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A multi-analytical approach for the archaeometric identification of a Roman period glass furnace in the central Nile delta

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ABSTRACT

A circular structure was excavated in a suspected industrial area of ancient Thmuis (Tell Timai), and due to heavy vitrification and discolouration of the inside walls, was suspected to be a glass furnace. The excavated furnace provides a unique example to further understand the mechanisms of primary and secondary glass manufacture in Roman Egypt. Samples were subjected to a number of archaeometric investigations in order to characterise the furnace, and identify its purpose. Following attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy and scanning electron microscope energy dispersive spectroscopy (SEM-EDS), we conclude that the furnace was used for glass. We propose that it is most likely that the furnace represents a small-scale, secondary glassmaking centre, shaping glass manufactured at Wadi el-Natrun, and recycling glass objects from the local area.

1. Introduction

There is limited archaeological evidence for glass-making in Egypt during the Roman period (Nenna et al., 2000), in particular, to the authors' knowledge, no excavations of secondary glass furnaces or workshops in Graeco-Roman Egypt have been published.

A number of primary glass production centres dating to the 4th to 8th centuries have been identified in Syro-Palestine and Egypt (Brill, 1988, 1999; Nenna et al., 2000; Foy and Nenna, 2001; Foy et al., 2000; Freestone et al., 2002, 2000; Picon and Vichy, 2003). Seventeen 8th century rectangular glass furnaces were excavated at Bet Eli'ezer in Israel (Freestone et al., 2000, 2002; Gorin-Rosen, 2000). Four 6th–7th century glass furnaces were also excavated in Israel, at the site of Apollonia (Freestone et al., 2008; Gorin-Rosen, 1995, 2000; Tal et al., 2004). 1st to 2nd century primary production glass furnaces have been excavated at Wadi el Natrun in Egypt (Nenna, 2003, 2007; Nenna et al., 2000, 2005). Later primary glass production furnaces dating from the Imperial period to the 8th century have been excavated near Alexandria, Egypt, at Lake Maryut (Nenna et al., 2000).

Secondary production workshops have been excavated at Sagalassos, Turkey, dating from Imperial to early Byzantine times (Degryse et al., 2006). Glass chunks, slag and kiln fragments were used as evidence to identify the site as a glass workshop. Fragments of a secondary glass furnace (Fig. 1), and evidence for primary glass production have been excavated near Bet Eli'ezer, at the site of Horbat Biz'a (Gorin-Rosen, 2012).

There are many examples of secondary glass production workshops in the western part of the Roman Empire (Foster and Jackson, 2010), but far fewer documented in the eastern part of the Empire (Stern, 2002).

1.1. Egyptian glass during the Roman Empire

In terms of glass manufacture and distribution, it appears that a number of sites around the Roman Empire were primary production centres, distributing glass products or ingots to more prolific secondary production centres for further shaping into finished products. It has been posited that primary production centres, supplying glass to secondary production centres around the empire, were located in the Levant in Israel, based on the discovery of glass furnaces at Bet Eli'ezer and Apolonia (Gorin-Rosen, 2000) and at Alexandria in Egypt (Lucas, 1962).

The archaeological evidence and compositional evidence of glass artefacts and artefacts associated with glass production support this model, based on the uniformity in both form and composition of these artefacts (Freestone et al., 2005; Scott and Degryse, 2014).

Primary glass production sites in Egypt have been identified at Malkata (Keller, 1983), Gurob (Tatton-Brown and Andrews, 1991), Qantir (Rehren and Pusch, 1997), Akhmim (Newberry, 1920), Lisht

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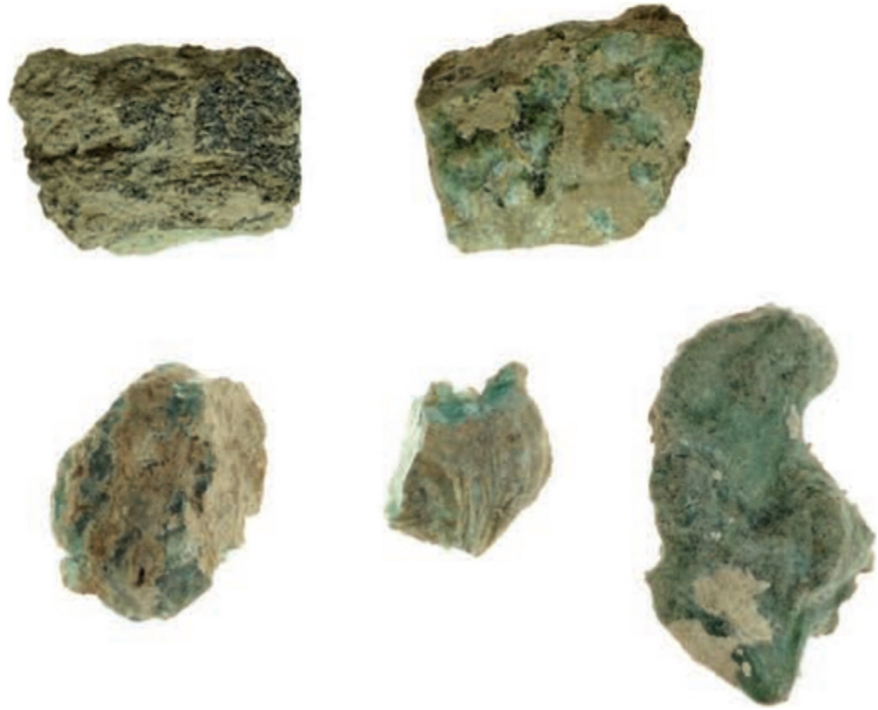

Fig. 1. Fragments of a Roman glass furnace excavated at Horbat Biz'a, Israel (Gorin-Rosen, 2012).

(Keller, 1983), Lake Maryut near Alexandria (Nenna et al., 2000) and Wadi el-Natrun (Nenna et al., 2000).

Secondary production sites would have focussed on creating finished products from ingots produced at primary production sites, as well as recycling glass items from the surrounding areas. As such, secondary production centres were smaller, required lower temperatures for softening glass, and are less frequently associated with other high temperature industries such as metal working and faience, as primary production centres were.

It has been established that, during the Roman Empire, glass from across the Mediterranean, despite being spatially and temporally widely distributed, has a relatively uniform elemental composition (Freestone, 2006; Wedepohl et al., 2011). However in the past ten to 15 years, it has been demonstrated that while the major elemental composition of glasses in the Mediterranean area are relatively uniform, trace elemental composition can be indicative of provenance within the Mediterranean area (Rehren, 2014; Shortland et al., 2007b). Roman glass consisted principally of silica sand ($\approx$75%), natron ($\approx$15%) in order to lower the melting point of the sand, and lime or magnesia ($\approx$10%) to stabilise the naturally soluble glass (Fleming, 1999; Mirti et al., 1993). During Roman times, lime as a stabiliser was found in a large enough quantity in beach sand, due to naturally occurring calcareous materials, to not always require the addition of a stabiliser as a separate ingredient (Freestone, 2006). A number of minerals could be added to the finished glass in order to colour or decolour the final product (Fleming, 1999; Nicholson and Henderson, 2000).

In terms of compositional characterisation of glass from Roman Egypt, very limited work has been produced, and this only recently (Degryse and Schneider, 2008; Rosenow and Rehren, 2014). In their study of Roman and Late Antique glass from Bubastis in northern Egypt, Rehren and Rosenow produce four compositionally distinct groups of glasses from the 87 samples analysed. From the data generated, they conclude that Bubastis, and likewise other glass production centres in Egypt, were integrated into a network of primary and secondary glass production centres throughout the Roman world, but with a preference for locally produced glass. However it is suggested that despite Bubastis' close proximity to Wadi el-Natrun, the glass at Bubastis is not consistent with glass originating at Wadi el-Natrun (Rosenow and Rehren, 2014).

With very little direct evidence of Roman glass production centres, (i.e., identifiable glass furnaces), interpretation on the method of glass production and distribution throughout the Roman Empire is based on surrogate archaeological evidence such as volume of glass artefacts found. Interpretation of a site as a glass production centre can therefore be somewhat problematic. We propose that the structure excavated at Tell Timai has been successfully identified as a Roman period glass furnace for secondary glass production, and as such, is one of very few examples of definitive evidence for a glass production centre in Graeco-Roman Egypt.

1.2. Archaeological context

Tell Timai is the ruins of the Graeco-Roman city of Thmuis in the eastern Nile delta. It is located on the extinct Mendesian branch of the Nile, where it had been established as a port, replacing the nearby port city of Mendes (Tell el-Ruba) when the Mendesian branch of the Nile shifted. The city flourished, reaching its peak in the second century CE, but was abandoned during the Arab period in the 10th century CE (Littman and Silverstein, 2008; Blouin, 2014). Today, Tell Timai survives as a rare example of a relatively well preserved Graeco-Roman city covering an area of approximately 90 ha in the central Nile delta. Survey and excavation have been conducted at the site by the Egyptian Minister of State for Antiquities (formerly the Supreme Council of Antiquities), the University of Hawaii since 2007 and also the Canadian Mission to Thmuis since 2013.

During the 2012 excavation season, a partially subterranean cylindrical brick structure was found in an area tentatively identified as a manufacturing area in the urban core during the 2007 site survey (Littman and Silverstein, 2008), and interpreted as a furnace (Fig. 1). The furnace has a diameter of 115 cm and a depth of 94 cm, cutting through an earlier plaster floor of a domestic structure (Fig. 2). In terms of its size and shape, the structure is consistent with glass furnaces excavated at Tell el-Amarna, which, while they represent glass

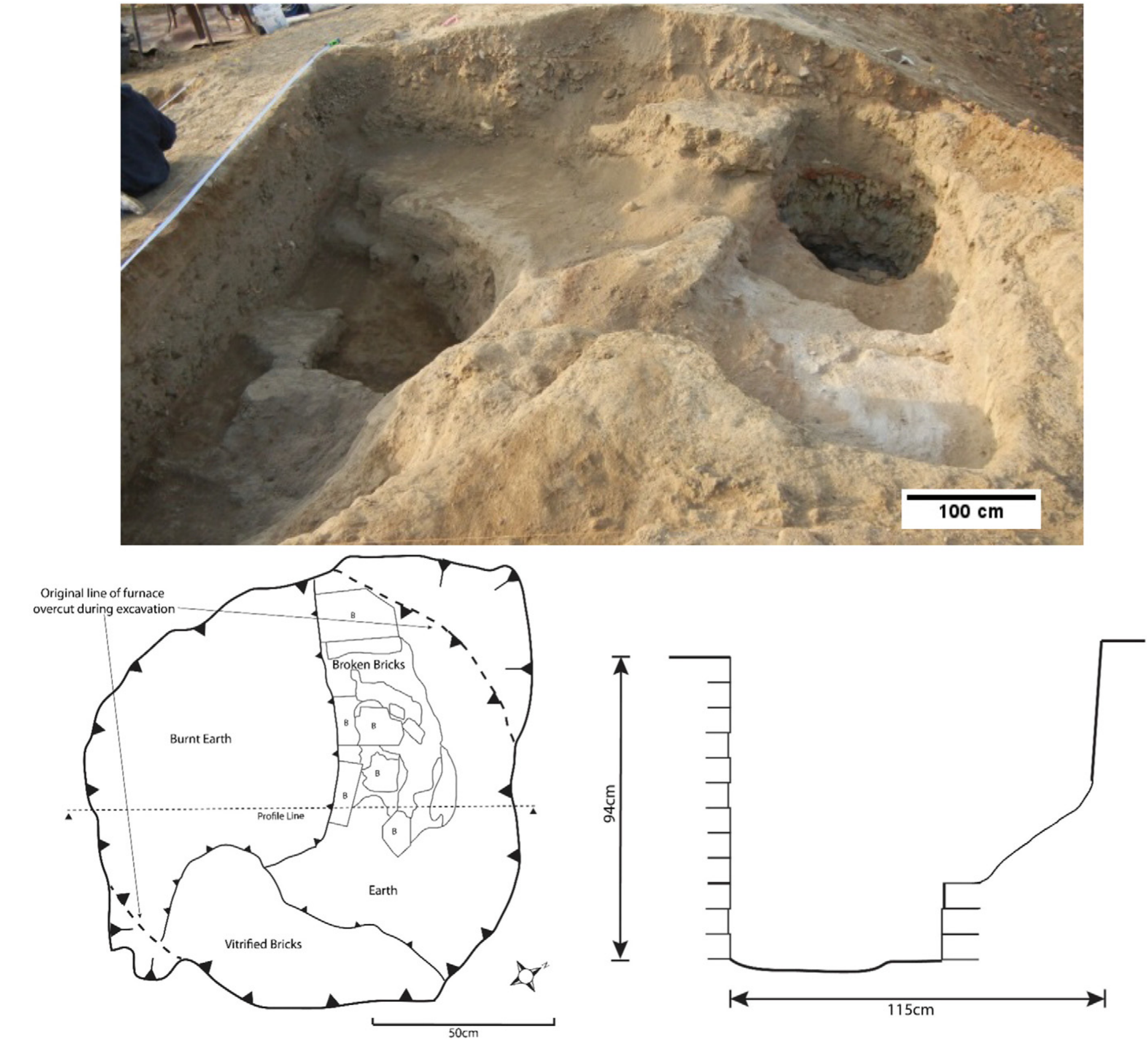

Fig. 2. The excavated structure in context; plan and profile of the furnace.

furnaces from New Kingdom Egypt, are some of the only examples of intact glass furnaces excavated in Egypt (Fig. 3).

The structure was discovered in a context which dates to the 3rd century CE based on ceramic analysis from sherds discovered in the same stratigraphic layer and from associated channels which were interpreted as having been dug to assist in disposal of industrial waste, specifically slag (Bonnette, 2013; Winter, 2013). Fifty centimetres from the top of the furnace a 'shelf' of fired bricks was discovered in the north-east third of the furnace, below that was burnt earth.

The baked brick walls of the furnace were noted to be heavily vitrified and discoloured, ranging between blue and green (Fig. 2).

In order to conclusively determine the function of the furnace, a sample of the vitrified brick (Fig. 4) was taken for analysis at the laboratories of the National Research Centre, Cairo. The sample was subjected to attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy and scanning electron microscope – energy dispersive spectroscopy (SEM-EDS).

2. Experimental procedure

2.1. Sample preparation

A sample was carefully removed from the walls of the structure using a trowel and then transported to the laboratories of the National Research Centre in Cairo for analysis.

The sample was photographed at the National Research Centre before being prepared for analysis.

Due to the inhomogeneous nature of the samples, preparation for all techniques initially involved carefully grinding the samples into a homogenous powder, and pressing into pellets.

2.2. Instrumentation

ATR-FTIR spectroscopy was performed using a 6100 Jasco (Japan) ATR-FTIR spectrometer with a range from 400 to 4000 cm^{-1}, at a rate

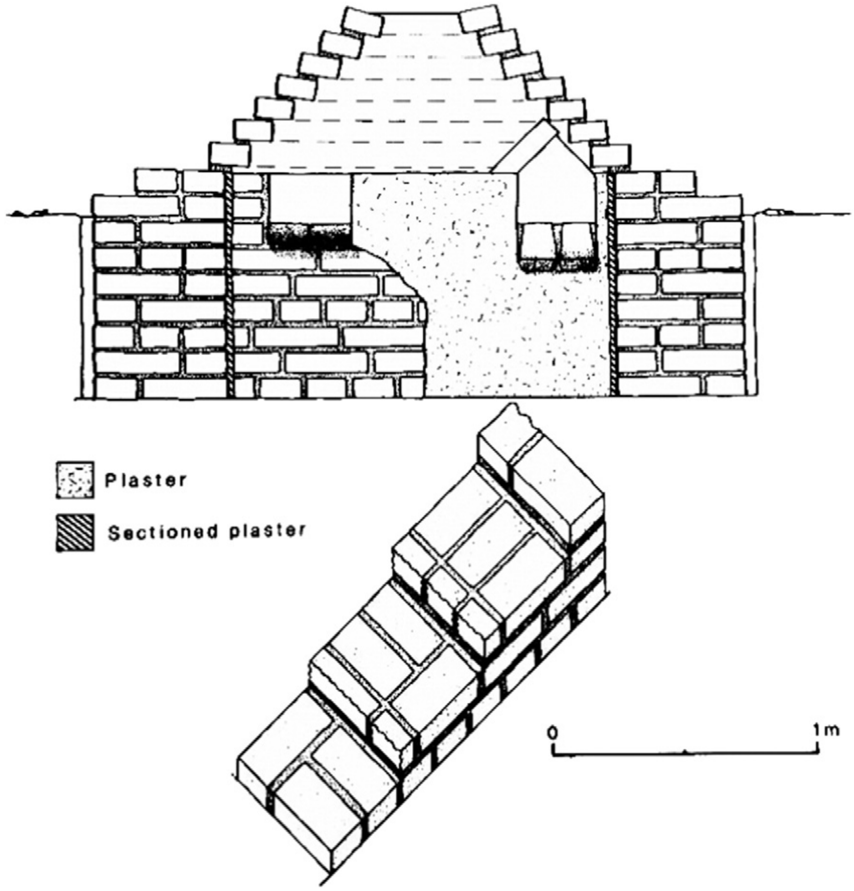

Fig. 3. Schematic cross section of 'kiln 3', excavated at Tell el-Amarna by Nicholson and Jackson (1998).

of 2 mm/s, with a resolution of 4 cm^{-1}. Each spectrum was obtained in the transmission mode with TGS detectors.

SEM-EDS analysis was performed using a FEI Quanta™ 3D 200i (Netherlands) scanning electron microscope. Analysis was carried out in environmental SEM mode, allowing the sample to be analysed without carbon coating. Accelerating voltage was 20 kV with the beam current set to 0.8 nA, a working distance of 10.6 mm and an acquisition time of 10 s. Quantification of results was achieved using built in software in the instrument to calibrate the EDS spectrum results against reference spectra.

3. Results

3.1. Attenuated total reflectance Fourier transform infrared (ATR-FTIR)

The ATR-FTIR spectrum for the furnace wall sample is shown in Fig. 5. The spectrum was compared to a standard spectrum for amorphous silica.

The broad band at 3430 cm^{-1}, as well as the peak at 1620 cm^{-1}, corresponds to the O—H stretching vibration of hydroxyls in adsorbed water in the sample.

Fig. 4. Vitrification and discolouration of the furnace interior wall; the furnace in context, in the top right of the image; a bird's eye view of the excavated furnace.

Fig. 5. Photomicrograph of the sample.

The strong transmission band at 1035 cm^{-1} can be attributed to the symmetrical Si—O—Si stretching vibration, and can also indicate the formation of a gel layer, indicative of glass alteration. Asymmetric stretching modes from SiO_2 molecules can be observed at 780 cm^{-1} as well as the bending mode at 470 cm^{-1}, as a weak band.

The peaks at 2915 cm^{-1} and 2850 cm^{-1} represent asymmetric and symmetric stretching vibrations, respectively, of methyls, which indicate the presence of organic structures on the silica's surface.

There is a distinct lack of bands which would indicate silanol Si—OH bending around 940–960 cm^{-1}, which would be associated with organic structures on the silica's surface, represented by the peaks at 2915 and 2850 cm^{-1}.

3.2. Scanning electron microscope – energy dispersive spectroscopy (SEM-EDS)

The composition of the sample indicates that the majority of the sample is made up of silica, indicative of glass. The other elements present may be indicative of the raw materials used in manufacturing and modifying the finished product (Table 1).

Fig. 6 and Table 2 show a comparison between the compositions of the Tell Timai sample and several examples of the composition of the interface between glass and glass-making. GT175 is a sample from the layer of glass on the interior of a ceramic, glass-making crucible discovered at the New Kingdom site of Qantir, in the eastern Nile delta (Schoer and Rehren, 2007). Samples with the prefix “AM” are from the site of Amarna, and are a selection of vitrified items associated with glass-making at the site (Shortland et al., 2007a), detailed in Table 2.

4. Discussion

4.1. Identification of a glass furnace at Tell Timai

ATR-FTIR analysis indicates that the material under consideration is glass, and that the spectrum produced is characteristic of silica with some organic substances present. These organic substances could well be from the bricks of the furnace or from materials used during glass production.

SEM-EDS analysis again confirms the presence of silica, as the sample is made up of 24.77% silicon and 48.53% oxygen. The minor elements identified from SEM-EDS analysis could be indicative of the raw materials used in the manufacture of the glass.

The composition of the sample taken from the structure at Tell Timai has confirmed that the structure was used as a glass furnace. When compared to examples of other vitrified items associated with glass manufacture in Egypt, such as ceramic crucibles and samples from the walls of other glass furnaces, the composition of the structure at Tell Timai would suggest that it too is associated with glass manufacturing. The size and shape of the structure would further indicate that it is a glass furnace.

There are many similarities between the composition of the sample from Tell Timai and the typical composition of Roman glass established by Brill (1999), however there are also some differences which can be explained by the sample from Tell Timai originating from the wall of the structure, as a point of interface between glass product and glass manufacturing processes. Of note, higher levels of alumina, potash and iron oxide combined with lower levels of lime are also consistent with a material which is fundamentally a ceramic. High aluminium content could be a consequence of contamination from the sample being adhered to the mud brick wall of the furnace. A sample of glass from Tell el-Amarna which was discovered adhered to a ceramic vessel was found to have a similarly high aluminium content of 9 wt%, and this high content was attributed to contamination from the ceramic vessel (Shortland et al., 2007a). The similar compositional traits of the Tell Timai and Qantir samples, in particular of high aluminium, iron, and to some extent potassium, would demonstrate that the two samples represent the interface between glass and glass-making: in the case of GT175, a ceramic glass-making crucible, and in the case of the sample from Tell Timai, a glass-making furnace.

Table 1
Composition of the Tell Timai sample in both percentage weight and percentage oxide.

% Oxide	
SiO_2	54.12
Al_2O_3	9.23
Fe_2O_3	5.94
MgO	3.16
CaO	5.62
Na_2O	4.18
K_2O	2.77
CO_2	15.1
Total	100.12

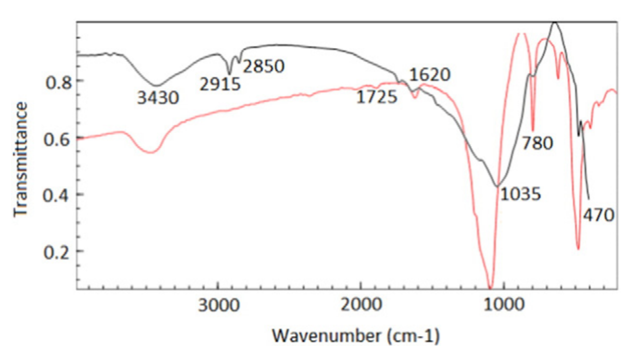

Fig. 6. Infrared spectrum of amorphous silica (red) (after National Institute of Standards and Technology (NIST) Chemistry WebBook) compared to infrared spectrum of sample (black).

Table 2
Chemical composition of the sample from Tell Timai and examples of comparable vitreous materials.

Sample	Material	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	Sb_2O_5	CuO	PbO	CoO	BaO	SnO_2	SrO	ZnO	Cl	SO_3
Tell Timai	Kiln wall	63.9	10.9	7.0	3.7	6.6	4.9	3.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
GT175	Ceramic	54.9	9.9	4.7	2.5	5.0	12.1	3.2	0.1	0.8	0.9	0.3	3.1	0.0	0.0	0.0	0.2	0.0	0.0	0.3	0.1
AM22	Ceramic	56.4	15.6	9.2	0.3	10.6	3.4	2.7	0.0	0.0	1.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM23	Fused clay	59.2	15.9	10.8	1.5	8.6	0.0	1.7	0.0	0.0	2.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM24	Fused clay	59.6	16.4	12.9	2.0	4.9	0.0	1.7	0.0	0.0	2.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM25	Fused clay	64.2	15.1	10.4	1.5	5.0	0.0	1.7	0.0	0.0	2.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM26	Fused clay	60.3	17.6	10.8	1.6	5.6	0.0	1.7	0.0	0.0	2.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM29	Fused clay	58.5	15.7	10.4	0.9	5.7	1.2	3.5	0.0	0.0	4.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM31	Ceramic	60.5	17.4	11.2	1.0	2.8	5.2	1.4	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM33	Ceramic	57.6	12.6	8.9	1.7	8.9	6.9	1.4	0.0	0.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM42A	Kiln wall	62.9	14.3	10.2	3.0	4.6	1.5	1.6	0.0	0.0	1.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM42B	Kiln wall	63.1	13.6	9.5	2.9	6.2	1.5	1.4	0.0	0.0	1.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AM42C	Kiln wall	60.0	15.7	11.3	3.2	4.9	1.6	1.3	0.0	0.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

4.2. The Tell Timai glass furnace in terms of glass manufacture in Graeco-Roman Egypt

When the atomic percentage elemental composition of typical Roman glass is compared to the composition of the sample found at Tell Timai, it can be seen that they are very similar (Fig. 7). However, there are some notable differences. The sample from Tell Timai is lower in sodium, the presence of higher levels of potash (K_2O) indicates the use of potash as an alternative flux agent (Parodi, 1908; Turner, 1956). It would appear that the Tell Timai sample is not an example of typical Roman 'natron' glass, which has concentrations of MgO and K_2O lower than 1.5% (Scott and Degryse, 2014). However it has also been argued that higher levels of potash may also be explained by contamination from the fuel source in the glass furnace (Caley, 1962; Geilmann, 1955).

The Tell Timai sample is also lower in calcium than typical Roman glass. It may be that sand used at Thmuis for glassmaking was desert sand rather than beach sand, which would not contain as much calcium carbonate. It could also be due to the addition of magnesia as a stabiliser, rather than lime (CaO), during the glass-making process. It has also been posited that the presence of lime is an incidental inclusion in the sand used for glassmaking, rather than a deliberate addition as a stabiliser (Nicholson and Henderson, 2000).

Also, the Tell Timai sample is higher in magnesium and aluminium. The difference in magnesium may again be due to the use of magnesia as a stabiliser rather than lime. In a study undertaken in 1961, Sayre and Smith found that Egyptian glass could be characterised as belonging to a group of soda-lime-silica glass with high magnesia (Sayre, 1965). However glasses which are high in both magnesium and potassium can be indicative of a plant ash based glass, common in Late Bronze Age Egypt and Mesopotamia (Shugar and Rehren, 2002). It has also been found that high levels of magnesium with high levels of aluminium in Roman glass are indicative of the recycling of glass, with these elements being added in the form of magnesia and alumina from the use

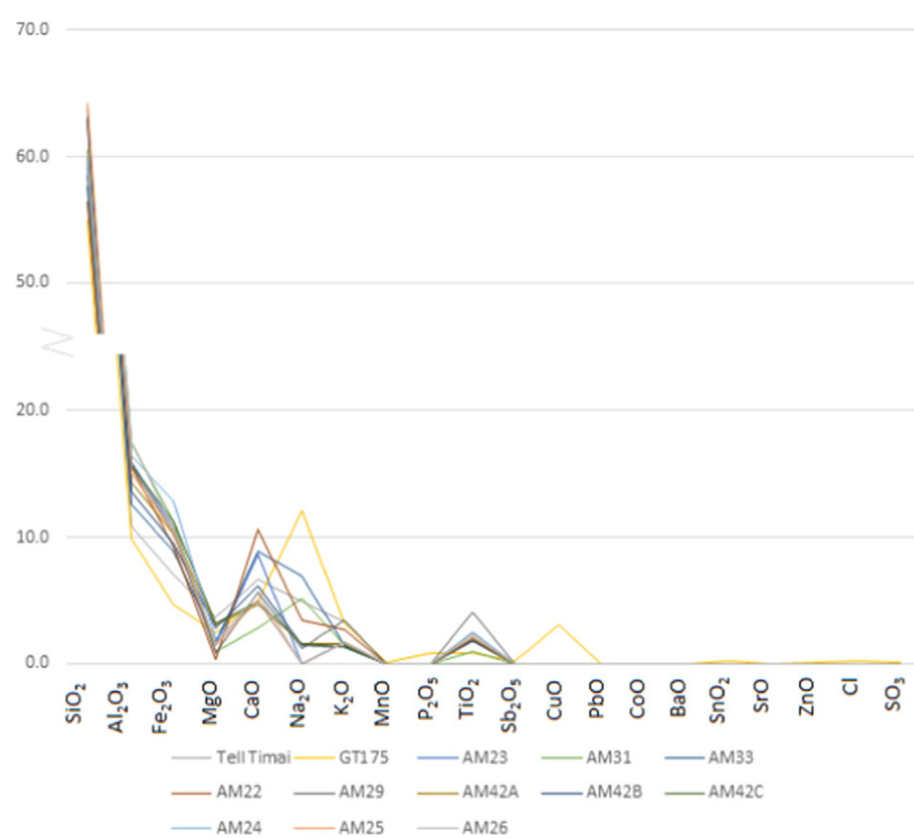

Fig. 7. A comparison of the elemental composition of the sample from Tell Timai, and the interface surface of a number of vitreous materials associated with glass-making.

of soda plant ash to improve the viscosity of the recycled material (Dussart et al., 2004).

Roman glass is thought to have been produced at several primary glass manufacturing workshops throughout the empire, after which it was dispersed in ingots amongst smaller, secondary workshops across the empire, to be worked into specific objects (Freestone et al., 2002; Davison and Newton, 2008). Principally among these primary manufacturing workshops are those on the Syro-Palestinian coast and the Egyptian Nile delta at Wadi el-Natrun, due to their proximity to raw materials, specifically good quality beach sand and natron, respectively (Ceglia et al., 2013; Degryse and Schneider, 2008; Davison and Newton, 2008). Tell Timai is roughly 140 km from Wadi el-Natrun, which supports the theory that the furnace discovered at Tell Timai is a small manufacturing enterprise, supplied with ingots of glass from the larger, primary manufacturer at Wadi el-Natrun, and possibly recycling glass from the local area into new objects.

5. Conclusion

The furnace discovered at Tell Timai has been demonstrated to be a glass furnace based on ATR-FTIR spectroscopy and SEM-EDS analyses. Due to its structure, proximity to major glassmaking workshops at Wadi el-Natrun, as well as the presence of high levels of magnesium and aluminium, we posit that this furnace was a small scale glass workshop. In this furnace, glass was probably worked into specific objects from ingots of glass imported most probably from Wadi el-Natrun, or recycled glass objects from more local sources. Since Thmuis was a major centre for the production of perfume, especially the famous Mendesian perfume that was exported throughout the Mediterranean (Blouin, 2014), it is likely that there was a thriving local industry for the production of perfume jars, both in glass and ceramic, of which this glass furnace is an example.

When the chemical composition of the sample of vitrified brick from Tell Timai is compared to that of the internal surface of a glass-making crucible from Qantir, it can be seen that the vitrified brick, like the crucible, represents the interface between glass and glass-making.

The implications of this discovery are significant. The evidence presented here further demonstrates that glass was made in Roman Egypt at primary production centres and then shaped and worked at secondary production centres. Locally, a single glass furnace such as the one discovered at Tell Timai was likely used to shape ingots provided from primary production centres, and to recycle glass from the surrounding area.

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