16th century azulejos excavated in Lisbon: a tile with arabesque designs found at *Terraços do Carmo*

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ABSTRACT

Counter to the notion that the production of azulejos in Portugal during the 16th century was scant, recent excavations are bringing to light shards that point to the opposite. The variety of fragments recovered from the ground will need many years of study to return a clear notion of what actually was produced at the time.

In some instances, we come across particularly interesting shards which are in a condition good enough to allow attribution based on previous research. A recent excavation at an area once part of the grounds of the ancient *Convento do Carmo* (Carmo Convent) recovered a large fragment that offers stylistically an immediate connection with productions of Antwerp. This paper includes the results of an analytical research of that fragment and discusses its provenance, significance and likely chronology.

RESUMO

Contrariando a noção de que a produção de azulejos em Portugal durante o século XVI era escassa, escavações recentes têm trazido à luz fragmentos que apontam para o oposto. A variedade de fragmentos recuperados do solo precisará de muitos anos de estudo para oferecer uma noção clara do que realmente foi produzido na época. Por vezes, deparamo-nos com casos particularmente interessantes e que se encontram numa condição suficientemente boa para poderem ser estudados.

Numa escavação recente, na envolvente do antigo Convento do Carmo, em Lisboa, foi recuperado um importante fragmento de um azulejo que sugere a nível estilístico uma ligação imediata a produções pouco divulgadas de Antuérpia. Este artigo inclui os resultados de um estudo analítico desse fragmento e discute a sua proveniência, significado e provável cronologia.

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1. INTRODUCTION

Between 2013 and 2015, during the archaeological work carried out at an area once part of the grounds of the ancient *Convento do Carmo* in Lisbon¹, a vast collection of tile fragments was recovered, which, in most cases, must have been once part of the internal arrangement of this important Lisbon convent. One of the fragments (Figure 1) caught our attention because a substantial part of a single tile had been preserved in good condition and because we recognized the sort of pattern it depicted from azulejos related with the productions of Antwerp [1-3] but which was, until now, unknown from the workshops of Lisbon.

The construction of *Convento do Carmo* started in 1389. Throughout its existence it had successive phases of new construction and reconstruction, either for natural reasons (as would have been the case of the 1531 and 1755 earthquakes) or because of new edifications within its old fence, also known as *Terraços do Carmo* [4].

Although other major works are documented in this area, as well as, at an earlier period, inside the building, in the case of the shard under study² (Figure 1) the context where it was found resulted from the remobilization of the land, on the occasion of the construction of the *Ordem Terceira do Carmo* Hospital, between 1703 and 1706, which collapsed completely during the 1755 quake. In association with the present fragment, a vast and important set of earthenware, majolica, porcelain and glass (as well as other archaeological materials) that chronologically point to the second half of the 16th and to the 17th centuries was also exhumed, constituting a coherent and well defined unity, which still needs to be studied in detail.

Therefore, it is believed that this tile would be part of an order made at the end of the 16th century, for the decoration of some internal space of the current building, or of some new construction that is known to have been erected during this period, such as the designated "New Dormitory", built between 1571 and 1582. In fact, according to Frei José Pereira de Santa Anna's description of the *cenobium* towards the middle of the 18th century, there are numerous references to the existence of tile panels on the walls of the building's various dependencies and worship spaces [5].

Tiles with related patterns have been reported by C. Dumortier [1], F. Caignie [2] and M. Archer [3] from the workshops of Antwerp. Dumortier connects these designs with Francesco di Pellegrino's book *La fleur de la science de pourtraicture* where his exotic patterns are collected and which was published in Paris in 1530 and republished in Antwerp in 1543 by Cornelis Bos as *Livre des Moresques*. Figure 2 illustrates two plates from the original edition, showing how the decoration of the azulejo may be assembled from simpler designs extracted from the plates. It is interesting to note that in the Flemish productions the design corresponding here to the stepped blue ribbon is usually made up of two, three or even four parallel thin lines. Here the ribbon is a single, thick, continuity after the suggestion in the plate illustrated, resulting in a pattern much easier to paint. The full lining, maybe a low sill, could have been made of a repetitive fleuron with a module of four tiles, or else be constituted by a more complex sequence as suggested by any of the plates in figure 2.

¹ The archaeological work was part of the implementation of an urban regeneration project by Architect Álvaro Siza Vieira.

² Exhumed from the Stratigraphic Unit 146.



Figure 1. A large fragment from a single tile (ca. 13 cm across) recovered from the ground at *Largo do Carmo* in Lisbon (the arrow indicates the sampling point)

This paper further includes the results of an analytical research of the fragment towards an attempt to determine its provenance and possible chronology.



Figure 2. Two designs of arabesques (plates XII and XVIII) from *La fleur de la science de pourtraicture* showing how the pattern used in the azulejo may be composed from sketches adapted from the book (source of the images: gallica.bnf.fr / Bibliothèque Nationale de France)

2. EXPERIMENTAL

2.1. Sampling and methodology

The tile was sampled at the point indicated by the red arrow in figure 1, allowing the acquisition of a scale of white faience with blue and yellow colours. The sample (with the reference Az327) was stabilized in resin, lapped and polished to obtain a cross-section for observation and analysis by scanning-electron microscopy coupled with an X-ray energy-dispersive spectrometer (SEM-EDS).

The optical acquisition of images of the sections was obtained with a Leica DFC295 digital camera coupled to a M205C stereomicroscope of the same brand.

SEM-EDS observations and analyses were made at the HERCULES Laboratory in Évora using a Hitachi S3700N SEM with a coupled Bruker XFlash 5010 EDS. The specimen was uncoated and the observations were made in backscattered electrons mode (BSE) in variable pressure mode at 40 Pa and at an accelerating voltage of 20.0 kV. The acquisition of X-ray spectra was done with the detector at ca. 10 mm working distance.

The selection of areas for EDS analysis avoided inclusions in the glaze or biscuit representing more than ca. 5% of the full area analyzed. Whenever possible area sizes of at least ca. $200 \times 200 \mu m$ for glazes and $500 \times 500 \mu m$ for biscuits were used but acceptable repeatability was verified in areas four times smaller. For comparison purposes, only the elements usually representing the major components were considered, excluding tin (Sn) in the glaze and lead (Pb) in the biscuit due to their variability with the area chosen (in the case of Sn in the glaze because of crystal aggregations and in the case of Pb in the biscuit because the content increases with proximity to the interface). The results of the EDS analyses are given in weight % of each element considered.

Principal Component Analysis (PCA) of EDS results was made using the SPSS© software platform by IBM Analytics.

2.2. Results

2.2.1. Morphological characteristics

Figure 3 illustrates a microscopic image of the section of the sample after preparation, compared to a SEM image of the same general area. Although some hair-thin microcracks are apparent in the glaze, including around the sand inclusions, the gas bubbles are still empty, meaning that the lixiviation of the components of the glaze has not yet started in a noticeably manner (otherwise the bubbles would not be clean inside, because of deposition) and therefore the results of analyses of the glaze should not be particularly affected by the long burial. No *coperta* (a transparent glaze layer sprinkled on top of the painted glaze) was used over the painting.

Figure 4 illustrates SEM images of the sample under study in which it is noticeable that the glaze is rather devoid of inclusions except for a few grains of sand, and that its interface with the biscuit has conspicuous outgrowths of individualized crystals formed over the firing cycle.



Figure 3. Paired images of sample Az327 in optical microscopy (top) and scanning electron microscopy (bottom)



Figure 4. SEM images of sample Az327 depicting the main micro-morphologic characteristics of the tile (a – glaze; b – biscuit)

2.2.2. Glaze composition

Table 1 includes the semi-quantitative results of the sectional analysis of the glaze by EDS in weight %. Eight different areas of the glaze were analyzed and the values of the averages and standard deviations were also determined. Sn was excluded for the reason pointed out in section 2.1. The amount of oxygen was calculated through the remaining elements stoichiometry of their most commonly considered oxides and the results were normalized to 100%. The ratio between Si and Pb (the main components of the glaze) was calculated and is included in the table.

| Determination nr. | Na | Mg | Al | Si | К | Fe | Pb | 0 | Si/Pb |
|-------------------|------|------|------|-------|------|------|-------|-------|-------|
| 1 | 1.20 | 0.72 | 3.22 | 18.05 | 1.57 | 0.92 | 45.75 | 28.57 | 0.39 |
| 2 | 1.10 | 0.65 | 3.57 | 17.98 | 1.54 | 1.01 | 45.42 | 28.73 | 0.40 |
| 3 | 1.07 | 0.59 | 3.98 | 18.37 | 1.55 | 1.23 | 43.75 | 29.45 | 0.42 |
| 4 | 1.03 | 0.45 | 2.73 | 17.83 | 1.41 | 0.63 | 48.25 | 27.68 | 0.37 |
| 5 | 1.13 | 0.60 | 3.01 | 17.79 | 1.48 | 0.81 | 47.14 | 28.03 | 0.38 |
| 6 | 1.08 | 0.56 | 2.56 | 17.46 | 1.24 | 0.78 | 49.01 | 27.29 | 0.36 |
| 7 | 0.91 | 0.61 | 2.31 | 17.56 | 1.18 | 1.23 | 48.88 | 27.32 | 0.36 |
| 8 | 0.95 | 0.74 | 2.45 | 17.31 | 1.22 | 0.82 | 49.37 | 27.14 | 0.35 |
| Average | 1.06 | 0.61 | 2.98 | 17.79 | 1.40 | 0.93 | 47.20 | 28.03 | 0.38 |
| St. Deviation | 0.09 | 0.09 | 0.58 | 0.34 | 0.16 | 0.22 | 2.04 | 0.83 | 0.02 |

Table 1.Semi-quantitative composition of the glaze of sample Az327 determined by SEM-EDS
(wt.% of oxygen and main elements, excluding Sn, normalized to 100%)



Figure 5. Relevant part of the analytic spectrum of the glaze of Az327 obtained by SEM-EDS (the peak marked "*" is an artefact)

2.2.3. Biscuit composition

Table 2 includes the semi-quantitative results of the EDS analysis, in weight %, of four different sectional areas of the biscuit. Pb was excluded for the reason pointed out in section 2.1. The amount of oxygen was calculated through the remaining elements stoichiometry of their most commonly considered oxides and the results were normalized to 100%. The ratio between Ca and Si (the main components of the biscuit) was determined and is included in the table as well as the averages per element and the corresponding standard deviations.

| Determination nr. | Na | Mg | Al | Si | К | Ca | Ti | Fe | 0 | Ca/Si |
|-------------------|------|------|------|-------|------|-------|------|------|-------|-------|
| 1 | 1.12 | 1.19 | 8.20 | 29.32 | 2.94 | 6.80 | 0.60 | 2.96 | 46.86 | 0.23 |
| 2 | 1.05 | 1.26 | 8.13 | 27.34 | 2.70 | 9.18 | 0.88 | 3.56 | 45.90 | 0.34 |
| 3 | 1.16 | 1.32 | 8.78 | 27.09 | 3.38 | 8.19 | 0.71 | 3.48 | 45.88 | 0.30 |
| 4 | 1.12 | 1.25 | 8.28 | 25.13 | 2.65 | 12.49 | 0.79 | 3.51 | 44.78 | 0.50 |
| Average | 1.11 | 1.26 | 8.35 | 27.22 | 2.92 | 9.16 | 0.75 | 3.38 | 45.86 | 0.34 |
| St. Deviation | 0.04 | 0.05 | 0.30 | 1.71 | 0.33 | 2.42 | 0.12 | 0.28 | 0.85 | 0.11 |

Table 2.Semi-quantitative composition of the biscuits of sample Az327 determined by SEM-
EDS (wt.% of the main elements corrected to 100%)

2.2.4. Yellow pigment

A grain of yellow pigment (left side of figure 6) was analysed and the pigment found to be, not Naples yellow, but rather tin-antimony-lead yellow [6] as shown by the semi-quantification by EDS (Table 3) and spectral counterpart (right side of figure 6). Discounting the elements present in the matrix, the simultaneous high contents in Sn, Sb and Pb confirm the use of a tin yellow pigment, with an approximate ratio between Sn and Sb of 1:2.8.



Figure 6. SEM image showing the selection of a grain of yellow pigment (left side); and relevant part of the EDS spectrum of the yellow pigment in logarithmic scale confirming the substantial presence of tin in it (the peak marked "*" is an artefact)

Table 3.Semi-quantitative composition of a grain of yellow pigment (wt.% of the elements
normalized to 100% in the same conditions as before).

| Az334/04 | Al | Si | Fe | Sn | Sb | РЬ | 0 |
|----------|------|------|------|------|-------|-------|-------|
| Yellow | 0.57 | 7.58 | 0.83 | 4.58 | 12.70 | 54.61 | 19.13 |

3. DISCUSSION

3.1. Technology

Figure 7 illustrates SEM images of the sample under study compared with 16th century Portuguese azulejos from *Igreja da Graça* in Lisbon (identified by the sample reference Az013/L1) [7] and the panel *Nossa Senhora da Vida* (Az032/01) [8]. In all samples, it can be noticed the same general glaze morphology, characterized by few inclusions, mostly grains of sand, and outgrowths of neo-formed crystals in the glaze-biscuit interface.



Figure 7. SEM images of the sample under study Az327 (top) compared to *Igreja da Graça* Az013/L1 (middle) and *Nossa Senhora da Vida* Az032/01 (bottom)

On the other hand, the glaze morphology of Az327 is very different from Antwerp tiles which are almost devoid of interfacial outgrowths at the scale studied [9]. For the same reason, it is also different from Portuguese 17th century tiles [9] and from Seville majolica productions - see also [10]. As an example, in figure 8, are presented SEM images of the glazes of samples from Antwerp (Az031/A), Portuguese 17th century (Az024/00) and Seville (Az040/02) glazed tiles.



Figure 8. SEM images of samples Az031/A ca. 1560 from the productions of Antwerp (top), Az024/00 from the 17th century productions of Lisbon (middle), and Az040/02 from Seville ca. 1580 (bottom)

The interfacial crystalline development is a very important distinguishing characteristic of 16th century Portuguese azulejos manufactured by the workshops of Lisbon [11] derived from a peculiar firing cycle, as was experimentally observed by the study of replicates [12]. The use of the unusual Sn+Sb+Pb yellow pigment has also been found in other panels stemming from the circle of João de Góis [11, pp. 129].

Therefore, as respects the micro-morphology and the use of a tin-antimony-lead yellow, the tile under study can be clustered with the productions of the João de Góis circle.

3.2. Glaze

In figure 9 are presented the results of a log-based PCA of the glaze of sample Az327, considering the analytical results in table 1, together with samples from *Igreja da Graça* (identified by the code Az013) [7; 11], the panel *Nossa Senhora da Vida* (identified by the code Az032) [11], as well as samples from Antwerp (Az030/01, Az031 [9] and Az311/02), Portuguese 17th century (Az024/00, Az100/02, Az052, Az198/AR2 and Az197/01) [9] and Seville 16th to early 17th centuries (Az040/01, Az192b+r, Az306/01, Az338/01 and Az345/04) [to be published]. The plot depicts a projection in the plane of the two principal components, PC1 and PC2. PC1 explains 49% of the variation and is controlled in the positive sense by the contents in Na, Si and K, and in the opposite sense by the content in Pb. PC2 explains 22% of the variation and is controlled in the positive sense by the contents in Na, Mg and Pb and in the opposite sense by the content in Al.



Figure 9. Score plot of the PCA of the glazes of: the shard under study (Az327- LgCarmo); Portuguese 16th century panels by João de Góis (Graça I and the panel *Nossa Senhora da Vida* - Sra Vida); Antwerp ca. 1560 (Antw); Portuguese 17th century (PT17) and early Seville majolica azulejos (Sev)

In a previous comparative study [9], we have shown that the composition of the glazes is a particularly important differentiating characteristic of 16th century Portuguese azulejos, which are distinguishable from other productions by their simultaneous low contents in Na and K. The PCA analysis shows that, in this respect, Az327 fits within such productions.

The direct comparison of spectra typical of the different productions (Figure 10) also confirms that the pattern of the spectrum of Az327, with simultaneously low peaks of Na, Mg and K and a relatively high peak of Pb, is only similar to the 16th century Portuguese productions, here represented by sample Az013/L2.



Figure 10. EDS spectra of the glazes of the shard under study (Az327), Portuguese 16th century sample of a panel by João de Góis (Az013/L2), Antwerp (Az031), Seville (Az040/02) and Portuguese 17th century (Az100/02) azulejos

3.3. Biscuit

Figure 11 shows the results of a log-based PCA of the biscuit of Az327 through a plot in the plane of the first two principal components (PC1 and PC2) considering the analytical

results in table 2, together with samples from the same tiles used for the PCA of the glazes. PC1 explains 58% of the variation and is controlled in the positive sense by the contents in Al, Si, K and Fe and in the opposite sense by the contents in Mg and Ca. PC2 explains 17% of the variation and is controlled in the positive sense, in varying degrees, by the contents in Na, Al and Fe, while the opposite sense is controlled by the content in Si (as shown by the loadings plot depicted in vector form in figure 12).



Figure 11. Score plot of the PCA of the biscuits of: the shard under study (Az327- LgCarmo); Portuguese 16th century panels by João de Góis (Graça I and the panel *Nossa Senhora da Vida* - Sra Vida); Antwerp (Antw); Portuguese 17th century (PT17); and Seville (Sev)



Figure 12. Loadings plot of the PCA analysis of the biscuits

The result shows that, as in the case of the glazes, Az327 is compatible with known productions of João de Góis, particularly with Graça I, the first phase of *Igreja da Graça*, presumably dated 1560–70, and to a lesser degree with the panel *Nossa Senhora da Vida*, dated ca. 1580 [11].

4. CONCLUSION

The micro-morphology and compositional results indicate that the tile is a 16th century production by the workshops of Lisbon. They further suggest that the tile is a production of the workshop of João de Góis tentatively datable from the 1560s to the early 1580s. Although the attribution to the workshops of Lisbon is well supported by the distinctive morphology, the comparative position of Az327 must be considered with caution because, contrarily to the panels with which it was compared, only a single tile fragment is available. Furthermore, a long burial in a damp environment may alter the composition. However, as referred above, the sample under study did not present any visible signs of degradation and so it was very likely buried in a relatively dry place.

The arabesques pattern was probably inspired by prints published in Antwerp, possibly the *Livre des Moresques* published by Cornelis Bos in 1543, although it is not an exact copy of any pattern in the book (for the original published in Paris in 1530 see [13]). The composition preserved in the fragment suggests a fleuron of four tiles that formed a repeating pattern, probably for parietal lining.

The study presented shows that, once a background of data is established for comparative purposes, important conclusions bearing on the early production of azulejos in Portugal may be drawn from a single excavated fragment.

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