Beirut and of about the same period as those analysed here have also been analysed by Thirion-Merle (2005) and Paynter (2006) and our results are broadly comparable. Evidence of the production of glass vessels, particularly the earlier linear-cut and ribbed bowls in deep yellow/amber, has been found elsewhere in the city (BEY 002: Foy 2005), although there is no evidence for the production of the forms post-dating the middle of the first century C.E. It seems likely that the locally fabricated vessels were made from the locally produced primary glass.

4.2 Colour in Levantine glass

The Levantine glass shows a range of manganese contents from background (c. 0.02%) up to around 2% MnO (Fig. 3). Colourless glass typically has MnO > 0.7% but colourless glass may be formed at lower MnO concentrations, and two of the colourless bowls from Beirut have 0.17% and 0.44% MnO. Only a single example of purple glass was analysed, which has high MnO at 1.51%, reflecting the very oxidising conditions needed to produce the Mn^{3+} ion. Weakly tinted blue, green and brown shades occur across the full range of MnO (Fig. 3). Deep blue glasses coloured by cobalt additions (c. 0.04% CoO) also have elevated Fe_2O_3 (> 0.8%) and CuO (> 0.05), reflecting the character of the cobalt ore used.

Comparing the relationship between colour and MnO content in the present dataset with that of glass from the fourth-century production site at Jalame (Freestone et al. 2023) it appears that while the general pattern is comparable, in the Beirut glasses the ranges of MnO producing each colour tend to be larger. Hence at Jalame colourless glass occurs in the range MnO = 0.7–1.7% and very few samples in this range are recorded as weakly coloured. For Beirut

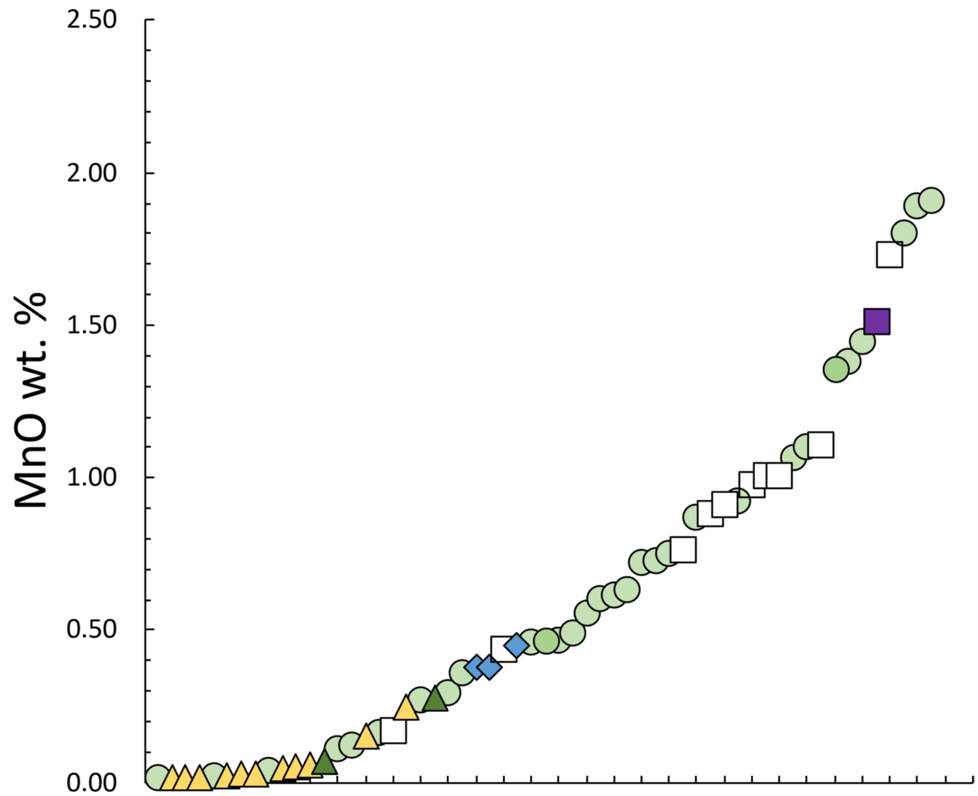


Fig. 3 Manganese contents of Levantine glasses analysed, labelled according to colour. Yellow triangles = amber; dark green triangles = olive; pale green circles = weak colours; blue diamonds = cobalt blue; white squares = colourless; purple square = purple (Color figure online)

however, colourless glass *mostly* occurs in the range of 0.7–1.7% MnO but can have as little as 0.17% MnO. In the Beirut glass, weakly coloured glass occurs throughout this range (Fig. 3) but at Jalame, is mainly confined to MnO concentrations below 0.7%. The wider spectrum of colours associated with a particular concentration range of MnO in the Beirut assemblage may be because of the longer time-span encompassing the production of the Beirut glasses; production at Jalame was more limited. Caution is needed here because the analytical samples in both studies were selected in part on the basis of colour; they cannot be considered as representative of the true distribution of glass compositions on either site.

Twelve vessels described as brown, yellow or amber in colour have low MnO, typically at background level (< 0.03%) but with three samples which have 0.06–0.25% MnO. Two olive green vessels also have low MnO (Fig. 3; Suppl. Table 2). Generation of amber and olive colours required particularly reducing conditions in the glass melt, to form the amber ferri-sulphide complex responsible for the colour (Schreurs and Brill 1984; Arletti et al. 2013). Under slightly less reducing conditions, the weaker amber combines with the blue tint of ferrous iron to give olive (Schreurs and Brill op. cit.). For this reason, amber and olive-green glasses usually have low Mn and Sb, which were added to oxidise the iron in glass and modify the blue colour imparted by Fe^{2+} ions (Sayre 1963). Amber vessels are likely to reflect the use of glass derived directly from a primary glassmaking furnace, rather than a secondary workshop, where they would have tended to oxidise in repeated remeltings (Freestone and Stapleton 2015; Paynter and Jackson 2018).

The measurement of sulphur by electron microprobe allows the technology to be explored in some detail. It should be noted that in the following analysis, it has been assumed that the amber, yellow, yellow–brown and olive colours are due to the ferri-sulphide complex, this has not been confirmed by spectroscopy.

Figure 4 shows EPMA data for the Beirut glass plotted with data for amber and other glass colours from other Levantine assemblages, measured in the same laboratory. There is a strong correlation between Na_2O and (total sulphur as) SO_3, reflecting the likely formation of sodium sulphate complexes in the molten glass. Given that amber is a sulphur-based colour, the observation (Fig. 4) that SO_3 tends to be lower in the amber and olive glasses than the weakly-coloured and colourless glasses across the full range of soda concentrations may seem counter-intuitive. However, it is a reflection of the speciation of the sulphur ions in the melt. X-ray absorption studies on sulphur in silicate glasses indicate that it can be present as two species, SO_4^{2-} and S^{2-} (e.g. Beerkens 1999; Nash et al. 2019; Vaishnav et al. 2020). The strong correlation observed between SO_3 and Na_2O in Fig. 3 is therefore likely to relate to a control of the

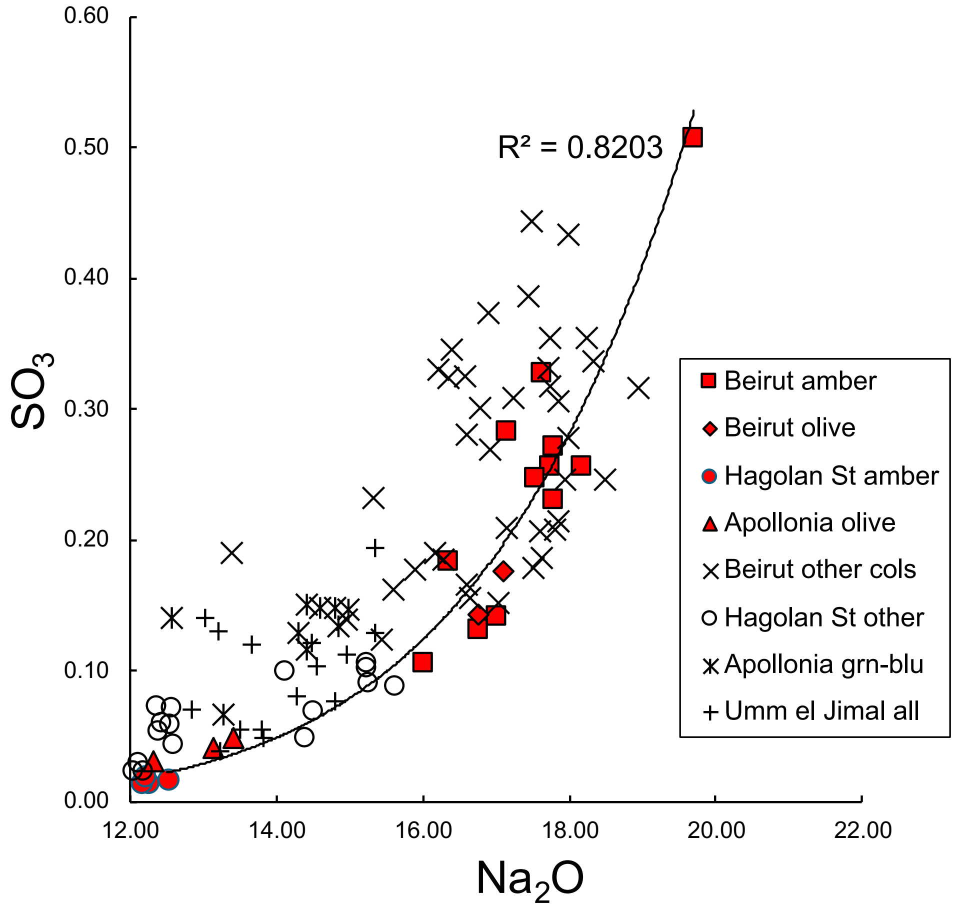


Fig. 4 Total sulphur as SO_3 versus soda for Levantine-type glass from Beirut and other sites analysed by EPMA in the UCL labs. Filled red symbols are amber and olive glasses where the colour is likely to be due to the ferri-sulphide chromophore. Other symbols are colourless and weakly coloured glass. The curve is a fit to the Beirut amber glasses. Additional data: Hagolan Street (Freestone et al. 2015), Apollonia (Tal et al. 2004), Umm el-Jimal (Al-Bashaireh et al. 2016) (Color figure online)

solubility of sulphur in the glass melt through the formation of complexes involving both sulphur and sodium. When the glass is reduced, as in the formation of the ferri-sulphide amber colour, more of the sulphur will be present as sulphide. However, sulphide appears less soluble in soda-lime-silica glass than sulphate, so maximum sulphur solubility of total sulphur species is attained in oxidised melts (Beerkens 1999). Hence the glasses showing reduced colours in Fig. 4 have lower total sulphur (expressed as SO_3) contents than the oxidised glasses. The power curve fitted to, and extrapolated from, the Beirut amber glasses in Fig. 4 is effectively delineating the solubility of sulphur in reduced natron glass. More oxidised compositions lie above the curve.

The significance of this finding is to confirm that a sulphur-bearing material was not added to the glass batch to generate an amber colour, but that the sulphur was intrinsic to the glass (Paynter 2006; Freestone and Stapleton 2015; Paynter and Jackson 2018), added in the form of sodium sulphates in the natron flux (Shortland 2004). It demonstrates that sulphur contents measured in early glass are meaningful, in that they in part reflect the redox conditions in the melt. Further, the iron contents of the glasses are, if anything, *lower* in the amber glasses than in the more oxidised colours (Fig. 5). Fe_2O_3 tends to be higher in the glasses with higher MnO, as a small amount of iron was added as a component of the manganese ore. While amber colour occurs in some glass with low concentrations of manganese (up to 0.25%) it is not possible to determine from the present data whether these were produced in a primary furnace in which the manganese distribution was heterogeneous, as proposed by Freestone et al. (2023), or in a secondary furnace due to the recycling of old manganese-containing glass into a manganese-free amber batch. Furthermore, it is still unclear as to whether amber glasses were an incidental consequence of uncontrolled variations in melting conditions or batch composition (e.g. the presence of more organic matter causing reduction) or the result of a deliberately induced reduction by adding charcoal or some other carbon-rich component.

4.3 Egyptian glass

As anticipated, the data indicate that manganese-decolourised Levantine glass was worked and probably made in Beirut in the late Hellenistic period. Antimony-decolorised glass from Egypt is first detected in the second half of the first century C.E. The absence of Sb-decolourised glass from either the slumped or the blown vessels in the cistern group assemblage indicates

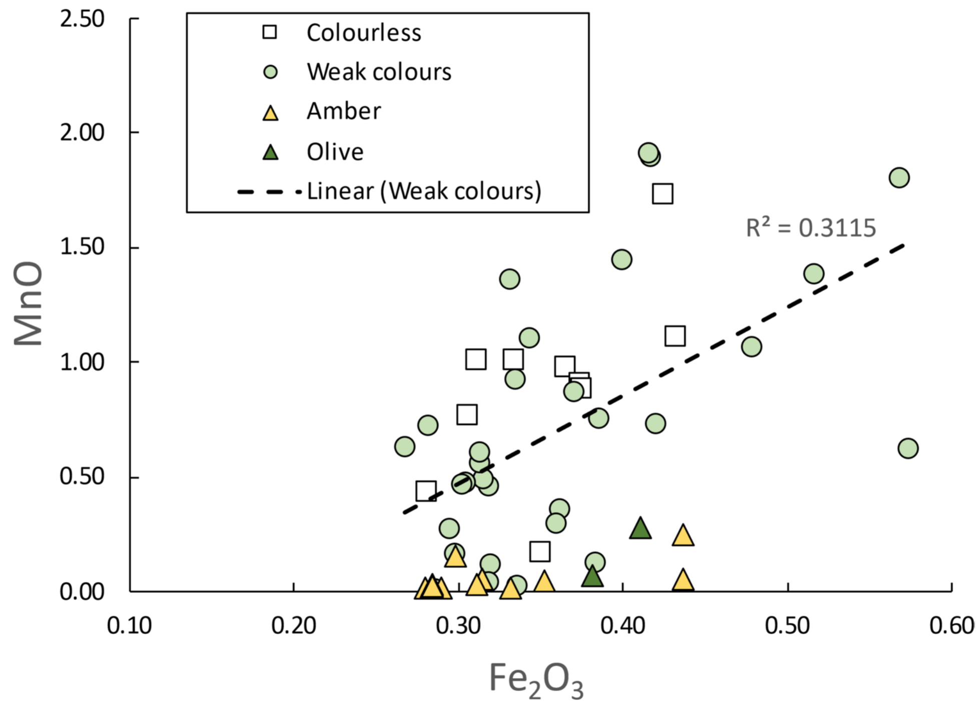


Fig. 5 Manganese versus iron oxides for various colours of Levantine glass, showing that iron tends to increase with manganese and that most amber glasses typically have relatively low Fe_2O_3

that it was not introduced before 60 C.E. This is consistent with recent work on the glass from Pompeii by Boschetti et al (2024), where Egyptian Sb-glass (before 79 C.E.) is rare. Furthermore, it seems from the present study that soon after Sb-decolourised glass production began in Egypt, glass vessels made from Egyptian colourless glass were in use in the Levant, in spite of the existing strong local production of manganese colourless glass. There was a high and growing demand for glass tableware in this period, reflected for example by the progressive increase in cast bowls in Beirut (Jennings 2006; Larson 2019) and this would have encouraged the procurement of glass, and particularly of the highly valued colourless glass, from non-local sources.

At about the same time as Egyptian glass appears in our sample, antimony-decolourised and manganese-decolourised glasses were mixed, melted and used to form colourless glass vessels. It seems likely that these Mn-Sb compositions are due to the mixing of broken vessels in a recycling process, as there is evidence of recycling going back into the earlier Hellenistic period (Smirniou et al. 2018). However, it is possible that the mixed compositions could be the result of mixing raw chunks of Egyptian and Levantine glass. It may be relevant that the resulting mixed vessels are also colourless, and have not developed tints due to reduction, unlike many recycled Roman glasses, which is consistent with the idea that the glasses being mixed were relatively pristine and had not previously been extensively recycled.

It is unclear where the bowls in Egyptian colourless glass were made. Grose (1986) emphasised the growth of slumped vessel production in Italy in the first century. The Italian industry is likely to have imported both Egyptian and Levantine glass and, therefore, Italy may have been where Egyptian antimony-decolourised and Levantine manganese-decolourised glasses were mixed, suggesting that these vessels were imported from Italy, but manufacture in the Levant or Egypt are possible alternatives and cannot be dismissed at this stage as the extent to which raw glass was imported from one region into the other is not well understood.

4.4 Glass blowing

The inflation of glass by blowing was introduced sometime in the second half of the first century B.C.E. but does not seem to have been taken up as rapidly in the East as in the West (Jennings op. cit.). The earliest blown vessels we have analysed in the present study are from the cistern group dated 60–70 C.E. Their compositions appear typical of the slumped Levantine glass analysed and there appears to be no significant change in glass composition with the change in forming method, in contrast to earlier suggestions of a compositional change (Fischer and McCray 1999).

5 Conclusions

The microprobe analysis of a series of glass vessels, mainly slumped bowls, from Beirut shows that throughout the first century B.C.E. and through to the middle of the first century C.E., glass from Beirut was made from typical "early Roman" Levantine glass, with around 2.4% Al_2O_3 and 8% CaO. It has a more-or-less continuous range of MnO compositions from the background levels in the glassmaking sand through to two weight percent. Antimony-decolourisation does not occur in Levantine glass production at this time. Amber glasses contain low or minor manganese and their sulphur contents tend to be lower than the other colours. This reflects the lower solubility of S^{2-} complexed with iron, relative to $SO_4{}^{2-}$ anions, the solubility of which is strongly dependant upon the soda content of the glass. The total sulphur content is therefore related to the intrinsic redox state of the glass. Small amounts of iron appear to be added with the manganese ore used to decolourise the glass. No additional inorganic components were added to generate amber, although it is possible that organic material was added to reduce the glass and cause the formation of the ferri-sulphide chromophore. This is consistent with the view that amber was produced at the primary glassmaking stage. There is no technologically significant compositional difference between the compositions of the earliest blown vessels analysed and the slumped vessels. Antimony-decolourised slumped bowls made of Egyptian glass appeared after 60 C.E., and at about the same time similar vessels were made in mixed Egyptian-Levantine glass. The origins of these vessels are unclear.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s12210-025-01311-x.

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Author contributions IF and PD conceived the project. DB carried out the analysis and processed the data. IF, DB and PD interpreted the data. IF drafted the text and the figures. IF, DB and PD produced the final manuscript. IF and PD provided resources. All authors edited the text and reviewed the manuscript.

Data availability No datasets were generated or analysed during the current study.

Declarations

Conflict of interest The authors declare no competing interests.

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