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Glass finds from the Museu de Lisboa – Teatro Romano (Lisbon):
historical and chemical approach of an archaeological set from the Modern Period

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Resumo Durante as recentes escavações arqueológicas no local do Museu do Teatro Romano em Lisboa (Portugal), foi descoberto um conjunto de fragmentos de vidro pertencentes a várias tipologias e que foram datados entre os séculos XVII e XVIII. Este conjunto de vidros foi descoberto num contexto habitacional e foi considerado de caráter utilitário. Na sua maioria, foi possível identificar-se garrafas de vinho em vidro verde e preto, copos e frascos em vidro incolor e pequenos frascos cilíndricos de vidro azul-turquesa.

Com o objectivo de se estudar e caracterizar este conjunto, foram seleccionados 56 fragmentos de vidro a partir de um total de 97 para posterior análise pelas técnicas μ-PIXE e LA-ICP-MS. Foi então possível observar-se e identificar-se uma relação entre os diferentes tipos de composição química do vidro e as tipologias dos objetos, onde todas as garrafas de vinho analisadas têm baixos teores de elementos alcalinos e elevados teores de óxido de cálcio (vidro HLLA) e todos os fragmentos de vidro incolor são constituídos por vidro do tipo potássio. Esta formulação rica em potássio é típica de regiões da Europa Central e predomina nos mercados europeus a partir da segunda metade do século XVII em diante. A composição química dos vidros escavados do local do Museu de Lisboa - Teatro Romano (Lisboa) foi comparada com composições de vidro coevos encontrados em Portugal e noutros centros Europeus de produção de vidro. Esta comparação veio destacar as diferenças e semelhanças entre estes, ampliando assim o conhecimento sobre o vidro que circulava em Portugal durante os séculos XVII e XVIII, e também sobre as relações comerciais portuguesas com a Europa.

Abstract During the recent archaeological excavations at the site of the Teatro Romano Museum in Lisbon (Portugal), a set of glass fragments belonging to several typologies were found and dated between the 17th to the 18th centuries. The glass set was unearthed from a habitational context

and was considered of utilitarian character. In its majority it was possible to identify wine bottles in olive green and black glass, beakers and flasks in colourless glass, and small cylindrical flasks in turquoise blue glass.
With the aim of studying and characterizing this assemblage, 56 glass fragments from a total of 97 were selected for further analysis by means of μ-PIXE and LA-ICP-MS.
A close relation was observed between the glass chemical types and the objects types, where all the analysed bottles have low contents on alkali elements and high contents on lime (HLLA glass), and all the colourless glass fragments are of a potassium-rich glass type. This potassium-rich formulation is typical in Central European regions and predominates in the European markets from the second half of the 17th century onwards. Comparison with published coeval compositions from Portugal and from other European glass production centres highlighted differences and similarities, extending our knowledge on the glass circulating in the country during the 17th and 18th centuries, and also on the Portuguese commercial relations with Europe.

1. Introduction

In the present study, a set of 56 fragments of glass unearthed from an archaeological excavation that occurred in the Teatro Romano area (Lisbon) and carried out in a context dated to the Modern period (17th and 18th centuries), was analysed. The fragments were identified as belonging in their great majority to wine bottles in olive green and black glass. In less quantity, objects of different typologies in colourless and turquoise blue glass were also identified.
The aim of this work is the study and chemical characterisation of the glass fragments, comparing them with coeval European glasswork, and finally, discussing their provenance. As a matter of fact, the attribution of glass objects found in Portugal purely on the base of stylistic and historic considerations is problematic because of the simultaneous presence in the national territory of glass manufactured in the country (often by foreign glassworkers producing glass according to their traditions), and of glass from several European centres.

1.1. European glass production in the 17th and 18th centuries

From the end of the 17th century onward, the supremacy of the soda-rich Venetian and *façon de Venise* glass started to decline and other types of glass produced on the bases of new recipes and raw materials began to conquer the European glass market. At the end of the 17th century, the desire of creating new glass types led to the development of two different compositions: a pure potassium glass matrix developed in central Europe, and a pure lead glass matrix developed in the British Isles (Lanmon, 2011, p. 25).
In what regards the potassium-rich glass, during the first half of the 18th century in central Europe three types of glass were produced: common glass, white chalk glass, and potassium crystal glass. The ordinary glass was the successor to the medieval forest glass formulation (Kunicki-Goldfinger & *alii*, 2005). The white chalk glass[1] was achieved by adding chalk to the batch instead of limestone and by using purer raw materials. For the crystal glass formulation, high quality raw materials were needed and saltpetre could replace almost totally the use of potash (Kunicki-Goldfinger & *alii*, 2005). During the 18th century, Bohemian glass exportation to all Europe was very well organized with established offices in important trade centres as Spain and Portugal (Lukás, 1981).
Other transformation concerns the bottle production. It was an evolution that began in the middle of the 17th century with the need of developing better and more resistant containers to improve wine and other beverages transportation. A relevant innovation made by the industry of the British Isles was to produce bottles with thicker walls: the increase of glass thickness made the bottles much more resistant mainly during their transport (Lanmon, 2011, p. 19). Wills (Wills, 1974, p. 45) believes that Portuguese Port wine was one of the propelling factors for the evolution of the shape of the bottle. As far as the glass production in Portugal is concerned, according to documentary evidence,

[1] It is mentioned in the paper Kunicki-Goldfinger & alii (2005, pp.259) the terminological problem when referring to the diverse glass formulations that emerged on the Central European region during the end of 17th century/ beginning of 18th century. This note serves to clarify that white chalk glass is referent to an uncoloured transparent glass called white chalk to equate to the Venetian soda-rich formulation of vitrum blanchum (Eng. White glass).

(a) (b)

Fig. 1 – a) Area excavated in 2001, which is coincident with the stairs belonging to the house. b) Example of some objects from the LTR glass assemblage.

several glass factories were active in the country during the 17th and the 18th centuries (Mendes, 2002, p. 39; Custódio, 2002, pp. 24, 43, 45, 51). In 1719, D. João V (1698–1750) ordered the installation of a Royal Glass Factory in Coina (Mendes, 2002, p. 56; Custódio, 2002, p. 71). This manufactory employed glassmakers from Catalonia, England, Ireland, Flanders, Italy, and Germany, and from 1731 to 1747 it was managed by foreign administrators, as the Englishmen Joam Butler (1731–1737) and Joam Poutz (1737–1741), and the Irishman John Beare (1741–1747) (Custódio, 2002, p. 101). The production at Coina glass manufactory included bottles, lead glass and uncoloured glass. The items produced followed the style and technology of the productions dominating the market, and bottles were made in French and English style, and goblets and other vessels in Venetian and Central European style (Custódio, 2002, p. 215).

Some of the glass fragments recovered from the Coina glass manufactory have been analysed, and only preliminary results were published (Lopes & *alii*, 2009).

1.2. Archaeological context and the glass findings

A major archaeological investigation into the Teatro Romano in Lisbon was initiated in 2001 as result of the opening of the Teatro Romano Museum. The archaeological intervention was focused in the southern area of the region excavated in the previous campaigns (Rua de S. Mamede, no. 3), with the intent of rehabilitating this space to a museum (Fernandes, 2013).

The glass assemblage here studied came from the area where nowadays the reception of the museum is located. This area below the museum's reception (building of the beginnings of the 19th century) is coincident with an ancient house dated to the 17th century and destroyed by the 1755 earthquake (Fig. 1 a).

The glass assemblage was gathered along 9 meters of excavated sediments. The two meters situated at an inferior level can be related with the context of the 1755's cataclysm, and the remaining can be related with different contexts, such as the placing of the rubbles gathered from the involving area and rubbles caused by the earthquake of 1755 (Fernandes, 2013).

The assemblage from the Teatro Romano Museum in Lisbon (LTR) is composed by fragments of utilitarian glass (see figure 1 b). Based on the glass colour, the selected set may be divided into three broad categories: natural coloured green bottles (40 fragments) with different body shapes (circular and square body sections); coloured glass where it can be distinguished cylindrical flasks in turquoise blue glass (4 fragments), one dark blue fragment and one light green fragment of a vessel rim; and colourless glass from diverse typologies as drinking beakers, an octagonal flask with enamel paintings, and some vessel fragments (9 fragments).

The glass assemblage was dated according to the archaeological context. It is worth noting that the deposits deriving from the destructions provoked by the 1755's earthquake and by the fires that followed provide a precise *terminus ante quem* for this set. The majority of the fragments date to the first half of the 18th century, while just a small fraction of the finds dates to the 17th century.

2. Method

Fragments were chosen according to their typology, glass coloration, and presence of some decorative features (such as the presence of enamels). Special attention was given to wine bottles since these compose circa 70% of the glass assemblage. In what regards the bottles, the fragments were selected giving priority to bottoms and necks. The shape (square or cylindrical bodies), the glass colour (black and green glass) and the glass thickness (some bottles presented thinner walls than others) were the next characteristics considered.
The chosen methodology implied the sampling of all the selected objects, in order to avoid erroneous results by analysing and quantifying corrosion layers instead of the uncorroded bulk glass. Small samples of 2-4 mm^2 were dry cut from the selected 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.
To obtain the glass chemical composition a few analytical methods can be used. In this current investigation, particle induced X-ray emission (PIXE) was chosen to obtain data on the major and minor glass components, and laser ablation induced coupled plasma mass spectrometry (LA-ICP-MS) was used to obtain information on the trace and rare earth elements.

2.1. μ-PIXE

Produced by the 2.5 MV Van de Graaff accelerator installed at Polo de Loures from IST, MeV proton beams were used to perform μ-PIXE sample analysis using an Oxford Microbeams OM150 type nuclear microprobe. The proton beam was focused down to 3×4 μm^2 and the produced X-rays detected by a 145 eV resolution Si(Li) or SDD detector. In order to avoid or detect possible local glass heterogeneities, imaging (2D elemental distribution) and X-ray spectra were obtained from an irradiated sample area of 750x750 μm^2. Operation and basic data manipulation was achieved through the OMDAQ software code (Grime & Dawson, 1995), and quantitative analysis done with GUPIX program (Campbell & *alii*, 2010). The results in oxides weight percentage form were normalized to 100%. In order to validate the obtained results, two glass reference standards were also analysed, Corning B and Corning C.

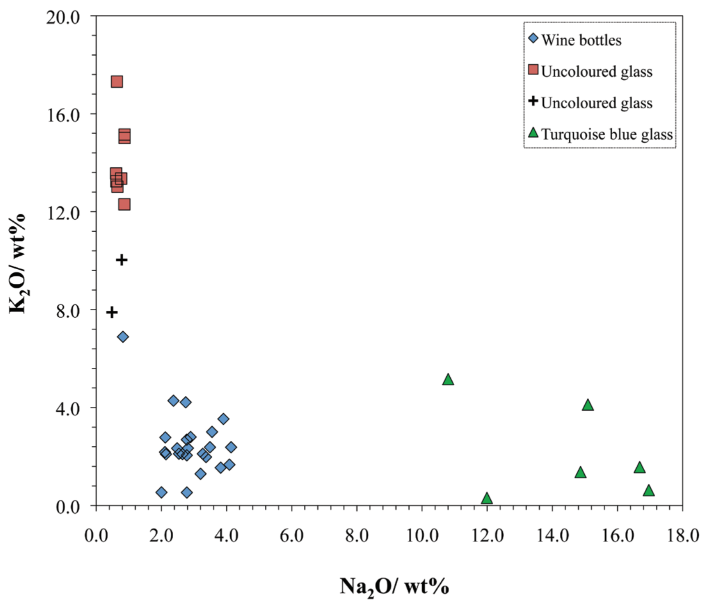

Fig. 2 – Binary plot of Na_2O vs. K_2O in weight percent of oxides. Marked regions for HLLA glass, soda-rich glass, potassium-rich glass and mixed-alkali glass (Dungworth & *alii*, 2006). The values obtained for the two lead glass fragments are also plotted.

2.2. LA-ICP-MS

The LA-ICP-MS (laser ablation-inductively coupled plasma-mass spectrometry) analysis (located at the National Centre of Scientific Research (CNRS) in Orleans, France) was carried out on the embedded glass cross-section. It consists of a Resonetics M50E excimer laser working at 193 nm coupled with the Thermo Fisher Scientific ELEMENT XR mass spectrometer. The excimer laser was operated at 3mJ with a repetition rate of 8 to 10 Hz. The beam diameter was adjusted to 80 μm. A pre-ablation time of 20 s is set in order to eliminate the transient part of the signal, which is then acquired for 55 s corresponding to 20 mass scans from lithium

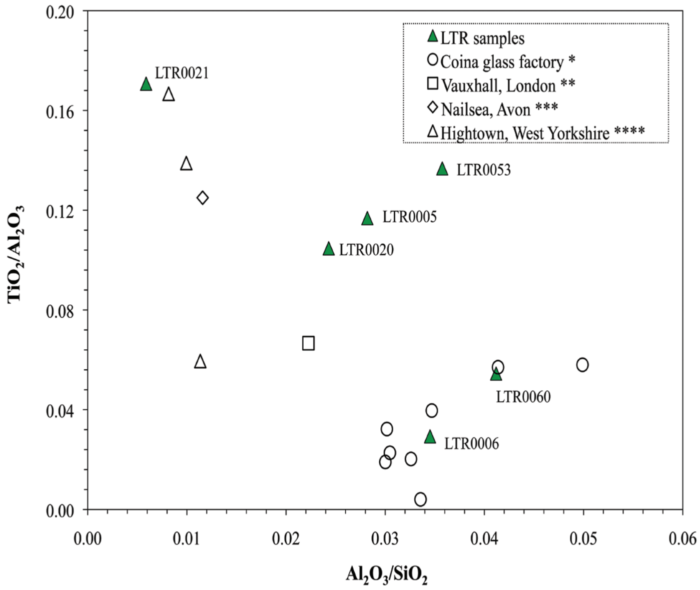

Fig. 3 – Binary plot of the ratios of Al_2O_3/SiO_2 *vs.* TiO_2/Al_2O_3 for the soda-rich glass. * Lopes & *alii*, 2009; ** Dungworth, 2006; *** Hatton, 2004; **** Lucas, 2010.

to uranium (the signal in counts/second is measured in low resolution mode for 58 different isotopes). Calibration was done by using NIST610 and Corning B, C and D glass reference material, as well as an home-made glass reference material for chlorine determination (Gratuze, 2013, 2014). The detection limits range from 0,1 to 0,01% for major element and from 20 to 500 ng/g for other. The composition is calculated from the averaged signal obtained from a single ablation of the sample. In order to validate the obtained concentration results, glass reference standards Corning A and Nist 612 are regularly analyzed as unknown samples throughout all the analytical sequence.

3. Results and discussion

The results obtained for the glass composition were plotted in a Na_2O vs. K_2O chart, that allows one to distinguish the nature of the employed fluxes (soda or potash), and also to identify the mixed alkali fragments and the low alkali ones that can be rich in lime (HLLA) or lead. In the chart represented in figure 2, one can distinguish between: i) a soda-rich glass group with a Na_2O content ranging between 11 to 17 wt%, mainly composed by the cylindrical turquoise blue small flasks; ii) the high lime low alkali (HLLA) group composed only by wine bottles; and iii) potassium-rich group, with K_2O ranging between 12 to 17 wt%, composed only by colourless glass (fragments of beakers and flask). Two lead glass fragments were also identified. The composition of these lead glass fragments will not be discussed here, only the major compositional groups will be considered.

3.1. Soda-lime silica glass

Soda-rich glass (from plant ash) has high levels of Na_2O and lower levels of K_2O.
Analysing Fig. 3, that allows one to look at important silica traces (alumina and titanium oxides), three different sources of silica for the LTR fragments can be seen, which might imply three different production locations. Comparing with compositions of coeval soda-rich glass from the UK (Dungworth, 2006; Hatton, 2004; Lucas, 2010) and from Portugal (Lopes & *alii* 2009), it is observed that two samples (LTR0006 and LTR0060) are consistent with the Coina Glass Manufactory production (Lopes & *alii*, 2009), and one sample (LTR0021) which belongs to the light green fragment of a rim (probably a bottle lip) is consistent with the glass produced in Hightown, New Yorkshire, UK (Lucas, 2010). The remaining three fragments share the same silica source, but it was not possible to find a relation with coeval published compositions, known so far.

3.2. Potassium-rich glass

High levels of K_2O and low levels of Na_2O characterise potassium-rich glass. In figure 4 b), the relation between the LTR fragments and coeval potassium-rich fragments drawn from the literature is presented (Herremans & *alii*, 2012; Kunicki-Goldfinger & *alii*, 2005; Lopes & *alii,* 2009; Müller & Stege, 2006; Smrcek, 1999; Van der Linden & *alii*, 2005). Fragments from the LTR assemblage and the fragments from the Coina glass manufactory appear highly related in the plot represented in figure 4, then allowing one to propose that the potassium rich fragments from the LTR set were made in the Coina glass manufactory.
As far as the shapes are concerned, a fragment worth noting is LTR0014, which belongs to a colourless octagonal flask decorated

with polychrome enamels, identified as being a typical feature of Central European glass dated to the 17th to 18th centuries (see for example: Metropolitan Museum of Art, Accession Number 13.179.70a). Flasks with the same shape and similar decorations were identified among the objects produced at the Coina Glass Manufactory. Furthermore, the shapes of the goblets LTR0063 and LTR0064 are described by J. Custódio as a Coina glass manufactory design and production (Custódio, 2002, p.164).

3.3. HLLA glass

High levels of CaO (>20 wt%) and low levels of alkaline oxides, usually from forest plants or wood ash (K_2O + Na_2O < 10 wt%), generally characterise the High Lime Low Alkali (HLLA) glass. This glass composition was widely used for the production of bottles, mainly in the British Isles. It is generally accepted that HLLA glass was taken to the British Isles c. 1567 by immigrant French glassmakers, since this composition was being made in France during the 16th century (Barrera & Velde, 1989; Dungworth, 2010; Dungworth & Clark, 2004).
The current knowledge on bottles chemical composition comes mainly from several reports and papers concerning bottles from archaeological excavation in the British Isles (Blakelock, 2007; Dungworth, 2005, 2006, 2007, 2010; Dungworth & Clark, 2004, 2010; Dungworth & Mortimer, 2005; Dungworth & *alii*, 2006; Farrelly & *alii*, 2014; Gartner, 2009, among others). More recently, some insights on French and Belgium bottles composition were published (Gratuze & Serra, 2010; Herremans & *alii*, 2012). The composition of the LTR fragments was compared with these published results. To better understand the relation between the bottles composition, the major oxides will be regarded all together instead of only analysing two oxides at a time. The major oxides as well as the trace elements and REE were normalised to concentration of the continental earth's crust (Wedepohl, 1995; Yanagi, 2011).
The analysis of the results allowed one to divide the data into two main groups (Group A and B). Group A is characterised for having higher contents in MnO, P_2O_5, and chlorine comparing with fragments from Group B. Observing the chart represented in figure 5 a), three sub-groups from Group A are perceptible and were represented with different colours. The main differences between the identified sub-groups from Group A are the contents of phosphorous oxide and chlorine. Looking to the contents of MnO, it is observed that it varies a little randomly, not following the sub-groups tendency. Despite the observed differences, bottles from Group A are still very similar between each other. Comparing with compositions from the literature, a relation between Group A and a group of bottles found in the Cistercian nunnery in Clairefontaine, Belgium was observed, as well as with the bottles from the production centre

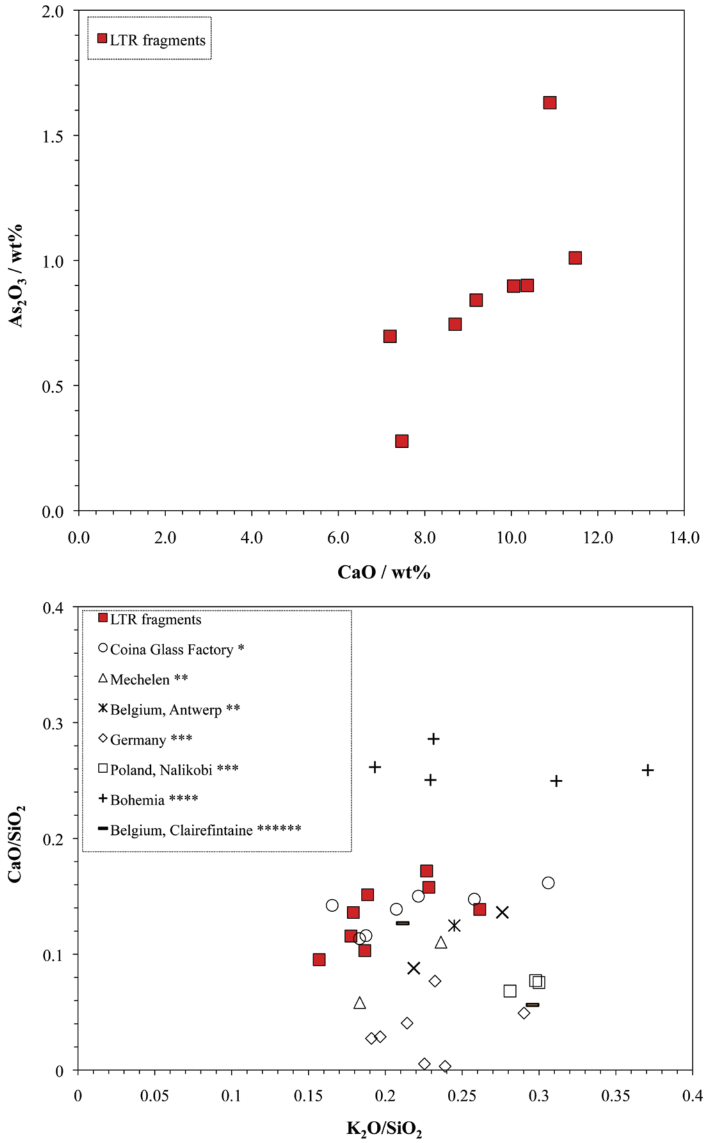

Fig. 4 – Binary plot of a) CaO vs. As_2O_3, and b) the ratios of K_2O/SiO_2 vs. CaO/SiO_2; both plots for the potassium-rich glass. *Lopes & *alii*, 2009; **Van der Linden & *alii*, 2005; ***Kunicki-Goldfinger & *alii*, 2005; ****Smrcek, 1999; *****Herremans & *alii*, 2012.

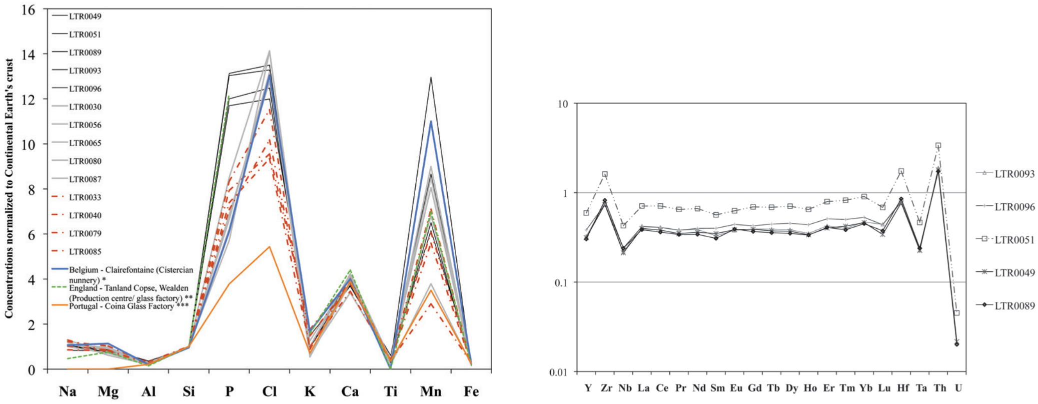

Fig. 5 – (a) Chart with the major elements pattern from the samples of Group A, divided by the identified sub-groups with different colours and normalized to concentration of the continental earth's crust (Wedepohl 1995; Yanagi 2011); (b) chart represented is the trace elements and REE pattern of samples from Group A analysed by LA-ICP-MS.

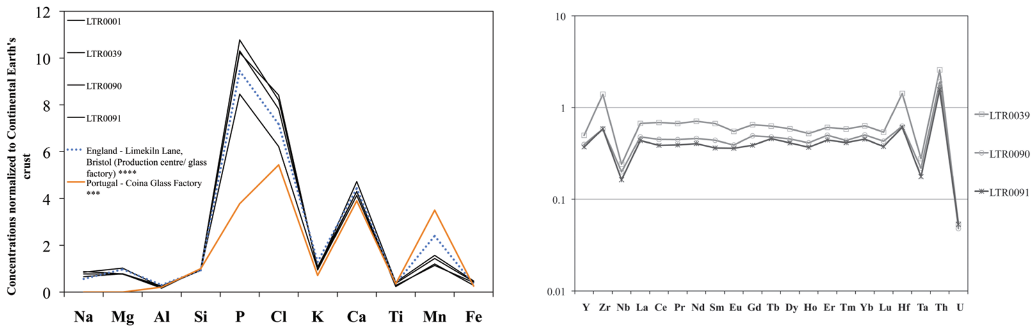

Fig. 5 – (c) chart with the major elements pattern from the samples of Group B, divided by the identified sub-groups with different colours and normalized to concentration of the continental earth's crust; and (d) chart represented is the REE pattern of samples from Group B analysed by LA-ICP-MS. *Herremans & *alii*, 2012; **Dungworth & ClarK, 2004; ***Lopes & *alii*, 2009; ****Dungworth, 2005.

in Tanland Copse, The Wealden, England. Concerning this last comparison, despite the absence of values for chlorine in the literature for Tanland Copse glass (probably because of the analytical method used to analyse the glass), it does not mean the glass does not contain it and for that reason, regarding all the remaining oxides, it is possible to attest the similitude between LTR and these ones. Before analysing the trace elements and REE signature for some of these bottles it is important to mention that bottle LTR0051 has a seal from Bad Pyrmonter Wassers. Thus it is assumed that this bottle was imported into Portugal from abroad. Despite the German origin of this SPA waters, the bottles might have been made elsewhere: this type of seal was found among archaeological remains from Gawber glasshouse near Barsnley, Yorkshire (Ashurst, 1970, p. 125, fig. 38-1). Its composition in major oxides fits well with the other fragments from Group A. In the trace elements and REE chart (figure 5 b), the similitude between samples is evident. Again, sample LTR0051 has a trace elements and REE line shape comparable to the other samples, however in different concentrations. This can indicate that very similar raw materials (containing more minerals bearing REE) were employed on the batch but in different proportions. It is proposed that this can represent the employment of raw materials from different origins, but still geographically close to each other. This hypothesis is proposed under the idea that for bottle production the cheapest and accessible raw materials were the ones to be used, and that in the same production centre glassmakers could use different sources of the same raw material (e.g. sand) depending on its availability and price.

For fragments belonging to Group B (Fig. 5 c), a similarity was found with glass produced in Limekiln Lane (Bristol) in England. In figure 5 d), the trace elements and REE signature of all samples belonging to Group B was represented. Observing the chart represented in Fig. 5 d), it confirms the close relation between these bottles and the origin of the raw materials employed in their production.

No similitude was found between the LTR bottles and the bottles from the Coina Glass Manufactory[2].

[2]A more complete study about wine glass bottles found in Portugal can be found in Coutinho & *alii* (2017).

4. Conclusion

The different but complementary analytical methods chosen allowed to characterise the glass chemical composition, discussing the employed raw materials, and finally studying the historical materials used for the objects production.

According to their chemical composition, the archaeological glass samples were divided into three major glass groups: soda-rich glass, potassium-rich glass and HLLA glass.

Soda rich is present only in a small amount of fragments, mainly related with small cylindrical flasks, used probably to storage pharmacy goods. Two out of the six analysed fragments are consistent with the Coina Glass Manufactory production and one fragment has a comparable composition with analysed fragments from The British Isles (Hightown, New Yorkshire).

All the colourless glass was identified as potassium-rich glass. For some shapes it was possible to find comparisons among the glass produced at the Coina Glass Manufactory. The obtained composition for all the potassium-rich glass type is consistent with the Coina Glass Manufactory unpublished compositions. One can conclude that the colourless glass produced in the Coina Glass Manufactory was then made following a Central European tradition (potassium-rich glass), which represents a change comparing with what is known for the 17th century, when colourless glass circulating in Portugal was, as far as we know, made following the Mediterranean tradition (Coutinho, 2016; Coutinho & *alii*, 2016a, 2016b; Lima & *alii*, 2012). Relating this information with the fact that soda-lime-silica glass was also being produced in the Coina Glass Manufactory (Lopes & *alii*, 2009), it is very interesting to observe that in the beginning of the 18th century this manufactory was producing different types of glass compositions.

The HLLA glass was identified as the glass type used for wine bottles, which is consistent with what was being used in Europe to produce this type of containers. HLLA glass was divided into two groups, none being compatible with the Coina Glass Manufactory glass. For each group it was possible to relate to a different foreigner provenance (Clairefontaine, Belgium; Tanland Copse and Limekiln Lane (Bristol), England). Finally, despite the similitude found and some degree of correlation established between the major oxides contents of bottles excavated in Lisbon and bottles produced in different European sites (values obtained from literature), the available data are not enough to confidently attest their provenance.

This comparison between obtained compositions for a Lisbon glass set and (whenever possible) coeval published compositions from European sites, allows one to suggest provenances or similarities in the employed raw materials, recipes and also furnace conditions around Europe for all the identified glass types. It served also to have a glimpse on the complex circulation of glass and glassmakers in Portugal.

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