A glass workshop in ‘Aqir, Israel and a new type of compositional contamination

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ABSTRACT

Materials associated with a secondary workshop of early Byzantine date (4th-5th centuries) were unearthed in excavations by the Israel Antiquities Authority in ‘Aqir, central Israel. Fragments of furnace structure, production debris and glass vessels have been analysed by scanning electron microscopy with energy dispersive X-ray analysis (SEM-EDS) and thin-section petrography.

The results suggest that the workshop melted raw glass chunks of similar composition to the primary glass made at Apollonia, Israel, to produce secondary glass products. Some glass vessels associated with the furnace are of different composition, and some of these may represent material brought in as cullet for recycling. The furnace was built with ceramic bricks comprising alluvial-type clay with inclusions of quartz sand, probably added as temper. It was fired by potash-rich fuel to approximately 1100°C. Lime mortar was used either to cement the gaps between mudbricks or to line the furnace as a parting layer, and it has introduced a previously unrecognised type of contamination in glass of the period, mainly of Fe₂O₃ and CaO. The contamination may be identified in glass vessel assemblages elsewhere but is not ubiquitous. As its origin relates to the furnace structure, its occurrence may depend upon chronology or geography and further work is needed to resolve this issue.

Keywords: The Levant; Byzantine; Glass-working; Glass furnace; Technology; SEM-EDS; Thin-section petrography; Provenance

1. Introduction

The trade and technology of ancient glass have attracted significant attention in the literature but the investigation of glassmaking and glassworking furnaces is largely limited to their forms. With a few notable exceptions (e.g. [Rehren, 1997](#); [Jackson et al., 1998](#); [Jackson et al., 2003](#); [Paynter, 2008](#) as well as the pioneering study into LBA materials by [Turner, 1954](#)) plus some work on post-Medieval materials (e.g. [Eramo, 2005](#); [Eramo, 2006](#); [Dungworth, 2008](#)), the structural and container materials have not been well investigated, and their interactions with the bulk glass have received limited attention. However, the selection and preparation of structural and container materials offers potential insights into the practices of the craftspeople and the diffusion and transfer of technological know-how, while compositional modification of the glass may affect the interpretation of provenance and workshop processes such as recycling ([Jackson and Paynter, 2016](#); [Barfod et al., 2018](#); [Al-Bashaireh et al., 2016a](#)). The present paper offers a contribution to this under-researched area.

The eastern Mediterranean coastal strip was an important centre of glass production in the first millennium CE. Levantine glass, identified by its chemical and isotopic composition ([Freestone et al., 2000](#); [Ganio et al., 2012](#)), is found across the ancient world (e.g. [Foster and Jackson, 2009](#); [Schibille et al., 2017](#); [Ares et al., 2019](#)). In spite of extensive excavation of both primary (glass-making) and secondary (glass-working) production sites, the materials science investigation of the furnace installations, debris and associated process has been limited. In the present study, we investigate the debris from a Byzantine glass-working furnace in Israel. The results pose interesting questions about the structure of the furnace and the possible use of a lime-rich release agent or protective layer, and also indicate a previously unreported source of contamination of the glass, which can explain compositional features of glass vessels from some consumer assemblages.

2. Materials and Methods

The installation under investigation was uncovered in a salvage excavation carried out in 'Aqir Village, Qiryat 'Eqron, Central District of Israel (License number: A-6758, [Fig. 1](#)) in the year 2013 ([Marmelstein, 2016](#)). In the Roman Period 'Aqir village was named Accaron and inhabited by Jews, according to Eusebius of Caesarea (around 324 CE, translated by [Wolf, 1971](#)); it was recorded as a settlement called Akkar on the Madaba Map in Jordan in

542 CE (Donner, 1992). The larger area, Palaestina Prima was lost by its Byzantine rulers to a joint army of Sassanians and Jews during 614–628 CE (Greatrex and Lieu, 2005). Although the Byzantine rulers took their territory back in 628 CE, they gradually and irreversibly lost it to the Arabs in 634–636 CE.

Fig. 1 The location of 'Aqir. **Left:** 'Aqir in the Eastern Mediterranean Region; **Right:** 'Aqir and several other ancient glass industrial sites in Israel. Made by Chen Chen using Folium python library (Filipe et al., 2018), base maps from Stamen Toner (left) and Stamen Terrain (right), are built-in to Folium.

Although the site had been damaged by development in the seventeenth century, deposits dating from the late Roman period to the early Islamic period were excavated (Marmelstein, 2016), among which were remains of an ancient glass working installation. The structure has a rectangular shape and was built of ceramic (Marmelstein, 2016, Figs. 9–12). Collapsed ceramic (Gorin-Rosen, 2016, Fig. 21), probably from the roof, filled the structure together with glass lumps and glass working debris (Gorin-Rosen, 2016, Fig. 22). Glass vessel fragments (Gorin-Rosen, 2016, Fig. 24) as well as ceramic sherds of Byzantine jars and a Gaza jar were found closely associated, but not inside the furnace (Marmelstein, 2016, Figs. 6:23–25). Similar debris was also discovered nearby, indicating the possible existence of more glass working installations. No crucibles with glass coatings were retrieved, so it seems probable that the glass was melted in a tank furnace, as appears typical for the region (e.g. Gorin-Rosen, 2000).

The excavated area was fairly small and was damaged in the past, and it seems that the craftsmen took away almost all reusable materials after the workshop was abandoned, so the

amount of waste is relatively small. Twenty-one samples were selected for analysis, comprising four glass chunks, one moil (the most diagnostic blowing waste; excess glass circling the tip of the blowpipe and knocked off after blowing) as well as twelve fragments of ceramic building material with and without adhering glass. Four glass vessels found close to the furnace were also analysed. All the glass samples are transparent to translucent, decolourised or naturally coloured by iron oxides. Details and illustrations of materials sampled are provided in the appendix.

Two small ceramic samples as well as fragments of all glass samples were mounted in transparent epoxy resin blocks, ground using 600–4000 grit carborundum abrasive and then polished with diamond pastes down to 1 μm . The polished blocks were vacuum-coated with carbon. Four samples have relatively different compositions from the majority (samples 12, 15, GV-1 and GV-4), so repeat samples of these were taken and analysed.

Fragments from seven ceramic samples were prepared into both polished blocks and petrographic thin-sections (Quinn, 2013, p.23-33). A fragment was first ground flat to expose the sample then mounted on a glass microscope slide and ground down to 30 μm thickness with carborundum powder.

The thin sections were examined at magnifications of 50–200 $\times$ in both plane polarised light (PPL) and between crossed-polars (XP). The thin sections were characterised in terms of their inclusions, matrix and voids. Quantitative modal and textural data was collected by point counting using the ‘single intercept’ method (Middleton et al., 1985; Quinn, 2013).

Polished samples were examined using a ZEISS EVO 25 scanning electron microscope (SEM), fitted with an Oxford Instruments X-Max^N 80 Silicon Drift Detector and Aztec Energy Dispersive X-ray Spectrometer (EDS) system. Working conditions were 20.0kV accelerating voltage, 8.50 mm working distance, and 100 s counting time, with a typical yield of c. 3,000,000 counts on metallic cobalt. Bulk analysis for glass samples was taken at 600 $\times$ magnification at 5 different areas, and that for ceramics was typically at 30–150 $\times$ magnification for 5 different areas. Oxygen was determined by stoichiometry and results are normalised to 100 wt %.

EDS analysis results for Corning Museum of Glass archaeological reference glasses A, B and D are presented in Table 1. For elements with a concentration greater than 5% the relative errors (RE) were all within 5% and the relative standard deviations (RSD) were all

within 2%. For most minor elements (0.1–5%) the relative errors were within 10% of the accepted values. The relative standard deviations of most minor elements were within 10% but deteriorated as the detection limits of c. 0.1% were approached.

Table 1 Accuracy and precision for measurements of glass standards by SEM-EDS

	Content	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	Sb ₂ O ₅
Corn A	Expected wt%	14.30	2.66	1.00	66.56	0.08	0.14	0.09	2.87	5.03	0.79	1.00	1.09	1.75
	Mean (n=3)	14.54	2.50	0.91	67.08	<0.1	0.17	0.09	2.94	5.20	0.89	1.03	1.10	1.85
	SD	0.03	0.01	0.01	0.10	-	0.04	0.02	0.03	0.05	0.07	0.06	0.01	0.12
	Precision=RSD%	0.20	0.50	0.90	0.15	-	22.09	18.21	0.89	1.05	8.00	6.19	0.85	6.26
	Accuracy=RE%	1.70	-6.14	-9.00	0.79		19.05	3.70	2.56	3.31	12.66	3.00	1.22	5.52
Corn B	Expected wt%	17.00	1.03	4.36	61.55	0.82	0.49	0.16	1.00	8.56	0.09	0.25	0.34	0.46
	Mean (n=3)	17.19	1.02	4.16	61.36	0.91	0.60	0.16	1.11	8.88	0.14	0.24	0.36	0.49
	SD	0.06	0.03	0.07	0.01	0.02	0.01	0.00	0.01	0.08	0.03	0.02	0.01	0.04
	Precision=RSD%	0.36	2.56	1.67	0.02	2.69	2.36	2.89	0.74	0.93	23.72	8.68	3.50	7.82
	Accuracy=RE%	1.12	-0.65	-4.66	-0.31	10.98	22.45	2.08	11.00	3.70	61.05	-5.33	4.90	7.25
Corn D	Expected wt%	1.20	3.94	5.30	55.24	3.93	0.23	0.16	11.30	14.80	0.38	0.55	0.52	0.97
	Mean (n=3)	1.34	3.87	5.09	54.87	4.22	0.22	0.17	11.58	15.12	0.39	0.55	0.53	1.13
	SD	0.06	0.02	0.09	0.03	0.05	0.01	0.00	0.06	0.04	0.05	0.04	0.03	0.03
	Precision=RSD%	4.27	0.63	1.77	0.06	1.17	4.22	0.00	0.53	0.29	11.80	6.80	5.44	2.32
	Accuracy=RE%	11.67	-1.78	-3.90	-0.66	7.29	-2.90	6.25	2.45	2.16	3.51	0.00	1.28	16.84

SD=standard deviation, RSD=relative standard deviation, RE=relative error = (Measurement value -True value)/True value. Errors which exceed our preferred limit of 10% are shown in bold, and typically occur as values approach 0.1% element. Expected values are mainly from Brill (1999) with additional information from Adlington (2017).

Table 2 SEM-EDS analyses of ceramic samples from ‘Aqir, normalised to 100%

Sample	Material Type	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃
1	Clay ceramic	0.8	1.5	9.5	74.3	<0.1	<i>0.1</i>	0.2	2.3	4.9	1.0	<0.1	5.4
2	Clay ceramic	1.2	1.9	10.8	65.6	<0.1	0.4	1.1	2.3	9.9	1.3	<0.1	5.7
3	Clay ceramic	0.4	0.5	7.6	83.9	<0.1	<i>0.2</i>	0.3	1.9	1.2	0.6	<0.1	3.4
4	Clay ceramic	0.6	1.0	10.0	78.1	<0.1	<0.1	<0.1	1.5	2.9	0.9	<0.1	5.1
5-black area	Clay ceramic	0.7	1.1	8.5	77.3	<0.1	<i>0.2</i>	<i>0.1</i>	2.3	3.9	0.9	<0.1	5.0
5b-vitrified	Vitrified surface	3.5	2.2	8.2	62.0	0.3	<0.1	<0.1	7.3	9.6	0.9	0.2	5.9
5-red area	Clay ceramic	0.5	1.3	9.8	76.5	<0.1	<i>0.2</i>	0.2	1.0	4.2	0.9	<0.1	5.4
6	Clay ceramic	0.8	1.5	9.1	75.5	<0.1	<0.1	0.2	2.0	4.6	1.1	<0.1	5.4
7	Clay ceramic	0.5	1.2	8.1	79.7	<0.1	<i>0.1</i>	0.2	1.4	3.8	0.8	<0.1	4.1
7-vitrified	Vitrified surface	3.2	1.9	7.1	69.8	0.7	<0.1	<0.1	8.0	5.1	0.7	<0.1	3.7
8c	Lime-rich	1.3	3.0	3.6	55.7	0.3	0.3	<i>0.1</i>	0.3	33.2	0.3	<0.1	1.8
10	Clay ceramic	0.4	0.8	8.6	82.2	<0.1	<i>0.1</i>	0.2	1.1	1.5	0.6	<0.1	4.6
10-vitrified	Vitrified surface	2.3	1.6	8.2	69.2	0.5	<0.1	<0.1	7.4	7.1	0.5	<0.1	3.1
13c	Soda-rich clay	5.8	0.3	3.8	83.5	<0.1	<0.1	0.3	1.1	4.8	<0.1	<0.1	0.4
14-Area A (Fig. 3)	Lime-rich	<0.1	1.9	2.5	50.9	0.4	0.7	<0.1	0.4	39.2	0.7	<0.1	3.3
14-Area B	Lime	<0.1	0.4	1.0	4.0	0.3	0.7	0.3	<0.1	89.4	<0.1	<0.1	1.2
14-Area C	Glass on lime-rich	4.9	0.8	2.6	72.7	<0.1	<i>0.2</i>	0.2	1.2	15.6	0.2	<0.1	1.8
14-Area D	Glass on lime-rich	8.1	0.7	2.7	65.1	<i>0.1</i>	<i>0.2</i>	0.5	1.0	20.8	<0.1	<0.1	0.9
14-Area E	Clay ceramic	2.8	0.7	7.2	77.7	0.6	<i>0.2</i>	0.4	2.3	3.5	0.5	<0.1	4.0
16c	Lime-rich	1.5	3.7	5.1	45.6	1.8	0.4	<i>0.1</i>	0.3	39.2	0.3	0.3	1.9

Chemical compositions are given in wt%. Mean of five area analyses, except sample 2 (average of two) and sample 3 (average of two). The letter ‘c’ denotes the ceramic part of a composite glass-ceramic sample. The figures in italic are close to the limit of detection and have a higher uncertainty.

Table 3 SEM-EDS analyses of glass samples from ‘Aqir, normalised to 100%

Sample	Type	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	Sb ₂ O ₅
<i>Glassworking debris</i>														
8g	glass with ceramic	13.0	0.64	3.1	71.3	<0.1	<0.1	0.74	0.60	10.0	<i>0.1</i>	<0.1	0.53	<0.3
9	lump	13.7	0.71	3.1	70.4	<0.1	<i>0.2</i>	0.72	0.85	9.7	<i>0.1</i>	<0.1	0.51	<0.3
11	moil	13.8	0.69	3.1	70.6	<0.1	<i>0.1</i>	0.74	0.93	9.6	<0.1	<0.1	0.50	<0.3
12_1	lump	14.8	0.73	2.9	64.6	0.2	<i>0.1</i>	0.58	0.71	14.7	<i>0.1</i>	<0.1	0.60	<0.3
12_2	lump	14.9	0.74	3.0	64.7	0.2	<i>0.1</i>	0.59	0.69	14.5	<i>0.1</i>	<0.1	0.60	<0.3
13g	glass with ceramic	13.3	0.62	3.1	72.5	<0.1	<i>0.2</i>	0.66	0.70	8.4	<0.1	<0.1	0.46	<0.3
15_1	lump	9.7	0.71	3.1	72.4	<0.1	<0.1	0.36	0.61	12.3	<i>0.1</i>	<0.1	0.67	<0.3
15_2	lump	11.8	0.66	3.1	72.1	<0.1	<0.1	0.53	0.62	10.6	<i>0.1</i>	<0.1	0.55	<0.3
16g	glass with ceramic	14.3	0.67	3.1	70.7	<0.1	<i>0.2</i>	0.73	0.63	9.3	<0.1	<0.1	0.47	<0.3
17	lump	15.2	0.69	3.1	66.8	0.2	<0.1	0.68	0.99	11.7	<0.1	<0.1	0.65	<0.3
<i>Glass vessels</i>														
GV-1_1	vessel	19.4	0.55	1.9	68.1	<0.1	0.4	1.06	0.62	6.8	<0.1	0.10	0.52	0.6
GV-1_2	vessel	19.8	0.50	2.0	68.0	<0.1	0.3	1.07	0.62	6.6	<0.1	0.10	0.47	0.6
GV-2	vessel	19.0	0.56	2.4	66.4	<0.1	0.3	1.09	0.59	8.0	<0.1	0.99	0.74	<0.3
GV-3	vessel	14.1	0.66	2.6	70.3	<0.1	<i>0.2</i>	0.73	1.10	8.6	<0.1	1.30	0.54	<0.3
GV-4_1	vessel	13.9	0.53	2.5	70.7	<0.1	<0.1	1.08	0.25	9.8	0.3	<0.1	0.96	<0.3
GV-4_2	vessel	14.1	0.55	2.5	70.7	<0.1	<i>0.1</i>	1.04	0.28	9.6	0.3	<0.1	0.97	<0.3

Chemical compositions are given in wt%. Mean of 5 values, sample_1 and _2 means two pieces of the same sample. The letter ‘g’ denotes the glass part of a composite glass-ceramic sample. The figures in italic are close to the limit of detection and have a higher uncertainty.

3. Results

3.1. Furnace materials

The structural furnace materials are mostly black to dark brown, some are brownish-red (samples 3 and 5), some with black glassy surface layers (samples 5-black part, 7 and 10). They are seen to comprise a quartz-rich fabric with c. 30% inclusions, predominantly sub-angular to sub-rounded fine quartz sand grains (<0.90mm, mode = 0.19mm) with rare feldspar (Fig. 2, in some cases molten to pools of glass with included acicular crystals, probably mullite) and chert. Rare wollastonite and secondary calcite are also present. The matrices, making up about 50% of a section, are typically isotropic; however, the red area of sample 5 is slightly birefringent in thin section. The remaining c. 20% comprises voids, some are very coarse bloating pores (>100 μm ; Freestone and Tite, 1986). The quartz sand grains are well sorted and do not grade into a fine fraction; they are assumed to have been added to the clay.

Fig. 2. Thin-section photomicrograph of ceramic sample 6 in plane polarised light (PPL) showing two molten feldspars, the light particles are quartz; spheroidal gas bubbles are also visible (voids labelled with 'v').

The bulk compositions of the clay ceramic (samples 1–7 and 10) are given in Table 2; they are silica-rich and contain 8–11% Al_2O_3 and 3–5% Fe_2O_3 , with CaO typically below

5%. Their $\text{Fe}_2\text{O}_3/\text{Al}_2\text{O}_3$ and $\text{Fe}_2\text{O}_3/\text{TiO}_2$ ratios are c. 0.5 and 5 respectively, which are close to those of typical alluvial clay (Kamber et al., 2005). These materials appear to represent an alluvial-type low-refractory clay with a sand temper and are interpreted as the remains of the furnace bricks.

Only a single sample (13c, Table 2; labelled soda-rich clay in Fig. 3) appears to unambiguously represent the result of contamination of furnace brick by soda-lime-silica glass, with elevated soda but low in the ash constituents, potash and phosphate.

Fig. 3 Bivariate plot of lime and alumina contents of furnace materials.

The ceramics frequently comprise complex samples with areas of different composition. The compositional relationships between them are summarised in Fig. 3. In addition to the clay-based ceramics described above we identify several types of material. Relatively smooth glaze-like surface layers, which are rich in K_2O , occur on samples 5, 7 and 10 (Table 2). They have elevated K_2O (7–8%), Na_2O (2.0–3.5%) and P_2O_5 (0.3–0.7%) and are also slightly higher in CaO than the respective ceramic bodies (Fig. 3), suggesting that potash-rich fuel ash and vapour (and possibly sodium-rich vapour from the glass) had reacted with and fluxed the clay (Paynter, 2008; Rehren and Perini, 2005). These are therefore bricks from the furnace wall or roof above the tank which held the glass, rather than samples which were in contact with the glass itself.

A second group of ceramic materials are rich in lime. For example, Sample 14 is a complex fragment which comprises both clay and lime-rich areas, as shown in Fig. 4, where area E is the typical clay ceramic fabric described above (Fig. 4vi). Area B is essentially lime with low silica, it contains a few quartz grains which show reaction rims with the matrix (Fig. 4iii). Area A consists of c. 51% SiO₂ and 39% CaO (Table 2) and contains sparse quartz grains which again show reaction rims with the calcium silicate matrix (Fig. 4iv). Area C has sparse quartz in a glassy matrix, and contains 4.9% Na₂O and 15.6% CaO; it appears to be the product of a reaction between soda-lime-silica glass and sand-tempered lime. Area D is glass containing 8.1% Na₂O and 20.8% CaO; it contains extensive wollastonite (CaSiO₃) crystals (Fig. 4v) and appears to represent soda-lime-silica glass which has dissolved significant amounts of lime.

Other lime-rich ceramics were found as part of composite samples 8 and 16. They are relatively thin lime-rich layers adhering to the pieces of the typical soda-lime-silica glass associated with the furnace. They comprise complex materials with c. 39% CaO and 50% SiO₂ (samples 8c and 16c, Table 2). They include areas of glass and a neo-formed crystalline phase with a composition (c. 4% Na₂O, 6% MgO, 10% Al₂O₃, 42% SiO₂, <0.1% K₂O, 33% CaO and 2.5% Fe₂O₃) which corresponds closely to the melilite group mineral alumoåkermanite (Weidenmann et al., 2009) in a matrix consisting of domains of a fibrous silica-rich phase with interstitial voids which sometimes contain calcareous material (Fig. 5). Morphologically these domains closely resemble the texture of the mineral larnite (Ca₂SiO₄), formed during combustion metamorphism of oil shale; compare Fig. 5 of this paper with Gross, 2016 (pp 67), Sokol et al., 2019 (figures 7 & 8) and Fukuda and Maki, 1989 (Fig. 1). As one of the reactive phases in portland cement, larnite is well understood to be unstable and breaks down in water (e.g. Sokol et al., 2019). The presence of larnite pseudomorphs in high temperature archaeological calc-silicates exposed to post-depositional processes is therefore to be expected. The inferred loss of CaO during the alteration process suggests that our measurements of the compositions of these layers (Table 2) are likely to be low in CaO relative to the pristine materials.

Fig. 4 Sample 14. **i:** thin section photomicrograph (XP). A & B: calcareous materials, C: glass penetrated into ceramic, D: wollastonite plus glass, E: clay ceramic; **ii:** polished block in ordinary light (different fragment of sample 14); **iii:** thin section photomicrograph (XP) of area B, showing rare grey quartz crystals with dark-brown reaction rims in a yellow-brown calcareous matrix; **iv-vi:** back-scattered electron (BSE) images of different areas. **iv:** area A, showing grey quartz sand inclusions with reaction rims in a calcareous matrix, growth of secondary calcite crystals is observed on its bottom left; **v:** area D, showing light-grey blocky wollastonite crystals formed in a glassy matrix; **vi:** area E, showing grey quartz sand inclusions in porous matrix, the black areas are voids.

Fig. 5 BSE image of sample 8c. The flat dark-grey phases at the top are high-Al, Fe soda-lime-silica glass (g) with light-grey acicular wollastonite (wo) crystals inside, the mid-grey rounded phases are calcite (cal), and the light-grey irregular areas are aluminosilicate (ak). The fibrous matrix comprises domains consisting mainly of crystallites of a silica phase with interstitial voids; these are interpreted as pseudomorphs after larnite (see text).

3.2. Glass

The compositions of the glass samples are given in [Table 2](#). For those that have been sampled and analysed twice (12, 15, GV-1 and GV-4), a ‘_1’ or ‘_2’ has been added after the sample names to distinguish the two analyses. The glass samples are all low-magnesium, low-potassium soda-lime-silica (natron) type.

Most of the glassworking remains (samples 8, 9, 13, 16 and the moil, sample 11) have Na₂O in the range 12–15%, CaO 8–11% and Al₂O₃ around 3% with TiO₂ close to the detection limit. These samples match in many respects the glass analysed from the primary

production furnaces at Apollonia (Freestone et al., 2000; Tal et al., 2004; Freestone et al., 2008; summarised in Freestone, 2020). However, glass with CaO greater than 11% has not been analysed from the Apollonia furnaces, but samples 12, 15 and 17 have lime ranging from 11 to 15%. Furthermore, while the average lime content of Apollonia raw glass is 8–8.5% (Freestone, 2020), the mean lime content of the present samples is 10.7% (median value is 9.9%). In addition, both analysed pieces of sample 15 resemble Apollonia-type glass but they have low Na₂O and Cl and are zoned with respect to these components.

The number of primary glassmaking centres at any period was limited, on the basis of the production sites which have been excavated to date and the compositional groups which have been identified (Freestone et al., 2000; Gorin-Rosen, 2000; Foy et al., 2003; Schibille et al., 2017). In contrast to the glass waste from the furnace, none of the four glass vessels are of the Apollonia type. GV-1 has high soda and low alumina and was decolourised using antimony (Table 3). It is typical of Roman-Sb glass of the first-fourth centuries, probably originating in Egypt (Gliozzo 2017; Schibille et al., 2017; Barfod et al., 2020). GV-2 which contains c. 1% MnO is higher in Na₂O (19.0%) than typical Roman-Mn type of the 1st-4th centuries CE (up to 17.5% according to the data of Silvestri et al., 2008) but of the recognised glass groups (Freestone, 2021), this appears to be the best fit and the glass is similar to that of Jalame (Brill, 1988). It is unlikely to be one of the later 5-6th century groups: Al₂O₃ is too high for Foy série 3.2 and MgO and Fe₂O₃ are too low for HIMT or Foy 2.1. GV-3 is part of a beaker with solid base, a well-known form dating back to the fourth century CE (Gorin-Rosen, 2016, Fig. 24:6), and its composition is also typical of Roman Mn-decolourised glass of the period and region (e.g. Brill, 1988). GV-4 has high TiO₂, indicating a likely Egyptian origin, and accords with Egypt II type (Phelps et al., 2016) dating to the late-8th to 9th centuries (Schibille et al., 2019). This piece, which was sampled and analysed twice, is somewhat anomalous with respect to the rest of the assemblage but other characteristics, particularly the low K₂O (Table 3), match Egypt II very well.

4. Discussion

4.1. Furnace, fuel and operational temperature

The structure of the furnace was built using bricks made from an alluvial-type clay, with a sand temper. The glazed surface layers of the bricks from the furnace superstructure show

elevated potash, soda, phosphorus and lime, suggesting contamination from potash-rich fuel ash (Paynter, 2008; Rehren and Perini, 2005). In addition to the available plants and woods, the use of olive-pressing residue or ‘pomace’ (Rowan, 2015) as a fuel in glass furnaces was suggested by Gorin-Rosen (2000) in Byzantine Bet She’an, Israel and by Barfod et al. (2018) in Byzantine and early Islamic Jerash, Jordan, and the latter authors note that the ash of olive residue is especially high in K_2O . The vitrified layers are contaminated by soda as well as potash and while this may represent vapour from the glass, pottery surfaces glazed by soda-rich fuel vapour have been reported by Freestone et al. (2001) at Deir ‘Ain ‘Abata, Jordan and the fuel must be considered a possible soda source.

Although one ceramic sample (13c; Table 3) shows evidence of contamination of the clay furnace brick by glass, most of the ceramic samples which show interaction with the soda-lime-silica glass show elevated lime. Lime appears to have present as a mortar containing varying amounts of a sand aggregate, similar to the sand used to temper the brick clay. There appears to have been extensive high-temperature interaction between the lime and the quartz and where present, the quartz grains have well developed reaction rims (Fig. 4iv). In two examples, samples 8c and 16c, the reaction between quartz sand grains and lime matrix appears to have gone to completion, so that no relict sand grains are present and a complex phase assemblage has resulted. This may have been accelerated due to contamination by minor amounts of glass (Fig. 5); Sample 14 (Area A) where quartz grains may be observed, is lower in Na_2O than samples 8c and 16c, which are otherwise similar in composition.

Hunault et al. (2017) estimate the working temperatures of soda-lime-silica glass to be in the range 750–1050°C. Experiments with a wood-fired pot furnace by Taylor and Hill (2008) indicate maximum temperatures in the melting chamber of around 1175°C, but typically in the range 1100–1150°C. The molten feldspars identified in both thin-section microscopy (Fig. 2) and SEM analysis are consistent with these figures, as the minimum melting temperature in the alkali feldspar phase diagram is c. 1080°C (Roedder, 1959) suggesting a temperature higher than this. In addition, the clay matrix of sample 10, which does not appear to be significantly contaminated by fluxes, shows continuous vitrification with very coarse bloating pores ($>100\text{ }\mu\text{m}$), indicating a temperature over 1100°C for most clay types (Freestone and Tite, 1986; Tite, 1999). It should be noted that the temperatures inside the ceramics were zoned, as might be expected of furnace wall, as the matrix of the red area of

sample 5 is slightly birefringent in thin section suggesting a temperature $<850^{\circ}\text{C}$ (Quinn, 2013, pp. 191).

4.2. Compositional variation of glass

While both analysed pieces of sample 15 match Apollonia-type glass, they show zoning with respect Na_2O and Cl , which are low, suggesting that this sample lost sodium mainly in the form of sodium chloride (NaCl) due to vaporisation during prolonged heating in high temperature (Gerth et al., 1998).

The lime contents among most ‘Aqir glass production materials are higher than the raw glass chunks produced in Apollonia, and slightly higher than those of the Islamic period Levantine natron glass assemblage from Raqqa, Syria (Henderson et al., 2004; Henderson et al., 2005), which has been noted (Phelps et al., 2016) to be characterised by relatively high CaO concentrations (typically 8–10%). This observation of elevated CaO is only marginally affected by the minor deviation of the present analyses from the Corning standards of 0.2–0.3% (Table 1).

Fig. 6 illustrates a strong correlation of Fe_2O_3 and CaO contents among eight ‘Aqir glass waste samples ($R^2=0.98$). Sample 12 has been neglected, as both analyses lie off the trend defined by the other glasses; this sample has other characteristics, such as slightly elevated Na_2O , MgO , and possibly P_2O_5 which suggest it may have been additionally contaminated by the fuel (Table 3). The strong correlation suggests that the additional iron oxide and lime in the majority of the samples have a common origin and that an explanation for the elevated CaO should take this into account. This Fe_2O_3 - CaO relationship does not occur among Apollonia raw glass chunks (Freestone et al., 2000; Tal et al., 2004; Freestone et al., 2008; Abd-Allah, 2010) and most other published Apollonia type glass products (e.g. Al-Bashaireh et al., 2016b; Ali and Abd-Allah, 2015; Barfod et al., 2018; El-Khoury, 2014; Phelps et al., 2016; Rehren et al., 2010; Tal et al., 2008), suggesting that a contamination was introduced into the ‘Aqir glasses during the glassworking campaign, rather than inherited from the raw glass.

Fig. 6 Bivariate plot of iron oxide and lime content with error bars (error = standard deviation of 5 analysis) among glass production materials, 12_1 and 12_2 are outliers.

Six of the samples analysed (8, 9, 12, 13, 15, 16) are debris containing both glass and ceramic, suggesting that they came from the walls or floors of glass furnaces. Among them, the ceramic areas of samples 8 and 16 are notably high in CaO (33.2% and 39.2% respectively). Areas A and B of complex sample 14 are also high in CaO (39.2% and 89.4% respectively). Sample 13 is not as high in lime in its ceramic area (4.8% CaO), and the CaO concentration in the attached glass is the lowest among the eight samples (8.4%) and within the typical Apollonia range (7–9%). These observations suggest that the calcareous structural materials (mortar or plaster) of the furnaces are likely the source of additional lime. The wollastonite-rich glass in sample 14 (Area D; Fig. 4v) has a Na₂O content of c. 8% along with c. 21% CaO; the high Na₂O indicates that the source of the silica was mostly the soda-lime-silica glass, rather than quartz sand. Thus, we may conclude that these calcareous materials were in contact with the glass during the melting process. They may have been used to cement the gaps between the mudbricks or to line the furnace as a parting layer (a calcium-rich coating allowing easy release of glass after working, Merkel and Rehren, 2007). While it is possible that the lime might be present in the furnace structure as mortar between the bricks, the apparent prevalence of lime in contact with the glass (as opposed to being in contact with the clay ceramic) leads us to suspect that the tank holding the glass indeed had a lime parting layer, which would also have served to insulate the clay bricks and molten glass

from each other. For reaction of the glass with the structural lime materials to explain the slope of the correlation observed between iron oxide and lime, a $\text{CaO}/\text{Fe}_2\text{O}_3$ ratio of around 20 would be required (Fig. 6, neglecting outlier sample 12). Lime-rich ceramic samples 8c and 16c have $\text{CaO}/\text{Fe}_2\text{O}_3$ ratios of c.19 and 21 respectively and are consistent with this.

The low numbers of moils recovered at 'Aqir and at other workshops in the region (e.g. Hagolan Street, Tel Aviv; Freestone et al., 2015) suggests that even when located close to the centres of raw glass production, the waste materials of secondary workshops were carefully collected for recycling. For comparison, a Roman glass workshop in London, UK which appears to have been abandoned without a final collection of the waste yielded 6400 moil fragments (Wardle et al., 2015, pp. 54). The evidence for the transportation of secondary production waste in the Ma'agan Mikhael B wreck (Natan et al., 2020) supports this view. The glass workers are likely to have recycled most of the waste glass material which was not incorporated into vessels. However, the glass that stuck to the furnace floor and walls could not easily be separated without serious contamination. It is therefore reasonable to suggest that the waste glass that we have analysed would have been more severely contaminated than the glass in the centre of the tank which was blown into products. In view of this we have looked for evidence in the literature that this form of contamination has affected vessel compositions. While such evidence cannot be recognised in every assemblage, the compositional distribution of vessels in Apollonia-type glass from Cyprus, analysed by Ceglia et al., 2015, Ceglia et al., 2019, shows a marked elongation in the Fe_2O_3 -CaO direction, which corresponds to the Fe_2O_3 -CaO trend seen in the 'Aqir samples (Fig. 7 left). There is also a second, higher Fe_2O_3 trend evident in Fig. 7 (left); this, however, is related to elevated alumina (not illustrated), indicating contamination by clay ceramic. Ceglia et al. (2015) analysed their vessels using electron microprobe (EPMA) and repeated the analyses with LA-ICP-MS (Ceglia et al., 2019) and the effect is shown by both datasets (Fig. 7 left). Furthermore, glasses that had low levels of transition elements and were therefore considered by Ceglia et al. (2019) to show least evidence for recycling, also show least contamination by iron oxide and lime (Fig. 7 left). This is consistent with an increase in contamination through repeated re-melting and contact with the furnace materials.

We have also looked for indication of contamination in the trace element contents of the glass from Cyprus. If the mortar in the furnace had been made by burning limestone, it would have had a lower Sr/CaO ratio than the glass in which the lime was derived from marine shell

fragments in the glassmaking sand (Freestone et al., 2003). The contaminated glass might therefore be expected to show a lower Sr/CaO ratio. However, while Fig. 7 (right) suggests that most of the glass with high CaO and showing evidence for recycling also has lower Sr/CaO, it is far from conclusive. This may be because the effects of the contamination are hidden by the scatter of the Sr/CaO ratios of the glass but, alternatively, the mortar may have been produced by burning shell or *kurkar* (the regional name for lithified beach sand on the Levantine coast). More work would be needed to investigate the trace elements in the mortars of the furnace, to resolve this issue.

Fig. 7 Left: Bivariate plot of iron oxide and lime contents of glass production samples from 'Aqir (this study) compared with Apollonia type glass from Cyprus, data of Ceglia et al., 2015, Ceglia et al., 2019. Samples prefixed 15 are EPMA data, samples prefixed 19 are LA-ICP-MS data. R indicates glass with evidence for recycling in the form of elevated transition metals and N indicates glass with no evidence for recycling. It is observed that the glasses with no evidence for recycling (dark symbols) typically have lower Fe₂O₃ and CaO contents; **Right:** Bivariate plot of strontium and lime contents of Apollonia type glass from Cyprus, data of Ceglia et al. (2019). 19R = glass with evidence for recycling; 19N = glass with no evidence for recycling.

4.3. Chronology

The furnace is dated by the excavator to be no earlier than the late third century CE on the basis of an associated burnt *folles* (coin) dated to 294–298 CE. The presence of bag-shaped

jars dated to the late fourth – early fifth century in the furnace structure suggests that the furnace was operating in this period (Marmelstein, 2016). This at first appears to be at variance with the identification of the glass-working waste as Apollonia type glass, as the Apollonia furnaces which have been excavated are dated to the sixth-seventh centuries. However, Apollonia-type glass is known in the fifth century from elsewhere (e.g. Série 3.3 of Foy et al., 2003, Levantine I of Rehren and Cholakova, 2014) and at present there is a chronological gap incorporating the fifth century between the known Levantine glass making sites of Jalame and Apollonia (Freestone, 2020). Therefore, the Apollonia-type glass associated with the ‘Aqir furnace is not inconsistent with a late fourth – early fifth century date. It may represent glass made in the Apollonia region earlier than the excavated installations or glass of similar composition from elsewhere on the Levantine coast.

Glass vessels GV-1, GV-2 and GV-3 found close to the furnace are of well-known compositional groups broadly consistent with their typological dating to the fourth century or earlier; they are of compositions which differ from the Apollonia-type of the waste products. One possibility is that they were pieces collected to be recycled but this is speculative.

5. Conclusion

The present study confirms the potential of the analysis of glass workshop debris to provide useful information but also the complexities of interpretation of this type of assemblage.

The presence of glass vessels associated with the furnace which were of a different compositional type to the glass being melted indicates the need for caution in relating the production of glass forms to associated furnace structures without supporting analytical evidence.

Although made of a clay which was not particularly refractory, the ceramics contain several tens of percent of quartz, probably added to reduce thermal shrinkage and the associated stresses, and were able to operate at temperatures of around 1100°C. Mortar was applied either to fill the gaps between mudbricks or to line the furnace as a parting layer, but unfortunately the recovered debris does not allow the unambiguous discrimination of these two possibilities, although given the relative abundance of fragments with both high lime ceramic and glass, a parting layer seems most likely. The use of a lime parting layer would be paralleled in glass making materials recovered from Late Bronze Age Egypt (Merkel and

Rehren, 2007; Smirniou and Rehren, 2016) and might suggest a long-lasting regional tradition. At 'Aqir, the glass interacted with the lime and allowed the identification for the first time in the Levant of a distinctive type of contamination. A review of available compositional data in the literature indicates a similar compositional pattern in contemporary glass vessels on Cyprus. It may be characteristic of a particular furnace technology common to the two regions, or alternatively indicate the export of cullet for recycling from Israel. In view of the association of the contamination in the Cypriot assemblage with evidence for glass recycling, the latter seems likely. Failure to recognise similar trends in other data for Apollonia glass (e.g. Phelps et al., 2016) may reflect chronological or geographical issues. It may be that other glass furnaces were lined with lime which had a low content of Fe_2O_3 , so that the trend observed here does not occur in every assemblage. It is likely that contamination of the glass with lime did not only occur due to diffusion from the walls of the furnace into the tank; thin layers of parting material attached to glass broken out of failed furnaces for re-use would have added significant amounts of lime to batches of glass in which they were included. Where it occurs, this type of contamination is likely to complicate attempts to subdivide vessel assemblages by composition.

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Appendix A: List of Samples

Table A.1 List of Samples

No.	Location	Area	Fig. No. on Report	Type	Colour	Description	Sample Preparation
1	L39	A2	21:9	ceramic	blackish-grey	Chunk of brick from the hard accumulation inside the furnace, probably the roof.	thin section, polished block
2	L39	A2	21:10	ceramic	yellowish brown, inside red	Drop of very light chunk of grey spongy material from the hard accumulation inside the furnace.	polished block
3	L39	A2	21:3	ceramic	red	Red brick similar to those used in construction, from the hard accumulation inside the furnace.	polished block
4	L39	A2	21:4	ceramic	blackish-grey	Fired brick with protruding edges (probably from the corner) of the hard accumulation inside the furnace, was baked in high temperature.	thin section, polished block
5	L37	A2	21:6 bottom right	ceramic	Two pieces. 5b: blackish-grey; 5r: red	Glazed mud-brick, one side glazed, inside red.	thin section, polished block
6	L37	A2	21:6 top left	ceramic	blackish-grey	Very heavy black chunk.	thin section, polished block
7	L37	A2	21:8 top right, big chunk	ceramic	dark grey	Big chunk partly glazed, looks like it was fired several times. White layer outside.	thin section, polished block
8	L2	A	22:4 top right	glass with ceramic	8c: light brown; 8g: yellowish-green	Glass with furnace debris, yellowish-green glass at the edge of the chunk.	polished block

9	L2	A	22:4 top left second	glass (with ceramic)	greenish-blue	Chunk of greenish-blue glass with debris.	polished block
10	L23	A2	21:13	ceramic	dark grey	Glazed brick, probably part of the furnace's ceiling.	thin section, polished block
11	L22	between A2A3	23:1	glass (moil)	colourless	Moil, found next to Samples GV-3 and GV-4.	polished block
12	L11	A	22:3 top left second	glass	greenish-blue	Mixed with the kiln's waste.	polished blocks (12_1, 12_2)
13	L11	A	22:3 top left first	glass with ceramic	13g: creamy-white; 13c: greenish-blue	Complete chunk: floor + glass.	polished block
14	L47	A	22:7	glass with ceramic	light brown & light green	Debris from furnace with glass penetrated and filled the cracks in the brick.	thin section, polished block
15	L4	A	22:6	glass (with ceramic)	yellowish-green	Glass with furnace debris.	polished blocks (15_1, 15_2)
16	L3	A	22:5 top left	glass with ceramic	16g: greenish-blue; 16c: light brown	Glass with small wall fragment.	polished block
17	L49	A	23:5	glass	greenish-blue	Glass chunk.	polished block
GV-1	L44	A2	24:1	glass (vessel)	colourless	Beaker body fragment with white creamy weathering layer on surface, its appearance looks like of the 2nd-3rd centuries CE.	polished blocks (GV-1_1, GV-1_2)
GV-2	L11	between A2A3	24:8	glass (vessel)	greenish-blue	Fragments from the upper part of a bottle with black pitted weathering.	polished block
GV-3	L22	between	24:6	glass	slightly greenish-blue	A solid base with silvery and black weathering, found	polished block

		A2A3		(vessel)		together with Samples 11 and GV-4.	
GV-4	L22	between A2A3	24:7	glass (vessel)	greenish-blue	Fragment of a small bottle that was distorted by the heat, found next to Samples 11 and GV-3.	polished blocks (GV-4_1, GV-4_2)

Appendix B: Illustration of Samples

Fig. B.1 Ceramic samples

Fig. B.2 Ceramic samples with glass or vitrified surface

Fig. B.3 Glass chunks

Fig. B.4 Glass vessel fragments

Photo by Chen Chen.

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