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Preliminary results of the first archaeometric study of three medieval glass assemblages from Almada, Portugal

Resultados preliminares do primeiro estudo arqueométrico de três conjuntos de vidros medievais de Almada, Portugal

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Abstract

In the late 1990s and early 2010, archaeological excavations were conducted in three neighbouring streets of the old part of the city of Almada (Lisbon South bay), where medieval residential complexes were brought to light. Among the materials found in the Pátio Prior do Crato and Rua Latino Coelho, a group of glass fragments is dated between the 14th and 16th centuries. In addition, the glass assemblage of Rua da Judiaria counts more than 1,200 fragments covering a time span from the 12th to the 19th century, including some objects that have been identified as luxurious due to their decorations. This work integrates archaeological and archaeometric research and focuses on glass dating between the 14th and 16th centuries from these excavations in Almada. The combination of results intends to provide a first glimpse of glass usage and circulation in the southern area of Lisbon during the late medieval period.

KEYWORDS
Middle Ages
Glass
Portugal
Archaeology
Archaeometry

Resumo

No final da década de 1990 e início de 2010, foram efetuadas escavações arqueológicas em três ruas contíguas da zona antiga da cidade de Almada (baía Sul de Lisboa), onde foram postos a descoberto conjuntos residenciais medievais. Entre os materiais encontrados no Pátio Prior do Crato e na Rua Latino Coelho, um conjunto de fragmentos de vidro é datado entre os séculos XIV e XVI. Por outro lado, o conjunto de vidros da Rua da Judiaria conta com mais de 1200 fragmentos, abrangendo um período que vai do século XII ao século XIX, incluindo alguns objetos que foram identificados como de luxo devido às suas decorações. Este trabalho integra investigação arqueológica e arqueométrica e incide sobre vidros datados entre os séculos XIV e XVI provenientes destas escavações em Almada. A combinação dos resultados pretende fornecer uma primeira visão do uso e circulação do vidro na zona sul de Lisboa durante o período tardo-medieval.

PALAVRAS-CHAVE
Idade Média
Vidro
Portugal
Arqueologia
Arqueometria

Introduction

This study examines a collection of glass fragments recovered from three adjacent archaeological contexts in Almada, a Portuguese town located south of Lisbon along the Tagus River. To shed light on the use and circulation of glass in this region between the fourteenth and sixteenth centuries, this research combines the archaeological contextual analysis with stylistic assessment and chemical characterisation of the glass. This integrated approach will aim to inform hypotheses regarding the fragments' provenance and broader patterns of trade and use.

In the late 1990s and early 2010, archaeological excavations were conducted in *Almada Velha*, the historic part of the city, across three neighbouring streets: Pátio Prior do Crato, Rua Latino Coelho and Rua da Judiaria (Figure 1). The excavation at Pátio Prior do Crato revealed that the earliest human activities in the area involved the construction of residential structures and silos between the fourteenth and fifteenth centuries, with their use ceasing by the fifteenth century. In the sixteenth century, the area was abandoned as a residential space and repurposed as a courtyard. Five glass objects related to liquid consumption were discovered and during the excavations and linked to the initial period of occupation and residential construction (fourteenth- fifteenth century) [1]. All fragments were subsequently subjected to chemical analysis.

Following the renovation of a building facing the street, the excavation of Rua Latino Coelho was conducted. The earliest anthropic traces date back to the fourteenth century with the opening of silos [2]. Their abandonment, between the late fourteenth and early fifteenth centuries, was probably due to the location being unfavourable for storage, as they were positioned in a valley with poorly drained soil. In the sixteenth century, cisterns were built perhaps for cooperage or the production of cereal yeast. Regardless of their function, they were abandoned, and the land levelled in the second half of the sixteenth century [1]. Twenty four glass fragments were retrieved from the filling of the silos abandoned in the first years of the fifteenth century. The recognised shapes mostly refer to the consumption of liquids, such as beakers and goblets. Two bracelets, one dark blue and one of dark glass, stand out in the assemblage. Eight fragments were selected for chemical characterization.

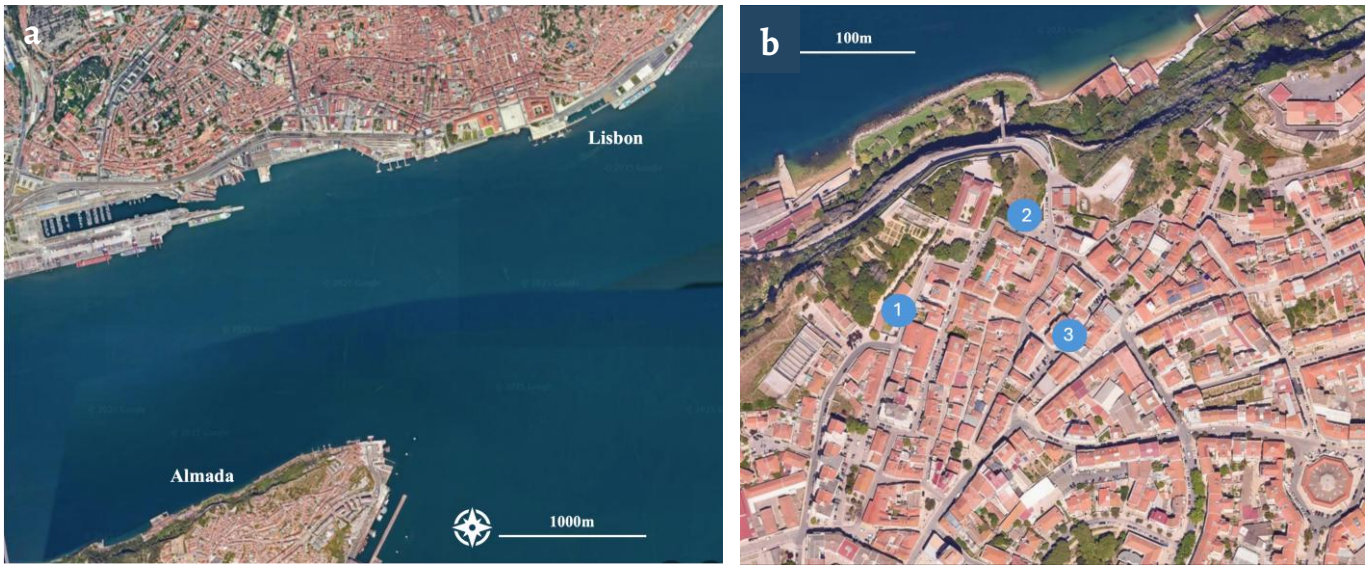


Figure 1. Maps: *a)* Lisbon south bay; *b)* Location of the three excavations in Almada: (1) Pátio Prior do Crato (APPC), (2) Rua Latino Coelho (ARLC), and (3) Rua da Judiaria (ARJ), Google Earth.

Archaeological excavations in Rua da Judiaria unearthed 22 silos carved in the Miocene rock, marking the earliest evidence of occupation in the area. They were probably part of a twelfth-century mercantile workshop, developed during the Islamic occupation of the city. Their filling has yielded various types of materials that can be dated between the twelfth and fifteenth centuries. By the fifteenth century, the silos were sealed, and residential buildings were constructed on the same site. These structures were later abandoned when the entire area was converted into a courtyard in the sixteenth century [3]. Of the various materials found, 1,200 glass fragments (twelfth-nineteenth centuries) were unearthed related to 400 objects [4]. This work includes 31 utilitarian vessels, mostly related to the table, dated between the fourteenth and fifteenth century, along with four personal adornments from the sixteenth century. The objects are described in detail in Medici, 2005, whose stylistic insights are integrated into this study. Additionally, 27 fragments were chemically characterised to support discussions of their provenance.

Glass production in Portugal in the Middle Ages

Reconstructing the history of Portuguese glass presents a difficult challenge due to the absence, to date, of archaeological findings attesting the presence of glass workshops after the fall of the Roman Empire. The earliest furnace finds that testify with certainty to the presence of glass production are from the excavation of the *Real Fábrica de Vidros Cristalinos de Coina*, active between the eighteenth and nineteenth centuries [5].

Excavations in the Islamic cities of Mértola and Silves, in the area known as Gharb al-Andalus, have returned glass fragments dated between the eleventh and twelfth centuries that suggest trade contacts with northern Syria, Egypt and other areas of al-Andalus [6-8]. Although little information is currently available on the presence and circulation of glass objects of the following centuries, some documentation and studies suggest an exchange of ideas and goods with the rest of Europe [9-10]. Documentary evidence refers to the activity of at least eleven glassmakers in and around the city of Lisbon from 1439 onwards, however, no reference is given to what type of glass was produced and these workshops have yet to be identified [5].

Studies conducted on glass assemblages from archaeological excavations dating between the fourteenth and sixteenth centuries have revealed contacts with Venetian production. The first evidence of the importation of Venetian glass into Portugal emerged from an excavation in the centre of Lisbon and is recognizable in a beaker with enamel decoration belonging to the group known as *Aldrevandino* dated to the fourteenth century [11-13]. Analyses conducted on a glass assemblage dated between the fourteenth and sixteenth centuries from Beja, in the southern region of the country, evidenced the import of a prunted beaker from Venice [14].

The chemical composition of other fragments from the collection from Beja indicated a shared origin, characterised by the use of alumina-rich silica and purified sodium-rich plant ash as a flux agent [14]. All fourteenth-sixteenth-century glass fragments studied so far belong to this soda-silica-lime type, linking Portuguese glass finds to contemporary Mediterranean production, where this type was common. A letter from King Afonso V, dated 1459, mentions the exclusive right of the Portuguese to harvest *barilla* – a plant whose ashes can be used as a flux agent – suggesting the possibility of local soda-rich glass production [15].

The archaeometric studies of a group of *façon de Venice* glass objects from the Monastery of Sta. Clara-a-Velha in Coimbra, Praça Miguel Fernandes in Beja and Monastery of São João de Tarouca, dated to the seventeenth century, have revealed the use of a silica source characterised by high levels of alumina. These objects were attributed to an unknown production centre leading to the hypothesis of local production [16-17]. Recent studies on silica sources, together with the stylistic features and the chemical composition of seventeenth-century glass fragments from the Monastery of Sta. Clara-a-Velha, led to conclude that high-quality glass,

which implied the use of feldspathic silica with a low level of impurities, was produced in the area of Coimbra [18].

Stylistic analysis

The five glass fragments found in the excavation in Pátio Prior do Crato are associated with the construction of a residential building in the fifteenth century. A coin of King Afonso V, found in the same layer, suggests a dating between 1449 and 1457 [1]. In Rua Latino Coelho, the assemblage consists of 24 fragments corresponding to ten objects, recovered from silos and pits dated between the fourteenth and sixteenth centuries [2]. At Rua da Judiaria, a total of 35 glass objects dated between the fourteenth and sixteenth centuries have been identified. These were retrieved from pits and rubbish deposits and have previously been documented in detail [4].

Across the three assemblages, the range of identified shapes can be related to objects used for the consumption of liquids, such as beakers, goblets, and bottles, alongside urinals and personal ornaments, like bracelets and a ring. While the weathering of some fragments makes it difficult to determine their original colour, most are colourless, with occasional blue, green, or yellow hues.

Out of the coloured objects, the most common colour is green, with shades ranging from blue-green and light green to dark green and olive green. The personal ornaments are notable for their dark glass, which appears almost black. Typically, such an appearance was achieved with an intense colouration of purple, brown, dark blue, or dark green [19].

Of the 45 vessels, 26 were produced using the free-blowing technique. The remaining objects feature decorative patterns on the body, such as ribs or rice grain patterns, that indicate manufacture by blowing the glass into a mould.

Just a few glass fragments were found in the contexts dated between the fourteenth-fifteenth century: in Rua da Judiaria, two beakers with pinched threads applied made of the same glass as the body of the object (Figure 2, ARJ0015, ARJ0039). This decorative technique is common in Medieval Europe between the thirteenth and sixteenth centuries [11, 20]. In Portugal, beakers with the same decoration have been found in fifteenth-century contexts of Beja [14] and Évora [21]. In Rua Latino Coelho and Pátio Prior do Crato, goblet bases with tubular base-ring and the neck of an elongated flask were yielded (Figure 2, ARLC0011-APPC0001).

As for the objects referable to the fifteenth century, the assemblage from Rua da Judiaria returns cylindrical beakers with mould-blown decoration, in low relief on the wall. A group of beakers presents a large and flat applied trail on the rim, melted with the rim itself (Figure 2, ARJ0058). Similar objects have been identified in Portugal, specifically in Beja [14] and in the assemblage of Largo do Corpo Santo in Lisbon (fragments currently under study), but no parallels have been documented outside Portugal to date.

The Rua Latino Coelho assemblage includes a fragment of a beaker with a 'grain of rice' mould-blown decoration (Figure 2, ARLC0001), of which an example is also recorded in the Rua da Judiaria assemblage dated to the sixteenth century (Figure 2, ARJ0501). Three goblets dated to the fifteenth century stand out for their decoration (Figure 2, ARJ0099, ARJ0452, ARJ453; Figure 3 and Figure 4 that allow them to be placed in the category of luxurious goods [4]. They are made starting from a single glass gather and then shaped by tooling. ARJ0099 has a large truncated-conical bowl and a low pushed-in base and is decorated with a mould-blown pattern of ribs in very low relief and a blue frill (Figure 2, ARJ0099 and Figure 3). The same decoration was detected on a colourless wall fragment presumed to be part of a goblet, from the excavation of Praça da Figueira in Lisbon (fragment currently under study). ARJ0452 has a shallow hemispherical bowl with applied spiralling blue trails and a higher pushed-in base (Figure 2, ARJ0452). Both are made of a very similar thin glass, colourless with a yellow tinge. The decoration with blue threads applied can be found in the production located in the south of

France between the fifteenth and sixteenth century [20], in Savona and in Monte Lecco in Italy in objects dated between the end of the fourteenth and the beginning of the fifteenth century [22].

Figure 2. Drawings of the analysed fragments (drawings of the fragments from Pátio Prior do Crato and Rua Latino Coelho, author: A. Cristoforetti; drawings of the finds from Rua da Judiaria, author: T. Medici, adapted from Medici, 2005).

Figure 3. Fragment of goblet decorated with a mould-blown pattern of ribs in very low relief and a blue frill-ARJ0099 (photograph: A. Cristoforetti).

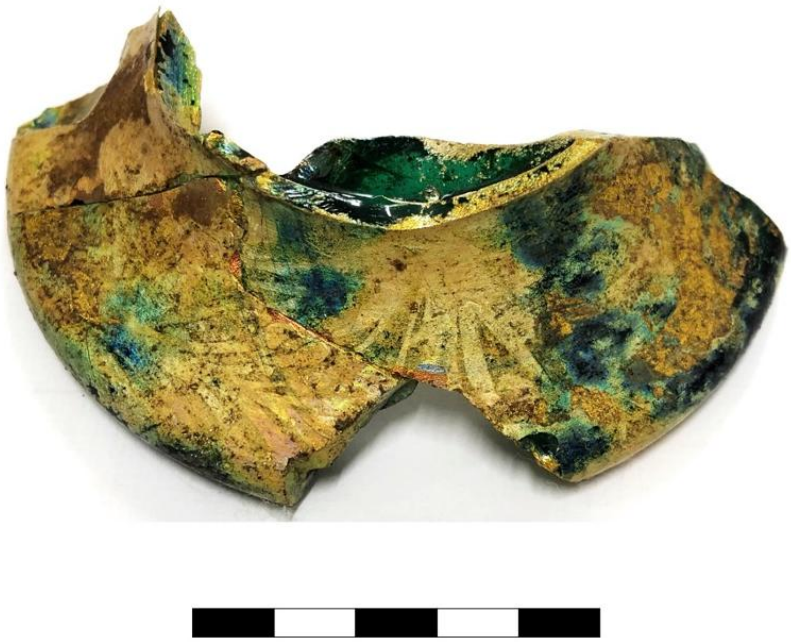


Figure 4. Dark green base of goblet with enamelled or gilded decoration-ARJ0453 (photograph: A. Cristoforetti).

ARJ0453 is a dark green-coloured goblet with a truncated-conical bowl and a larger pushed-in base with a tubular base (Figure 2, ARJ0453 and Figure 4). It shows a badly preserved enamelled decoration for which a floral/vegetal gilded pattern was proposed [4]. The corrosion layers that cover the fragment prevent an extensive iconographic study of the motif and the possibility that it may be a gilded decoration cannot be ruled out. Several fragments of feet, most of them with pushed-in bases and tubular base ring, free-blown or with a mould-blown

pattern of ribs, are recorded, together with two fragments of disk-shaped feet. A few fragments were probably part of a bottle (Figure 2, ARJ0410). A pushed-in tubular ring base (Figure 2, ARLC0005) from the assemblage of Rua Latino Coelho likely belonged to the truncated cone base of a beaker.

The characteristics of the objects dated to the sixteenth century are fairly constant and consistent. The goblets are usually characterized by a foot with a ring base (Figure 2, ARJ0138) and the stem consists of one or two solid or hollow knobs (Figure 2, ARLC0017). Only a few fragments of rims of bowls or plates have been identified (Figure 2, ARJ0051). The bottles show similar features from the fourteenth to the sixteenth century, the only detail in the later objects being the rim underlined by an applied thread.

A fragment of convex base can be attributed to a urinal, vessel whose shape remains almost unchanged over time from the fourteenth to the eighteenth century (Figure 2, ARJ0251). This shape is a widespread object throughout Europe, frequently used in medicine for uroscopy [11]. As for the other forms, there are containers with handles (Figure 2, ARJ0450) and a fragment of what would appear to be a spout perhaps from a jug (Figure 2, ARJ0054).

Excluding the goblets with blue and enamelled/gilded decoration for which an import was suggested, for most of the utilitarian vessels composing the assemblages a first hypothesis of Portuguese production was advanced based on the stylistic characteristic and the quality of the glass [4].

In the assemblages of Rua da Judiaria and Rua Latino Coelho, examples of bracelets are found, most of them of a black appearing colour, with circular or D-shaped sections (Figure 2, ARLC0012-ARJ0572). Only one of the bracelets is blue, twisted, with a circular section (Figure 2, ARLC0018). They find comparisons with objects dated between the fourteenth and seventeenth centuries from contexts such as Portillo (Valladolid) [23], Toledo [24] and Malaga [25]. In Portugal, the same types are found in Beja [14].

The first sporadic records of glass bracelets are found in Egypt, dated to the second millennium BCE. They became popular in Europe in the Iron Age and their presence is attested during the Roman period in western Europe [26], including Portugal [27-29]. After the fall of the Roman Empire, the use of this personal ornament seems to be restricted to Palestine and the Byzantine world. A new diffusion of glass bracelets occurred in the seventh century after the Islamic Conquest of Palestine and the subsequent expansion of Muslim rule. In Europe, after the Roman age, such bracelets are found in areas that have had Islamic influence [25, 30].

In the Iberian Peninsula, they represent an object that connects the kingdoms of Portugal and Castile in the absorption of the Islamic customs [31]. Several fragments found in the archaeological excavation in Beja, demonstrate the Portuguese use of glass bracelets between the fourteenth and fifteenth centuries [14]. Furthermore, this type of object was also found in a Portuguese colony in Morocco, Alcácer Ceguer, where a Christian tomb dated to the fifteenth century was unearthed with a female adolescent wearing bracelets on both wrists [32].

Experimental

In order to obtain the chemical composition of the glass under investigation, to discuss the raw materials used, and to advance hypotheses on the origin, a total of 39 fragments (Table 1; Figure 2) from the three assemblages were analysed using particle-induced X-ray emission (μ-PIXE). This is an analytical technique widely used in the field of cultural heritage and is increasingly applied to glass material for the detection of chemical composition [33-34]. The selection favoured those with decoration as stylistic analysis identified them as possible imported objects. It should be noted that objects with decoration were probably produced with greater care in selecting the raw materials, as they were intended for the highest strata of society. Furthermore, at least one representative of each shape identified in the chrono-typological study was selected.

Table 1. List of the analysed fragments.

Sample	Type	Colour	Dating*
ARJ0015	Beaker with applied impressed trail	Green	14th /15th cent.
ARJ0039	Beaker with applied impressed trail	Green	
ARLC0005	Beaker	Colourless	
ARLC0011	Goblet	Colourless	
APPC0003	Goblet	Light green	
ARLC0006	Goblet	Colourless	
APPC0005	Goblet	Colourless?	
APPC0001	Flask	Light blue	15th cent.
ARLC0001	Beaker	Colourless	
ARJ0501	Beaker with mould-blown pattern of rice grains	Colourless -green hue	
ARJ0058	Beaker with flat ribbon applied trail	Colourless	15th/16th cent.
ARJ0176	Goblet with mould-blown pattern of ribs	Colourless	15th cent.
ARJ0138	Goblet	Colourless	16th cent.
ARLC0017	Goblet	Colourless	15th/16th cent.
ARJ0099	Goblet decorated with mould-blown pattern of ribs and an applied blue frill	Colourless	16th cent.
ARJ0452	Goblet with mould-blown pattern of ribs and applied spiralling dark blue trails	Colourless	
ARJ0453	Goblet with enamelled decoration - perhaps gilded	Green	15th cent.
ARJ0001	Bottle	Colourless	
ARJ0410	Bottle	Green	
ARJ0051	Plate	Colourless	16th cent.
ARJ0450	Vertical handle	Light green	
ARJ0022	Goblet	Colourless- yellow hue	15th cent.
ARJ0027	Beaker?	Colourless	
ARJ0054	Spout	Colourless -yellow hue	15th/16th cent.
ARJ0251	Urinal	Green	15th cent.
ARJ0029	Bracelet	Dark glass	
ARLC0012	Bracelet	Blue	
ARLC0018	Bracelet	Dark glass	
ARJ0572	Bracelet	Dark glass	16th cent.
ARJ0574	Bracelet	Dark glass	
ARJ0573	Bracelet	Dark glass	
ARJ0571	Ring	Dark glass	
ARLC006	Goblet?	Colourless?	15th cent.
APPC005	Goblet?	Colourless?	
ARJ0172	Beaker with mould-blown decoration pattern of ribs	Colourless	
ARJ0156	Goblet?	Light blue	
ARJ0089	Bottle?	Colourless	
ARJ0077	Goblet?	Colourless	
ARJ0175	Goblet?	Colourless -blue hue	

*The proposed dating of the objects is based on the archaeological reports submitted by the archaeological team of Câmara Municipal de Almada and Museu Municipal de Almada, supported by the study of ceramic objects and the presence of coins [1-3].

Special attention is given to personal adornments since these objects are not often part of archaeometric studies and there are few publications to compare the results. The idea is to start building a basis for further research on this type of object, its origin and its connection to their tradition. In the analysis of the bracelets, it must be borne in mind that the choice of raw materials may be dictated by the fact that their production does not require processing at such high temperatures, unlike blown objects [19].

To avoid erroneous results by analysing and quantifying corrosion layers instead of the uncorroded bulk glass, it was decided to sample all selected objects. Small glass samples of 2-4 mm^2 were dry cut from the selected glass fragments with a diamond wire. Samples were embedded in an epoxy resin and polished with SiC sandpapers down to 4000 mesh. This sampling procedure was performed only in broken objects and on individual fragments without possible connections. The analyses are carried out in collaboration with the Instituto Superior Técnico, Universidade de Lisboa (C2TN-IST/UL) using an Oxford Microbeams OM150 type scanning nuclear microprobe setup, with the in vacuum configuration which allows increased sensitivity in the detection of light elements. The samples were irradiated with a in vacuum to detect efficiently lighter elements such a Na, with low-energy X-rays first -1 MeV proton beam for the detection of lighter elements as silicon and alumina. For the quantification of elements with a higher atomic number, such as lead, a higher energy was used, a 2 MeV proton beam.

The results were manipulated using GUPIX program to obtain oxides concentrations and, to help validate the results a glass reference standard, Corning B, was also analysed. The results are expressed in weight percentage of oxides and were normalised to 100 % (Table 2).

Results and discussion

According to the results of the μ-PIXE analysis presented in Table 2 all fragments can be classified as soda-lime-silica glass. The main component, silica, has a concentration ranging between 54.2 and 68.2 wt%, Na_2O varies between 14.9 wt% and 21.4 wt% and CaO varies between 2.4 wt% and 9.6 wt%. The use of halophytic coastal plant ash (*Halogeton sativus* or *Salsola kali*) as flux agents is suggested by the amount of MgO and K_2O above 2 wt%, and the presence of chlorine close to 1 wt% [35]. Throughout the Roman Empire, the most common alkali source used by glassmakers added to lower the melting temperature of silica (1700 °C) was the soda contained in natron blocks from Egypt. Due to supply issues around the ninth century, glass production in the Islamic Near East began to replace natron with soda obtained from alternative sources, such as the ashes of coastal halophytic plants, which grow in saline deserts, marshes, and along seashores [36].

Table 2. Composition of the analysed samples determined by μ-PIXE, in weight percent of oxides.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	Fe_2O_3	CoO	CuO	PbO
ARJ0015	21.3	4.6	8.2	55.4	0.48	0.12	1.04	2.61	3.43	0.09	0.33	0.53	< DL*	0.01	0.01
ARJ0039	17.9	3.5	5.7	58.6	0.52	0.16	0.97	2.96	7.00	0.33	0.57	1.07	< DL	0.01	< DL
ARLC0005	16.3	3.6	2.8	67.1	0.26	0.06	0.98	1.54	4.42	0.07	0.94	0.76	< DL	< DL	< DL
ARLC0011	16.8	3.9	3.0	66.5	0.27	0.10	1.09	1.71	4.78	0.08	0.81	0.65	< DL	0.01	< DL
APPC0003	18.6	2.3	3.7	63.1	0.62	0.10	1.30	3.40	4.50	0.12	0.79	0.69	0.01	0.01	< DL
ARLC0006	16.6	3.4	4.4	59.5	0.21	0.18	0.83	5.73	6.34	0.17	0.48	0.67	0.01	0.01	0.10
APPC0005	20.5	4.7	2.7	64.4	0.40	0.10	1.25	1.83	2.45	0.21	0.12	0.56	< DL	0.01	< DL
APPC0001	19.7	3.7	4.7	62.3	0.40	0.12	1.10	2.42	2.83	0.27	0.67	0.94	< DL	0.01	< DL
ARLC0001	17.2	3.7	2.9	65.0	0.28	0.09	1.03	1.79	4.90	0.08	1.09	0.80	< DL	0.05	0.05
ARJ0501	16.3	3.1	4.4	57.7	0.29	0.17	0.79	5.47	8.77	0.17	0.54	0.83	< DL	0.01	< DL
ARJ0058	17.3	3.4	2.6	67.1	0.37	0.08	1.07	1.94	4.38	0.05	0.67	0.29	< DL	< DL	0.01
ARJ0176	18.5	4.7	4.1	62.4	0.67	0.06	1.00	2.43	3.76	0.12	0.63	1.10	0.01	0.01	< DL
ARJ0138	15.5	3.3	4.1	59.7	0.31	0.15	0.72	4.40	9.11	0.11	0.31	0.60	0.01	0.01	0.02
ARLC0017	16.9	3.5	4.5	59.8	0.26	0.16	0.86	5.59	6.30	0.17	0.47	0.66	0.01	0.01	0.10
ARJ0099	17.3	2.4	3.5	64.4	0.44	< DL	1.03	3.61	4.16	0.42	0.29	1.48	< DL	0.21	< DL
ARJ0452 (core)	18.2	2.3	3.5	64.7	0.53	0.07	1.12	3.29	4.03	0.42	0.29	1.00	< DL	0.01	< DL
ARJ0452 (decoration)	16.8	2.2	3.4	65.1	0.48	< DL	1.18	3.47	4.21	0.44	0.30	1.37	0.25	0.26	< DL
ARJ0453	18.8	2.4	4.1	59.4	0.71	0.11	1.11	3.60	5.35	0.58	0.18	1.53	< DL	2.53	< DL
ARJ0001	14.9	3.1	2.9	64.0	0.57	0.18	0.92	2.90	8.07	0.15	0.52	0.92	< DL	0.01	< DL
ARJ0410	15.2	3.1	2.9	64.3	0.54	0.12	0.87	2.85	7.69	0.37	1.07	2.25	0.01	0.01	< DL
ARJ0051	21.1	5.6	2.5	60.7	0.25	< DL	1.13	1.60	5.29	0.06	0.65	0.47	< DL	0.01	< DL
ARJ0450	15.0	3.2	3.6	59.1	0.24	0.15	0.70	6.43	9.61	0.19	0.47	0.59	0.01	0.01	< DL
ARJ0022	21.1	4.8	4.7	58.3	0.68	0.10	1.14	2.73	3.96	0.18	0.44	1.48	0.01	0.01	< DL
ARJ0027	18.0	3.2	4.5	62.5	0.48	0.08	1.09	2.71	4.19	0.45	0.43	1.66	< DL	0.01	< DL
ARJ0054	17.1	4.5	2.3	68.2	0.29	0.08	0.99	1.36	3.32	0.10	0.32	0.79	< DL	0.01	0.02
ARJ0251	15.3	3.1	5.7	57.8	0.23	0.11	0.71	6.29	7.63	0.25	0.95	1.27	0.02	0.05	< DL
ARJ0029 (bracelet)	18.4	4.6	6.8	54.9	0.29	0.38	0.47	3.10	8.72	0.25	0.04	1.36	< DL	< DL	< DL
ARLC0012 (bracelet)	19.1	3.3	5.8	57.3	0.65	0.70	1.19	2.26	5.97	0.28	0.07	2.42	< DL	0.03	< DL
ARLC0018 (bracelet)	15.7	3.4	3.6	64.1	0.60	0.15	0.88	2.71	6.28	0.10	0.34	0.94	0.08	0.05	0.11
ARJ0572 (bracelet)	20.4	4.3	3.2	59.8	0.55	0.15	0.90	2.90	4.03	0.17	1.38	0.82	0.01	0.05	< DL
ARJ0574 (bracelet)	19.8	6.0	7.0	54.2	0.31	0.42	0.42	3.38	6.34	0.19	0.05	1.04	0.01	0.01	< DL
ARJ0573 (bracelet)	16.5	4.2	6.8	57.1	0.36	0.52	0.52	2.95	8.71	0.24	0.03	0.90	< DL	< DL	< DL
ARJ0571 (ring)	18.5	4.0	9.3	52.6	0.62	0.69	1.01	2.53	5.06	0.66	0.01	3.73	< DL	0.01	0.10
ARLC006	16.6	3.4	4.4	59.5	0.25	0.18	0.81	5.72	6.34	0.17	0.48	0.67	0.01	0.01	0.10
APPC005	20.5	4.7	2.7	64.4	0.40	0.10	1.24	1.82	2.45	0.21	0.11	0.56	< DL	0.01	< DL
ARJ0172	16.6	2.8	5.0	61.1	0.50	0.09	0.97	2.94	7.27	0.15	0.66	0.97	0.01	0.01	< DL
ARJ0156	21.4	4.4	4.9	60.3	0.33	< DL	1.22	2.12	2.81	0.16	0.77	0.74	< DL	< DL	< DL
ARJ0089	16.3	4.0	3.1	66.8	0.27	0.07	0.91	1.68	4.39	0.07	0.78	0.66	0.01	0.01	< DL
ARJ0077	16.1	3.9	3.0	67.2	0.30	0.07	0.91	1.73	4.43	0.07	0.83	0.61	0.01	0.01	< DL
ARJ0175	19.3	4.4	2.5	65.4	0.44	0.08	1.15	1.67	3.22	0.10	0.19	0.66	0.01	0.01	< DL
CMOG B	16.5	0.99	4.2	62.6	0.71	0.59	0.16	1.01	8.40	0.11	0.25	0.35	0.04	2.89	0.51
Reference [37]	17.0	1.03	4.36	62.3	0.82	0.49	0.16	1.00	8.56	0.09	0.25	0.34	0.04	2.66	0.61
St. D.	0.3	0.09	0.1	0.2	0.01	0.04	0.03	0.01	0.1	0.01	0.004	0.006	0.005	0.07	0.01

*DL = Detection Limit

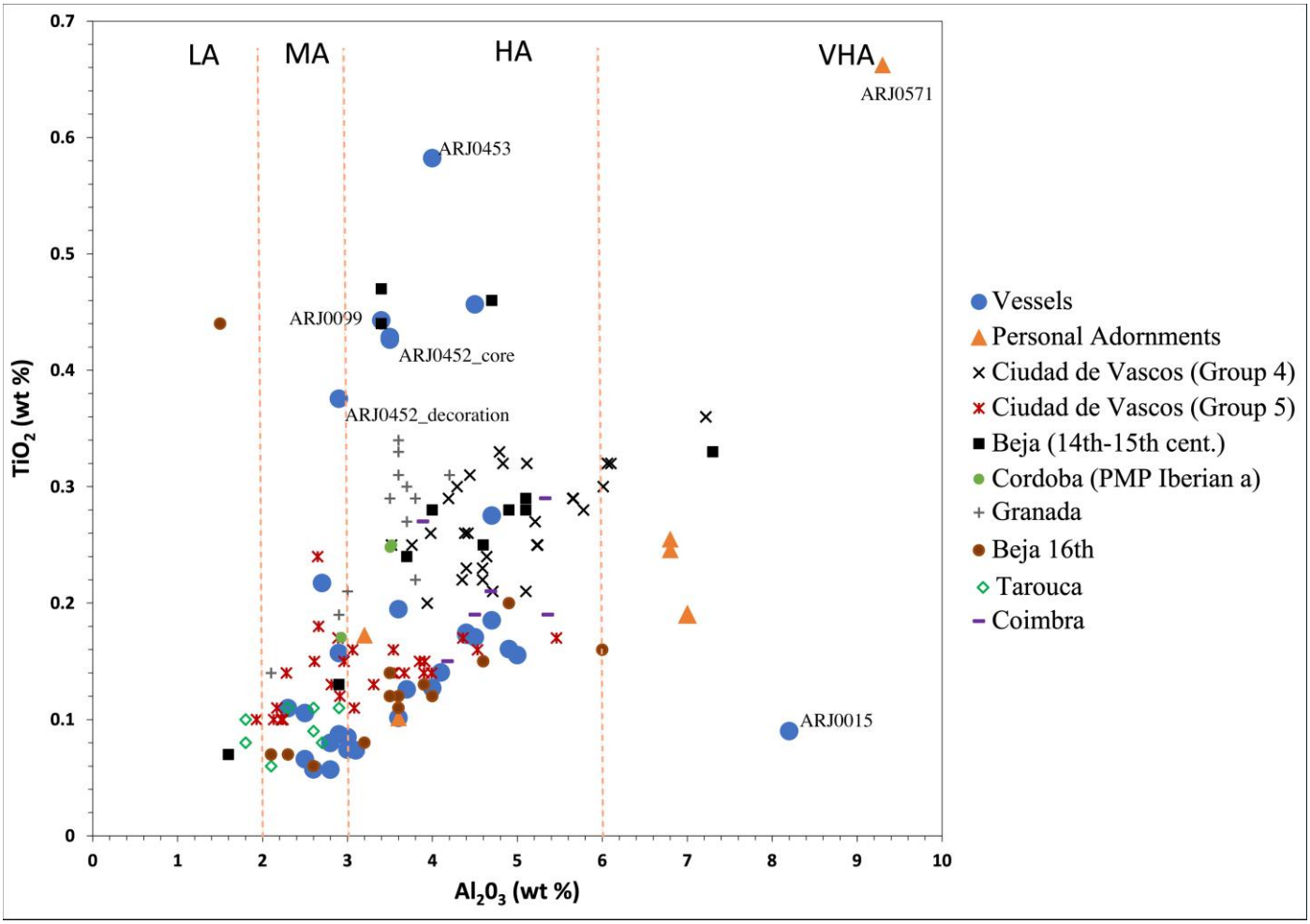


Figure 5. Binary plot of alumina *vs*. titanium oxide. The division based on alumina levels is proposed by Lima et al. [16]. The plot reports values from glass fragments from coeval archaeological excavations in the Iberian Peninsula: Ciudad de Vascos (Groups 4 and 5) [38], Córdoba (Iberian a) [39], Granada [40]; Beja dated to the 14th/15th century [15] and 16th century [17], and Coimbra [16].

Silica is the main component of glass, the so-called vitrifying agent. When added to the batch, silica unintentionally brings contaminant compounds in small amounts, such as alumina or titanium and iron oxides. Their presence in the sand silica source depends on the nature of the local geological setting, giving information to discuss the possible origin of the raw materials involved in the glass production [41-42]. Taking this in consideration, the contents of alumina and titanium oxide of the fragments under study were plotted (Figure 5).

Most of the samples have a TiO_2 concentration below 0.35 wt% and fall into the categories identified as Medium and High Alumina (MA: 2-3 wt%, HA: 3-6 wt%).

Comparing the levels of titania and alumina with available literature data on glass objects in the Iberian Peninsula (Figure 5), similarities can be observed with Islamic plant ash glass fragments in al-Andalus from Ciudad de Vascos [38] and Cordoba [39], dated between the tenth and the twelfth century. In these studies, the fragments with higher concentrations of alumina are proposed to be produced with local raw materials [38-39]. Furthermore, for the fragments with high alumina levels, correspondence is recorded in coeval glass objects (fourteenth-fifteenth century) unearthed in Beja, in the south of Portugal [14]. On the other hand, no parallels are found with the production remains and objects dated between the sixteenth and seventeenth centuries from *Calle Horno del Vidrio*, Granada [40]. Outside the Iberian Peninsula, similar concentrations of alumina are also found in studies of objects dated between the thirteenth and the sixteenth centuries from Italy, in Liguria [43] and Tuscany [44].

Considering the impurities present in the silica sources, iron-alumina-titania, the group that falls into the MA boundaries finds affinity with the cluster identified as "low-lead" by Duckworth 2016. No relationship can be advanced due to the absence of lead found in the

objects examined [45]. Fragments with MA have iron concentrations below 1 wt%, indicating the use of high-purity feldspathic silica sources, suggesting an attempt to produce high-quality items using local sources. Similar values, for both group MA and HA, in terms of silica sources emerge in comparison with compositions of *façon de Venice* objects between the sixteenth and the seventeenth century from Coimbra, Tarouca and Beja, for which a common origin from a hitherto unknown location has been proposed, not excluding they could be part of a local Portuguese production [16-17].

A group of fragments with high titania (> 0.40 wt%) and alumina (3.4-4.4 wt%) stands out. For the goblets with applied blue thread decoration (ARJ0099, ARJ452), a possible provenance from Altare or Monte Lecco has been proposed for stylistic reasons, however, the comparison with the chemical composition from these locations could not confirm the hypothesis [43, 46]. Looking at the chemical compositions of ARJ0452 and ARJ0099, one can note that they are almost identical, suggesting that the two objects were highly likely produced in the same workshop. It cannot be ruled out that the same recipe and raw materials were used, probably from the same batch. Furthermore, their silica source composition corresponds to a colourless beaker with blue decoration, unearthed in Beja [14] suggesting the hypothesis of a common origin. ARJ0453 differs from ARJ0099, and ARJ452 by higher TiO_2 concentration (0.58 wt%), leading to the proposal that it was produced with a different silica source. Four of the six fragments related to personal adornment fall in very high alumina (> 6 wt%), implying the use of a silica source richer in impurities, probably from sands richer in feldspar. The ring ARJ0571 stands out for the very high levels of alumina (9.3 wt%), titanium (0.66 wt%), and iron (3.79 wt%).

The only vessel that falls in VHA (Very High Alumina) is ARJ0015, revealing a different source of silica, if compared with the other objects, both personal adornment and vessels.

The presence of magnesium oxide, above 2 wt%, as well as the levels of K_2O (1.36-6.43 wt%), P_2O_5 (0.07-0.68 wt%), and Cl (0.47-1.33 wt%) implies the use of coastal plants from halophytic species as a flux [47]. The type of vegetable ash used in the batch can be discussed by correlating sodium and potassium normalized to the content of all alkaline and alkaline-earth oxides (Figure 6), to identify whether it was purified ($Na_2O^*+K_2O^*= 0.75$) or used without purification treatments (impure) ($Na_2O^*+K_2O^*= 0.6$) [48].

Figure 6 shows that most fragments fall in the section that corresponds to the *cristallo* boundaries [48]. It is assumed that purified ashes with characteristics comparable to Levantine ones were used, showing a careful selection of raw materials. All the fragments with a silica source characterised by MA and levels of titania below 0.3 wt% fall in this area, supporting the idea of a careful selection of raw materials for these objects. Part of this group are also the three decorated goblets (ARJ0099, ARJ0453, ARJ0452). A second substantial cluster lies between the two correlation lines, some fragments are close to the boundaries that correspond to *vitrum blanchum I* [48] or *vitrum blanchum II* [47], probably implying the use of less pure ashes or ashes for which the purification process was not completed.

Most of the fragments of personal adornment objects fall close to the area of *Vitrum blanchum I*, and two within the *cristallo* boundaries, which given the silica sources, are the ones falling into the VHA alumina category. This indicates the involvement of two different technologies in the preparation of the alkaline component, corresponding to two distinct silica sources.

Most of the fragments show an unintentional greenish or bluish colouration caused by impurities introduced with the silica source, such as iron [41], the presence of which is higher or close to 1 wt% in several objects. The fragments where Fe_2O_3 content is <1 wt% are colourless. In this latter group, most have alumina levels above 3 wt% and fall in the High Alumina category defined by Lima et al. [16] (Figure 7a). For these objects, the use of silica sources of felspathic origin of high purity, with low iron contents is proposed. This might suggest that they were selected carefully to produce high-quality objects.

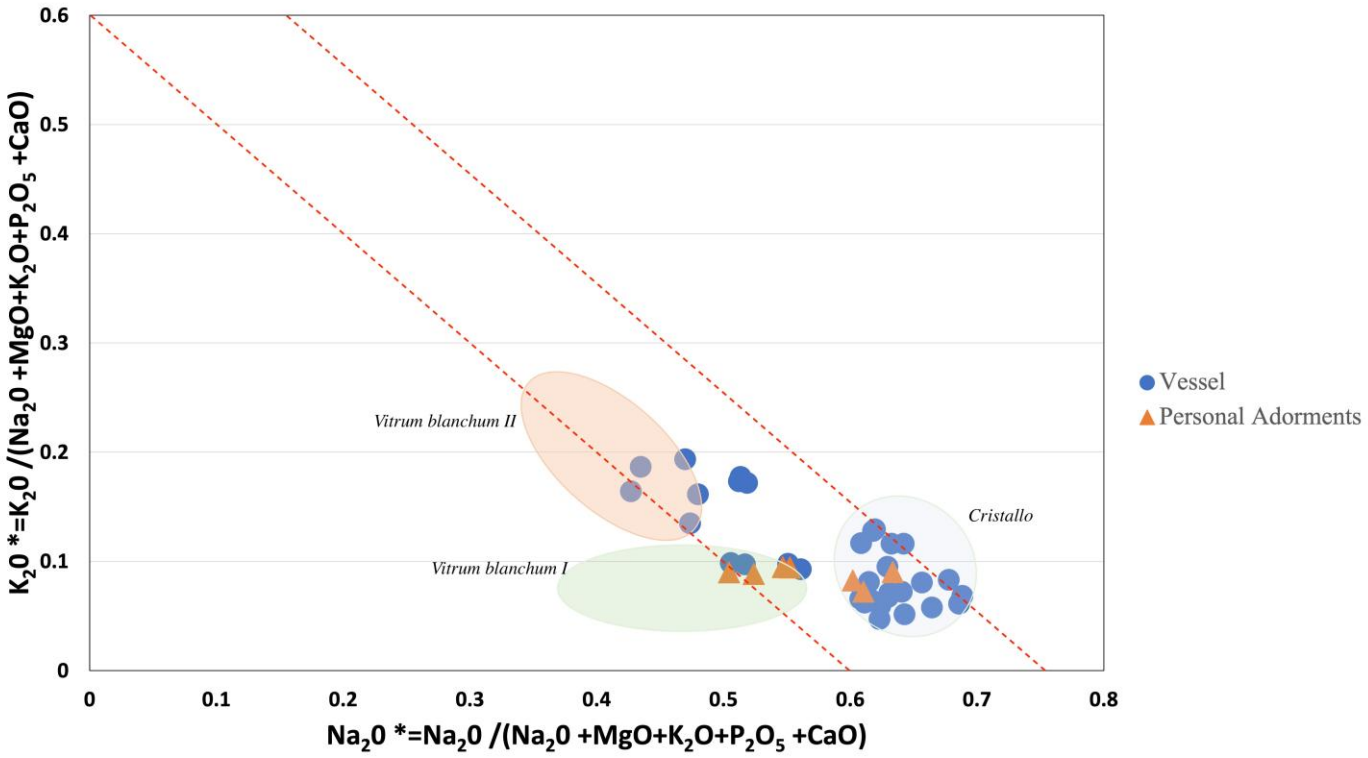
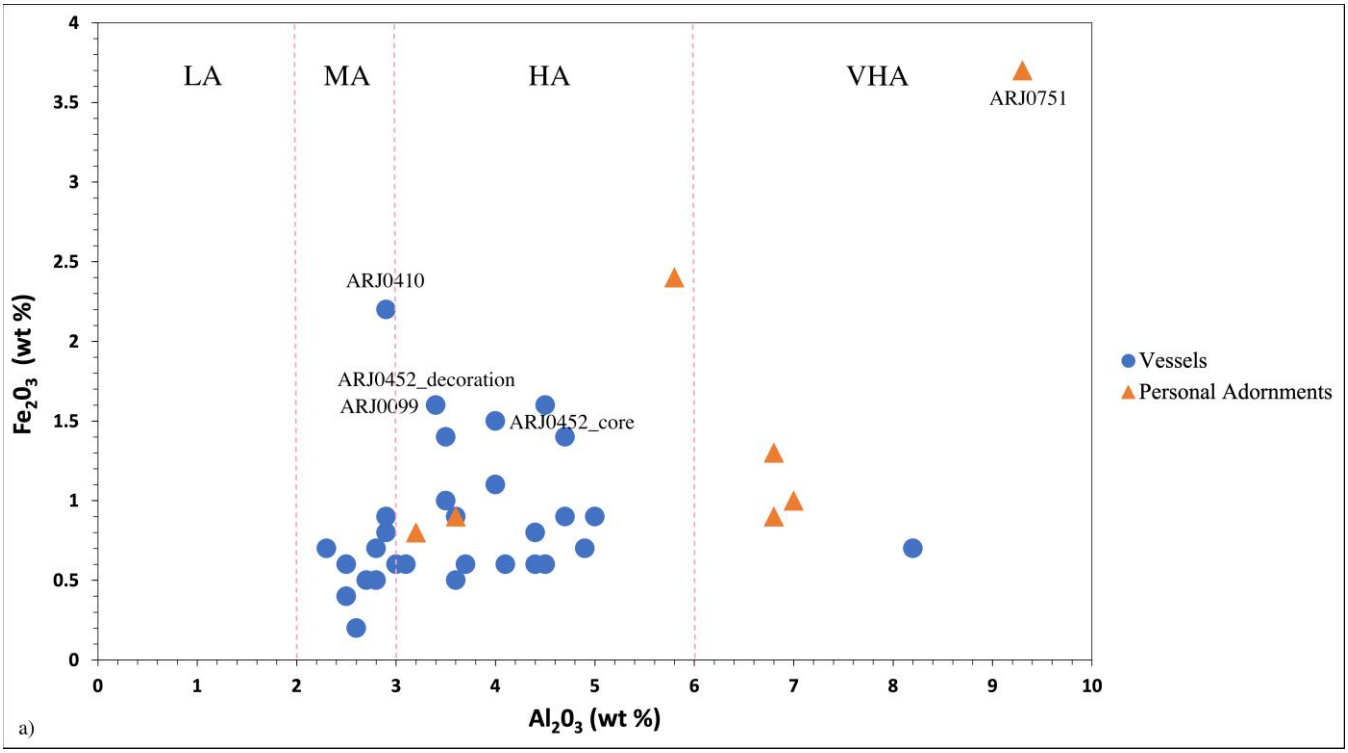


Figure 6. Binary plot of Na_2O^* vs. K_2O^*. Na_2O^* and K_2O^* values are obtained through the division of the respective oxide by every component introduced by the ash (Na_2O, MgO, P_2O5, K_2O and CaO). It is possible to observe the Venetian *cristallo* boundaries, as well as the two known *vitrum blanchum* areas [47].

Intentional colouring is recognisable in some objects, such as ARJ0410 and ARJ0291 with a light green colour due to the co-presence of iron (1.2-2.2 wt%) and cobalt oxides (0.01-0.02 wt%). ARJ0453 is distinguished by its deep green colour indicative of an intentional addition of copper (2.53 wt%). As for the blue colouring of the ARJ0452 applied decoration, it is due to the addition of cobalt at 0.25 wt%. The decoration and the body glass of ARJ0452 have an almost identical composition in terms of silica sources and flux agents, meaning that the same glass was probably used for the body and the decoration.

For fragments with MnO concentrations below 0.5 wt%, it was likely not added intentionally for its colouring or decolourising properties but instead is present naturally in the glass mixture and raw materials used in production. On the other hand, objects with a higher MnO content (between 0.5 and 1.5 wt%) indicate that the final colour of the objects is the result of decolourisation [49]. Fragments ARJ0410 and ARJ9251, with MnO concentration around 1 wt%, present a green colouration. The colouring agent of the personal adornments is still under discussion. Considering the fragments with VHA, these all have MnO levels below 0.15 wt%, leading to the exclusion of manganese being the colouring agent. In this group, ARLC0012 and ARJ571 stand out for their high concentration of iron (2.42 wt%, 3.73 wt% respectively), suggesting it was deliberately added to achieve colouration. As for ARJ0574 the addition of iron (1.04 wt%) and cobalt (0.01 wt%) oxides is probably responsible for the dark colouring. Considering the fragments that are part of the MA group, it can be observed that different oxides were added as colouring agents: cobalt to ARLC0018 (0.08 wt%) and manganese (1.38 wt%) to ARJ0572. These discrepancies reveal that a multiple approach was employed for the choice of raw materials and in the selection of colouring agents.



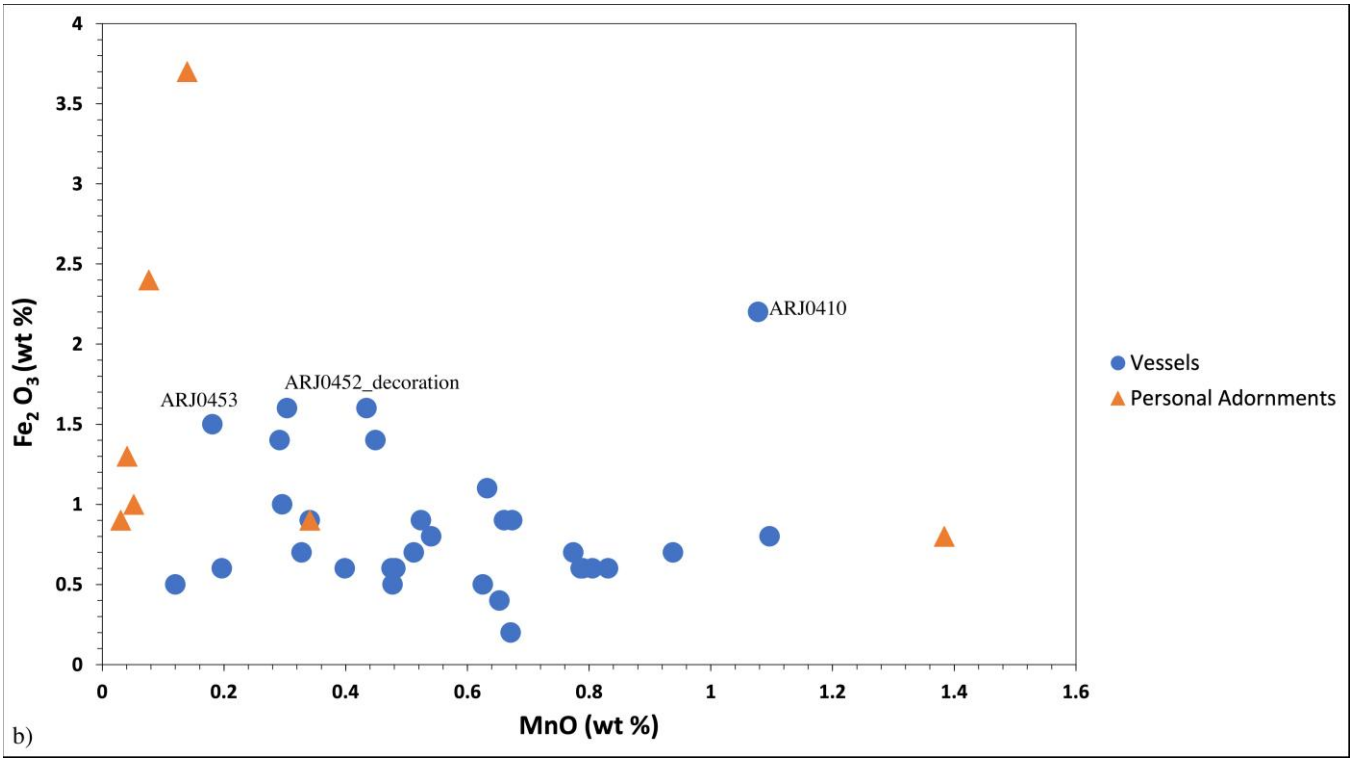


Figure 7. Binary plot of: *a)* alumina *vs.* iron oxide; *b)* manganese oxide *vs.* iron oxide.

Conclusions

The μ-PIXE analysis of selected fragments from three late medieval archaeological excavations in Almada allowed for the quantification of major and minor elements, which enabled the discussion of the type of glass and the origin of the raw materials involved in the production. The results of this first archaeometric study show that the assemblages are composed of soda-silica-lime glass type, disclosing a link with the Mediterranean production tradition.

For most of the vessels, an initial stylistic investigation identifies a possible common origin due to their quality and simplicity. These are utilitarian objects, mostly related to the consumption of liquids, but also indicate the presence of activities related to medical practices, as in the case of the urinals. The goblets with decoration suggest the presence, in the southern area of Lisbon, of high social classes who could afford to acquire luxury goods. However, based on the currently available evidence, it is not possible to propose definitive hypotheses regarding their origins. The function or cultural significance of the associated bracelets remains undetermined.

Looking at the chemical analysis, some preliminary observations can be advanced on the objects analysed. Most of the fragments show a similar composition in terms of silica source with medium or high alumina concentrations implying the use of a feldspathic silica source and the use, as a flux agent, of purified or pure plant ashes. Considering the studies on Portuguese glass, similar compositions are found with analysis conducted on late medieval glass objects from Beja. Thus, a recurrent pattern can be observed in the composition of glass circulating in Portugal between the fourteenth and sixteenth centuries. These characteristics were also recognized in the analysis of *façon de Venice* glass dated to the seventeenth century, unearthed in different areas of the country.

For the group of fragments related to vessels with medium alumina levels and iron below 1 wt%, the use of purified ashes as a flux agent was detected, suggesting an attempt to produce quality items with the available resources. The group of objects consisting of bracelets and a ring present high or very high levels of alumina. Archaeometric studies on this type of object are very limited and no correlation with materials of Spanish provenance is found at present. However, different colouring technologies and raw materials implied in their production can be distinguished. Concerning the two goblets with blue decoration (ARJ0099 and ARJ0452) it is proposed that they were made in the same workshop. Moreover, as they share the same compositional and stylistic characteristics with a coeval object found in Beja, they may originate from the same location, currently unknown.

Excluding ARJ0099, ARJ0452, ARJ0453 and ARJ0015, for most of the analysed fragments, a common origin in a currently unknown production centre is proposed. The similarity with the chemical composition of other assemblages, coeval and later, from other Portuguese excavations, for which a local origin has been proposed, leads us to propose the same hypothesis for these group of fragments.

This is a preliminary work that aims to contribute to the construction of the history of Portuguese glass in the medieval and late medieval periods. Further studies are certainly needed to have a more exhaustive view of the presence and circulation of glass in the country.

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