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Chronology of early Islamic glass compositions from Egypt

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ABSTRACT

Compositional data of archaeological glasses offer an opportunity to trace the movement of materials in the ancient and medieval world. The lack of a comprehensive record of well-dated samples from Egypt, one of the major producers of glass throughout the first millennium CE, has limited the systematic application of a chronological and geographical model. Here, we report LA-ICP-MS results of Islamic glass weights and stamps from Egypt that provide a precise record of high temporal resolution of glass compositions from Abd al-Malik's monetary reform in 697 CE to the year 1020 CE. This period covers several radical changes in Islamic glass-making. After a significant hiatus in the production of glass weights between the last quarter of the ninth and the middle of the tenth century we see the advent of novel glassmaking recipes based on the use of soda-rich plant ash. Our study establishes a temporal model that serves as a tool for dating archaeological glass assemblages, as well as a geographical model that allows for a clear classification of Levantine and Egyptian plant ash glasses.

1. Introduction

Small glass discs with Arabic inscriptions present an important record of the early Islamic economic system as well as the organisation of the glass industry (Balog, 1976; Morton, 1985). For more than 150 years, scholars have debated the exact function of these artefacts (Fig. 1A) that were made in Egypt between the Umayyad (685–705 CE) and Mamluk (1250–1517 CE) periods (Fenina, 2016). While it is by now accepted that the Umayyad and Abbasid glass weights served as coin weights, their function during the Fatimid and later caliphates may be more accurately described as money weights (Bates, 1981). Based on the inscriptions that in addition to the exact denomination often carry the name of the official sponsor, director of finances or caliph, many of these glass weights can be dated with relative precision and as such are extremely well suited to trace the chronological developments of glass compositions. Egypt was one of the most important glass manufacturing centres from the Late Bronze Age through to the early modern period and may thus hold the key to understanding the changes that led to new glass making recipes in the latter part of the first millennium CE. Chemical analysis and archaeological excavations showed that the primary production of Roman and late antique glasses was centrally organised in a limited number of workshops close to suitable silica sources either on the Palestinian coast or in Egypt itself (Foy, 2017),

and relied on the use of mineral soda sourced for the most part from the Wadi el Natrun in the western Nile Delta of Egypt (Shortland et al., 2006). In the ninth century CE, this system of production disintegrated. The use of mineral soda as the fluxing agent was gradually abolished in favour of soda-rich plant ash at different rates in the Levant and in Egypt (Picon et al., 2008; Whitehouse, 2002). Exactly when and how these transformations of the glass industry in the eastern Mediterranean took place is still unsolved. A continuous chronology of glass compositions throughout the Umayyad, Abbasid and into the Fatimid periods in Egypt can therefore yield new insights into the technological transitions in the history of glass making.

Over the past decades, first millennium glass types have been increasingly refined into at least nine primary production groups with distinct compositional characteristics and attributed to specific chronological ranges and geographical origins (Freestone et al., 2018; Picon and Vichy, 2003; Schibille et al., 2016). Of these, the Levantine groups are by now well-established (Phelps et al., 2016 and references therein). Owing to restrictions on the export of archaeological materials from Egypt, however, the definition of the Egyptian glass categories still relies on very limited data obtained more than 30 years ago. At the time, a preliminary analytical study of early Islamic glass weights and stamps was realised using Fast Neutron Activation Analysis (FNAA) (Gratuze, 1988; Gratuze and Barrandon, 1990). This investigation that

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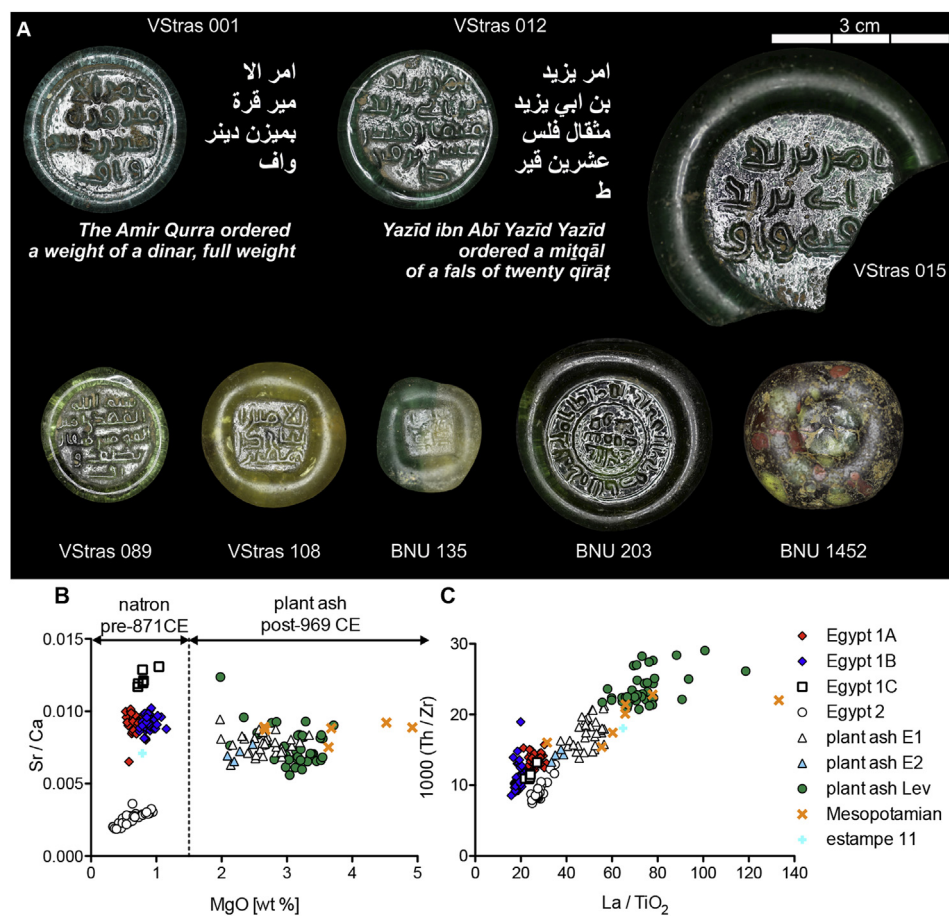


Fig. 1. LA-ICP-MS analysis of Islamic glass weights and stamps dating to 700–1020 CE. (A) Selection of glass weights from the Bibliothèque nationale et universitaire in Strasbourg, representing different compositional groups and dates; (B) Sr/Ca ratios and MgO concentrations distinguish different groups of natron and plant ash glasses; (C) Th/Zr and La/TiO₂ ratios separate Egyptian from Levantine glasses based on their heavy element concentrations.

built on the earlier studies of Sayre and Smith (1974) revealed four successive compositional groups that have been widely used as a benchmark, even though the analytical data have not been published. It has however become apparent that this characterisation of Egyptian glass groups is no longer sufficient for detailed socio-economic and geopolitical analysis. Problems include the limitations of the analytical method, the small number of samples and trace elements considered, as well as the inherent challenges of a finite archaeological record.

To refine the chemical composition of Egyptian glasses at high temporal resolution, we analysed 171 glass weights and stamps ranging from the reign of the Umayyad caliph Abd al-Malik (685–705 CE) to the reign of the Fatimid caliph al-Hakim (996–1020 CE). No workshops are known, but it is thought that the glass weights and stamps originated ultimately from Egypt, since they bear the names of Egyptian officials and most of the samples with known provenance were in fact retrieved from Fustat (ancient Cairo) (Ollivier, 2019). There is no evidence about how or by whom these glass weights, vessel stamps and large commodity weights were produced or under what circumstances. What seems certain is that their manufacture was strictly controlled by the official mint and officials of the treasury (Noujaim-Le Garrec, 2004). The samples for this study stem from two of the largest European collections of Islamic glass weights and stamps, from the Bibliothèque nationale et universitaire (BNU) in Strasbourg and the Department of Islamic Arts in the Musée du Louvre in Paris. The samples further include one object (estampe 11) from a private collection dating to the reign of Abd al-Malik (685–705 CE), representing the earliest glass weight analysed in this study. Our investigations offer a temporal and geographical model for the use of glass in Egypt and by extension a good approximation for the developments in glass production that can be applied to archaeological glass assemblages from consumer sites. The high temporal resolution of the artefacts allowed us to examine the

correlation between the compositional glass groups and archaeological and historical hypotheses. Our results thus elucidate how the changing political, social and cultural landscape in ninth-century Egypt affected long-distance exchange of raw materials and glass, and how this may have driven technological innovations and the development of new glassmaking recipes.

2. Materials and methods

2.1. Glass weights and stamps

170 samples of glass weights and vessel stamps were selected from the collections at the Bibliothèque nationale et universitaire in Strasbourg (BNU, $n = 137$; Ollivier, 2019) and the Department of Islamic Arts in the Musée du Louvre ($n = 33$; Noujaim-Le Garrec, 2004). The number of individual analyses amount to 220 because of the inclusion of some polychrome samples and the separate analysis of the vessel fragments still attached to the back of the vessel stamps (Fig. 1A, Table S1). To capture the onset of Islamic glass production, one extra sample from a private collection was included because it coincides with the reign of the caliph Abd al-Malik (685–705 CE) and his monetary reforms. Contextual details and complete analytical data of all the samples are given in Table S1 in the supplementary material. 32 of the objects from the BNU (29 weights and 3 stamps) had been analysed previously by Fast Neutron Activation Analysis (FNAA) thirty years ago (Gratuze, 1988; Gratuze and Barrandon, 1990).

2.2. LA-ICP-MS analysis

Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) was performed at IRAMAT-Centre-Ernest-Babelon (Orléans)

using a ThermoFisher Element XR combined with a Resonetic UV laser microprobe (ArF 193 nm) as detailed previously (Gratuze, 2016; Schibille et al., 2016). This analytical technique was chosen as it enables the quasi non-destructive determination of a large number of elements (major, minor as well as trace elements) in a single analysis. Using our analytical protocol, we routinely analyse fifty-eight elements, including all major and minor constituents of glass (Si, Na, Ca, K, Al, Mg, Pb, Cl, P, Fe) that identify the raw materials and recipes; most of the colouring and opacifying agents and their associated impurities (Co, Cu, Sb, Sn, Mn, Zn, Ni, As, Ba, Cr, V, In, Bi, Au, Ag, Se, Cd); as well as a large number of trace elements ranging from lithium to uranium that are important for provenancing glass (Li, B, Ga, Ru, Sr, Cs, Zr, U, Th, REE). Single-point analyses of the unprepared solid samples were carried out with a laser beam diameter ranging from 40 μm to 100 μm in order to avoid saturation of the detector by mono-isotopic elements such as manganese or elements present in the form of inclusions such as tin oxide. The laser was operated at 5 mJ with a pulse frequency of 10 Hz. A pre-ablation time of 20 s was occasionally increased to 40 s to ensure the removal of possible corrosion layers or other surface contaminations. Surface enrichment with potash from fuel ashes was observed in some cases. Collection time was set at 30 s. Blanks were run every ten samples to determine the offset. The response coefficient (K) for each element was calculated based on a set of five glass standards (NIST SRM610, Corning B, C, D, and the in-house standard APL1) to convert the signals into quantitative data. Corning A and NIST 612 glass standards were systematically measured at regular intervals to determine accuracy and precision. The analytical precision as reflected in the relative standard deviation (SD) is better than 5% for most elements and the accuracy was generally better than 10% (Table S2).

3. Results

Our analytical data confirm that the overwhelming majority of the Islamic glass weights and stamps analysed in this study correspond to Egyptian glass groups. Specifically, we have identified eight different base glasses: four natron-type groups, dating to the eighth and ninth centuries, and four plant-ash categories that date to the second half of the tenth and the early eleventh century. Strontium to calcium ratios in relation to magnesia contents give a broad overview of the different base glass categories (Fig. 1B). The earlier natron-type glass weights (pre-871 CE) that were produced using a mineral soda are easily distinguished from soda-rich plant ash glasses (post-969 CE) on account of their lower MgO (< 1.5%) levels. The differential Sr/Ca ratios of the natron glasses reflect the carbonate fractions in the silica source. Higher strontium levels separate Egypt 1A, B and C (Sr/Ca \approx 0.008–0.013) clearly from Egypt 2 with significantly lower strontium contents relative to calcium (Sr/Ca \approx 0.003). The strontium in the plant ash glasses (MgO > 1.5%) derives mostly from the plant ash and are relatively constant (Sr/Ca \approx 0.008). To further classify the glasses into Egyptian and non-Egyptian groups, we compared trace elements associated with the silica source such as thorium, zirconium, lanthanum and titanium (Fig. 1C). Egyptian natron glasses are usually characterised by high heavy element contaminations. The ratios of Th/Zr and La/TiO₂ of Egyptian glasses accordingly differ from those of natron glasses originating from the Levantine coast that have overall lower heavy element impurities (Freestone et al., 2018). Even though Islamic plant ash glasses have thus far eluded a clear distinction between Levantine and Egyptian origins, the criteria differentiating natron-type glasses support a separation into Egyptian and Levantine plant ash groups (Fig. 1C).

3.1. Characteristics of Umayyad and Abbasid natron-type glass groups

Principal component analysis (PCA) of six base glass elements (Na₂O, MgO, Al₂O₃, CaO, TiO₂, ZrO₂) clearly separates the four main compositional groups of natron-type glasses (Fig. 2A). Egypt 1A forms a tight cluster in the principal component space of PC1 and PC2 that

account for over 80% of the total variance, and differs from Egypt 1B due to its lower Al₂O₃, MgO, TiO₂, and ZrO₂ contents, while the two groups match each other in their CaO (Fig. 2B) and Na₂O concentrations (Table 1). The trace element profiles of Egypt 1A and 1B follow very similar overall patterns, but those of Egypt 1A are on average by a factor of 1.5 lower. A small group of five samples is tentatively referred to as Egypt 1C because it has a clear compositionally affinity with Egypt 1A and 1B in terms of most major, minor and trace elements except for higher CaO and Sr and lower Al₂O₃ levels and higher heavy elements relative to REE contents (Figs. 1B, 2A–D). These characteristics suggest the use of a different silica source and thus different production location in the coastal region of Egypt, even if the contamination by trace elements associated with colouring agents (e.g. Cu, Zn, Sn, Pb) is somewhat elevated in the samples of group Egypt 1C (Table S1). The main distinguishing features of Egypt 2 are its comparatively high CaO levels coupled with low Sr and Al₂O₃, and high Zr to Ti ratios (Fig. 2C). Egypt 2 has overall lower trace and rare earth elements (REE) except for the heavy elements zirconium and hafnium. The Na₂O concentrations separate Egypt 2 into two subgroups, one with on average 16.5% Na₂O, the other with about 13.5% Na₂O (Table 1). While the silica levels remain constant, all other elements associated with the silica source are substantially higher in the sub-group with lower soda contents (Fig. S1), reflecting changes in the raw materials and/or production technologies. All four natron glass groups have exceptionally low variability in their trace element concentrations, which corroborates group affiliation.

Three samples can be considered outliers, including the oldest sample (estampe 11) that is attributed to the turn of the century (685–705 CE). With high Al₂O₃ and low TiO₂ values and elevated levels of manganese and colouring agents such as copper and lead (Table S1), this sample exhibits the characteristics of a recycled natron-type Levantine base glass (Fig. 1B (Barfod et al., 2018)).

3.2. Characteristics of Fatimid plant-ash glass groups

Multivariate principal components 1 and 2 of nine base glass elements (Na₂O, MgO, Al₂O₃, P₂O₅, K₂O, CaO, TiO₂, Cr₂O₃, ZrO₂) distinguish four compositional plant ash categories (Fig. 3A). The largest group (plant ash Lev) has moderate Al₂O₃ (< 2%), low TiO₂ and ZrO₂ and somewhat higher MgO and K₂O levels than the plant ash glass groups E1 and E2 (Fig. 3A–C). Direct comparison with published data of 81 plant ash glasses attributed to the Levant (Phelps, 2018) shows that the former agree well with Levantine plant ash glasses (Fig. S2). Conversely, an Egyptian provenance can be assumed for plant ash glass groups E1 and E2 due to the elevated heavy element concentrations (Fig. 1C) and higher TiO₂ to Al₂O₃ ratios (Fig. 3B). These different ratios proved effective in the separation of Levantine and Egyptian natron glass groups (Schibille et al., 2017), and they appear to account in part also for regional variations in plant ash glasses from these regions. A group of samples with low Al₂O₃ contents (< 1%) and high TiO₂ to Al₂O₃ ratios (encircled in Fig. 3B) seem at odds with this interpretation, suggesting that these criteria are only applicable to plant ash glasses produced from common sands and not to glasses made from quartz pebbles or quartz rich sands that are poor in mineral impurities. Plant ash E2 has particularly high Al₂O₃, TiO₂, and ZrO₂ and trace elements akin to those of natron glass group Egypt 2 (Fig. 1C). Both Egyptian plant ash groups exhibit elevated levels of colouring and opacifying elements (e.g. Cu, Zn, Sn, Sb, Pb) that are indicative of recycling procedures. This is not entirely unexpected given the existence of polychrome and multi-compositional weights and stamps among the plant ash glasses, some of which might have ended up in the melting pot for recycling. What is surprising is that the Levantine plant ash glasses were not affected by recycling processes to the same extent, suggesting a relatively fresh consignment of raw glass from the Levantine coast. A fourth plant ash group can be ascribed to a Mesopotamian provenance identified based on their high MgO (> 3.5%) and low P₂O₅ (< 0.3%)

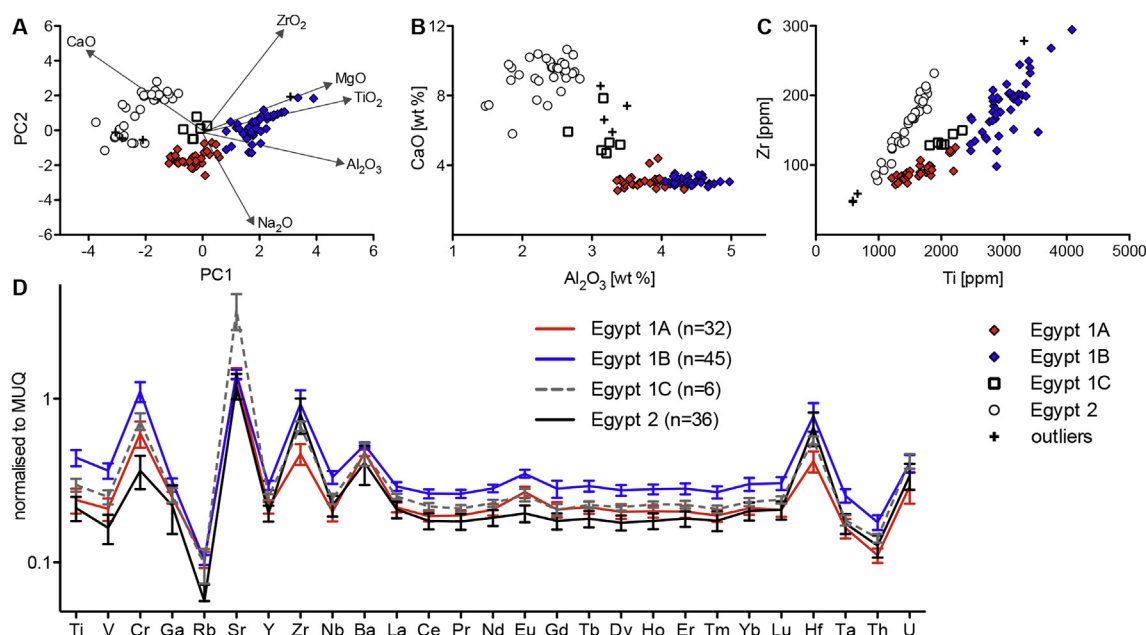


Fig. 2. Base glass characteristics of the natron-type glass weights and stamps. (A) Principal components (PC) 1 and 2 separate the four main compositional groups; coefficient vectors are given on an enlarged scale (10x); (B) lime versus alumina levels highlight the main differences between Egypt 1A, B, C and Egypt 2 glasses; (C) zirconium to titanium concentrations display different correlations between the groups; (D) average trace element and REE patterns of the four identified natron-type glass groups normalised to the upper continental crust (MUQ (Kamber et al., 2005)); error bars represent the standard deviations (SD) of the individual groups.

Table 1

Average compositional characteristics of the main glass groups. Metal oxides and chlorine concentrations are given in wt %, strontium and zirconium are given as elements [ppm].

	Other names	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	Cl	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	Sr	Zr
Egypt 1A (n = 32)	Egypt I (Freestone et al., 2000)	16.6	0.64	3.75	72.4	0.09	1.02	0.59	3.08	0.28	0.03	1.13	201	91.5
SD	Group 9 (Foy et al., 2003) N2-a2 (Kato et al., 2009)	1.1	0.07	0.26	1.7	0.05	0.09	0.14	0.36	0.05	0.01	0.15	15	13.6
Egypt 1B (n = 45)	Egypt I (Freestone et al., 2000)	16.0	0.87	4.38	71.6	0.083	0.98	0.50	3.07	0.50	0.042	1.82	201	185
SD	Groupe 8 (Foy et al., 2003) N2-a2 (Kato et al., 2009)	1.0	0.08	0.21	1.2	0.026	0.08	0.07	0.17	0.06	0.002	0.35	13	40
Egypt 1C (n = 6)		15.9	0.81	3.14	70.3	0.16	0.96	0.73	5.64	0.34	0.44	1.27	497	136
SD		0.4	0.12	0.25	1.0	0.07	0.08	0.09	1.16	0.03	0.47	0.11	123	9
Egypt 2 < 815 CE (n = 12)	Egypt II (Freestone et al., 2000)	16.5	0.47	2.00	69.7	0.10	1.08	0.33	8.51	0.20	0.045	0.84	139	120
SD	Groupe 7 (Foy et al., 2003) N2-b (Kato et al., 2009)	1.0	0.09	0.31	1.9	0.05	0.13	0.09	1.32	0.03	0.083	0.31	17	25
Egypt 2 > 815 CE (n = 24)	Egypt II (Freestone et al., 2000)	13.4	0.70	2.52	70.1	0.11	1.04	0.51	9.57	0.27	0.44	1.18	187	181
SD	Groupe 7 (Foy et al., 2003) N2-b (Kato et al., 2009)	0.6	0.15	0.20	1.4	0.05	0.10	0.25	0.54	0.03	0.47	0.32	21	27
plant ash E1 (n = 30)		12.7	2.68	2.23	67.8	0.39	0.76	2.19	8.86	0.14	1.00	0.87	490	64.7
SD		0.7	0.32	0.21	1.3	0.14	0.06	0.31	0.98	0.02	0.66	0.15	60	10.2
plant ash E2 (n = 4)		15.3	2.26	2.88	66.9	0.31	1.03	1.65	7.61	0.24	0.44	1.19	388	126
SD		0.5	0.16	0.13	0.8	0.03	0.06	0.84	0.28	0.02	0.06	0.06	40	10
plant ash Lev (n = 40)	Levantine plant ash (Phelps, 2018)	13.2	3.09	1.62	67.6	0.32	0.84	2.64	8.96	0.083	0.96	0.48	492	36.1
SD		1.3	0.39	0.41	1.1	0.05	0.12	0.36	0.89	0.014	0.57	0.09	96	3.9
Mesopotamian (n = 8)		13.9	3.95	2.05	66.5	0.22	0.70	2.71	6.77	0.13	0.87	1.53	423	73.6
SD		1.2	1.14	1.21	3.1	0.07	0.11	0.56	1.04	0.08	0.65	0.95	60	32.4

contents. It is highly variable and represents different Mesopotamian glassmaking traditions (Schibille et al., 2018). The samples with low Al₂O₃ and CaO were made from a relatively pure quartz-rich silica source, the others show higher silica-related contaminants. Mesopotamian plant ash glasses often but not always contain higher chromium levels.

The separate analysis of some white and yellow patches in a number of samples indicate the combined use of tin and/or antimony and lead as colorant and opacifier (Table S1). The yellow and green phases decorating samples BNU 1450, 1451 and 1453 are high lead glasses (PbO > 60%) *sensu stricto*, meaning that they represent a simple mixture of lead, tin and silica and only negligible amounts of soda and lime.

In contrast, the reduced and normalised compositions of some of the white decorations match the base glass compositions of the respective artefact as in the case of three samples attributed to Egypt 2 (VStras 119, 123, 124). Two bulky multi-coloured Fatimid samples (BNU 1452, 1453) present an intriguing example of multiple glass types combined in the same object. For example, the greenish amber coloured body of sample BNU 1452 (Fig. 1A) is made from Levantine plant ash glass, whereas the red, yellow and green patches show the characteristics of Egypt 1A that pre-dates the manufacture of the weight by at least three centuries (Table S1).

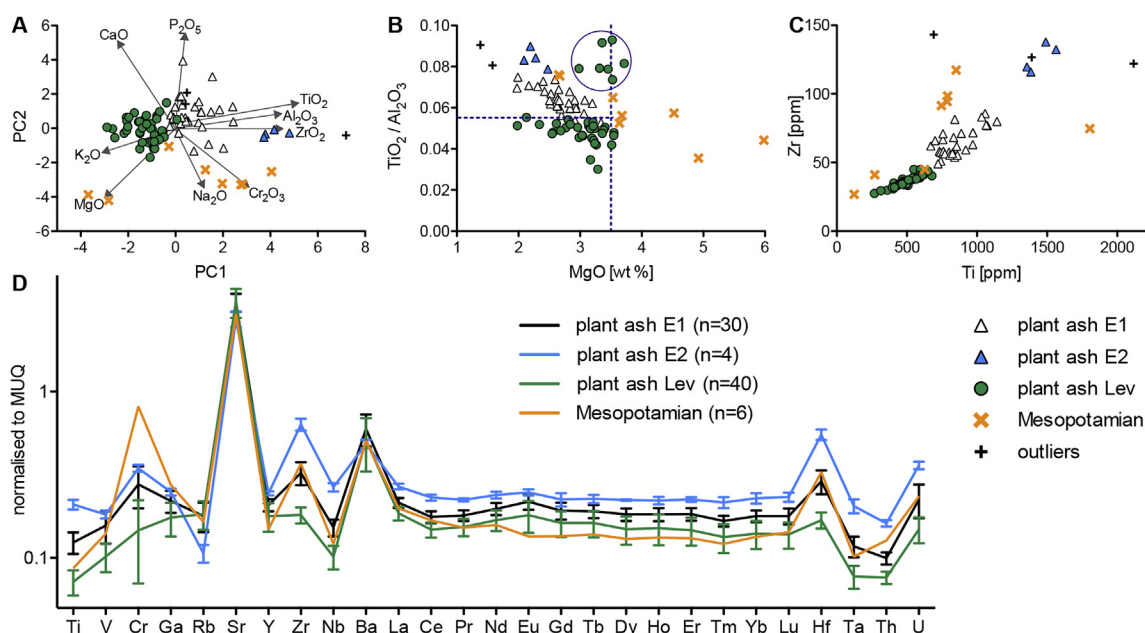


Fig. 3. Base glass characteristics of the soda-rich plant ash glass samples. (A) PC1 and PC2 account for 60% of the variance and distinguish between four compositional groups based on nine elements; coefficient vectors are shown on a 10x scale; (B) titanium to aluminium oxide ratios and magnesia levels suggest a subdivision into Levantine (lower left, $TiO_2/Al_2O_3 < 0.055$), Egyptian (upper left, $TiO_2/Al_2O_3 > 0.055$) and Mesopotamian plant ash glasses (right, $MgO > 3.5$ wt %); (C) zirconium and titanium contents reveal differential contamination levels; (D) average trace element and REE patterns of the four main plant-ash groups normalised to the upper continental crust (MUQ (Kamber et al., 2005)); SD are represented by error bars.

3.3. Spatio-temporal model

Next, we examined the chronological attribution of the samples. A temporal model based on the names given on the weights and stamps identified a remarkable match between the compositional groups and their chronology at least during the Umayyad and Abbasid periods with minimal overlap (Fig. 4). The glass weights of the Egypt 1A group date to the first quarter of the eighth century (< 725 CE) with an average temporal precision of 5 years across 18 samples, calculated using the time spans during which the officials named in the inscription were in office. The average precision across 30 Egypt 1B artefacts is likewise 5 years, while the average precision across Egypt 2 is in the order of 20 years. This chronological precision allows for a more accurate interpretation of analytical glass data than ever before. Our analytical model thus provides exact dates for the changes in the use of primary glass groups for the manufacture of official glass weights and by extension a very good temporal estimate for the technological transitions in Egyptian glass production.

The transition from Egypt 1A to Egypt 1B happened between 720 and 725 CE (Fig. 4), during which time a certain Yazid ibn Abi Yazid was the director of finances (Noujaim-Le Garrec, 2004). While one of

the weights with his name (VStras 015) corresponds to Egypt 1A, the others (e.g. VStras 012) already show an Egypt 1B signature (Fig. 1A). Between 720 and 780 CE, Egypt 1B was the principal base glass employed for the production of official glass weights and stamps. Three out of the five Egypt 1C samples (VStras 138, 140, 141) belong to an anonymous series with a short and simple pious legend (*In the name of Allah. My Lord is Allah*) (Ollivier, 2019). Samples similar to VStras 138 in the British Museum have been attributed to the eighth century based on the overall frequency of half- and third-dinar weights during this period (Morton, 1985, no. 512). The same die appears to have been used for VStras 139 and VStras 140, despite different base glass compositions. The other two Egypt 1C samples (VStras 120, MAO 1134) are exceptional with no known unambiguous parallels. Sample VStras 120 is tentatively assigned to the middle of the eighth century (737–747) (Ollivier, 2019), while MAO 1134 is attributed to the beginning of the ninth century (Noujaim-Le Garrec, 2004). Given the small number and uncredited nature of the samples, and the compositional characteristics that deviate from the official types, Egypt 1C may have been produced outside the official run of glass weights, perhaps in a provincial centre (Morton, 1985). After 780, Egypt 2 was virtually the only glass type used for weights and stamps up to and including the third quarter of the

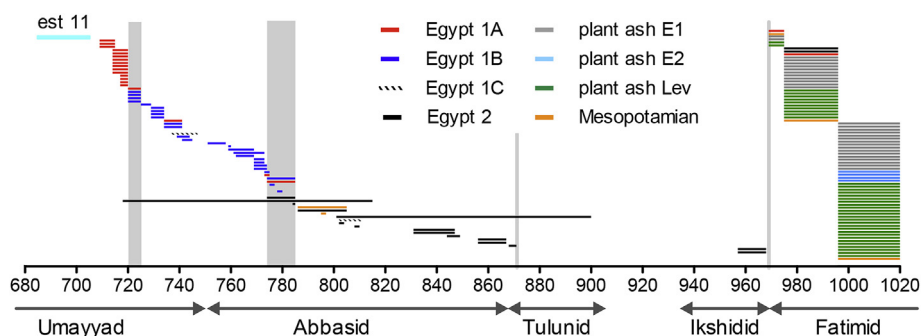


Fig. 4. Temporal model of compositional glass groups. The chronological sequence of compositional data based on well-dated objects exposes four specific production transitions (highlighted in grey).

ninth century. Egypt 2 is in fact the last natron-type glass manufactured in Egypt. A decrease in soda levels sometime in the early ninth century CE suggests that during the production of Egypt 2 the availability of soda might already have been an issue. One Egypt 2 sample (VStras 170) has been given a provisional date of 758–760 CE (Table S1) due to its similarities with an object in the collection of the American Numismatic Society (Ollivier, 2019). However, the inscription on the sample from the BNU is illegible and its date is thus not secured. It is therefore not taken into account for the temporal model. The epigraphic evidence places two vessel stamps with an Egypt 2 signature (VStras 187 & 188) to the middle of the ninth century or later (Morton, 1985, no. 527–528). If we accept the isolated *kāf* barely visible on VStras 187 to be identified with the name of Kafur, chamberlain and then regent of Egypt (966–968 CE), these two vessel stamps may be attributed to after the middle of the tenth century instead (957–968 CE, Fig. 4) (Bacharach, 2006). In this case, the material likely represents recycled glass, which could also explain the elevated contents of copper and lead in sample VStras 188 (Table S1).

There is a remarkable interval of 98 years between the period of the Tūlūnids and the beginning of the Fatimid caliphate (969 CE) to which no glass weight or stamp from Egypt has yet been attributed based on a survey of 65 catalogues and over 5000 entries (Ollivier, 2019). The appearance of plant-ash glasses in the manufacture of weights and stamps coincides with the reign of the first Fatimid caliph al-Mu'izz (953–975 CE) who moved from Tunisia to Egypt after the foundation of Cairo in 969 CE (Fig. 4; Table S1). Glass weights bearing the name of the caliph al-Mu'izz as well as his successor al-'Aziz have been produced from multiple base glasses, including recycled Egypt 1A (MAO 999, 1148) and Egypt 2 (BNU 154, 165), Levantine and Egyptian plant ash glasses and two samples of possible Mesopotamian origin (Fig. 4). The commencement of plant-ash recipes was evidently characterised by a shift from a single principal glass group to multiple sources reflected in the simultaneous use of several glass types, some of which appear to have been imported. What is more, a larger number of glass weights (tens of thousands) survive from the Fatimid era, suggesting that production of these objects had increased dramatically.

To corroborate our temporal and geographical model, a comparison with published data of vessel glass from Tebtynis and Fustat (Foy et al., 2003) and the Sinai Peninsula (Kato et al., 2009) offers a promising strategy. Picon's group 7 and Kato's group N2-b correspond to our group Egypt 2, Picon's group 8 and Kato's group N2-a2 are equivalent to Egypt 1B, while Picon's group 9 is comparable to Egypt 1A (Fig. 5a, Table 1). To group 9/Egypt 1A belong some of the earliest Umayyad vessels such as three stemmed goblets from Tebtynis that are dated by the authors to the first half or the middle of the seventh century (Foy et al., 2003). This suggests that glass of the Egypt 1A composition was in circulation prior to its use in Islamic glass weights. The vessels from Fustat (Isabl'Antar) that are affiliated either with an Umayyad typology or

stratigraphy are, without exception, attributed to group 8/Egypt 1B. The N2-a2 samples from Raya are broadly dated to the eighth century, while group N2-b is associated with ninth-century contexts (Kato et al., 2009). The Abbasid vessels from Tebtynis and Fustat that match group 7/Egypt 2 are dated to the late eighth and ninth century CE. These comparisons demonstrate that vessel glass from Egypt follows the same temporal evolution, which is also confirmed by the fact that all the vessel stamps analysed in our study are invariably of the same compositional group as their supports (Table S1). It is worth pointing out that assemblages of Umayyad vessel glass from both the Sinai Peninsula as well as al-Hadir in Syria additionally include contemporaneous Levantine II compositions (Gratuzze and Foy, 2012; Kato et al., 2009). For Umayyad and Abbasid glass weights, in contrast, only Egyptian base glasses were used, suggesting a more local supply.

Our temporal and geographical patterns may now be employed as a dating tool and to provenance glass finds from consumer sites. For example, scholars have long debated the date of the palatial complex of Hirbat al-Minya on the shores of lake Tiberias in northern Palestine since its excavation in the 1930s (Ritter, 2017). The dating of the building and its decoration hinges on the interpretation of a dedicatory inscription referring to the caliph al-Walid who was variously identified either with al-Walid I (705–715 CE) or with al-Walid II (743–744 CE). Among the finds from al-Minya were the largest known corpus of coloured window glass from an early Islamic context as well as thousands of multicoloured glass mosaic tesserae scattered throughout the Umayyad palace, particularly in the three-aisled hall. The analytical investigation of a set of 60 mosaic tesserae and 14 window glass fragments showed that they are mostly made from either fifth-to seventh-century Levantine glass or from Egypt 1A (Fig. 5B). None of the samples can be attributed to Egypt 1B, C or Egypt 2. The presence of Egypt 1A and absence of any later material clearly supports a date prior to 725 CE for the completion and decoration of the palatial complex. The date gleaned from the analytical data is congruent with the observation that the mosaics from Hirbat al-Minya share technical similarities with those of the Dome of the Rock in Jerusalem (691/92 CE) and the Great Mosque in Damascus (705–715 CE) (Ritter, 2017). A contemporaneous execution in the early eighth century is therefore likely.

As for the plant ash glasses, the analytical evidence for Egyptian plant ash glasses in the early Islamic period did not yield a chronological sequence. We can, however, compare the plant-ash signatures of glass assemblages from firmly established origins to identify a geographical model instead. A substantial set of published data from the Monastery of Wadi al-Tur on the southwest coast of the Sinai Peninsula was attributed to a tenth- to eleventh-century Egyptian provenance (Kato et al., 2010). With few exceptions, the monastic finds exhibit TiO_2 to Al_2O_3 ratios that are above the proposed threshold separating Egyptian from Levantine glasses ($\text{TiO}_2 / \text{Al}_2\text{O}_3 > 0.055$, Fig. 5C). It should however be borne in mind that the published data from Wadi al-

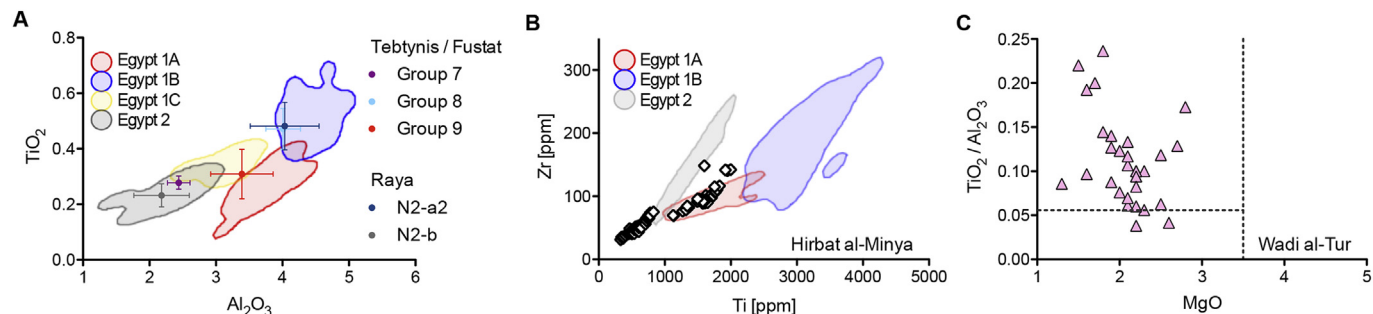


Fig. 5. Application of the temporal and geographical model. (A) 95% kernel density ranges for titanium and aluminium oxide concentrations of natron-type glass weights compared to published data of vessel glass from Fustat and Tebtynis (Foy et al., 2003) and Raya (Kato et al., 2009), represented by their means and standard deviations; (B) zirconium versus titanium contents of glass samples from Hirbat al-Minya in comparison with 95% kernel density estimates of Egypt 1A, 1B and 2 as defined in this study. (C) TiO_2 to Al_2O_3 ratios compared to MgO contents of published samples from Wadi al-Tur (Kato et al., 2010) support a likely Egyptian origin of the glasses. Kernel density outlines were generated using the open-access RESET statistical tools (<https://c14.arch.ox.ac.uk/resetdb/db.php>).

Tur were obtained by portable XRF spectrometry and that only a limited number of elements were measured. To match archaeological glass assemblages unambiguously will require a more detailed comparison of trace elements, especially of heavy elements such as chromium, lanthanum, thorium and zirconium (De Juan Ares and Schibille, 2017; Swan et al., 2017).

4. Discussion

Our new LA-ICP-MS data of Egyptian glasses are generally consistent with results obtained 30 years ago by Fast Neutron Activation Analysis (FNAA) for most of the elements that were originally used to define compositional glass groups (Al, Ca, Ti, Zr, Fe, Mn, Na, Mg, Si, K, Rb and Ba) (Gratuze, 1988). However, FNAA seems to consistently overestimate the titanium levels and the two analytical methods deviate notably in relation to rubidium, barium, and to a certain extent the sodium levels (Table S3). Despite these differences between the two datasets, only two objects had to be re-classified. In the previous study glass weight VStras 086 (Gratuze n° 18) had been attributed to group 1B due to its dating (774–785 CE), but it has a higher compositional affinity with group Egypt 1A (Fig. S3) and appears to have been manufactured from recycled glass. A bi-coloured stamp (VStras 120/Gratuze n° 66) that was originally an outlier now forms part of the newly identified group Egypt 1C (Fig. S3). Hence, the higher number of samples (23 objects for group 1A instead of 4; 33 objects for group 1B instead of 20), wider range of analysed elements and lower detection limits of our LA-ICP-MS data provide a robust basis for the identification and deep compositional profiling of Egyptian glass groups (Table 1).

The higher strontium levels in groups Egypt 1A, B and C are consistent with the use of maritime sands containing aragonitic seashell as lime-bearing component, and a likely production location in the coastal regions of northern Egypt. As suggested by the low strontium relative to calcium contents, a geologically aged calcitic limestone was the principal source of lime in the Egypt 2 group instead, pointing to the use of a landlocked sand source (Degryse, 2014; Freestone et al., 2003). An eighth- to ninth-century secondary glass-working site where Egypt 2-type glass was processed has been identified in el-Ashmunein in Middle Egypt (Freestone et al., 2009). A production location for Egypt 2 away from any viable natron source in the Wadi el Natrun may also explain the significant decrease in soda levels in Egypt 2 glasses after in the ninth century. This coincides with the period when plant ash-based glassmaking is introduced in the Levant, most notably in Tyre (Phelps, 2018) and Raqqa (Henderson, 2002; Henderson et al., 2004), suggesting limited supply of natron more generally.

Our data demonstrate that in Egypt itself, plant ash glasses appear only after the manufacture of Egypt 2 ceased in the late ninth century CE (Fig. 4). When exactly this occurred is difficult to determine as the earliest glass weight with a plant-ash signature dates to the second half of the tenth century. It is possible that glass was manufactured using plant ash as early as the late ninth century CE, but it is unlikely that plant ash glasses were produced on any significant scale prior to this, since we did not detect any local plant ash glass among the analysed weights and stamps. Only a single plant ash glass sample dates to the end of the eighth century, and it has clear Mesopotamian characteristics (MgO = 4.5 wt %). About half of the tenth-century plant ash glasses analysed have been made using Levantine imports, which supports a later development of the plant ash glassmaking technology in Egypt. This finding fits well with the historical evidence. The port cities along the Levantine coast had been conquered by the Fatimid troupes in 970–971 CE and remained under Fatimid control until the first quarter of the twelfth century (Bramoullé, 2011). The Cairo Geniza documents contain a power-of-attorney deed dated to the year 1011 according to which a merchant in Tyre was waiting for his payment for 37 baskets of glass that had been shipped to Fustat (Gil, 1997). Our study now presents analytical proof for the bulk exchange of glass between the Syro-

Palestinian coast and Egypt during the early Fatimid caliphate.

Based on the chemical characteristics of the glass groups, our temporal model offers a reliable blueprint for Egyptian glasses that can serve as an independent dating tool at least with respect to natron-type glasses. As has been shown, the presence of Egypt 1A and earlier glass groups as well as the conspicuous absence of later samples from the assemblage of al-Minya provides a reliable marker to attribute the glass from al-Minya to the first quarter of the eighth century (pre-725 CE). Hence, in spite of the fact that glass may have been stored over longer periods, recycled and/or re-employed at a later stage, the clearly defined sequence of the glass groups can provide a very good estimate for glass finds from consumer sites. Using this temporal model, the chronology of early Islamic glass assemblages from consumer sites may be further refined. For example, the majority of samples identified as Egypt 2 from ten different sites in Israel were attributed to the early to mid-eighth century Umayyad dynasty (Phelps et al., 2016). Our results together with published data of vessel glass from Egypt provide strong evidence that the Egypt 2 compositional group is an Abbasid glass type that was produced from the second half of the eighth century CE. The earliest securely dated Egypt 2 weight (VStras 089, Figs. 1A and 4A) dates, in fact, to the last quarter of the eighth century (774–785 CE) (Table S1).

The temporal sequence of glass groups breaks down during the Fatimid period when plant ash glass groups begin to multiply and are used simultaneously. The contemporaneous production and secondary working of several plant ash glasses has been observed in numerous other early Islamic contexts (Henderson, 2003; Schibille et al., 2018). Glass weight BNU 135 (Fig. 1A) exemplifies the concurrent use of different plant ash glasses, as it is compositionally split into two: a greenish half with higher heavy elements (Ti, Cr, Zr, Hf) that resembles Egyptian plant ash glass E1, and a colourless part corresponding to the Levantine group (Table S1). The combination of two base glasses in the same object rules out that the compositional variations are due to accidental contaminations through the crucible, and demonstrates that they truly reflect differences in the raw materials and by extension different production locations. Our detailed characterisation of plant ash glasses thus allows to establish a geographical model that distinguishes Levantine and Egyptian plant ash glasses based on the heavy element signatures (Figs. 1C, 3B and 5C), despite the Egyptian plant ash glasses exhibiting some evidence of recycling. This geographical model can be applied to glass assemblages from consumer sites but needs to be carefully tested against glass finds of reliable origin. Very few early Islamic glass assemblages have been analysed with high precision techniques to date, and even less is known about early plant ash glasses from Egypt. This study thus presents an adaptable framework into which future analytical studies can be integrated and evaluated.

Our temporal and geographical model of Islamic glass manufacture in the first three centuries (700–1020 CE) of Islamic rule in Egypt contains important information about the organisation and transformation of the glass industry in Egypt in relation to wider historical developments (Fig. 4). The high temporal resolution of the glass weights and stamps together with the large number of elements determined by LA-ICP-MS have brought to light four transitions in glass making technologies, some of which are seemingly independent of overt geo-political changes. The earliest glass weight analysed in this study is in the name of the caliph 'Abd al-Malik (685–705 CE). Even though it already reflects 'Abd al-Malik's monetary reforms of 697 CE in that its weight corresponds to the reformed weight of the half-dinar (Grierson, 1960), its base glass is a recycled Levantine natron-type glass that was circulated widely between the fifth and seventh century CE. The earliest Islamic weight with a new Egypt 1A chemical signature dates to the governorship of Qurra ibn Sharik (709–715 CE). This may well be happenstance, since it is from his period of office that the most extensive administrative evidence survives (Sijpesteijn, 2007). The first precisely determined transition from Egypt 1A to Egypt 1B can be attributed to the years between 720 and 725 CE. It is difficult to fathom to

what extent the tax rises of 725 CE (Wickham, 2005) may have prompted changes in the raw materials used for the production of glass and its weights. The manufacture of Egypt 1B outlasted the Umayyad caliphate in Egypt and continued to be used as the principal glass until about 775 CE. There is, however, a notable drop in the number of samples dating to the middle of the eighth century (Ollivier, 2019), when the first Islamic dynasty, the Umayyads, was overthrown by the Abbasids in 750 CE.

In Egypt, not much changed at first. The early Abbasid administration appears to have continued Umayyad policies. The same seems to be true for glassmaking. Judging from our data and publications on other glass finds from Egypt, a new production centre for glass was launched only in the second half of the eighth century. The change from Egypt 1B to Egypt 2 falls within the years of the caliphate of al-Mahdī (775–785 CE). Glass weights bearing his name were produced from Egypt 1B, Egypt 2 as well as from recycled Egypt 1A, such a multiplication of compositional types may be expected for a transitional period (Fig. 4). The end point of Egypt 2 coincides with the demise of Abbasid rule in Egypt. Egypt became *de facto* autonomous from the central caliphate government under the Tūlūnids (868–905 CE) and it is during this and the subsequent dynasty of the Ikhshīdids (935–969 CE) that there are no glass weights or stamps preserved. Presumably they were not produced during this period. Glass weights may have been reintroduced by the Ikhshīdīd Kāfūr when he was governor of Egypt (966–968 CE) and more extensively with the establishment of the Fatimid caliphate and the move of al-Muʿizz from Tunisia after the establishment of al-Qahira (Cairo) in 969 CE. Fatimid rule was a period of economic prosperity and from now on, glass weights and stamps were produced from plant-ash base glasses. Some of this material was imported from Syria-Palestine, reflecting the resurgence of Mediterranean trade between the Fatimid-ruled ports on the Levantine coast, Egypt and the Latin West. These wider socio-economic and geopolitical developments raise two main hypotheses. Firstly, the manufacture and administration of glass weights and stamps reflect a high degree of monetization of the Egyptian economy that was under the direct control of the local government, contingent on the central caliphate governments in Damascus (Umayyad), later Baghdad and Samarra (Abbasid). Secondly, it appears that the glass industry may also have been controlled by the local administration. The fragmentation of caliphate powers at the end of the ninth century has been interpreted as underlying the creation of new industrial complexes including glass workshops (Henderson, 2003). This could explain why glassmaking in Egypt developed at a different rate and independently from glassmaking centres in Syria-Palestine. It can furthermore explain the dwindling availability of mineral soda first in the Levant and then in Egypt with increasing distance of the production sites from the natron sources in the Libyan Desert. As such, the compositional groups of Egyptian glasses offer an independent spatio-temporal read-out of technological and administrative changes.

5. Conclusion

The significance of early Islamic glass weights and vessel stamps is their precise dating and Egyptian provenance in view of the dearth of analytical data from Egypt. Our analyses identified and characterised four natron-type and four plant ash glass groups, and allowed us to advance a temporal and geographical model for glass compositions between 700 and 1020 CE. The timeline for Islamic glasses produced in Egypt covers several changes in glassmaking technologies and recipes from the Umayyad and the Abbasid to the Fatimid periods. Differences in the trace element signatures provide compositional markers to distinguish between Levantine and Egyptian plant ash glasses, revealing the resurgence of Mediterranean wide trade under the Fatimid dynasty. The proposed ranges for the manufacture of Egypt 1A (< 725 CE), Egypt 1B (720–780 CE) and Egypt 2 (760/780–870 CE) are broadly consistent with the dating of Egyptian glass vessels that is rarely as

precise as that of the glass weights. An absolute chronology of glass compositions can ultimately serve as an additional dating tool for assemblages from consumer sites, while the geographical differentiation of plant ash glasses ascertains distribution patterns and networks of supply. This study thus presents crucial reference material for archaeological glasses and an adjustable framework for future analytical investigations.

Data and materials availability

All data needed to evaluate the conclusions in the paper are present in the paper and/or the Supplementary Materials. Additional data related to this paper may be requested from the authors.

Competing interests

The authors declare that they have no competing interest.

Author contributions

N.S., B.G. and E.O. conceived, designed and coordinated the project. E.B. provided access to the samples. N.S. and B.G. performed the analyses, processed and analysed the data, prepared the figures and tables and wrote the manuscript.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jas.2019.02.001>.

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