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## Islamic soda-ash glasses in the Christian kingdoms of Asturias and León (Spain)



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## ABSTRACT

The castle of Gauzón on the north coast of Spain controlled access to the Avilés estuary and was intimately linked to the Asturian monarchy. With the intention of studying the medieval glass trade networks in the kingdom of Asturias and León, the chemical composition of twenty-four glass samples from recent excavations at the castle were determined by LA-ICP-MS. The analytical results identified two distinct types of soda-rich plant ash glasses, one from the eastern Mediterranean, and the other from Mesopotamia. Similarly, most of the decorations correspond to luxury Islamic relief-cut glasses that are known from the eastern Mediterranean and Mesopotamia. The decorative features and compositional characteristics of the glass samples from Gauzón thus reveal close commercial links between the Christian kingdoms of Asturias and León on the Atlantic coast and the Islamic world, with al-Andalus, Mesopotamia and the Mediterranean Levant.

### 1. Introduction

Research into first millennium CE glass has mainly focused on ancient and Roman natron glasses, their compositional and typological features, their geographical and chronological attribution and dissemination (e.g. De Juan Ares and Schibille, 2017a; Foy, 2017b; Freestone, 2006; Gliozzo, 2017). Less is known about glass and its distribution patterns during the Middle Ages. Towards the late first millennium CE, the fluxing agents changed from mineral soda to potassium rich plant ashes in central Europe (e.g. Krueger and Wedepohl, 2003) and soda-rich plant ashes in the eastern Mediterranean (Phelps et al., 2016; Shortland et al., 2006). Some lead-rich glasses appeared at around the same time, both in Europe as well as the Islamic east (Mecking, 2013). Practically nothing is known about glass in the early Christian kingdoms in northern Spain and its relationships with other Christian territories such as France and central Europe or Islamic al-Andalus (Calleja, 2017; De Juan Ares and Schibille, 2017a). A few isolated medieval glass objects preserved in churches have been published and are generally assumed to originate either from al-Andalus or the Islamic east (García and Valdes, 1996; Velasco and Whitehouse, 2012).

The present article focuses on the decorative techniques and

chemical compositions of glasses from the Christian kingdoms of Asturias (718–910 CE) and León (910–1230 CE) in the northern Iberian Peninsula. The samples come from the archaeological excavations in Peñón de Raíces (Castrillón, Spain). According to historical and archaeological research, this was the location of the castle of Gauzón that was closely linked with the Asturian monarchy (García and Muñiz, 2013). It was here that in 908 king Alfonso III and Jimena ordered the making of the Victory Cross (García de Castro, 2008), a jewel offered to the Cathedral of Oviedo that was to become the symbol of the Christian reconquest and the Principality of Asturias. The castle controlled access to the estuary of Avilés, one of the routes into the centre of the Iberian Peninsula (Fig. 1). Archaeological research has revealed that the castle was constructed at the end of the sixth century, possibly over a Roman settlement, and rebuilt between the second half of the eighth and the first half of the ninth century, possibly in connection with Viking assaults on the Spanish coast. The castle was destroyed and abandoned in the second half of the fourteenth century (Muñiz López and García Álvarez-Busto, 2010).

In this study, we present the technological and compositional characteristics of a series of medieval glass objects that were recovered during recent excavation campaigns at the castle of Gauzón and analysed by LA-ICP-MS. The typological features and chemical signatures

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Fig. 1. Map of the Iberian Peninsula, highlighting the location of the castle of Gauzón and Iberian sites mentioned in the text.

of the glass-finds reflect long-distance trade networks and the supply of a relatively rare and precious commodity in the form of prestigious glass objects to the Christian kingdoms of Asturias and León. By comparison with other medieval glass assemblages, it is thus possible to gain insights into the site's connectivity and the cultural value of glass.

## 2. Materials and methods

Two-thirds of the 24 glass fragments that were selected for analysis came from stratigraphic contexts and can be dated based on associated materials and  $C^{14}$  data to between the eleventh and the twelfth century (Table S1) (García and Muñiz, 2013). One fragment (GA014) was found in an earlier, ninth- to tenth-century context, two (GA019 & GA023) came from thirteenth- to early fourteenth-century deposits and one sample (GA012) dates to the fifteenth or sixteenth century. Three samples derive from disturbed contexts of previous excavations (GA017, GA018 and GA021) and one was retrieved from a superficial layer (GA024) (Table S1). With the exception of three dark blue fragments, all samples are either colourless or weakly coloured (greenish, bluish, greenish-yellow).

The samples were selected to represent the entire range of identifiable forms and decorative techniques and include rims and body fragments. All those decorated are body fragments from bowls or bottles that were cut, engraved and ground (Table S1, Fig. 2). Two fragments have simple scratch-engraved ornaments (GA010, 015), one is a facet-cut vessel (GA003), and the rest are relief-cut glasses where both the background and interior of the motifs have been cut and ground so as to leave only the raised outlines. The glass finds tend to be too fragmentary to allow for the identification of the exact decorative patterns. Sample GA017 with relief cut discs has parallels among Sasanian and Islamic glass assemblages (e.g. Whitehouse, 2010, Cat. 124), while geometric motifs similar to those of samples GA008 and GA018 are generally widespread among glass finds from the Islamic Near East (Whitehouse, 2010). On the Iberian Peninsula, these types of decorations have only been sporadically documented, both in al-Andalus as well as in ecclesiastical contexts in the northern parts of Spain.

Small fragments of the glass samples were mounted in epoxy resin and polished (13 samples per block). Single bulk analyses were then performed on the cross-sections by Laser Ablation Inductively Coupled

Plasma Mass Spectrometry (LA-ICP-MS) at the Centre Ernest-Babelon of IRAMAT (Orléans) using a ThermoFisher Element XR combined with a Resonetic UV laser microprobe (ArF 193 nm) (following the protocol of Gratze, 2016). The laser was operated at 5 mJ energy with a pulse frequency of 10 Hz and a beam diameter set at 100  $\mu\text{m}$ . 20 s pre-ablation time was followed by 30 s collection time, the equivalent of 10 mass scans. The signals for 58 isotopes (Li to U) were measured in counts-per-second and converted into quantitative data by calculating a response coefficient (K) using a set of five glass standards (NIST SRM610, Corning B, C, D and APL1, an in-house standard). Detection limits for major elements are in the range from 0.01% to 0.1%, for minor and trace elements from 20 to 500 ppb. Standard reference material (NIST SRM612, Corning A) were regularly measured to ascertain the precision and accuracy of the measurements. The precision is typically better than 5% for most elements and accuracy is better than 10% across all elements (see Table S1 in De Juan Ares et al., 2018).

## 3. Results

### 3.1. Natron glasses

Three main compositional groups can be distinguished according to the fluxing agents: two different plant ash categories and a small set of natron-type glasses (Fig. 3, Table S1). The natron glasses have low potassium and magnesium oxide levels (< 1.5%), but differ in terms of the silica sources (Table S1). For example, one fragment (GA022) with elevated titanium and zirconium as well as manganese and iron exhibits the typical characteristics of so-called HIMT glass, dating to the fourth to fifth century CE (Freestone et al., 2018 and references therein). Sample GA015 corresponds roughly with group 2 of Foy et al. (2003) on account of elevated manganese, titanium, iron and zirconium levels but that are below those of HIMT glass. This type of glass (Foy-2) is believed to be ultimately of an Egyptian origin as well (Ceglia et al., 2017; Foy et al., 2003; Schibille et al., 2016). The sample shows signs of recycling in the form of elevated antimony and lead concentrations (> 100 ppm see, for example, Degryse, 2014). The remaining two natron glass samples (GA001 & GA014) are likewise the result of recycling given the contaminations through elements typically associated with colouring such as copper, tin, antimony and lead. Their mixed manganese and

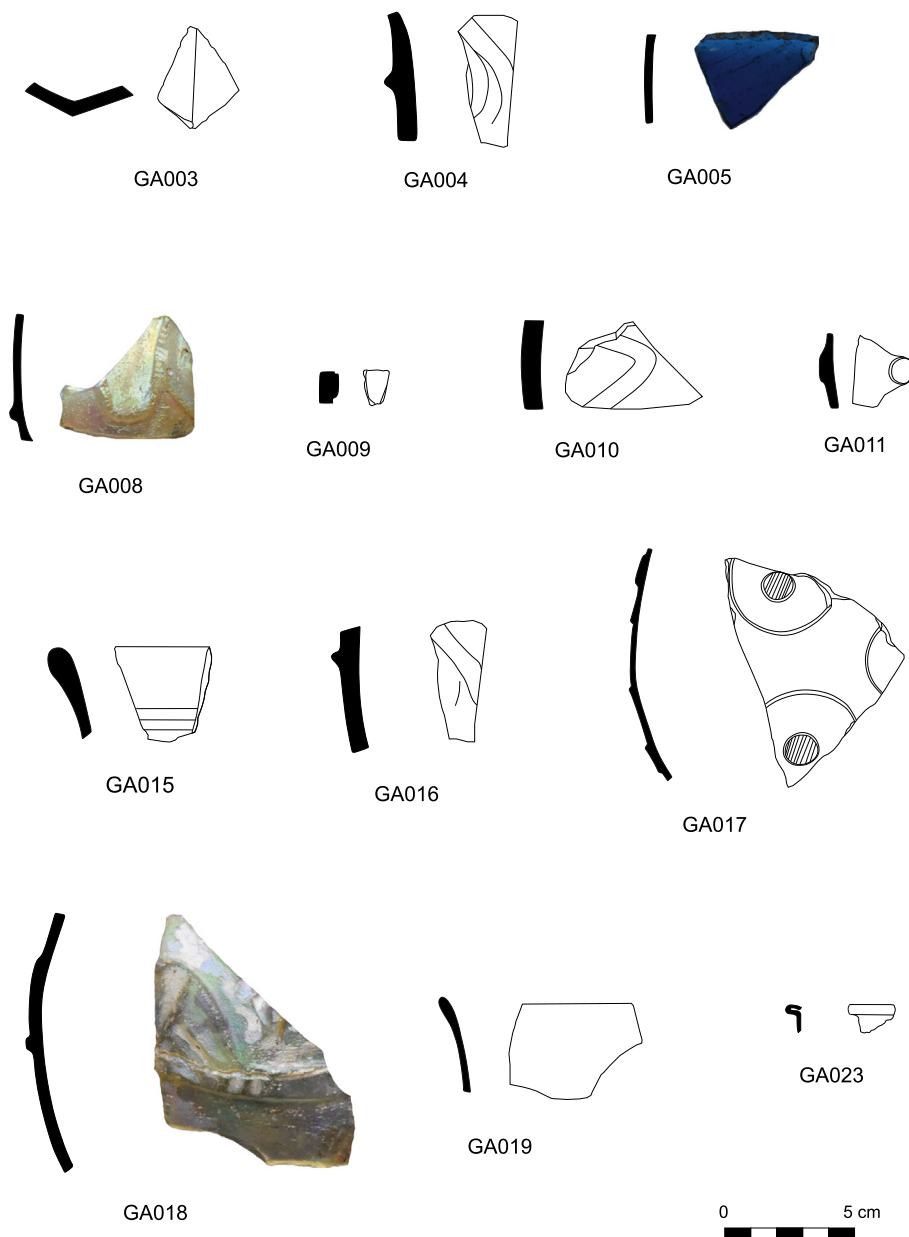


Fig. 2. Selection of glass fragments from the castle of Gauzón, representing different typologies and glass working techniques.

antimony signatures suggest that Roman antimony decolourised glass was combined with a manganese decolourised glass of Levantine origin (Table S1) (Jackson and Paynter, 2016).

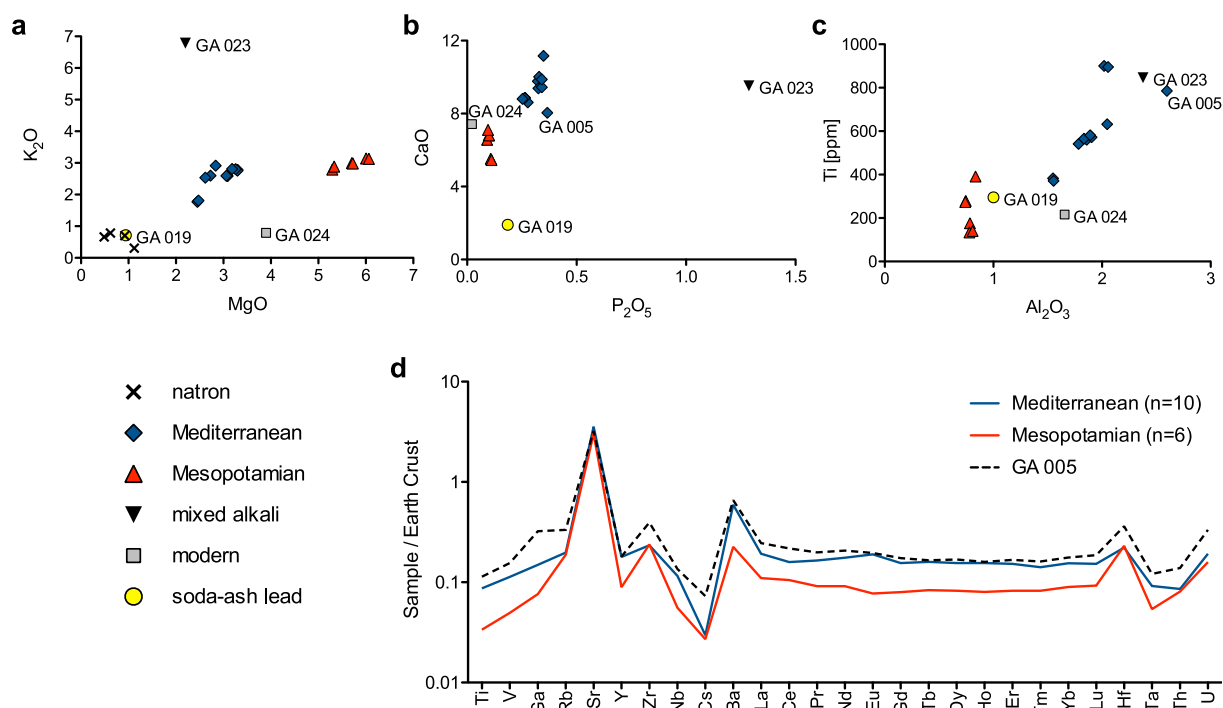
### 3.2. Plant ash glasses

Different magnesium and potassium oxide concentrations (Fig. 3a) indicate two distinct soda-rich plant ash glassmaking traditions. While the glasses with lower potash (< 3%) and magnesia contents (< 3.5%) may be associated with plant ash glasses of Mediterranean origin (Egypt, Syria-Palestine), the samples with higher MgO concentrations (> 5%) are more likely Mesopotamian (Freestone, 2006; Henderson et al., 2016; Phelps, 2018; Schibille et al., 2018). The two plant ash groups can be further separated based on their lime and phosphorus levels, suggesting different production methods and/or species of plants used for the preparation of the ashes. The group tentatively attributed to a Mediterranean provenance tends to have higher calcium and phosphorus oxide concentrations than glass from Mesopotamia

(Fig. 3b). The Mesopotamian glasses are furthermore characterised by very low silica-related impurities, having significantly lower aluminium and titanium oxide contents (Fig. 3c) and overall lower trace elements (Fig. 3d). Elevated levels of barium in the Mediterranean plant ash glasses (Fig. 3d) appear to be related to the significantly higher manganese contents of these fragments (Table S1).

Sample GA005 has been singled out, because it varies from the Mediterranean group due to its higher iron and heavy element concentrations (Fig. 3c, d) as well as exceptionally high lithium contents (Table S1). Whether or not these variations are related to the presence of cobalt in this sample is not clear. A zinc-rich cobalt source was used that is characteristic of ninth- to eleventh-century Islamic glass (Gratuze et al., 2018). The cobalt source of samples GA007 and GA012, on the other hand, has low cobalt to nickel ratios associated with a late antique cobalt colourant typically encountered in glasses dating to between the late fourth and seventh century CE (Gratuze et al., 2018).

Three pairs of samples (GA006/010; GA008/011; GA007/012) have a very similar composition with the concentrations across all elements within the experimental error as reflected in the standard deviation of



**Fig. 3.** Base glass compositions of the main glass groups and individual samples from Gauzón. (a)  $K_2O$  versus  $MgO$  [wt%] identifies three distinct groups in terms of the fluxing agents as well as soda-ash lead glass (GA 019), modern sample (GA 024) and the mixed alkali specimen (GA 023); (b)  $CaO$  versus  $P_2O_5$  levels confirm differences in the plant ash component between Syro-Palestinian and Mesopotamian glasses and the outliers singled out; (c) variations in the  $Al_2O_3$  and  $TiO_2$  contents separate the plant ash groups in relation to the silica source; (d) average trace element patterns of the two plant ash glass groups and sample GA005 normalised to the mean values of the upper continental crust (Kamber et al., 2005).

the repeated measurements of the glass reference material. This suggests that they may have derived from the same batch or object (Freestone et al., 2009). While the first pair came from the same stratigraphic layer, the latter two were retrieved from different contexts. These data are represented as separate samples (Table S1).

### 3.3. Individual samples

Sample GA019 contains 42%  $PbO$  (Table S1), corresponding to the soda-ash high lead glass type known from other ninth- to twelfth-century sites in al-Andalus (De Juan Ares and Schibille, 2017b; Duckworth et al., 2015). Hence, it can be assumed that sample GA019 may likewise date to this period even though it was recovered from a later thirteenth- or fourteenth-century layer. Sample GA023 has high potassium and phosphorus oxide concentrations (Fig. 3a, b) and potash to soda ratios that fall between European potash and Mediterranean soda plant ash glasses (Wedepohl et al., 2011). It derives from a thirteenth- or fourteenth-century context. Its relatively high levels of copper, tin, lead and antimony suggest the recycling and mixing of two different base glasses, most likely a potassium-rich glass of the central European tradition and an Islamic soda-rich plant ash glass (Table S1). Finally, sample GA024 is a modern soda-lime-silica glass with low levels of phosphorus, potassium and hardly any chlorine, evidently made from synthetic soda and a clean silica source. The relatively large amount of magnesium indicates a twentieth-century date (Dungworth, 2011), possibly from the glassmaking factory of Saint-Gobain in Avilés that has been active nearby since 1952.

## 4. Discussion

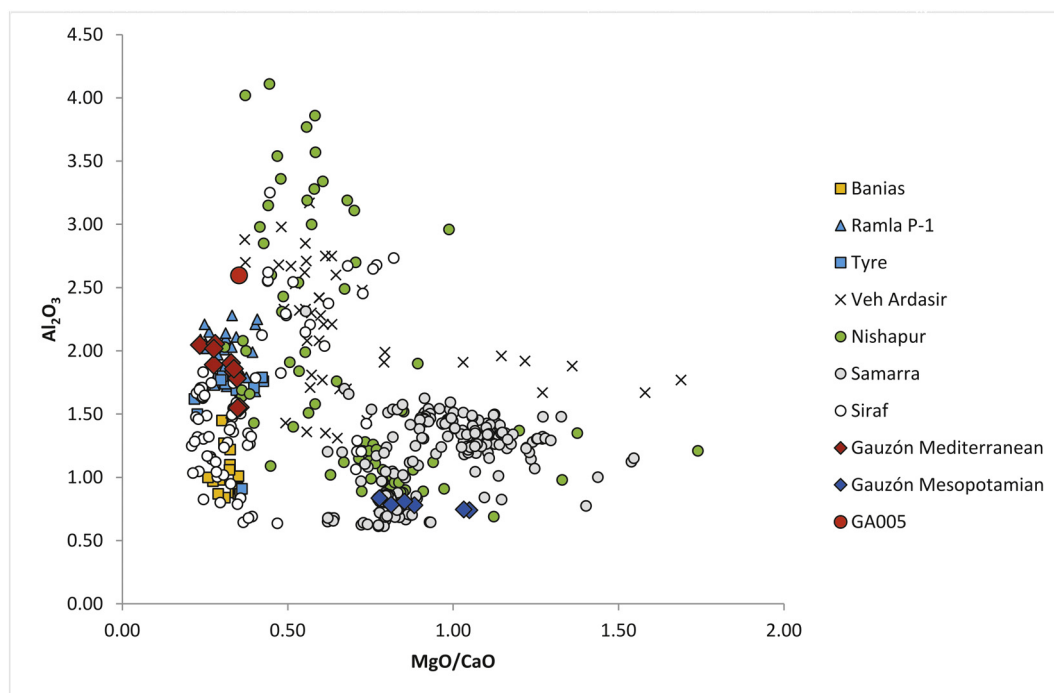
The analytical results of the glass samples from the castle of Gauzón reflect the presence of some natron glasses in medieval contexts, probably residual materials from an earlier occupation. The only natron glass with recognisable typology is a rounded and thickened edge with

horizontal, incised lines (GA015) that matches sixth- and seventh-century typologies of objects of the Foy-2 group to which it belongs (Foy and Bonifay, 1984; Sánchez, 2009) (Fig. 2). Some natron samples are the result of recycling, but no clear signs of mixing between plant-ash and natron glass was detected. The soda-ash lead sample can be likely attributed to the ninth to twelfth century CE based on its similarities with other glass finds from al-Andalus (De Juan Ares and Schibille, 2017b; Duckworth et al., 2015).

### 4.1. Mesopotamian and Mediterranean plant ash glasses

The presence of two distinct soda-rich plant ash glass groups at Gauzón is the most striking finding of this study. The binary graph comparing alumina contents to the ratio of magnesium to calcium oxide (Fig. 4) has proven effective to differentiate Mesopotamian from Mediterranean soda rich plant ash glasses (Henderson et al., 2016; Phelps, 2018; Schibille et al., 2018; Swan et al., 2017). One of the groups from Gauzón matches the compositional characteristics of Mesopotamian glass. It appears to be closely linked with Samarra group 1 (Schibille et al., 2018). Similar compositional groups were identified among the glass assemblages from ninth- to tenth-century Nishapur (Brill, 1995; Carboni and Whitehouse, 2001; Henderson et al., 2016; Wypyski, 2015) and Sasanian glass from Veh Ardasir (Mirti et al., 2009). This type of glass is characterised by high  $MgO/CaO$  ratios and surprisingly low phosphorus contents, as well as very low silica related impurities such as aluminium, titanium and zirconium (Table S1). These features point to the use of a relatively pure source of silica either in the form of quartz pebbles or a quartz rich sand (Brill, 1995; Schibille et al., 2018). Written sources indicate that quartz pebbles were used for Assyrian glass (Brill, 1970) and this has also been proposed for Sasanian glasses (Mirti et al., 2009) and ninth-century Islamic glasses from Raqqa (Henderson et al., 2005).

The Mediterranean plant ash glasses were produced from a different source of vegetable fluxes as well as from a silica source richer in



**Fig. 4.** Comparison of the data from Gauzón with soda-rich plant ash glasses from the Mediterranean and Mesopotamian regions.  $\text{Al}_2\text{O}_3$  versus  $\text{MgO}/\text{CaO}$  ratios separate Mediterranean from Mesopotamian soda-rich plant ash glasses. Data sources: Banias (Freestone et al., 2000), P-1 group from Ramla (Phelps, 2018), Tyre (Freestone, 2002), Sasanian glass from Veh Ardasir (Mirti et al., 2009), group 1 from Samarra (Schibille et al., 2018), Nishapur (Brill, 1995; Henderson et al., 2016), Siraf (Swan et al., 2017).

impurities (Fig. 4). This may be the reason why greater quantities of manganese were added to these glasses to mitigate the effects of the silica-related impurities and to obtain a colourless glass (Table S1). The samples from Gauzón appear closely related to the glass chunks from the primary production furnaces at Tyre (Freestone, 2002) and the glass vessels from group P-1 of Ramla (Phelps, 2018), dating to the middle of the eighth to the eleventh centuries, rather than to the samples from the eleventh- to thirteenth-century secondary workshops at Banias (Freestone et al., 2000). Recently it has been found that glasses from Iranian Sirāf are similar in their major element compositions to Mediterranean glasses making trace element analysis indispensable (Swan et al., 2017). Hence, the Gauzón glasses can be attributed to a Mediterranean origin with some certainty, given the close match with the finds from Ramla as regards also the trace element patterns.

#### 4.2. Relief-cut plant ash glass

Interestingly, the glasses from Gauzón do not have recognisable parallels with other contemporary Christian assemblages from France or northern Europe, and appear to support written sources that suggest that commercial and cultural contacts with France and other European regions remained limited until at least the end of the eleventh century (Calleja, 2017; Muñiz López and García Álvarez-Busto, 2016). Instead, the relief-cut samples can clearly be linked to Islamic finds from al-Andalus and the Near East, thus reflecting the economic and cultural hegemony of Islamic al-Andalus over the entire Iberian Peninsula, including to a certain extent the Christian kingdoms to the north (Valdés, 2007). Relief-cut glasses enjoyed great popularity in the Islamic world from al-Andalus and the Maghreb in the west to Khorāsān in the east (Carboni and Whitehouse, 2001; Foy, 2017a; Kröger, 1995). The presence of relief-cut artefacts in the early eleventh-century shipwreck of Serçe Limani (Bass et al., 2009) supports the impression that they were objects of an active and far-flung trade in the Mediterranean and beyond. In the ninth and tenth century, relief-cut vessels circulated widely in central Asia and the Near East. In the west, at Sabra al-Mansuriya in

Tunisia or in al-Andalus, for example, relief-cut vessels may have been introduced with a delay of about a century (Foy, 2017a). The origin of vessels with relief-cut decorations, therefore, is assumed to be either Egypt, Syria or Iran (Carboni and Whitehouse, 2001).

In terms of the chemical composition and by extension provenance of the vitreous material, relief-cut glasses have been made from both Mesopotamian and Mediterranean soda-rich plant ash glass. Relief-cut glasses of Mesopotamian composition have been identified among the finds from the Serçe Limani shipwreck (Bass et al., 2009), Nishapur, Samarra, and Ctesiphon (Brill, 1995; Henderson et al., 2016; Schibille et al., 2018; Wypyski, 2015), Ramla (Phelps, 2018) as well as Raya and Wadi al-Tur (Kato et al., 2010), and less frequently with a Mediterranean composition as in Wadi al-Tur (Kato et al., 2010). The analyses of the glasses from Gauzón confirm the different origins of the relief-cut glasses as they are represented among both the Mediterranean as well as the Mesopotamian plant ash groups (Table S1).

On the Iberian Peninsula, the most common decorative types are glasses with circles similar to sample GA017 that have been identified in Silves (Gomes, 2015), Córdoba (Rontomé, 2000), Mallorca (Capellà and Riera, 2015) or Santa Maria de Cap d'Aran (Velasco et al., 2011). Glass vessels with stylised vegetal, figurative or geometrical motifs such as samples GA008 and GA018 are less abundant. It is remarkable that all known examples come from sites that are closely related to the upper echelons of society. This is true for the specimens retrieved from the caliphal capital of Madīnat al-Zahrā (Rontomé, 2000), the citadel of Silves, seat of the regional government during the caliphate and short-lived capital of the Taifa kingdom in the eleventh century (Gomes, 2015), the citadel of Badajoz that was then seat of the Aftas dynasty in the eleventh century (García and Valdes, 1996), or the church of Sant Vicenç at Besalú (Velasco and Whitehouse, 2012). These archaeological contexts provide glimpses of the socio-economic patterns of exchange during the medieval period between the Christian kingdoms of Spain, Islamic al-Andalus and the wider Mediterranean. The social elites evidently controlled the movement of luxury goods, which in turn reflects the cultural value of these glass objects.



### 4.3. Trade routes

Traditionally, relief-cut vessels have been considered valuable objects that arrived in the Spanish Christian kingdoms as the result of looting of Muslim territories after the fall of the Caliphate at the beginning of the eleventh century. Other types of prestigious objects (relics, books, silks and jewellery) demonstrate the maintenance of commercial links between the south of the peninsula and the kingdoms of Asturias and León (Muñiz López, 2013; Muñiz López and García Álvarez-Busto, 2016). Unfortunately, the results from the investigation of the glass finds from Gauzón do not allow us to draw any definite conclusion as to whether they were the product of trade or looting. However, several textual sources such as numerous Arabic geographical texts (e.g. Ibn Hawqal, Idrīsī, al-Muqadasi, Yāqūt) describe the commercial links between al-Andalus and the Near East. Hence, even though no direct written records have been found that document the trade of glass between the eastern Mediterranean and Iberia during the early Middle Ages, some hypotheses can be put forward. An eleventh-century letter of the Cairo Genizah explicitly refers to the presence of Andalusian merchants in Jerusalem, Aleppo and Tyre (Valdes, 1991). Other letters testify to the trade of raw glass between Tyre and Egypt (Carboni et al., 2003) and several more reveal the intense connections of Denia, Almeria and Seville with the Egyptian ports (Constable, 1992). Therefore, it is not unlikely that eastern Mediterranean and Mesopotamian glasses were shipped from Levantine and Egyptian ports to al-Andalus, along the same path usually used by merchants, scholars or pilgrims from al-Andalus alike.

### 5. Conclusion

Analytical studies of archaeological glasses can evidently provide fundamental new insights into the mechanisms of medieval trade. However, more systematic studies are needed to determine the significance of this commerce, the degree of recycling activities or the impact of the crusades on the commerce and production of glass. New analytical studies of archaeological materials from the Christian and Muslim territories of the Iberian Peninsula and its border regions will make it possible to better establish the nature and scale of the commercial networks, their mechanisms of distribution and their evolution over time.

In medieval Spain, glass and especially relief-cut glasses were luxury objects that are associated with centres of power and that testify to a thriving long-distance exchange. The formal and compositional study of the glasses from Gauzón clearly shows that during the tenth and eleventh centuries supply networks of highly valuable and prestigious glass objects connected the Atlantic coast of the Iberian Peninsula under Christian rule with al-Andalus as well as with the Near East and Mesopotamia. This suggests that at least until the eleventh century Gauzón cultivated closer commercial links in terms of glass supplies with the Islamic world than with the Christian realm to the north (e.g. France). This phenomenon may be explained by the relative geographical isolation of Asturias and León from the rest of Europe and the economic power of al-Andalus during this period, the influence of which began to decline only in the following century.

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