ELECTRON MICROPROBE ANALYSIS OF 9TH-12TH CENTURY ISLAMIC GLASS FROM CÓRDOBA, SPAIN

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Twenty-six samples from domestic assemblages of 9th–12th century Córdoba were subjected to electron microprobe analysis. The results reveal two main compositional types. The first, encountered in 13 of the samples, seems to result from the combination of plant ashes with high-impurity sand, and has some contemporary parallels from Syria and Egypt. The second type is a lead–soda–silica glass, encountered in a relatively high proportion of the glasses (11 of the 26 sampled), possibly formed by the addition of lead metal to existing glasses and with very few known parallels. These are among a very small number of results available to date on the chemical composition of glasses from medieval Spain, and the presence of a high proportion of lead–soda–silica glasses is particularly interesting, possibly indicating a technological practice unique to, or originating in, the western Muslim world.

KEYWORDS: AL-ANDALUS, ISLAMIC GLASS, LEAD GLASS, LEAD–SODA–SILICA, EPMA, *SALICORNIA*, PLANT ASH, GALENA, *BARILLA*, CÓRDOBA

INTRODUCTION

Islamic glass—glass made in regions under the control of followers of Islam from about the 7th to the 14th century (Brill 2001, 25)—has lately received much deserved attention, and several important elemental and isotopic studies have been undertaken, improving our understanding of the technological development and provenance of such glasses (see, e.g., Henderson 2002; Freestone *et al*. 2003; Henderson *et al*. 2009). Islamic Spain, however, with its major centres in Córdoba and Seville, is rarely included in such studies because of its unique geo-historical evolution, and tends to be omitted from studies of European glass due to its political and religious isolation. Two collective volumes and a small number of individual papers have been published (Fernandez-Puertas 1998; Jiménez Castillo *et al*. 1998; Cressier 2000; Rontomé Notario 2006; Rontomé Notario and Pastor Rey de Viñas 2006), largely focusing on the results of archaeological excavation, typological analysis and a small amount of historical evidence, but to the authors' knowledge only one full programme of chemical analysis has been conducted and published to date, on 36 items of 12th century glass from a workshop in Casón de Puxmarina, Murcia, 23 of which were quantitatively analysed by XRF (Carmona *et al*. 2009, originally reported in García Heras 2008). The glasses from the Murcian workshop were found to be of HMG (high magnesia, plant ash) and high-lead compositions (though only two samples fall into the latter category), with sophisticated methods of coloration in the production of silver–yellow and copper ruby red glasses. Semi-quantitative chemical analysis has also been conducted on later, 15th century glass from a synagogue in Lorca (García Sandoval 2010, 264).

Twenty-six samples of late 8th/9th to 12th century glass vessels from sealed archaeological contexts in the city of Córdoba were subjected to electron microprobe analysis wavelength-dispersive spectrometry (EPMA–WDS), in order to determine their major and minor elemental constituents. This is part of a larger project investigating glass in al-Andalus, and is aimed at addressing several related research questions, as follow. Does glass composition reflect interaction with the rest of the Islamic world, and/or with Christian Europe? Which

raw materials were used to make the glass, and what are their provenances? What scale of production occurred within al-Andalus itself, and how does this change over time? All of these questions are relevant to our understanding of the Islamic societies that inhabited the Iberian Peninsula, and to our wider understanding of technological change and development; the broader picture of glass production and exchange; and the mechanisms by which this technological knowledge was passed from generation to generation and region to region.

ARCHAEOLOGICAL AND HISTORICAL BACKGROUND

The principal cities of al-Andalus are shown in Figure 1. In continuation of its former importance in Roman *Baetica*, Córdoba was made the political and administrative centre of al-Andalus soon after the ad 711–13 conquest, following—it seems—a decision taken by the first Andalusi governor, Abd al-Aziz, in around ad 716. This determined a rapid urban development from an early stage, as shown by the excavations of the *arrabal* of Shaqunda, and by sharp demographic growth, with the arrival in the city of an aristocratic, governing elite of Arab origin, which from the beginning occupied the high political echelons of the Andalusi state. The settlement of these aristocratic groups and the city's high population explain the concentration of the production of certain luxury goods in the city from as early as the 8th century: silk, jewellery and glass itself.

Figure 1 A map of the Iberian Peninsula with modern borders, showing the locations mentioned in the text.

The urban expansion of Córdoba towards the area of Poniente in the last decade of the 20th and the first decade of the 21st centuries has enabled the archaeological documentation of ample zones of the Islamic *arrabal*. The excavation of this sector of the city has yielded a high number of domestic structures and a complete assemblage of cultural materials associated with the everyday life of its inhabitants (Murillo *et al*. 1999, 2004, 2010).

Enormous progress has also been made regarding our knowledge of industrial activities carried out in the capital of al-Andalus. Traditionally, their study had been limited to the analysis of the scarce historical sources available for the period (Vallvé 1980; Córdoba 1991,

107–8), but the archaeological works published over the past two decades have considerably increased the volume of our evidence. The discovery of industrial facilities, such as pottery workshops and tanneries, and the analysis of tools and other technical equipment found during excavation, have shed light on hitherto little known activities. This analysis has permitted the detailed study of different industrial structures, mostly bread ovens and pottery kilns (Cano *et al*. 2010, 692–9; Salinas 2012, 580–690), but also some interpreted by the excavators as glass furnaces. In Avenida and the surroundings of Puerta de Sevilla, Vargas and Gutiérrez (2003) documented an industrial production structure including crucibles, metal and 'glass slag', and the remains of a furnace probably used for glass working, all dated to the 12th century. In a less central location towards Poniente, José Manuel Bermúdez documented a pit that was probably the remains of a small furnace for glass working; finally, in Cercadilla, Silvia Fuertes and Rafael Hidalgo excavated a glass furnace probably dating to the 9th century (Fuertes and Hidalgo 2001, 164; Vargas and Gutiérrez 2003; Cano *et al*. 2010, 690).

Despite the excavation of these furnace structures, the study of glass production in al-Andalus is still in its infancy. A good deal of the attention thus far has focused on several 12th century furnaces in a workshop in Murcia, one of which (Furnace 4) has been hypothesized as being used for primary glass production, as it contained a large chunk of semi-fused glass mixed with charcoal (see Jiménez Castillo *et al*. 1998, 441); and to a lesser degree on a glass-working furnace located at the site of Pechina, Almeria (Castillo Galdeano and Martínez Madrid 2000, 83–101). As Jiménez Castillo pointed out some years ago, most references to glass finds or glass production come from rescue excavation reports, and a systematic approach is clearly lacking (Jiménez Castillo 2006–7, 51).

A bibliographical survey has resulted in the identification of at least 10 workshops dedicated to glass working: in Murcia (Jiménez Castillo *et al*. 1998, 419–58), Seville (Tabales and Huarte 1997, 455), Pechina, Almeria (Castillo Galdeano and Martínez Madrid 2000, 83–101), Córdoba (see above), Malaga (Alba Toledo 2004, 681; Expósito Capilla 2006, 3363) and Jaen (Crespo 2006, 2650). Several more possible cases have been identified in Cadiz and Baza (Jaen), but the references are too partial to be absolutely certain. The furnaces have been irregularly studied and some are merely mentioned in excavation reports, although they seem to reflect a complex reality; the examples mentioned range from the large workshop with up to five furnaces near to a historical *medina*, in Murcia, to the much smaller, possibly single-furnace workshop in Pechina. It is not currently possible to determine when and where the primary production of glass began in al-Andalus on the basis of furnace remains alone.

GLASS TECHNOLOGY AND COMPOSITIONAL ANALYSIS

The second half of the first millennium ad marks a period of change and innovation in primary glass production, as is clearly evidenced by compositional analysis. During the Roman and immediately post-Roman periods, primary glass production seems to have occurred on a massive scale, but in a relatively limited number of locations (Freestone *et al*. 2002b). It is clear that a large amount of glass was made in the Eastern Mediterranean, and along the Syro-Palestinian coast in particular, as shown by the remains of glass-making tank furnaces (Gorin-Rosen 2000; Aldsworth *et al*. 2002, 64–6). Chemical evidence for the widely encountered HIMT (high iron, manganese and titanium) glass group seems to relate to

primary production in Egypt (Freestone *et al*. 2002a, 173), though recent evidence has shown that at least some glasses may have been produced outside these areas (Wedepohl *et al*. 1997; Degryse and Schneider 2008; Rehren and Cholakova 2010).

Sometime in the 8th century, the established ingredients for glass composition in the West and Near Eastern worlds began to change owing to an interruption in the supply of natron, resulting from political events in the Delta region of Egypt (Sayre and Smith 1961; Shortland *et al*. 2006) and/or environmental change (Henderson 2013, 98). Transition to a plant-ash, high-soda composition began as early as ad 805 at al-Raqqa, Syria (Henderson *et al*. 2004, 442), and occurred throughout the Islamic world (Sayre and Smith 1974, 13; Henderson 1999; Brill 2001): these glasses can be recognized by their high magnesia (HMG) content. This tradition also seems to have spread to Southern Europe by the 8th or 9th century, where plant ashes were eventually imported to be combined with local, often purified, sands (Silvestri *et al*. 2005; Cagno *et al*. 2010; Arletti *et al*. 2011, 380; Cagno *et al*. 2012a, 2196) or even produced locally (the case of Spanish *barilla* is discussed below). In Northern Europe, people began to produce glass using fern and wood ashes, which produce variable compositional signatures characterized by a high potassium content (see, e.g., Wedepohl *et al*. 1997). It may be possible to recognize the use of local plants, such as *Salsola kali*, in Western Europe by a mixed alkali signature found in some specimens (Tite *et al*. 2006, 1284), though it should be noted that geology and the ashing techniques can have an overriding effect on the final compositions of these plant ashes and the glasses made from them.

Recycling was also practised, but it can be difficult to detect, and to quantify (but see Silvestri 2008). In general, it is indicated by 'blurred' or intermediate compositional types, and by the accumulation of trace or minor quantities of certain elements—in particular, the transition metal elements (see, e.g., Henderson 1993; Freestone *et al*. 2002b, 266)—and is best detected using trace element analysis.

SAMPLE SELECTION AND ANALYSIS

The samples presented in this paper (Table 1) come from three different excavations in suburban residential areas of Córdoba, which abounded in the city from its 10th century expansion. Although some evidence of minor industrial activity has been detected in the suburbs in question, all samples analysed were excavated from domestic contexts.

Sample selection was aimed towards a diachronic perspective, and because many samples were from highly fragmentary glasses, the types of objects from which they derived could not always be determined. Where possible, colourless glasses (including glasses with a slight yellowish tint) were prioritized for the purposes of comparison, but coloured translucent glasses were also sampled in order to provide a broader picture. The colourless glasses in particular tended to have rather heavy corrosion crusts, and all samples were thus taken from thicker areas to minimize the effects of surface alteration on the results. The samples were analysed using a JEOL JXA-8200 electron microprobe at the Microanalysis Research Facility (MRF), Department of Archaeology, University of Nottingham. A more detailed outline of the analytical methods employed can be found in Appendix A (available as supplementary material from the publisher's website).

RESULTS AND DISCUSSION

The summarized results of electron microprobe analyses are presented in Table 2. The glasses fall into four groups, two of which are only represented by one sample each: soda–lime–silica glasses produced using halophytic plant ashes, high in soda, magnesia (HMG) and with a potash content averaging 1.9%; lead–soda–silica glasses; a single 9th century sample of mineral (natron) fluxed glass; and a glass with intermediate lead. The compositions of the four glass types are summarized in Table 3, with the low-lead glasses analysed by Carmona *et al*. (2009) also included for comparative purposes.

High-lead glasses

The desirability of lead as a major glass component is related to its visual and working properties: it is known to soften glasses and to lower their working temperature and extend their working period, while its high refractive index increases the brilliance of their appearance (Arletti *et al*. 2011, 378). The levels of PbO in the high-lead Córdoban glasses discussed here fall between 33.61% and 43.07%. These results—all of which are for 10th and 11th century glasses—show significantly higher quantities than those found in the two high-lead glasses from the Murcian workshop (17.08% and 27.18%) (Carmona *et al*. 2009, 441). Glasses from both sites, however, are similar in that they fall into the lead–soda–silica, rather than the lead–silica, group. Most of the Córdoban high lead samples (a total of seven) came from RondaWest (see Table 1), but they were represented at all three of the Córdoban sites from which material was taken.

Although Islamic lead glasses are a recognized category (Sayre and Smith 1961), the proportion of high-lead glasses encountered in this study—making up 11 out of the 26 analysed—is unusually high, and to the best of the authors' knowledge, presents the highest concentration of Islamic lead–soda–silica glasses from one site known to date. By comparison, of 357 Islamic glasses analysed by Brill (1999a,b), most of which are dated to between the 9th and 13th centuries, only 10 had PbO contents in excess of 10%, most of which were of the lead–silica system (which was also the basis for most European high-lead glass). Scattered examples of lead–soda–silica glasses from these publications include: sample 3121 of a 9th–13th century red opaque bracelet from Fustat, Egypt (22.4% PbO; 14.9% Na2O); sample 5399 of a 'late Islamic(?)' red opaque bracelet from Timna, Yemen (25.8% PbO; 9.4% Na2O); and sample 5603 from a 9th–10th century, turquoise bowl in the San Marco Treasury, Venice (42.7% PbO; 7.1% Na2O).

Some contemporary African material may also be comparable. In recent LA–ICP–MS analyses of glass beads from 9th–11th century Al-Basra, Morocco (Robertshaw *et al*. 2010), 14 of 30 samples analysed were found to contain high lead, but only two of these (PR876FM and PR877FM) were lead–soda–silica (Robertshaw *et al*. 2010, 366–7). These two glasses, which were green in colour, have higher lead and slightly lower soda than the high-lead group from Córdoba (52.9–56.6% PbO; 10.6–12.4% Na2O). Though as yet unpublished, the authors are also aware of some lead–soda–silica glasses from the site of Essouk in Mali, which prospered by the Saharan caravan trade. Of 20 glass beads dated to the 9th–14th centuries from this site analysed by LA–ICP–MS at the Field Museum, Chicago, five conformed to the lead–soda–silica type (Lankton 2008) though forthcoming results may add a few more to this number (P. Robertshaw, J. Lankton, L. Dussubieux and S. Nixon, pers. comm.).

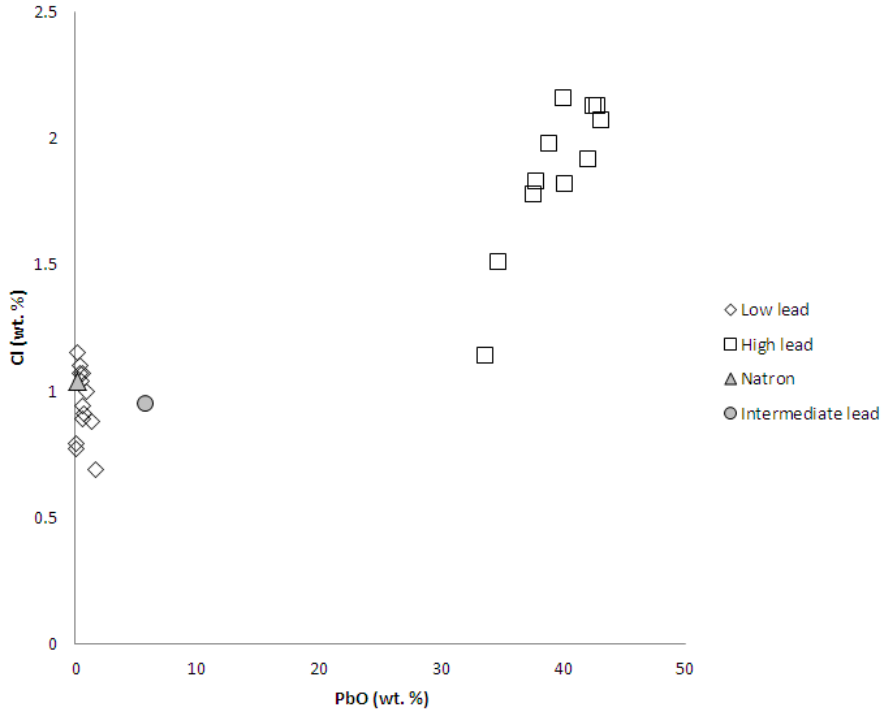

Figure 2 PbO versus Cl (wt%) in the four groups of Córdoban glasses.

The only element or oxide that was found to correlate with lead in the Córdoban glasses was chlorine, which is elevated in the high-lead glasses by comparison with the low-lead group (see Fig. 2). This suggests that the lead was added in a relatively pure form, without contaminants (though trace element contaminants may yet be detectable). In a discussion of historical sources on later medieval Venetian glass production, Arletti *et al*. (2011, 380) note that most give calcined lead litharge, or metallic lead as the source of lead. Lead metal was also thought to be the source of PbO in the high-lead samples from al-Basra (Robertshaw *et al*. 2010). Lead chloride occurs naturally as cotunnite ($PbCl_2$), a secondary alteration product found in association with galena (PbS), but it is unclear how this could have found its way into the finished glasses when the sulphides had been so successfully driven off as to remain below detection limits for all but one of the high-lead glasses. Galena from the mines in Sierra Morena was exploited between the Roman period (Linares and Cástulo) and the Late Middle Ages (Villanueva del Duque, Córdoba and Guadalcanal, Seville), and direct evidence of extraction has been found in Hornachuelos, barely 50 km from Córdoba (see Grañeda Miñón 2008). Associated with the glass production furnaces in the Murcian workshop discussed above was a structure (Furnace 1) containing abundant remains of galena, lead and copper oxides, melted lead metal and vitrified clay. Jiménez Castillo *et al*. (1998, 446–9) interpret this as a furnace for the extraction or the oxidation of lead. It would thus be very tempting to assume that this is evidence for the production of high lead glasses in Murcia, though it should be noted that the Murcian workshop also yielded fragments of glass mirrors with a lead backing, which could explain the presence of the lead-related structure, and that only two of the 23 samples from that site analysed by XRF were found to be high-lead glass (Carmona *et al*. 2009). In any case, it is unlikely that the Córdoban material had lead added to it in the same workshop as the high-lead glasses found in Murcia: neither of the two samples analysed by Carmona *et al*. (2009) had elevated

chlorine, either by comparison with the Murcian low-lead glasses or with our own data set (Cl in the high- and low-lead Murcian glasses fell below 1%).

As noted long ago by Sayre and Smith (1967, 303), it is possible that at least some Islamic high-lead glasses were formed by the addition of lead to preformed glasses, with the non-lead portion of the glasses having a composition comparable with that of low-lead glasses from the same site or region. Brill (2001, 29) also notes the addition of lead (25–35% PbO) to potash-based Western European window glasses, particularly from the 12th to the 15th centuries. In order to investigate the possibility that the Córdoban glasses were made in a similar way, the lead signature was removed and the results normalized to 100%, as presented in Table 4.

The results appear to indicate the use of different recipes, or different 'base' glasses, for the high- and low-lead glasses. As shown in Figure 3, the normalized high-lead group falls into the accepted region of 'HMG' glasses, but does not significantly overlap with the low-lead group. Similarly, the calcium content is significantly lower in the normalized high-lead glasses, which may indicate purification of the plant ashes or sand, or the use of materials from different sources. The two high-lead glasses from Murcia reported by Carmona *et al*. (2009) are also shown in Table 4, with their PbO contents removed. They are rather different in composition to one another, especially in their manganese and iron (Px-7), and in their alumina (Px-5) contents, possibly suggesting the use of different sand sources; they do not provide a close match for the Córdoban samples. Copper is also elevated in these glasses, and may have been brought in with the lead (Carmona *et al*. 2009, 444). It is thus unlikely that the Córdoban and Murcian glasses are from the same production location, either in terms of the 'base' glasses or raw materials, or of the lead which was added to these.

COR23, taken from a fragment of green translucent, plant ash glass discovered in a domestic assemblage dated to the 11th century, was found to contain a significant, but much lower amount, of lead (5.76%) than the samples in the high-lead group. Two glasses from the Murcian workshop (Px 4, red; and Px 6, pale green) had comparable levels of lead (4.47% and 4.19% PbO, respectively), though they otherwise differed significantly from the Córdoban sample, having higher soda, magnesia, alumina and calcium oxide, and significantly lower manganese (see Carmona *et al*. 2009, 441). An opaque red sample (Vre 2) with intermediate lead (6.38% PbO) from a church in Vreden, Germany, is listed in Wedepohl *et al*. (1995, 76) under 'wood ash lead glasses', though its composition is also that of a soda glass (9.7% Na2O; 1.66% K2O).

Alkali source

As noted above, with one exception, the low-lead glasses—and the normalized base glasses to which lead was added to create high-lead glasses—show compositional characteristics associated with the use of soda-rich plant ashes, in particular in terms of magnesia and potash. The exception (COR15) has a very different signature, with low potassium and magnesia consistent with the use of a mineral alkali flux, presumably natron, and with lower levels of iron, manganese and titanium than the others: its colouring is discussed below. For the other low-lead glasses, the average Na2O:K2O ratio is 7.6:1, which is more compatible with Near Eastern glasses than the mixed alkali plant ashes common inWestern Europe

(0.3:1–1.8:1, according to Tite *et al*. 2006), though still rather higher than the Near Eastern 4:1 ratio also cited by Tite *et al*. (2006), or the 5:1 ratio given by Cagno *et al*. (2008, 2010); similarly, examination of the 9th–10th century coloured and colourless glasses from Nishapur and neighbours reported in Brill (1999b) revealed an average Na2O:K2O ratio of 5.6:1. Gallo and Silvestri (2012) found Na2O:K2O ratios of up to 6.7:1 in glasses from 7th–10th century Rocca di Asolo, northern Italy, and Cagno *et al*. (2012a, 2193–4) report Na2O ranging from 11% to 15% and K2O ranging from 1.5% to 3% in Italian glass of the 10th–16th centuries, in both cases taken to suggest the importation of Levantine ash (perhaps *Salicornia soda*, although there are other possibilities).

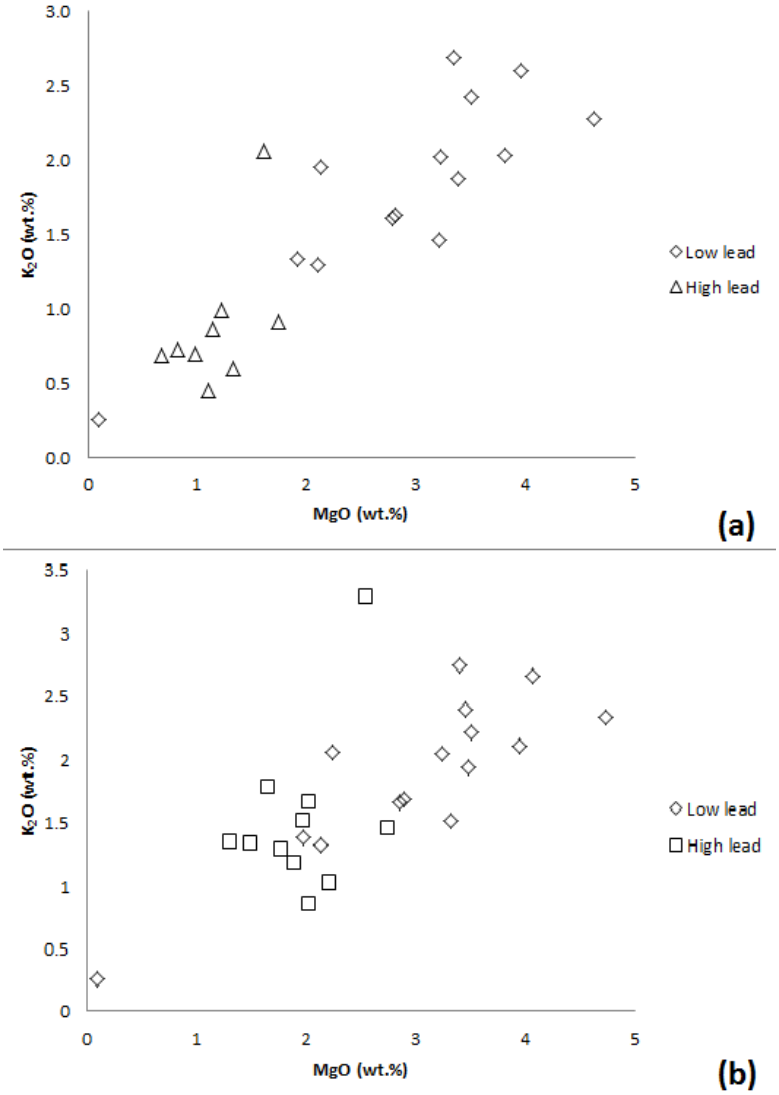

Figure 3 MgO versus K2O for (a) high- and low-lead glasses, and (b) for normalized high- and low-lead glasses following the removal of the lead signature.

Figure 4 shows Na2O and K2O normalized compositions for the low-lead plant ash glasses, obtained by dividing each of the two oxides by ash-introduced components MgO, P2O5, K_2O and CaO, following Cagno *et al*. (2012a,b). As can be seen, all but one of the low-lead glasses fall into the high-soda region occupied by the 'common'Venetian glasses (*vitrum blanchum*). One sample (COR1) falls closer to the region associated with Venetian *cristallo* glasses

(though with higher CaO levels): this may thus indicate the use of purified plant ashes. It should be noted that although the low-lead Córdoban glasses are generally discussed here as a single group, there is some variation within this, particularly in the MnO content, which is discussed further below with reference to alkali source and colourants.

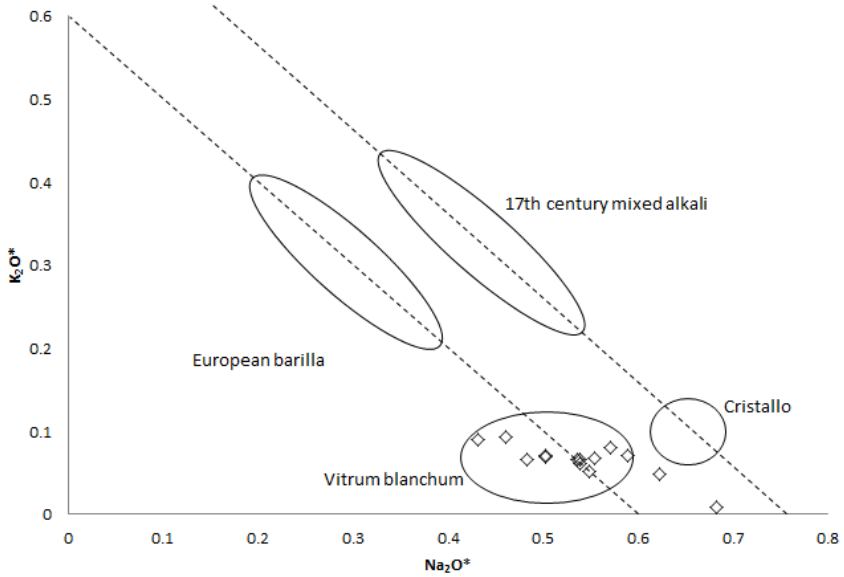

Figure 4 Na2O and K2O normalized compositions, following Cagno et al. (2012a,b) and obtained by dividing each of the two oxides by all ash-introduced components (Na2O, MgO, P2O5, K2O and CaO), with correlation lines Na2O + K2O = 0.6 and 0.75, indicating the use of unpurified and purified ash, respectively.

Figure 5 shows a comparison between magnesia and potash levels in the low-lead Córdoban glasses and those from Murcia analysed by Carmona *et al*. (2009), with the Islamic glass 'fields' given by Brill (2001). Several of these overlap with the Córdoban group, but most of the Murcian glasses have higher magnesia, falling outside both the area of the Córdoban group and Brill's Islamic glasses.

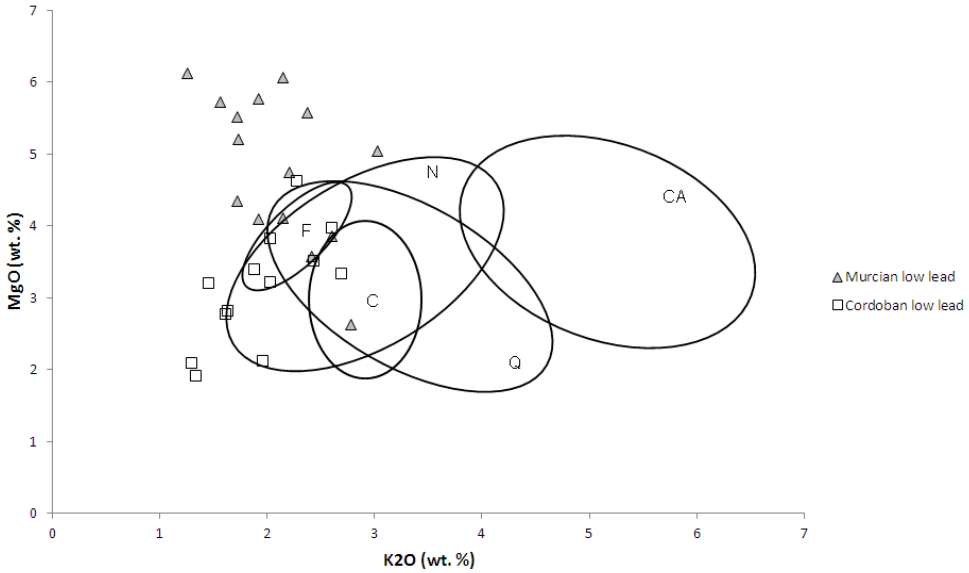

Figure 5 K_2O versus MgO (wt%) for the glasses from Córdoba presented in this study, and those from the Murcian workshop analysed by Carmona et al. (2009). Also shown are the Islamic glass 'fields' given by Brill

(2001) for various groups of Islamic glasses: CA, Afghanistan; N, Nishapur; Q, Qasr al-Saghir; C, Caesarea; F, Fustat.

It is certainly possible that the Córdoban glasses were made using imported Levantine ash, but the state of the evidence is currently incomplete. By the 14th century, all Venetian glass was mandatorily produced using imported Levantine plant ashes (Jacoby 1993; Verità and Zecchin 2009), but historical sources suggest that other Italian glass production may have relied in part on imports from the Iberian Peninsula (Frank 1982, 77; Lerma 2004, cited in Basso *et al*. 2008; Cagno *et al*. 2012b, 1547). The reference is to *barilla*, a soda plant ash produced from purified halophytic plants of unknown or various species: its composition has yet to be thoroughly investigated. Cagno *et al*. (2008, 2010) give Spanish ash a Na2O:K2O ratio of just 2:1, which would place it well outside the field of the Córdoban glasses. Although lower Na2O:K2O ratios are generally associated with Western Europe, Tite *et al*. (2006, 1289) were able to sample a high-soda plant ash from Sicily, dubbed 'Soda di Catania', which was a close match for Near Eastern compositions. We must also take into account here the natural variability in plant ash composition and its complex relationship with plant species: as noted above, local geology (Barkoudah and Henderson 2006; Henderson *et al*. 2009) and production processes (Misra *et al*. 1993; Rehren 2000; Santopadre and Verità 2000) can modify plant ash compositions markedly. Certainly local plant ashes were in use in the Iberian Peninsula by the 12th century: in Catalonia, it was recorded that the monastery of Poblet granted the glassblower Guillem the exclusive rights to gather local glasswort in 1189 (Glick 1979). In al-Andalus itself, it is apparent that by the Late Middle Ages, *Salicornia* plant ashes were mostly, if not exclusively, sourced more or less locally. They were abundant in the Ebro Delta, the Mediterranean coast (Alicante, marshes of Murcia) and, closer to Córdoba, the marshes in the lower Guadalquivir valley. For the 15th century, Enrique Otte documented numerous notarial records of sales of *Salicornias* from the Guadalquivir marshes to glassmakers in Seville and Jerez de la Frontera (Otte 1996, 88).

Silica and lime

As shown in Figure 6, there is a strong correlation in the Córdoban glasses between alumina and iron oxide in both the high- and low-lead groups. High iron and aluminium, as seen in these glasses, is characteristic of the use of relatively impure raw ingredients sources (typically sands, though plant ashes can also elevate these impurities). This is in contrast to many plant ash based compositions, which are frequently found to have been made with relatively pure, or purified, silica sources (see, e.g., Tite *et al*. 2006; Cagno *et al*. 2010, 2012a,b).

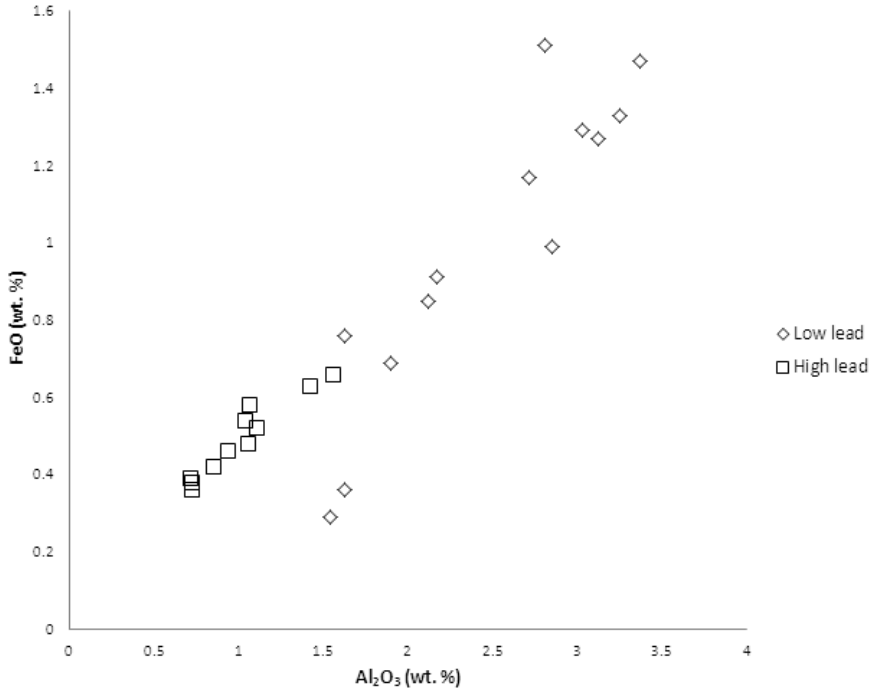

Figure 6 The correlation between alumina and iron oxide (wt%) in low- and high-lead glasses.

There are, however, some parallels for this 'dirty' composition. Among the Islamic glass groups summarized by Brill (2001, 43–4), the coloured glasses from Nishapur (9th–10th centuries) were found to have comparable compositions to the Córdoban glasses (i.e., within or very close to the standard deviations presented in Table 3), especially in relation to major and several of the minor oxides, including particularly iron oxide and alumina, indicative of high levels of impurities. As with the Córdoban glasses, the coloured glasses from Nishapur also display a high degree of variation in MnO content (between 0.36% and 4.28% in the Córdoban samples, and between 0.03% and 2.65% in those from Nishapur). The average Na2O:K2O ratio of the Nishapur glasses, however, was 5.56:1, lower than that of the Córdoban glasses. A similar composition to the Córdoban glasses has also been found by Cagno *et al*. (2012b) in a group of four samples from San Genesio dated rather broadly to between the 6th and 11th centuries, which they labelled 'SG3'. Four 9th century samples of raw furnace glass from al-Raqqa, Syria, also show comparable compositional features, with elevated iron, alumina and manganese (Henderson *et al*. 2004, table 1, samples 1, 5, 17 and 22), and four 10th and 11th century samples of Egyptian glass weights presented by Vaggelli *et al*. (2013, table 1, samples B7, B47, B53 and B60) are broadly comparable with the lower-manganese samples from Córdoba. The Córdoban glasses thus seem to have their strongest parallels with Near Eastern material. Their compositional features clearly arise from the use of relatively impure raw ingredients, most probably the sand source, though the plant ashes could also have contributed to these impurity levels.

A comparison between the average results and standard deviation for the low-lead Córdoban samples and those from Murcia analysed by Carmona *et al*. (2009) can be found in Table 3. In general, the glasses can be said to be of a similar plant ash tradition, with high iron and alumina impurities, but those from Murcia tend to have higher soda and magnesia, and do not show the same variability in manganese (all but one contain under 1% MnO).

As far as we can tell, the stabilizing lime (CaO) content is consistent with that of contemporary Syrian glasses (see, e.g., Freestone 1991, 40). As shown in Figure 7, in all of the Córdoban glasses save for the 'natron' glass, CaO is broadly correlated with K2O, and is somewhat more closely correlated with SO3, indicative of a direct relationship between it and the soda source. Barkoudah and Henderson (2006) suggest that a CaO concentration of 6–9% is consistent with the use of unrefined sodic plant ashes, though these plant ashes may not necessarily provide sufficiently high calcium oxide levels by themselves (Henderson 2013, 46). It is thus possible that in some cases a small amount of CaO was introduced along with the sand, which may have been naturally low in lime (as are inland sources such as desert and stream sands).

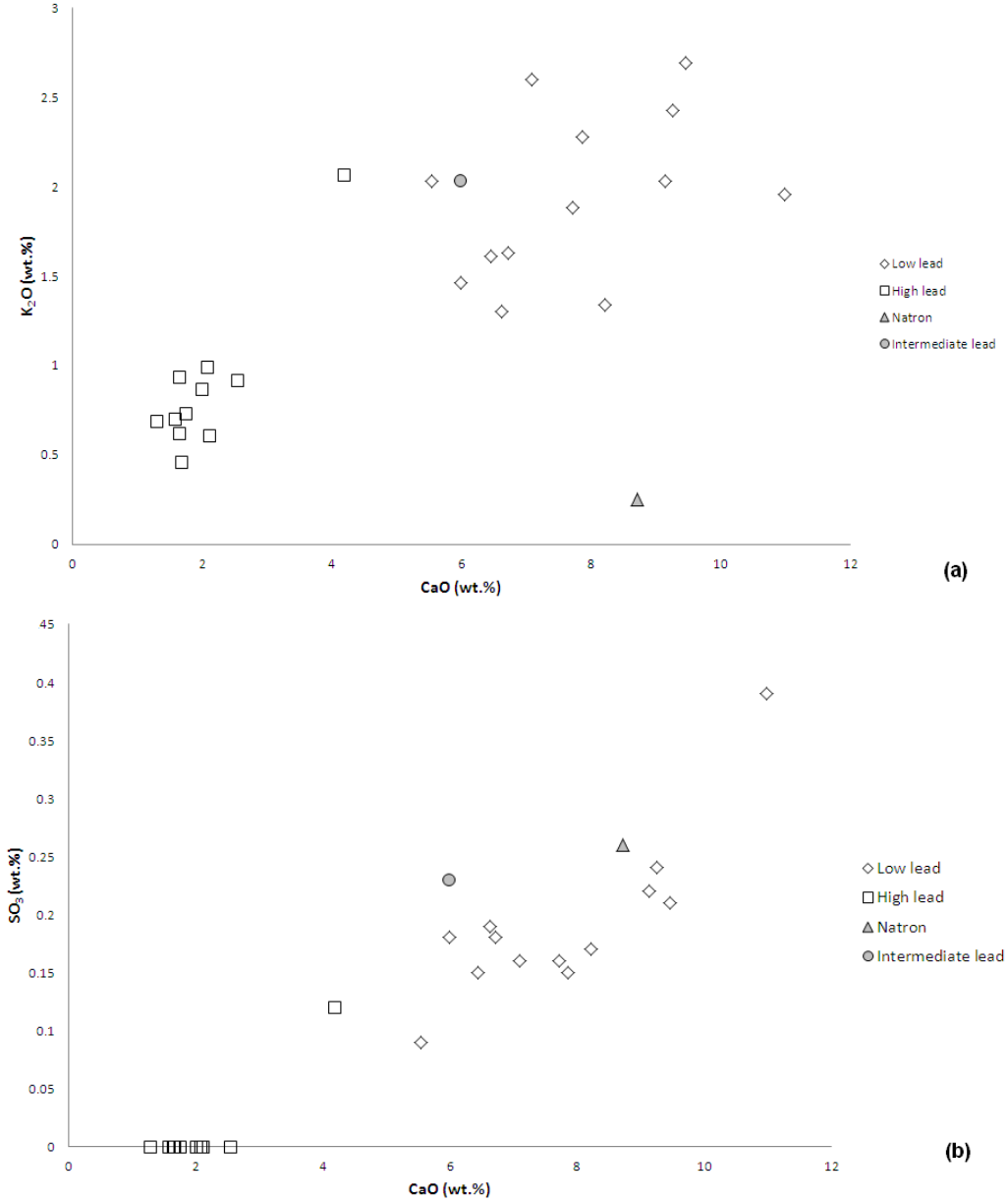

Figure 7 The correlation of CaO with (a) K2O and (b) SO3 for the four glass groups in this study. Note that the relationship between CaO and SO3 is not discernable in the high-lead glasses because the quantities of SO3 present were below the minimum detection limits of the instrument.

Colourants and decolourants

Samples COR9 and COR20 are green and turquoise, respectively. They contain higher copper contents than other samples, so it was presumably added deliberately as a colourant. The single example of natron glass (COR15) is a bright blue colour, which is due to CoO, present at 0.11% and also correlated with elevated arsenic, antimony and nickel. This sample, one of several excavated from the fill of a well, is of a typical 'Roman' natron composition, and may thus have been a relic at the time of burial.

Several of the glasses sampled were colourless or tinted, the latter usually yellow or light green, and a result of the impurities present. Manganese replaced antimony as a decolourant from the 2nd century bc. As noted by the 9th century scientist al-Jabir, manganese was added deliberately to Islamic glasses and this is borne out by the scientific investigations of factory glass from al-Raqqa (Henderson *et al*. 2004, 461). In the Córdoban glasses, manganese is present in higher quantities in the coloured glasses, suggesting that it is either an impurity or that it was used as a decolourant for the glasses with the highest impurity levels, but was not entirely successful. A similar situation might be hypothesized for the colourless and coloured glasses from Nishapur presented by Brill (1999b). The level of alumina might shed further light on this: as shown in Figure 8, the low-lead colourless glasses in general have lower alumina and manganese, suggesting the use of purer sand rather than a decolourant (this, however, was not found to be the case for the high-lead glasses). Also belonging to this group was COR20, a turquoise glass coloured by copper (the natron glass, COR15, was not included). As shown in Figure 8, the apparent positive correlation between manganese and alumina breaks down in the colourless glasses, which may suggest a different sand source, slight contamination from the addition of cullet to the glass batch (see below) or, for COR5, which has 2.03% MnO, the deliberate addition of extra manganese.

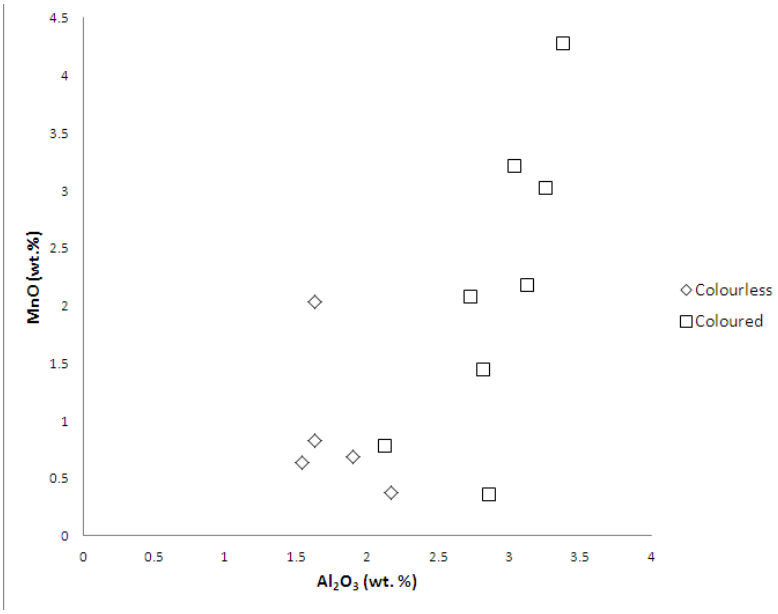

Figure 8 The correlation between MgO and Al_2O_3 (wt%) in the low-lead glasses, showing the difference between coloured and colourless glasses.

Regardless of any differences in absolute levels, both colourless and coloured glasses fall on to the same iron–alumina trend line, distinct from a large number of the Murcian samples (see Fig. 9), which could suggest that purification of the same sand source is the more likely cause of the different alumina quantities in the Córdoban glasses. For the high-lead glasses,

the compositional distinction between coloured and colourless (or tinted) glasses was much less apparent, and it is unclear by what process the glasses were rendered colourless.

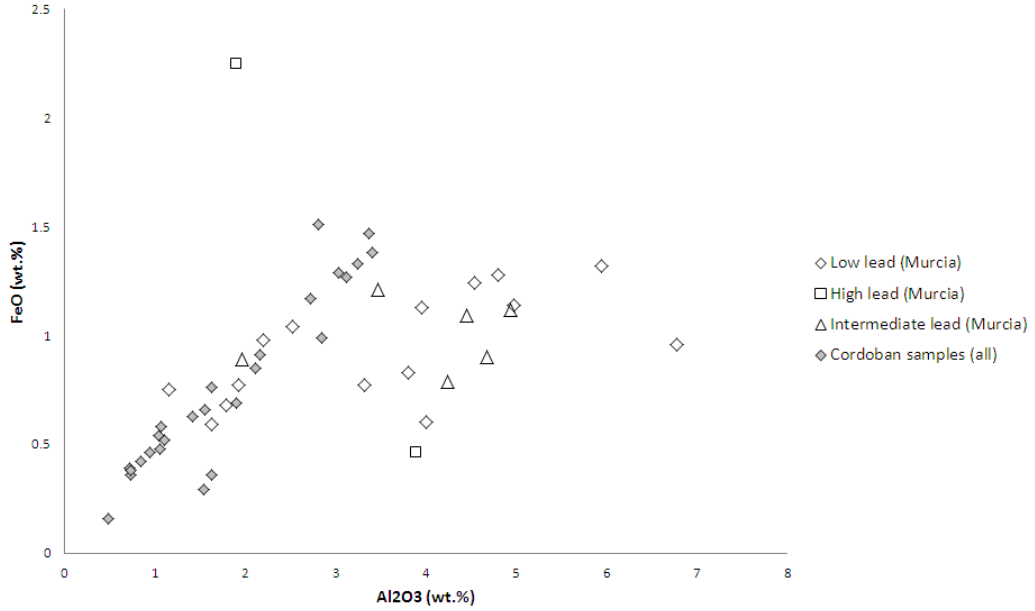

Figure 9 Al_2O_3 versus FeO for the Murcian samples analysed by Carmona et al. (2009), compared with the Córdoban samples analysed in this study. Note that the additional alumina in some of the Murcian glasses does not seem to bear any relationship to coloration or decoloration.

CONCLUSIONS

The results presented are among the first for Islamic glasses in the Iberian Peninsula, and include the earliest glasses yet analysed from al-Andalus. Both the low-lead glasses and the base composition to which the lead was (presumably) added in the high-lead group are consistent with contemporary Islamic glass compositions from Egypt and the Near East, though the low-lead group in particular shows markedly high levels of impurities.

While there is as yet no unambiguous archaeological evidence for primary glass production in al Andalus, some tantalizing historical evidence suggests the merit of investigating this as early as the 9th century, when the Córdoban polymath Abbas Ibn Firnas was reported to be 'the first who made glass out of clay', and to have established glass factories in al-Andalus (al Makkari 2002 [orig. 1600s], 148; for more historical sources, see Jiménez Castillo 2006–7, 52–3). It is interesting that the low-lead, high-impurity plant ash composition remains in evidence in Córdoba from the 9th to the 12th centuries—that is, over the entire time period included in this study—and it is perhaps not implausible to suggest that the elite echelons of the conquering Arab and Berber groups 'imported' glassmakers—who established production using imported or local ingredients—rather than glass itself. Weight is lent to this argument by the fact that, over the time period in question, Córdoba witnessed numerous changes in government with attendant shifts in external relations; if the material was imported by elites, we would expect some compositional changes in tandem with changes in external relations and consequently trade routes. On the other hand, the urban, domestic contexts of the sampled glasses—and in particular the high level of impurities within them might indicate that they are the product of a lower level of production and trade, perhaps reflecting the importation of raw materials or cullet, which would be less affected by overarching changes in government. One future avenue for this investigation

into raw ingredients sources is, of course, isotope analysis (including lead isotope analysis of the high-lead glasses).

As noted above, lead glass is known, but rare in the Islamicworld. The high lead glasses reported here are rarer still, as they are not lead–silica, but lead–soda–silica, and seem to have been made by the addition of lead to a base glass of the usual plant ash composition, or to the raw ingredients required for this. The precise reasons behind this practice are unclear, though it might have been intended to improve the working properties of the glass. Crucially, the North African connection now seems to be significant for glasses as well as for glazed ceramics, particularly as it is North African sites that bear some of the only contemporary parallels for lead–silica-soda glasses.

Until further studies are made of glasses in the Iberian Peninsula (including glass from both high- and low-status sites, and comparative material from the Christian periods), it will be difficult to firmly place these results in their broader context. They present, however, some of the first tentative steps to including this important area, which at various times acted as a bridge and barrier between the Muslim and Christian worlds, into a larger-scale picture of technological development, and glass production in particular. As already shown by research into their glazed ceramics (Coll Conesa 2003; Cruz Zuluaga *et al*. 2012), the shifting border zones of al-Andalus and the Christian kingdoms provide an ideal case study for the relationship between sociopolitical factors and technological change. The time is thus ripe for further diachronic studies of medieval and early modern glasses from the Iberian Peninsula.

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Table 1 Archaeological information.

	Site and context	Macroscopic description of glass
COR1	Ronda Oueste (Domestic collapse; 10th century)	Fragments of bottle or flask form; possible perfume vial. Lightly corroded. Green.
COR2	Ronda Oueste (Domestic collapse; 10th century)	Possible base of perfume vial or similar small vessel form. Corroded to white patina.
COR3	Ronda Oueste (Domestic collapse; 10th century)	Lamp fragments. Surface corroded.
COR4	Ronda Oueste (Public thoroughfare; 10th century)	Vessel fragment. Surface corroded.
COR5	Ronda Oueste (Domestic collapse; 10th century)	Vessel fragment. Surface corroded.
COR6	Ronda Oueste (Domestic collapse; 10th century)	Vessel fragment. Surface corroded.
COR7	Ronda Oueste (Domestic collapse; 10th century)	Fragments probably from lamp form. Surface corroded.
COR8	Ronda Oueste (Domestic collapse; 10th century)	Fragment from lamp or similar form. Surface corroded.
COR9	Ronda Oueste (Public thoroughfare; 10th century)	Fragment from base of lamp or goblet. Surface corrosion with green glass visible beneath.
COR10	Ronda Oueste (Public thoroughfare; 10th century)	Small rim fragment. Surface corrosion with yellowish/decoloured glass visible beneath.
COR11	Ronda Oueste (Public thoroughfare; 10th century)	Small vessel fragment. Surface corroded.
COR12	Ronda Oueste (Public thoroughfare; 10th century)	Fragments of moulded glass. Light surface corrosion (iridescence).
COR13	Peri9 (Domestic; 10th century)	Fragment of small vessel; possible perfume vial. Neck is blue; rim is yellow with surface corrosion.
COR14	Peri9 (Domestic; 10th century)	Small fragment with corroded surface.
COR15	Peri9 (Well fill; 8th-9th century)	Dark blue, uncorroded fragment.
COR16	Peri9 (Well fill; 10th century)	Small fragment corroded to silvery surface patina.
COR17	Peri9 (Well fill; 8th-9th century)	Vessel fragments. Light corrosion over yellowish/decoloured glass.
COR18	Peri9 (Well fill; 8th-9th century)	Vessel fragments. Lightly corroded. Blue.
COR19	Joaquin Sama/Musico Cristobal Morales (Domestic; 10th-11th century)	Vessel fragment. Surface corrosion with yellowish/decoloured glass visible beneath.
COR20	Joaquin Sama/Musico Cristobal Morales (Domestic; 12th century)	Vessel fragment. Surface corrosion with blue-turquoise glass visible beneath.
COR21	Joaquin Sama/Musico Cristobal Morales (Domestic; 12th century)	Vessel fragment. Surface corroded.

COR22	Joaquin Sama/Musico Cristobal Morales (Domestic collapse; 11th century)	Vessel fragment. Surface corroded.
COR23	Joaquin Sama/Musico Cristobal Morales (Domestic collapse; 11th century)	Small fragment corroded to whitish surface patina.
COR24	Peri9 (Well fill; 8th-9th century)	Moulded vessel fragment. Light corrosion. Green.
COR25	Peri9 (Domestic; 8th-9th century)	Vessel fragment. Light corrosion. Green.
COR26	Peri9 (Domestic; 11th century)	Vessel fragment with two applied trails in same colour. Light corrosion. Green.

Table 2 EPMA–WDS results of the chemical compositions (wt%) of the glass matrix: the results are an average of three areas analysed on each sample.

	Colour	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	FeO	CoO	NiO	CuO	ZnO	As_2O_5	ZrO_2	SnO_2	Sb_2O_5	BaO	PbO	Total	Group
COR 1	Green	16.9	2.15	2.85	65.52	0.22	0.19	1.15	1.3	6.63	0.25	0.36	0.99	<mdl	<mdl	0.07	0.03	<mdl	<mdl	<mdl	0.09	<mdl	0.06	98.73	low Pb
COR 2	Blue	6.87	0.82	1.07	39.71	0.18	<mdl	1.92	0.73	1.74	<mdl	1.04	0.58	<mdl	<mdl	0.14	0.03	<mdl	<mdl	0.11	0.11	<mdl	42.01	97.08	high Pb
COR 3	Colourless	6.13	1.01	0.73	39.28	0.26	<mdl	2.13	0.62	1.65	<mdl	0.5	0.36	<mdl	<mdl	0.17	0.04	<mdl	<mdl	<mdl	<mdl	<mdl	42.77	95.66	high Pb
COR 4	Colourless	7.67	1.1	0.85	38.57	0.22	<mdl	2.16	0.46	1.67	<mdl	0.5	0.42	<mdl	<mdl	0.1	0.05	<mdl	<mdl	0.52	<mdl	<mdl	39.95	94.24	high Pb
COR 5	Colourless	14.34	2.13	1.63	59.47	0.27	0.39	0.69	1.96	10.99	0.08	2.03	0.76	<mdl	<mdl	0.11	0.03	<mdl	0.04	<mdl	0.12	<mdl	1.62	96.66	low Pb
COR 6	Colourless	13.36	3.34	1.63	64.84	0.19	0.21	0.79	2.69	9.46	0.06	0.82	0.36	<mdl	<mdl	0.06	0.03	<mdl	<mdl	<mdl	0.17	<mdl	<mdl	98.01	low Pb
COR 7	Colourless	6.76	1.33	1.04	44.02	0.29	<mdl	1.78	0.61	2.11	<mdl	0.92	0.54	<mdl	<mdl	0.23	0.04	<mdl	<mdl	0.07	<mdl	<mdl	37.56	97.29	high Pb
COR 8	Colourless	5.53	0.87	0.72	39.38	0.31	<mdl	2.07	0.94	1.65	<mdl	0.35	0.39	<mdl	<mdl	0.16	0.04	<mdl	<mdl	<mdl	0.22	<mdl	43.07	95.69	high Pb
COR 9	Green	7.15	1.13	1.11	40.07	0.25	<mdl	1.82	0.87	2	<mdl	0.42	0.52	<mdl	<mdl	1.91	0.04	<mdl	<mdl	<mdl	0.17	<mdl	40.1	97.56	high Pb

COR 10	Colourless	6.88	0.98	1.06	39.08	0.27	<mdl	2.13	0.7	1.58	<mdl	1.35	0.48	<mdl	<mdl	0.13	0.03	<mdl	<mdl	<mdl	0.3	<mdl	42.41	97.4	high Pb
COR 11	Colourless	16.88	3.82	2.17	62.82	0.39	0.09	1.07	2.03	5.55	0.11	0.37	0.91	<mdl	<mdl	0.06	0.05	<mdl	<mdl	<mdl	0.13	<mdl	0.27	96.72	low Pb
COR 12	Colourless	11.68	3.51	1.54	70.64	0.19	0.24	0.77	2.43	9.26	<mdl	0.63	0.29	<mdl	<mdl	0.06	<mdl	<mdl	<mdl	<mdl	0.14	<mdl	<mdl	101.36	low Pb
COR 13	Yellow	8.95	1.22	0.94	42.52	0.33	<mdl	1.83	0.99	2.07	<mdl	0.47	0.46	<mdl	<mdl	0.1	<mdl	<mdl	<mdl	<mdl	<mdl	<mdl	37.73	97.61	high Pb
COR 14	Yellow	9.12	1.74	1.42	43.37	0.33	<mdl	1.51	0.92	2.54	<mdl	1.3	0.63	<mdl	<mdl	0.14	0.04	<mdl	<mdl	<mdl	<mdl	<mdl	34.65	97.72	high Pb
COR 15	Blue	19.58	0.1	0.48	67.83	0.03	0.26	1.04	0.25	8.72	<mdl	0.11	0.16	0.11	0.04	0.05	0.03	0.09	<mdl	<mdl	0.3	<mdl	0.16	99.34	natron
COR 16	Light green-blue	14.93	3.22	2.72	61.52	0.36	0.22	1.07	2.03	9.14	0.13	2.08	1.17	0.02	<mdl	0.14	0.03	<mdl	<mdl	<mdl	0.13	0.25	0.48	99.65	low Pb
COR 17	Yellow	13.08	2.78	3.37	62.32	0.38	0.15	1	1.61	6.45	0.17	4.28	1.47	<mdl	<mdl	0.11	0.03	<mdl	<mdl	<mdl	0.11	<mdl	0.78	98.09	low Pb
COR 18	Blue	14.26	1.92	2.81	63.5	0.23	0.17	0.88	1.34	8.22	0.17	1.45	1.51	0.05	<mdl	0.21	0.05	<mdl	0.03	<mdl	0.12	<mdl	1.24	98.15	low Pb
COR 19	Yellow	6.14	0.67	0.73	38.43	0.18	<mdl	1.98	0.69	1.29	<mdl	0.49	0.38	<mdl	<mdl	0.14	0.05	<mdl	<mdl	<mdl	0.15	<mdl	38.79	90.12	high Pb
COR 20	Turquoise	18.61	3.97	2.12	57.28	0.36	0.16	0.89	2.6	7.09	0.08	0.78	0.85	<mdl	<mdl	2.48	0.05	<mdl	<mdl	<mdl	0.14	<mdl	0.45	97.91	low Pb
COR 21	Colourless	18.84	4.63	1.9	58.76	0.39	0.15	1.1	2.28	7.86	0.07	0.68	0.69	<mdl	<mdl	0.09	0.04	0.05	<mdl	<mdl	0.13	<mdl	0.27	97.93	low Pb
COR 22	Green	8.85	1.61	1.56	40.78	0.3	0.12	1.14	2.07	4.19	<mdl	0.85	0.66	<mdl	<mdl	0.65	0.03	<mdl	<mdl	<mdl	0.17	<mdl	33.61	96.58	high Pb
COR 23	Green	14	3.24	3.41	56.85	0.35	0.23	0.95	2.04	5.98	0.17	3.26	1.38	<mdl	<mdl	0.08	0.03	<mdl	<mdl	<mdl	0.13	<mdl	5.76	97.86	intermediate Pb
COR 24	Green	12.84	3.21	3.03	63.66	0.34	0.18	0.94	1.46	6	0.15	3.22	1.29	<mdl	<mdl	0.09	0.04	<mdl	<mdl	<mdl	0.09	<mdl	0.45	96.98	low Pb
COR 25	Green	13.25	2.82	3.25	63.3	0.33	0.18	0.91	1.63	6.72	0.17	3.03	1.33	<mdl	<mdl	0.18	0.06	<mdl	<mdl	<mdl	0.14	<mdl	0.56	97.85	low Pb
COR 26	Green	13.49	3.39	3.12	62.28	0.36	0.16	1.04	1.88	7.72	0.14	2.18	1.27	<mdl	<mdl	0.09	0.04	<mdl	<mdl	<mdl	0.13	<mdl	0.43	97.72	low Pb

Table 3 Average compositions (wt%) with standard deviations for the low-lead group, the high-lead group, the two anomalous glasses, and—for comparison—the low-lead glasses reported by Carmona et al. (2009).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO
low lead group average n=13	14.81 ±2.29	3.14 ±0.79	2.47 ±0.66	62.76 ±3.35	0.31 ±0.08	0.19 ±0.07	0.95 ±0.14	1.94 ±0.46	7.78 ±1.59	0.13 ±0.06	1.69 ±1.25
high lead group average n=11	7.28 ±1.23	1.13 ±0.32	1.02 ±0.27	40.47 ±1.96	0.26 ±0.05	0.12 ±0	1.86 ±0.31	0.87 ±0.43	2.04 ±0.79	<mdl	0.75 ±0.37
intermediate lead (COR 23)	14	3.24	3.41	56.85	0.35	0.23	0.95	2.04	5.98	0.17	3.26
natron glass (COR 15)	19.58	0.1	0.48	67.83	0.03	0.26	1.04	0.25	8.72	<mdl	0.11
Carmona et al low lead average n=17	19.09 ±2.39	4.88 ±0.97	3.5 ±1.62	59.54 ±2.74	0.26 ±0.07	0.16 ±0.06	1.05 ±0.19	2.14 ±0.5	7.47 ±1.47	0.18 ±0.09	0.36 ±0.29
	FeO	**CoO**	**NiO**	**CuO**	**ZnO**	**As_2O_5**	**ZrO_2**	**SnO_2**	**Sb_2O_5**	**BaO**	**PbO**
low lead group average n=13	0.99 ±0.39	0.04 ±0.02	<mdl	0.29 ±0.66	0.04 ±0.01	0.05 ±0.00	0.04 ±0.00	<mdl	0.13 ±0.02	0.25 ±0.00	0.60 ±0.46
high lead group average n=11	0.49 ±0.10	<mdl	<mdl	0.35 ±0.54	0.04 ±0.01	<mdl	<mdl	0.24 ±0.25	0.19 ±0.07	<mdl	39.33 ±3.23
intermediate lead (COR	1.38	<mdl	<mdl	0.08	0.03	<mdl	<mdl	<mdl	0.13	<mdl	5.76

23)											
natron glass (COR 15)	0.16	0.11	0.04	0.05	0.03	0.09	<mdl	<mdl	0.3	<mdl	0.16
Carmona et al low lead average n=17	0.93 ±0.23	NA	NA	0.14 ±0.41	NA	<mdl	NA	0.10 ±0.01	<mdl	NA	0.34 ±0.41

Table 4 Average compositions (wt%), normalized to 100% and with standard deviations for the low-lead group, the high-lead group and groups corrected to remove PbO: the high-lead glasses from Córdoba and the high-lead glasses reported in Carmona et al. (2009). Prior to normalization, the low-lead group average totals 98.58% and the high-lead group average totals 96.46%.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO
low lead glass n=13	15.10 ±2.38	3.20 ±0.80	2.52 ±0.68	63.94 ±2.96	0.32 ±0.08	0.20 ±0.07	0.96 ±0.14	1.98 ±0.47	7.92 ±1.60	0.13 ±0.06	1.72 ±1.28
high lead glass n=11	7.57 ±1.21	1.18 ±0.32	1.06 ±0.27	42.12 ±1.65	0.27 ±0.05	0.13 ±0	1.94 ±0.34	0.91 ±0.44	2.12 ±0.80	<mdl	0.77 ±0.37
high lead glass - corrected n=11	12.76 ±1.44	1.97 ±0.43	1.78 ±0.36	71.47 ±3.03	0.46 ±0.08	0.19 ±0.00	3.32 ±0.73	1.52 ±0.64	3.55 ±1.10	<mdl	1.30 ±0.61
Carmona et al. (Px-7) - corrected	17.29	3.64	2.29	61.76	0.16	0.22	0.92	2.21	5.92	0.14	0.08
Carmona et al. (Px-5) - corrected	13.47	2.77	5.34	61.80	0.14	0.16	1.17	1.52	6.32	0.21	0.15

	FeO	CoO	CuO	ZnO	As_2O_5	ZrO_2	SnO_2	Sb_2O_5	BaO	PbO
low lead glass n=13	1.01 ±0.40	0.04 ±0.02	0.29 ±0.68	0.04 ±0.01	0.05 ±0	0.04 ±0	<mdl	0.13 ±0.02	0.25 ±0	0.62 ±0.47
high lead glass n=11	0.51 ±0.10	<mdl	0.36 ±0.55	0.04 ±0.01	<mdl	<mdl	0.25 ±0.27	0.19 ±0.07	<mdl	40.96 ±3.58
high lead glass - corrected n=11	0.86 ±0.13	<mdl	0.61 ±0.93	0.07 ±0.01	<mdl	<mdl	0.43 ±0.46	0.34 ±0.12	<mdl	NA
Carmona et al. (Px-7) -	2.71	NA	2.46	NA	0.04	NA	0.12	0.04	NA	NA

corrected										
Carmona et al. (Px-5) - corrected	0.63	NA	5.80	NA	0.33	NA	0.03	0.16	NA	NA

APPENDIX A: METHODOLOGY AND CONDITIONS OF ANALYSIS

The Cordoban glass samples were mounted in cold-setting epoxy resin, and then were ground and polished using standard sample preparation procedures down to a 0.02 micron final polishing solution. The samples were coated with a thin film of carbon prior to analysis, to allow the conduction of the electron beam.

The polished samples were analysed by EPMA-WDS using a JEOL JXA-8200 electron microprobe (Microanalysis Research Facility, Dept. Archaeology, University of Nottingham). The backscattered electron image was taken using a 20kV accelerating voltage and a 500pA beam current.

Quantitative compositional analyses were carried out using wavelength dispersive spectrometers (WDS) and an analytical set-up of a 20kV accelerating voltage and a 50nA beam current with a 50µm defocussed beam. A defocused beam is used to reduce the effect of the migration of alkalis within the samples. The EPMA-WDS was calibrated against a combination of certified mineral, pure metal and synthetic standards. The composition of 26 elements was analysed with the results presented as oxide weight percentage: Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, SO_3, Cl, K_2O, CaO, TiO_2, V_2O_3, Cr_2O_3, MnO, FeO, CoO, NiO, CuO, ZnO, As_2O_5, SrO, ZrO_2, Ag_2O, SnO_2, Sb_2O_5, BaO, PbO. Three areas of interest at x1000 were analysed for each sample.

To provide a check on the calibration of the EPMA-WDS, three repeat analyses of a secondary standard, Corning B glass, were included during the analytical run for the Cordoban glass (at the start and end of the analysis, and in between each polished block). The published composition of the Corning B reference material and the average of the three measured EPMA-WDS analyses are produced below (Table A.1).

The absolute difference between the measured data and the published data for Coring B varies from -1.56% to 0.53% at 20kV in all oxides, with a 1.02% deviation for the total composition. The relative difference of major and minor oxides (Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, MnO and CuO) varies from -5.74% to 4.94%. The relative difference of most trace oxides (P_2O_5, SO_3, Cl, FeO, NiO, ZnO, Sb_2O_5 and PbO) varies from -21.51% to 8.52%. Some of the trace elements were below the minimum detection limit of this analytical set-up (As_2O_5, SrO, ZrO_2, Ag_2O, SnO_2, Cr_2O_3 and BaO). Some of those measured were clearly problematic (TiO_2, CoO and, V_2O_3), but this was probably due to poor counting statistics on the Corning B glass using this analytical setup as the concentration of these elements in Corning B

is very low. The standard deviation of the measured data is good for most of the oxides, with the exception of those below the minimum detection limit (for which the standard deviation cannot therefore be measured) and those oxides previously mentioned as problematic on the Corning B glass.

Table A.1: Measured EPMA-WDS analyses and published composition of Corning B Glass, conducted at 20kV, 50pA and 50 micron defocused beam (wt%).

	Mean Corning B measured (n=3)	Corning B published	absolute difference	relative difference %
Na_2O	17.79	17.26	0.53	3.07
MgO	1.12	1.19	-0.07	-5.74
Al_2O_3	4.33	4.22	0.11	2.53
SiO_2	59.99	61.55	-1.56	-2.53
P_2O_5	0.66	0.84	-0.18	-21.51
SO_3	0.52	0.54	-0.02	-4.32
Cl	0.17	0.20	-0.03	-13.17
K_2O	1.09	1.10	-0.02	-1.36
CaO	8.83	8.71	0.12	1.42
TiO_2	0.07	0.10	-0.03	-29.67
V_2O_5	0.05	0.03	0.02	56.67
Cr_2O_3	<mdl	0.01	NA	NA
MnO	0.24	0.23*	0.01	3.57
FeO	0.29	0.31*	-0.02	-6.45
CoO	0.05	0.04	0.01	30.48
NiO	0.10	0.09	0.01	8.52
CuO	2.83	2.70	0.13	4.94
ZnO	0.21	0.20	0.01	6.67
As_2O_5	<mdl	NA	NA	NA
SrO	<mdl	0.01	NA	NA
ZrO_2	<mdl	0.03	NA	NA
Ag_2O	<mdl	0.01	NA	NA
SnO_2	<mdl	0.04	NA	NA
Sb_2O_5	0.47	0.46	0.01	2.32
BaO	<mdl	0.14	NA	NA
PbO	0.39	0.40	-0.01	-1.92
Total	99.20	100.49		

Notes: * FeO and MnO compositions for Corning B calculated from published value for Fe_2O_3 and MnO_2 respectively.

Table A.2 The standard deviation of the measured EPMA-WDS analyses of Corning B Glass, conducted at 20kV, 50pA and 50 micron defocused beam (wt%).

	Mean Corning B measured (n=3)	Std. dev.	Std. dev. %
Na_2O	17.79	0.145	0.82
MgO	1.12	0.018	1.64
Al_2O_3	4.33	0.029	0.67
SiO_2	59.99	0.947	1.58
P_2O_5	0.66	0.019	2.81
SO_3	0.52	0.032	6.10
Cl	0.17	0.001	0.33
K_2O	1.09	0.012	1.13
CaO	8.83	0.006	0.07
TiO_2	0.07	0.014	19.92
V_2O_5	0.05	0.000	0.00
Cr_2O_3	<mdl	NA	NA
MnO	0.24	0.009	3.88
FeO	0.29	0.003	1.03
CoO	0.05	0.005	9.87
NiO	0.10	0.006	6.58
CuO	2.83	0.015	0.54
ZnO	0.21	0.013	6.05
As_2O_5	<mdl	NA	NA
SrO	<mdl	NA	NA
ZrO_2	<mdl	NA	NA
Ag_2O	<mdl	NA	NA
SnO_2	<mdl	NA	NA
Sb_2O_5	0.47	0.009	1.93
BaO	<mdl	NA	NA
PbO	0.39	0.020	5.03
Total	99.20		