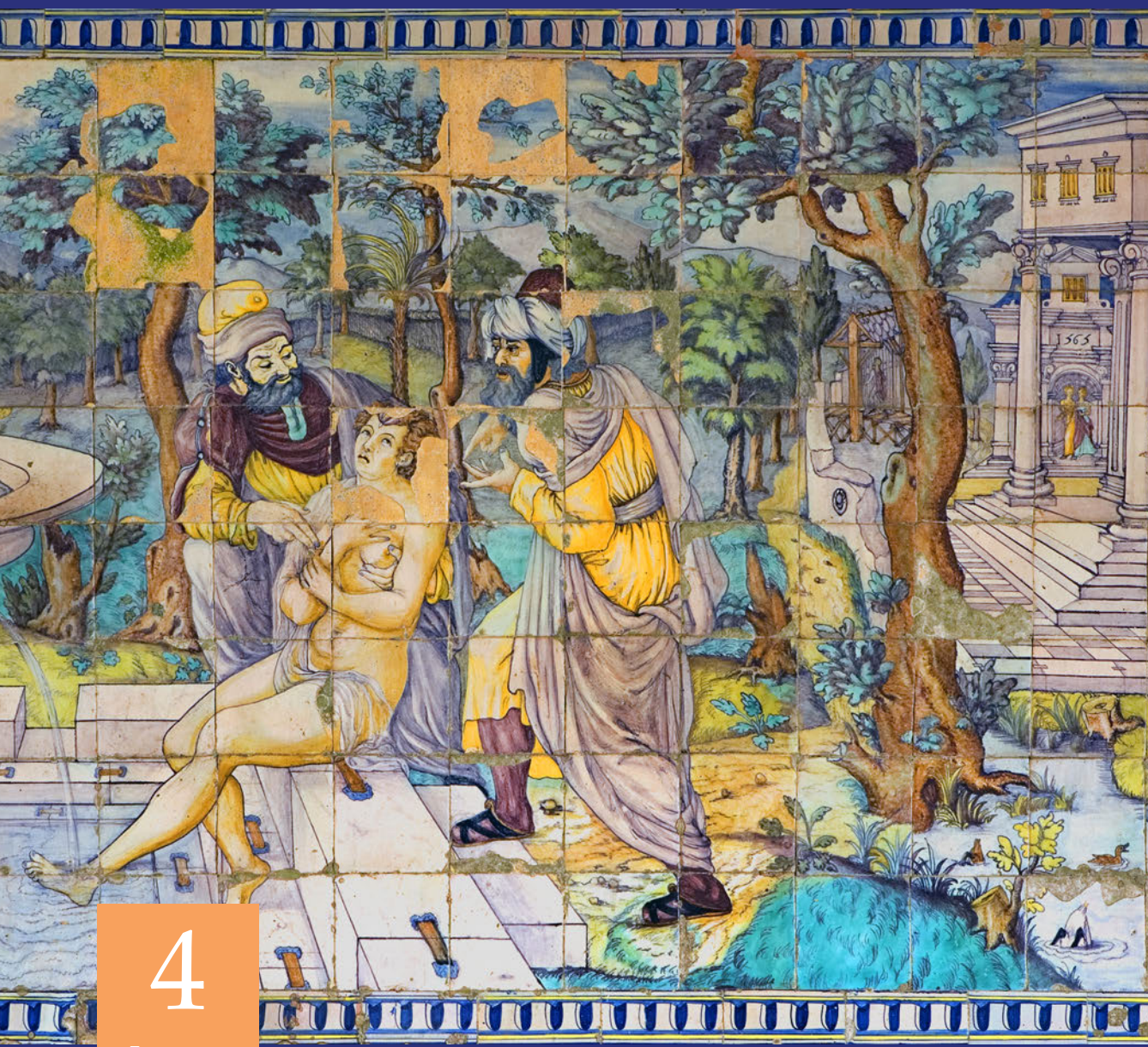
Studies in Heritage Glazed Ceramics

The majolica azulejo heritage of *Quinta da Bacalhôa*



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Volume II

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CONTENTS

PREFACE

This is the second of a special series of four volumes of *Studies in Heritage Glazed Ceramics* dedicated to the renaissance majolica azulejo heritage of *Palácio e Quinta da Bacalhôa* in Azeitão, Portugal.

The azulejos of Bacalhôa have a legendary status in the studies of renaissance majolica in the Iberian Peninsula in general, because of their extraordinary variety and quality and the fact that its most mythical panel, representing the biblical episode of *Susanna and the Elders*, is dated "1565" – a chronology hardly compatible with the then-recent production of azulejos in Portugal. Several hypotheses were advanced over the years to cope with this seemingly impossibility, almost always involving Flemish potters immigrated to the Peninsula which the present study finally confirmed.

The first volume of the series dedicated to Bacalhôa, issued in December 2021, published three papers that established the pillars supporting the subsequent detailed study of the panels and patterned tiles: a study of the estate, locating its 16th century majolica azulejos; a study of the career and productions of Jan Floris de Vriendt of Antwerp (known in Spain as *Juan Flores*) who, according to the results, was likely the main potter, painter and pattern designer connected with the lining of Bacalhôa with majolica azulejos around 1565; and finally a systematization of the main types of 16th century majolica azulejos still extant in the Palace, the Pleasure House by the lake and the garden. That first volume was complemented by a study of the panels and tiles that, according to the previous results, had been manufactured in Talavera (Spain) and imported to Portugal, probably the earliest painted majolica to be applied at Bacalhôa.

This second volume of the series starts the presentation of the research results connected to panels and patterned tiles that were mostly produced in Portugal. The four articles cover in detail: the central room of the Pleasure House, where *Susanna and the Elders* is applied; the five panels of the *Loggia of the River Gods*; the very interesting and often belittled *Rape of Europa*; and the according to our knowledge until now unpublished floor of the oratory of the *piano nobile* of the Palace.

The scientific production stands on several pillars, one of them the peer-reviewers of the authors' papers, whose names are often unknown but whose importance in the final output is singular. The editors wish to heartily thank the reviewers for this number: Doctor Alexandre Nobre Pais, Director of *Museu Nacional do Azulejo* and Doctor António dos Santos Silva of *Laboratório Nacional de Engenharia Civil* (LNEC) who have graciously accepted the hardship of the revisions.

Two more numbers of the journal dedicated to the azulejo heritage of Bacalhôa, with four new research papers in each, are expected to be published over the next 12 months.

LNEC thus presents No. 4 of *Studies in Heritage Glazed Ceramics*. Its 108 pages condense an important part of the results obtained over 20 months of multidisciplinary research, as befits the aims of this journal, aiming to clarify the early diffusion of majolica azulejos in Portugal.

The Editors

EDITORS

SCOPE

Studies in Heritage Glazed Ceramics is dedicated to the results of scientific studies in the field with a particular emphasis on analytical results, conservation issues and historical studies and very specially to multidisciplinary research in the domain.

The contents will include:

- Archaeometry studies, namely the application of analytic methods to the identification of materials and the establishment of technologies, provenance or the setting of chronologies;
- The artistic and historical context of productions, materials and evolving technologies, as well as the origin, preparation and trade routes of pigments and other raw materials;
- Decay of glazed ceramics, techniques and materials for conservation;
- Other innovative research results in the field.

The tile floor of the *Palácio da Bacalhôa* oratory

João Manuel Mimoso, Alfonso Pleguezuelo, Maria Augusta Antunes, Sílvia Pereira, Dória Costa, Álvaro Silva

ABSTRACT

The only surviving tiled floor at *Palácio da Bacalhôa* graces a former oratory and is interesting not only for its singularity in Portugal, but also because, unlike others, the tile elements that compose it may be tentatively dated with a reasonable accuracy.

Seven small samples were collected from the floor, of which five from the central area: two from the white rhombi elements and three from elements with different shades of blue. Analytical research revealed that some tiles could not be connected with a Portuguese origin, but four of the seven sampled were manufactured in Lisbon, likely by the workshop of João de Góis.

The dating of the floor itself is somewhat compromised by the use of tiles from several provenances and chronologies in its assembling, corresponding also to a variability of hues composing areas where a single shade of blue was expected to be used.

RESUMO

O único pavimento em azulejo que chegou aos nossos dias no Palácio da Bacalhôa adorna um antigo oratório e é interessante não só pela sua singularidade em Portugal, mas também porque, ao contrário de outros casos, os elementos que o constituem podem ser tentativamente datados com razoável exactidão.

Foram colhidas sete pequenas amostras do pavimento, das quais cinco da área central: duas dos losangos brancos e três de elementos com diferentes tons de azul. A investigação analítica revelou que alguns azulejos não terão origem portuguesa, mas quatro dos sete amostrados foram fabricados em Lisboa, provavelmente pela oficina de João de Góis.

A datação do pavimento propriamente dito é algo comprometida pela integração de azulejos com proveniências e cronologias diversas, donde derivam variações de tons de azul que fogem à simetria do desenho.

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KEYWORDS: Renaissance glazed floors; azulejos; Palace of Bacalhôa; João de Góis

Acknowledgements

We are thankful to Mr. Joe Berardo and to Mr. Renato Berardo, who authorized the sampling of the unique azulejo heritage of *Palácio e Quinta da Bacalhôa*; and to *Associação de Colecções | The Berardo Collection* and *Bacalhôa Vinhos de Portugal* for support to this project.

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1. INTRODUCTION

The tiled floor of one of the oratories of the *Palácio da Bacalhôa* [1, p. 36] is striking for its geometric composition, balance of colours and evenly application. Its singularity in Portugal makes it particularly interesting.

Figures 1 to 3 depict the floor as it was, seemingly until the late 20th century. At an unknown date, before the estate was acquired by the present proprietors, the altar was removed and a stone bathtub was installed, covering part of the floor to near the white central star. However, it is presumed that the tiles that were not affected by the installation are still in place under the tub.

Figure 1. The oratory at the first floor of the palace of Bacalhôa in the late 20th century (?), still with the altar in place (image © Associação de Colecções | The Berardo Collection).

Figure 2. A view of the floor as it originally was, seen from the door, showing the narrow strip of rectangular tiles around the step to the altar (image © Associação de Colecções | The Berardo Collection).

Figure 3. A view of the original floor from the top of the altarpiece (image © Associação de Colecções | The Berardo Collection).

This article makes a historical and stylistic introduction to the floor and reports the results of an analytical research aimed at determining its age and provenance of the tiles used.

2. PRELIMINARY STUDY OF THE FLOOR

2.1. On the use of the space

Given its location at the end of what may be considered to be the main sleeping room of the palace [1], the space whose floor we researched must have been what is usually called an oratory for private use, where the couple who owned the palace would say their daily prayers. In its private character it differs to some extent from what may be supposed to be the house oratory on the ground floor, located next to the exit to the garden, which was accessible to members of the family coming from any of the rooms on the upper floor, maybe even to possible guests or visitors.

In the oldest photographs we can see that the space was still used as an oratory in the mid-20th century, although the altarpiece, the altar table (at least its visible covering) and also the wooden platform in front of both pieces of furniture were not the original ones, but possibly those renovated at the end of the 18th or beginning of the 19th century. The chestnut-coloured paint that covered these elements may even have been applied much later than this and may have concealed a former decoration. However, the 20th century photos (as in Figures 1 and 2) show that the wooden platform occupies the same surface area as the original one, as it is perfectly visible that the floor slabs that surround it are the same ones that would have surrounded the original platform. The old photos do not show whether the wooden cladding we can see is simply a cover on a previous element that was to be hidden, perhaps because of its poor state of conservation, or whether it replaces entirely the older platform. It is also possible that, at the same time as the new altarpiece and altar were installed, repairs were made to the floor, although it is difficult without systematically resourcing to analytical means, to identify and date the pieces that may have been replaced on that occasion.

2.2. Stylistic notes

We do not know whether the original tile floor covered the entire circle, which is quite possible, although not everything would have been at the same level, since in front of the altar it was customary to place the table on a platform somewhat higher than the rest of the floor. In this case, given the small size of the oratory, it would probably have only a single step. We can imagine what this place would be like if we compare it with the image in Figure 4 of the chapel in the *Casa de Pilatos* in Seville [2]. The usual practice in these cases was to give a rectangular plan to this platform and to cover its floor with a somewhat different design to the rest of the oratory, often richer in geometry or colour as it was the area closest to the sacred image of the altarpiece or, where appropriate (certainly not in this case), to the Holy Sacrament.

If we begin the commentary on this interesting floor by its current appearance, the first problem we face is the number of chromatic nuances offered by the large number of pieces that compose it. The confusion is increased because it is also very evident that the original was indeed meant to be composed, as was usual, of various colours and various

hues of the same colour that were highlighted by contrast. However, the shades of blue present in this floor deviate from a rigorous geometric setting of repeating shapes and colours suggesting that the floor may have suffered alterations and restorations in the course of the centuries.

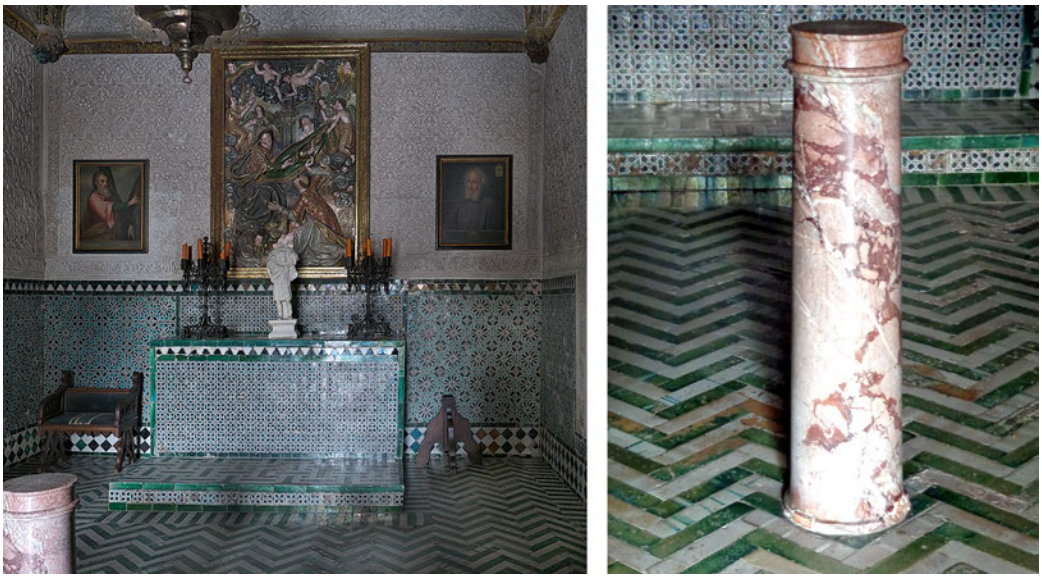


Figure 4. *Capilla de la Flagelación* (*Casa da Pilatos*, Seville- Spain) (images: Wikimedia Commons[1] by Jl FilpoC (left side) and Superchilum (right side).

The paving forms an almost complete circle made up of a central star design and eight radial sectors that correspond to two types of geometric patterns repeated four times each. One of the types (diamond sectors) is mostly formed of rhombi; the second is strikingly marked by a blue star (star sectors) - Figure 3. The major diagonals of the largest rhombi, such as those making up the eight-pointed white star, measure 22.5 cm, therefore the tile pieces were cut from 16 x 16 cm (or larger) square tiles.

Diamond sectors

These sectors seem to have been made up of rhombi in two different shades of blue, the dark ones alternating with the light ones. The dark ones show a rather cobalt-saturated glaze; the lighter ones have less cobalt pigment balanced with white (tin) pigment producing a tone somewhat close to grey. A few rhombi (in larger number in the sector that leads from the door to the altar) are of a sky-blue, again obtained from a balance between the blue and the white pigments.

Star sectors

The surface area of these four sectors is larger than that occupied by the diamond sectors and their geometry is rather more complex because they do not form an extensible mesh like the previous ones, but a centrifugal composition that is not well resolved, originating

1 https://commons.wikimedia.org/wiki/Category:Capilla_de_la_Flagelación,_Casa_de_Pilatos

irregular polygons when approaching the boundaries of the composition.

The four stars are formed by eight dark-blue glazed rhombi and are surrounded by eight light blue square tiles. These squares are surrounded by eight rhombi of the dark-blue glaze mentioned above. The square, rhombi and irregular polygonal tiles that surround the previous lozenges, now of very varied shapes, would correspond to the light blue sort, although the colour distribution is not regular, either because the colours of the tiles used were not steady, or because some tiles have already been replaced in past restorations. Therefore, it is difficult to establish exactly what the original intention in the distribution of the shades of blue was.

The curved side of the sector is topped by a series of alternating square and diamond-shaped tiles, and then a strip that may have originally been entirely dark blue, as most of it has been preserved.

The rectangular shapes bounding the four sectors containing stars, as well as those running parallel to the walls of the circular plan, must have been intended to be of the light blue type, although what we see today is a mix of shades (Figure 5) that may partially derive from later alterations and restorations. Interestingly, the rectangular shapes are not pieces cut from tiles in the *alicatado* manner, rather, after applying the tiles, a straight continuing cut of limited depth was made through the tiles to define a second line parallel to the boundary, enhancing the geometry and sense of complexity of the floor, without actually imposing the work of cutting the tiles clean (Figure 5). It is not evident whether the cuts were made at the time the floor was applied, but they were probably filled with mortar to hide the varying colour of the biscuits as seen in Figure 7.

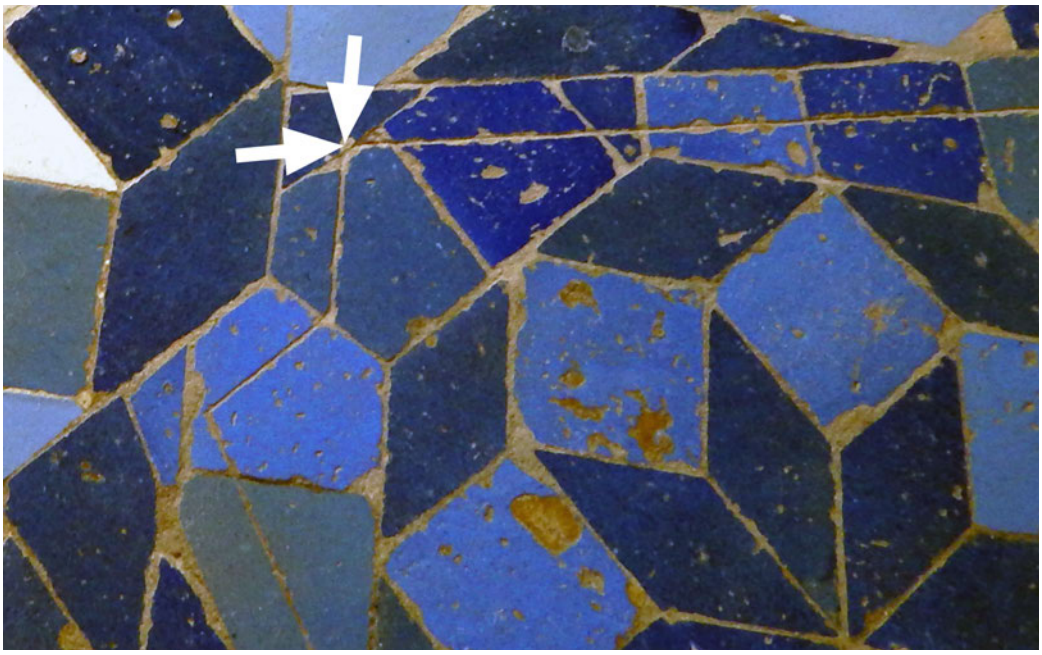


Figure 5. An enhanced view showing the variety of blue hues and the cuts through a continuity of tiles (two of them indicated by the white arrows), seemingly made with a small hatchet pick, giving the impression of more *alicatado* work than there really is in the floor (image © Associação de Colecções | The Berardo Collection).

The entire circle made up of the four diamond sectors and the four sectors of stars is surrounded by a band of tiles that must have been dark blue, set in a diamond pattern, and light-blue glazed triangles. There are five concentric perimetral strips between this band and the wall, one of which is made of dark-blue square tiles and the other four are made of light-blue squares or rectangles of two different widths. Here, too, several shades of blue are to be seen, set without a fixed rule as if remains of tiles of several hues had been used without a set rule, to cover this final continuance towards the wall.

Central star

In this mostly blue floor composition, the centre stands out chromatically, occupied by a star formed by eight white rhombi, possibly originally surrounded by eight dark blue tiles and eight light blue rhombi.

In this scheme, which we assume to be the primitive one, we can detect misplaced original pieces and others that seem to be later replacements. All that deviates from what we consider to be the four basic original colours, white, dark blue, light blue and sky-blue, could be interpreted as possibly later replacements after the original paving was installed.

In the colour white itself, two different hues seem to be distinguishable. One detail that is striking about one of the pieces is that it is contaminated with a drop of green glaze, evidence that it was fired in a kiln in which green-glazed ceramics were also fired.

In the blue tiles, the picture is even more complex than in the white tiles, as four or perhaps five different hues seem to be distinguishable, of which only three would maybe be expected to be the original ones. For example, some of the sky-blue elements have a chromatic purity and striking homogeneity that could point to a more recent manufacture than most of the rest. Even among the darker blues there seems to be a certain difference between those of the four stars and those used in the circle near the walls, although this difference is not evident in the photographs and would need to be verified *in situ*.

2.3. Precedents in Portugal

It is very likely that there were, and still remain, many other examples of Mudéjar floors in Portugal than has been published in the past. In fact, Portuguese painting offers numerous testimonies, although, admittedly, most of those we saw so far seem to be very early (e.g., in the *Mosteiro de Alcobaça* [3, plates I-V]) and very different from this case, mostly formed with Hispano-Moorish tiles. The two most important and best-known Mudéjar tiled floors in Portugal are at the *Palácio Nacional de Sintra*. Both are very different from each other and neither has been studied in detail, although their interest would recommend it. One of these covers the central space of the presbytery of the palace chapel [4, image p.28] and has been dated to the third quarter of the 15th century, given that, by constructional logic, it must have been installed before 1470, the year in which the altarpiece presiding over this space was commissioned. This dating is consistent with the style of the work. The glazes on this floor are quite worn from use, but, in general, the composition is well preserved and restored with pieces that were installed at a later date. In terms of its geometry and the colours of its glazes, it resembles some contemporary tiles made in Seville or by documented Sevillian potters who worked in other parts of Andalusia, Extremadura, Castille, Leon and Aragon. Some authors have also pointed to a possible Moroccan origin. Santos Simões mentioned a possible origin in Toledo [3,

pp.57-58], while Reynaldo dos Santos points to similarities with the floors of Andalusia [4, pp. 28-29].

The other tiled floor is that of the supposed bedroom of king Afonso VI (1643-1683) [3, plates VIII-VIIIA]. Although its application is much older than the chronology of that monarch, we have no arguments to date it, although the presence of tiles that seem to be decorated by the *cuerda seca* process could be dated to the second half of the 15th century. Reynaldo dos Santos atributes them to Fernán Martínez Guijarro [4, p. 29]. However, as the production of this Sevillian potter has not yet been analytically identified, but only by documentary means, it seems risky to venture such a hypothesis, or even a Sevillian origin, given that nothing similar to this floor has been found in western Andalusia, unlike the floor of the Sintra chapel.

A fragment of tiled paving has recently been found *in situ*, although in a very modified architectural context, on the site of the former convent of *Nossa Senhora da Graça*, in Tavira [5]. It is of the same type as the floor of the palace chapel in Sintra and can therefore be dated from the mid-14th to the 15th century. Although the above are only isolated examples, these facts prove that in Portugal there were floors in the Mudéjar tradition, although we do not actually know where they were made and who may have installed them.

However, the floor of the small oratory of the Palace of Bacalhôa should be much more recent and fits, in terms of style, into the late Mudéjar tradition which, in the case of Seville, persisted throughout the 16th century. But neither Santos Simões nor Reynaldo dos Santos seem to have been aware of its existence, maybe because of its secluded location n the Palace.

3. ANALYTICAL CHARACTERIZATION BY SEM-EDS

3.1. Samples

For technical reasons the number of samples was limited to seven. Since the centre of the floor is the place where more colours are to be found (two shades of white and three or four shades of blue) it was decided to concentrate the sampling there instead of dispersing it throughout the area. Also, the central area must have been the first to be laid and the condition of the tiles suggests that it is composed of mostly original elements, while other areas may have been restored or recomposed over the years. The two last samples were collected from the periphery at both sides of the room.

Figure 6 illustrates the sampling spots and the codes through which the samples were referenced. Sampling was done with a scalpel removing only very small scales, except in areas where the glaze had already been lost, allowing the collection of a slightly larger piece of biscuit. The tiles selected for sampling (representing two shades of white and three shades of blue) were already noticeably decayed which, besides making the sampling easier, ensured that the pieces selected could not have been applied in eventual 20th century restoration works.

Table 1 includes data on each sample and its analytical use.

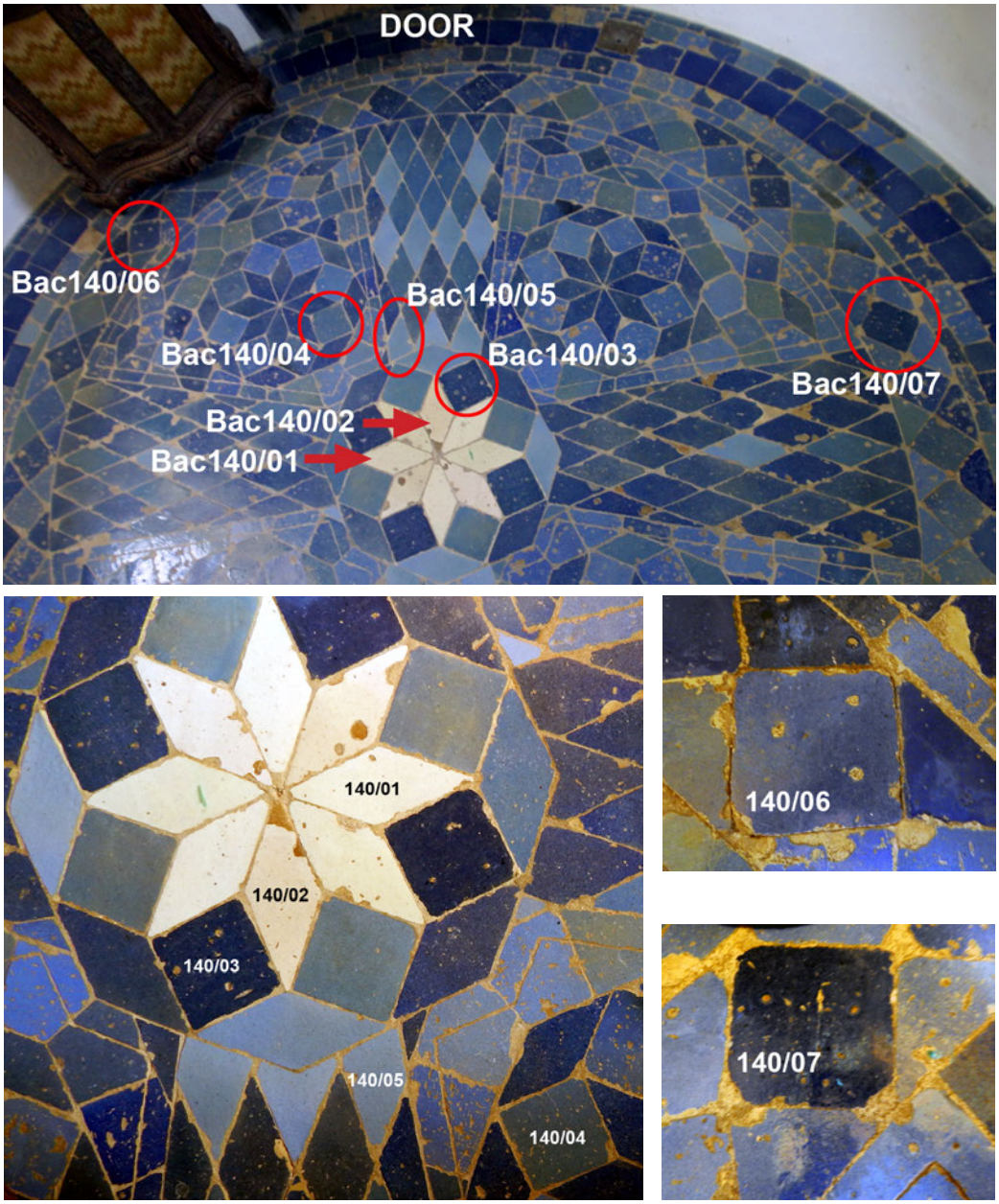


Figure 6. Sampled elements and reference codes.

Table 1. Identification of items and sample references

Identification	Sample Ref.	Number of analyses performed
White rhombus, with no stilt marks	**Bac140/01**	**glaze (1); biscuit (2)**
White rhombus, darker hue, with no stilt marks	**Bac140/02**	**glaze (2); biscuit (1)**
Deep blue square with stilt marks	**Bac140/03**	**glaze (2); biscuit (-)**
Medium blue square, with no stilt marks	**Bac140/04**	**glaze (2); biscuit (1)**
Light blue quadrilateral, with no stilt marks	**Bac140/05**	**glaze (2); biscuit (2)**
Dark blue square with stilt marks	**Bac140/06**	**glaze (3); biscuit (2)**
Dark blue square with smaller stilt marks	**Bac140/07**	**glaze (1); biscuit (1)**

3.2. Methods and instrumental means

The azulejo samples were stabilized in epoxy resin, lapped and polished to obtain a flat cross-section for observation and analysis by scanning-electron microscopy coupled with an X-ray energy-dispersive spectrometer (SEM-EDS).

The optical images of the sections were acquired with an Olympus DP20-5 digital camera coupled to an Olympus SZH stereomicroscope. SEM observations and EDS analyses were made at LNEC using a TESCAN MIRA 3 field emission microscope combined with a BRUKER XFlash 6|30 EDS system. The samples were uncoated and the observations were made in backscattered electrons mode (BSE), with a chamber pressure of typically 10 Pa, at an accelerating voltage of 20 kV with the sample sections at a distance of 14 ± 1 mm from the detector. SEM images were typically acquired at magnifications of 350 x and 700 x for the glaze and 1,000 x or over for inclusions in the biscuit.

The selection of areas for EDS quantification avoided large inclusions in the glaze or biscuit representing more than ca. 5% of the full selected area. From our previous experience, the adequate minimum measurement areas are 200 x 200 μm for glazes and 500 x 500 μm for biscuits. In general, multiple measurements were made and in such case the results are averages and smaller non-overlapping areas may be used to the same effect.

Minor elements, usually representing less than 1% of the compositions, such as magnesium (Mg) and iron (Fe) in the glazes, or titanium (Ti) in the biscuits were not included in the tables of results.

The quantification of tin (Sn) in the glazes may be problematic because the aggregation of crystals often results in a large variance. That problem was dealt with by using larger areas whenever aggregation was visually detected in the SEM images or, when that was not possible, averaging the results of multiple analyses on different areas.

The amount of oxygen (O) was calculated through the remaining elements stoichiometry of their most commonly considered oxides (Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, Fe_2O_3, SnO_2, PbO) and the result was normalized to 100 %.

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

3.3. Results

3.3.1. Optical microscopy of samples

Figure 7 depicts optical images of a selection of samples of which the terracotta colour of samples Bac140/04 and Bac140/05 is particularly remarkable. Samples Bac140/06 and Bac140/07 have a cream colour similar to those of the top row of Figure 7.

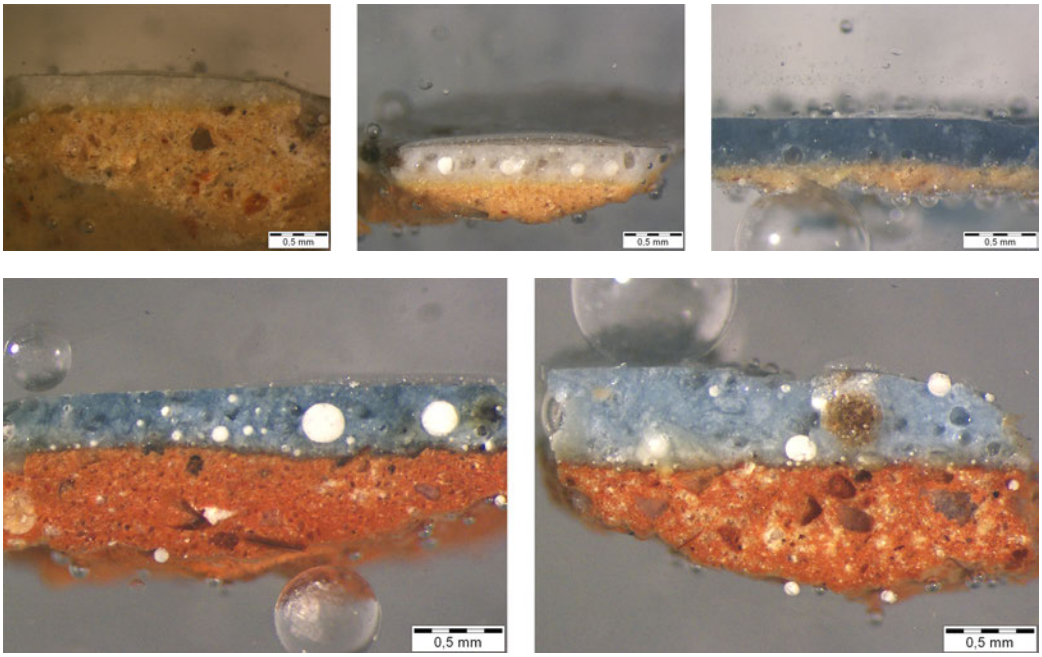
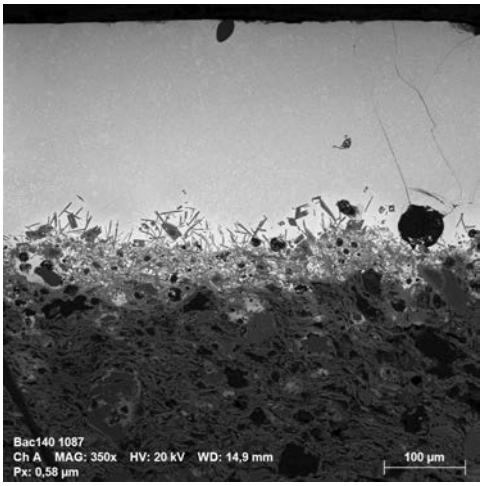
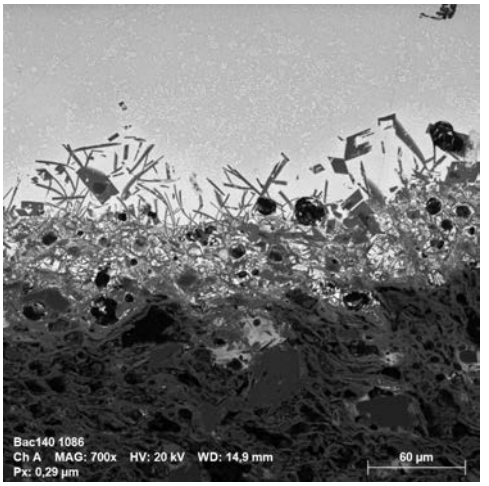


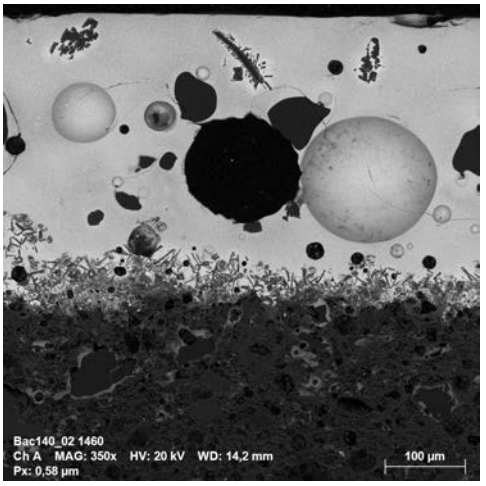
Figure 7. Top row, from left to right: Bac140/01; Bac140/02 and Bac140/03. Bottom row, from left to right: Bac140/04 and Bac 140/05 (images: LNEC).

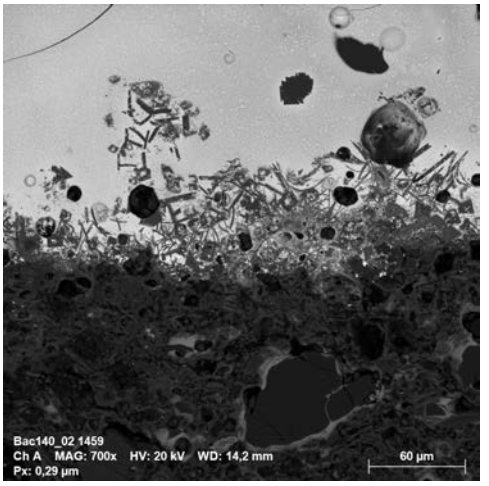
3.3.2. Morphology

Figure 8 depicts, at the same magnifications for comparison purposes, sectional SEM images of the samples showing the main micro-morphological characteristics generally associated with the glazes and their interfaces. The light grey area on top is the glaze, while the dark grey area corresponds to the biscuit. Because of its colour, the inclusions in the glaze are conspicuous: gas bubbles retained in the glass, grains of sand (larger compact dark inclusions, usually with rounded edges) and bits of feldspars, often in disaggregation. The white specks in the midst of the glaze are aggregations of crystals of the white pigment (tin oxide).


Bac140 1087
Ch A MAG: 350x HV: 20 kV WD: 14,9 mm
Px: 0,58 µm
100 µm


Bac140 1086
Ch A MAG: 700x HV: 20 kV WD: 14,9 mm
Px: 0,29 µm
60 µm


Bac140_02 1460
Ch A MAG: 350x HV: 20 kV WD: 14,2 mm
Px: 0,58 µm
100 µm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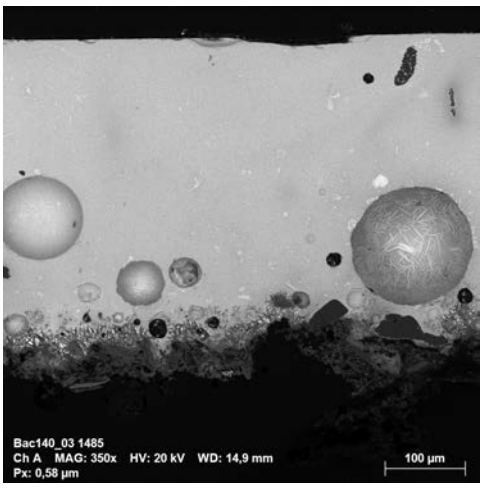
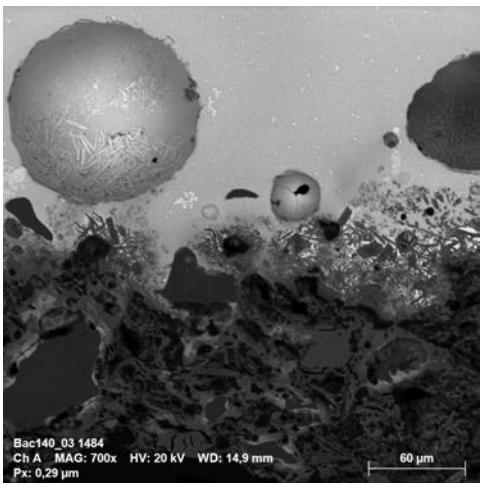
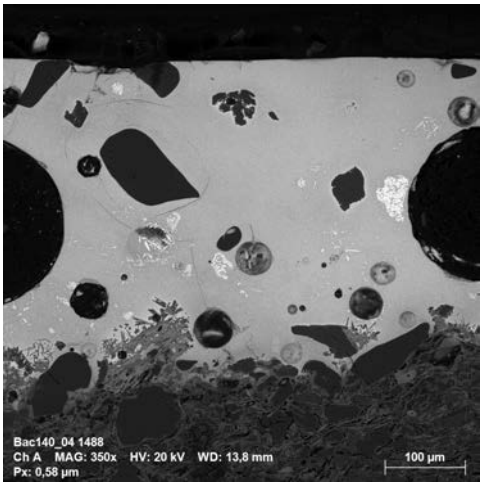
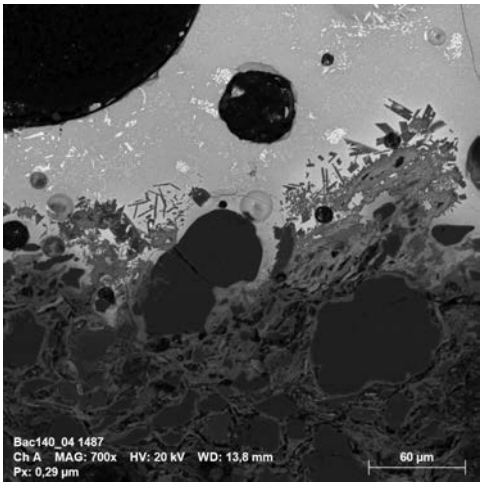
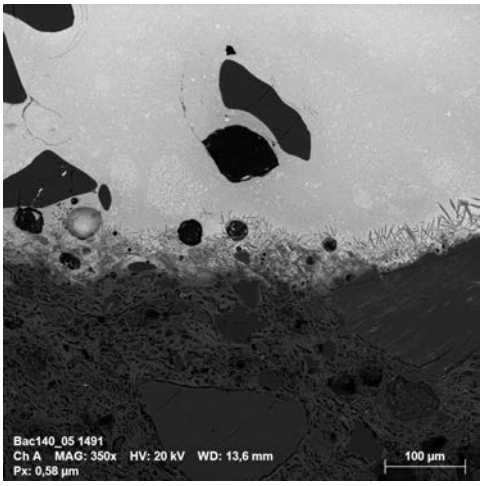
60 µm

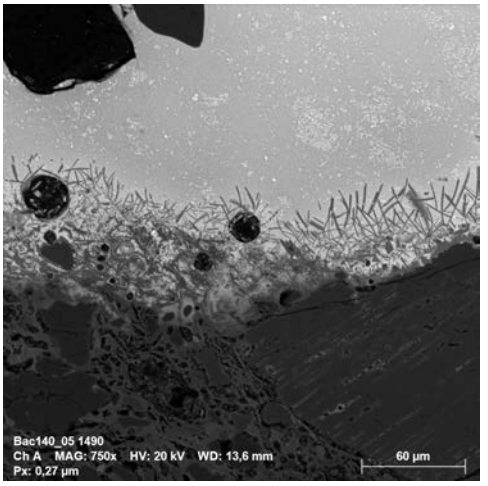

Bac140_03 1485
Ch A MAG: 350x HV: 20 kV WD: 14,9 mm
Px: 0,58 µm
100 µm

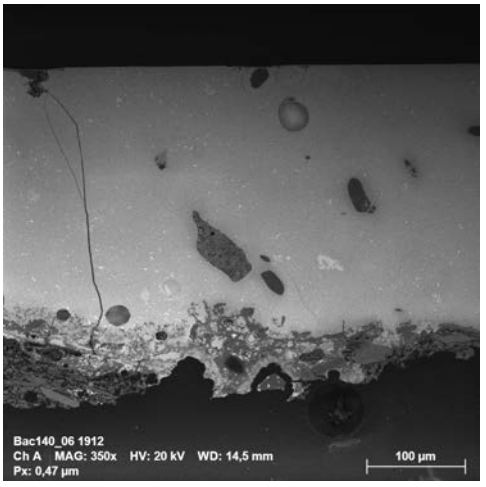

Bac140_03 1484
Ch A MAG: 700x HV: 20 kV WD: 14,9 mm
Px: 0,29 µm
60 µm

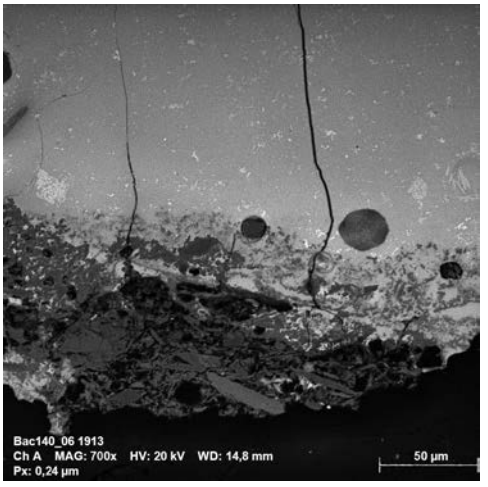

Bac140_04 1488
Ch A MAG: 350x HV: 20 kV WD: 13,8 mm
Px: 0,58 µm
100 µm


Bac140_04 1487
Ch A MAG: 700x HV: 20 kV WD: 13,8 mm
Px: 0,29 µm
60 µm


Bac140_05 1491
Ch A MAG: 350x HV: 20 kV WD: 13,6 mm
Px: 0,58 µm
100 µm


Bac140_05 1490
Ch A MAG: 750x HV: 20 kV WD: 13,6 mm
Px: 0,27 µm
60 µm


Bac140_06 1912
Ch A MAG: 350x HV: 20 kV WD: 14,5 mm
Px: 0,47 µm
100 µm


Bac140_06 1913
Ch A MAG: 700x HV: 20 kV WD: 14,8 mm
Px: 0,24 µm
50 µm

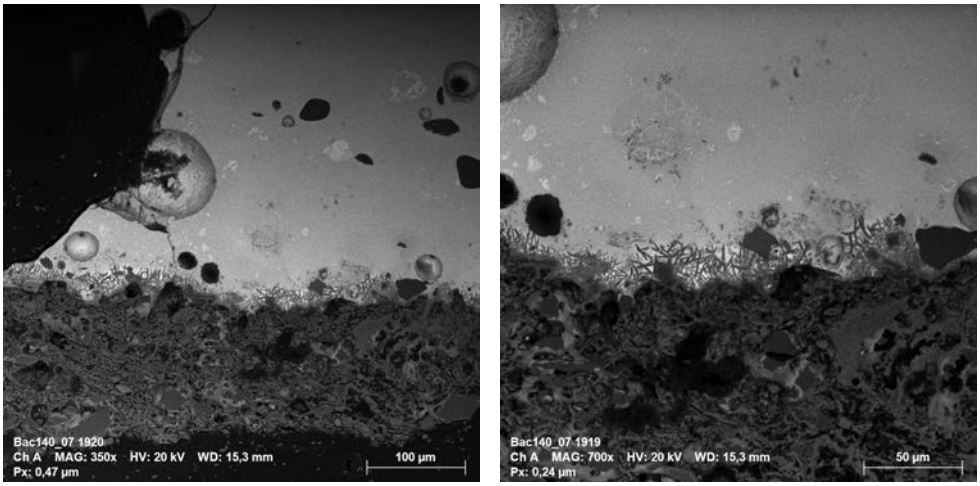
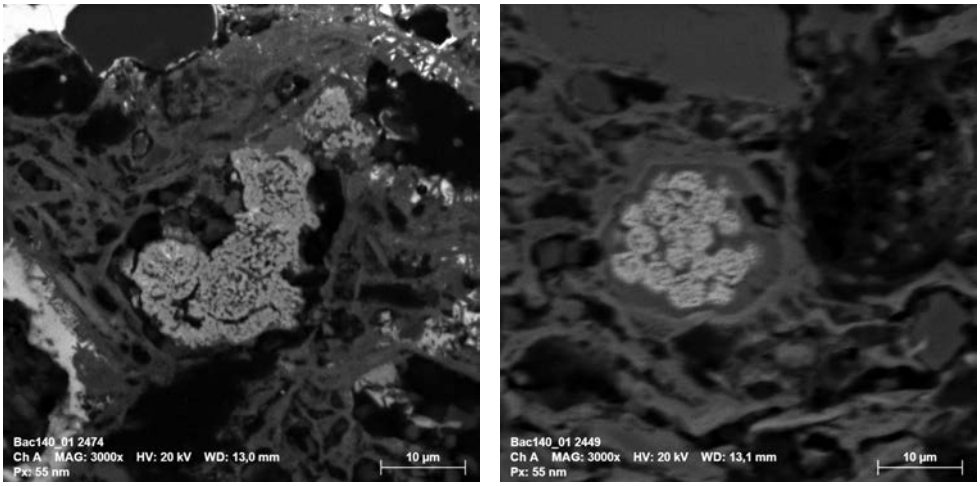


Figure 8. SEM-BSE images showing the main micro-morphological characteristics of, from top to bottom, Bac140/01, Bac140/02, Bac140/03, Bac140/04, Bac140/05, Bac140/06 and Bac140/07. Left side: glaze section at 350 x; Right side: detail of the biscuit-glaze interface at 700 x (images: LNEC).

Figures 9 and 10 depict images of morphological aspects or inclusions noted in the biscuits: framboidal crystallizations of pyrite [6] and rhombohedral dolomite crystals [7], not totally consumed over firing.



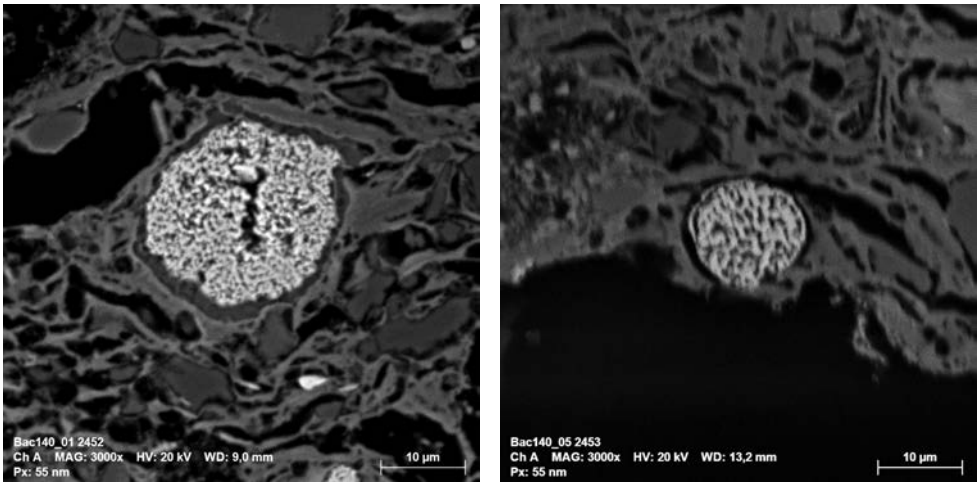


Figure 9. Top row: pyrite framboids in the biscuit of Bac140/01; lower row: more framboidal crystallizations of pyrite in the biscuits of Bac140/01 (left side) and Bac140/05 (right side) (images: LNEC).

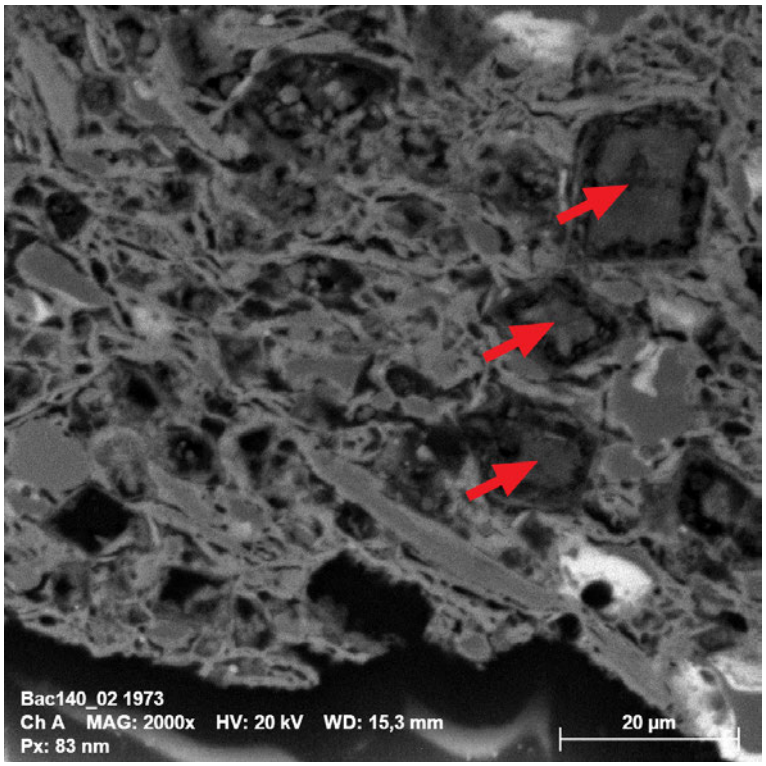


Figure 10. Rhombohedral dolomite crystals, not totally consumed over firing, in the biscuit of Bac140/02 (image: LNEC).

3.3.3. Glaze compositions

Table 2 includes the semi-quantitative results of analyses of the glazes by EDS in weight %. The silicon to lead ratios (Si/Pb) have been determined and are also included in the table. This ratio is a technological trait set by the glaze recipe and gives important information about the firing conditions in the kiln because the lower the ratio, the lower the temperature at which the glaze could be properly fired.

Table 2. Semi-quantitative composition of the glazes determined by EDS (values in wt. % with oxygen obtained by stoichiometry and sum of all elements normalized to 100%)

	O	Na	Al	Si	K	Sn	Pb	Si/Pb
Bac140/01	28.03	0.45	2.76	16.48	1.82	7.20	43.25	0.38
Bac140/02	26.14	0.35	2.01	14.57	1.77	11.41	43.74	0.33
Bac140/03	30.66	1.32	1.61	19.99	2.01	3.15	41.26	0.48
Bac140/04	29.13	0.56	2.57	17.26	1.54	8.76	40.17	0.43
Bac140/05	29.71	0.26	2.77	17.77	1.69	8.12	39.68	0.45
Bac140/06	34.47	2.03	1.50	22.70	3.77	4.10	31.43	0.72
Bac140/07	30.66	1.41	1.48	19.92	2.02	2.31	42.21	0.47

3.3.4. Biscuit compositions

Table 3 includes the semi-quantitative results of analyses of the biscuits in weight %. The calcium to silicon (Ca/Si) ratios, related with the suitability of the clay to tin-glazing, have been determined and are included in the table.

Table 3. Semi-quantitative composition of the biscuits determined by EDS (values in wt. % with oxygen obtained by stoichiometry and sum of all elements normalized to 100%)

	O	Na	Mg	Al	Si	K	Ca	Fe	Ca/Si
Bac140/01	44.71	1.15	1.71	8.95	23.67	1.78	13.84	4.20	0.58
Bac140/02	42.28	0.61	3.62	6.93	19.78	2.17	20.38	4.22	1.03
Bac140/03	43.71	1.28	2.29	7.08	23.03	2.29	16.49	3.84	0.72
Bac140/04	47.23	1.09	0.83	8.72	28.91	3.12	6.39	3.70	0.22
Bac140/05	45.64	0.65	1.35	9.85	25.23	3.27	9.38	4.62	0.37
Bac140/06	43.29	1.55	2.00	6.32	22.58	1.32	19.23	3.70	0.85
Bac140/07	44.32	0.76	1.97	6.05	25.02	1.83	16.79	3.26	0.67

4. DISCUSSION OF THE INSTRUMENTAL RESULTS

4.1. The glazes

The morphological characteristics of the glazes are broadly compatible with the productions of the Lisbon workshop of João de Góis and his circle, namely as respects the profusion of crystals at the interface between the glaze and the biscuit [8, fig. 7], especially remarkable in samples Bac140/01, -/02 and -05. Therefore, in this case, the interfacial outgrowths do not allow an aggregation in separate clusters.

Glazes from the 16th century use three main fusing agents: sodium (Na), potassium (K) and lead (Pb) which must be balanced in the recipes. Portuguese azulejo panels may be separated from known Spanish and Flemish productions, as well as from later Portuguese productions, by their very low contents in sodium and potassium, two chemical elements that are incorporated by the potter as weighted raw materials, as well as by the low Si/Pb ratios [8; 9]. For a graphical assessment based on those two characteristics, Figure 11 plots the ratio (Na+K)/Pb against Si/Pb for all samples (the normalization to the content in lead enhances the comparability by making all other contents relative to it).

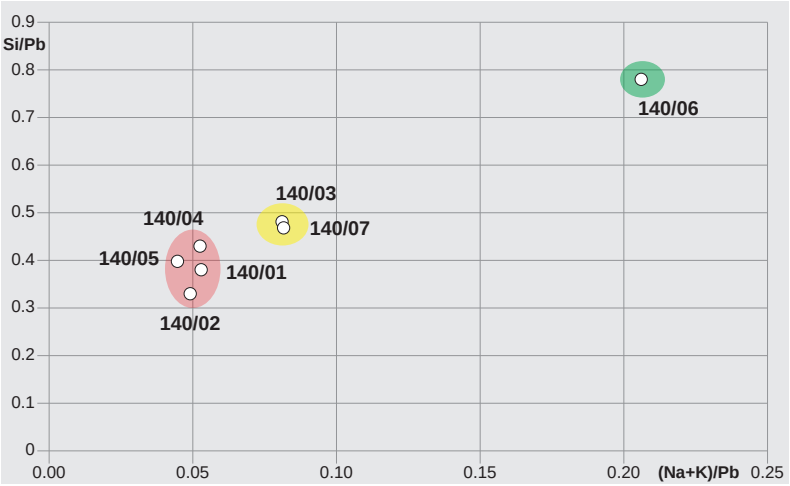


Figure 11. Comparison of the glaze compositions of all the Bac140 samples based on two characteristic content ratios.

Figure 11 clearly defines three clusters on the basis of the ratios compared: one is formed by samples Bac140/01; -/02; -/04 and -/05 (red cluster), while the remaining samples constitute two more clusters (yellow and green- this one with a single element).

Figure 12a compares the EDS spectra of Bac140/01 from the red cluster with one from the yellow cluster (Bac140/07) evidencing the very clear difference pertaining the low contents in sodium (Na) but also the higher contents in aluminium (Al) and tin (Sn) of the red cluster, extending to all of its four elements as may be verified from Table 2. In the yellow cluster, the contents in potassium are only slightly higher but in Bac140/06 (green cluster) the content is much higher.

The spectra may be compared with previously published reference spectra [8, p. 43] to confirm that the spectrum of Bac140/01, used to characterize the red cluster, conforms only with productions of the workshops of Lisbon, while those in the yellow cluster, with low contents in aluminium and tin and high Si/Pb ratios, are only similar, of the provenances studied, to mid-16th century Hispano-Moresque productions of the workshops of Seville. Figure 12b includes two such EDS spectra for comparison with the ones above them.

The composition of Bac140/06 is separated by its low content in lead compensated by higher contents in sodium and potassium. Following a graphical technique [10] Figure 13 offers a comparison of spectra of this sample with one from the yellow cluster, showing that the glaze formulations differ only markedly in the balance of fusing agents,

presumably aiming at a lower use of the expensive lead oxide by the workshop that manufactured the tile from which Bac140/06 was collected.

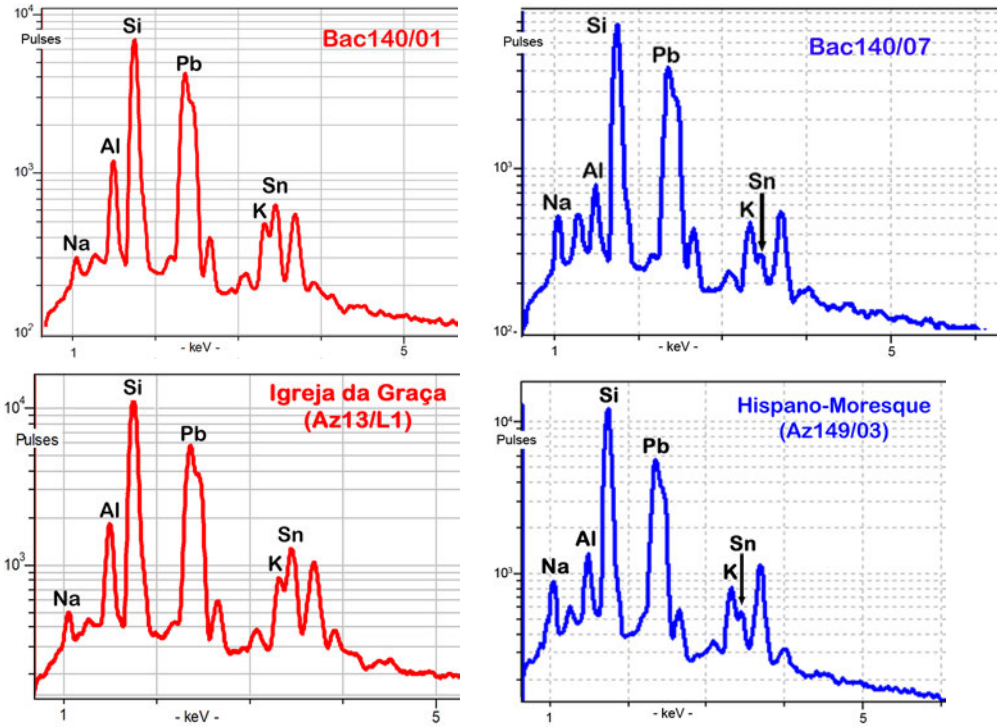


Figure 12. Top (12a) - Comparison of EDS glaze spectra of Bac140/01 (red cluster) and Bac140/07 (yellow cluster). Bottom (12b) - EDS glaze spectrum of one of the tiles bearing the monogram of João de Góis at *Igreja da Graça* and typical glaze spectrum of mid-16th century Hispano-Moresque Sevillian blue or green tiles.

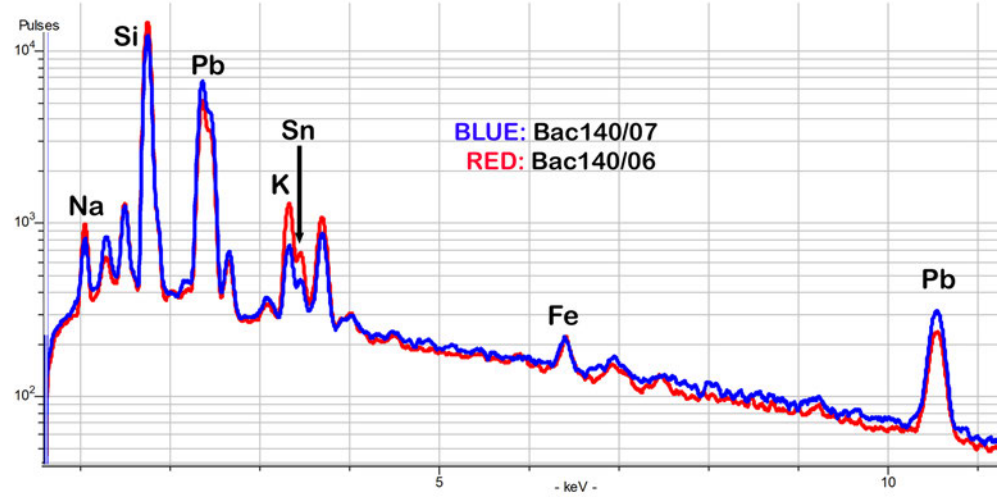


Figure 13. Superposition of the spectra of Bac140/07 from the yellow cluster and Bac140/06 from the green cluster for a graphical comparison of compositions.

The red cluster in Figure 11 includes two white tiles (samples Bac140/01 and -/02) and two blue tiles (samples Bac140/04 and -/05) and yet, from Table 2, the contents in tin (Sn) of the blue tiles are comparable to those of the white tiles. This is surprising, because a blue glaze needs much less tin than a white glaze, exactly as may be seen in the case of Bac140/03, and suggests that in both Bac140/04 and -/05, the blue pigment was added to a raw white glaze with a tin content compatible with majolica, while in Bac140/03 the tin content was low and finely balanced with the blue cobalt pigment as in Hispano-Moresque tiles manufactured in Seville. The images in Figure 7 are revealing: while in Bac140/04 and -/05 the glaze looks whitish, in Bac140/03 it looks blue and transparent, because its composition has little of the expensive tin, as befits a coloured glaze.

To support a better insight into the provenance of the tiles, a log-based principal component analysis (PCA) was performed comparing the elemental chemical composition of the glazes of the samples taken from the floor to those of other 16th century productions researched in the past, namely samples from the tiles in *Igreja da Graça* signed by João de Góis [8, figure 2; 11], the panel *Nossa Senhora da Vida* also by his workshop [8, figure 4], samples of Hispano-Moresque tiles ascribed to the workshops of Seville [8, figure 1], and the Albuquerque coat-of-arms in Bacalhôa which we concluded to be likely of foreign production [12] as well as the *Rape of Europa* panel, also in Bacalhôa, that we ascribed to a Portuguese workshop, likely that of João de Góis [13]. The semi-quantitative compositions obtained from [8] did not include the contents in tin (which, anyway, should not be used when comparing majolica with Hispano-Moresque tiles because these may have much less tin in their compositions) and therefore all elemental contents in the other cases were re-calculated for the PCA without considering the contents in tin.

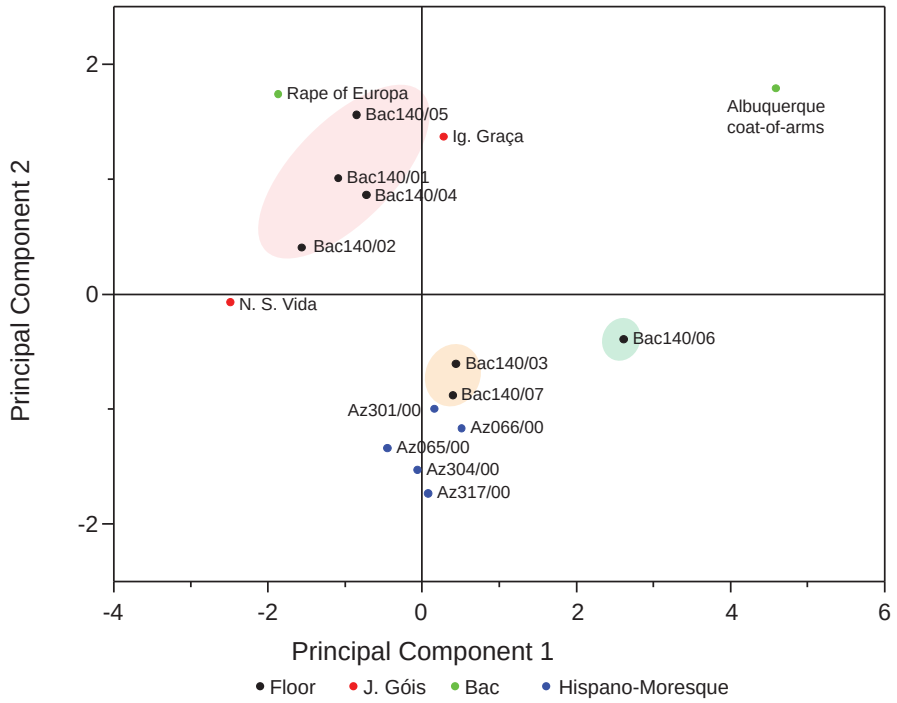


Figure 14. Score plot of the Principal Component Analysis of the glazes compared to those of known types.

The result of the PCA analysis of the glazes is presented in Figure 14 through a plot in the plane of the two first principal components (PC1 and PC2). PC1 explains 58 % of the variation and is controlled in the positive sense by the contents in sodium (Na), silicon (Si) and potassium (K), and in the opposite sense mostly by the contents in lead (Pb). PC2 explains 31 % of the variation and is controlled in the positive sense mostly by the contents in aluminium (Al), and in the opposite sense by the contents in sodium and lead, as seen in Figure 15, where the loadings plot is represented as a vector graph. Given the percentages of variation explained by each principal component, the planar projection of Figure 14 accommodates graphically ca. 90% of the compositional variation of the glazes of all test items included in the PCA analysis.

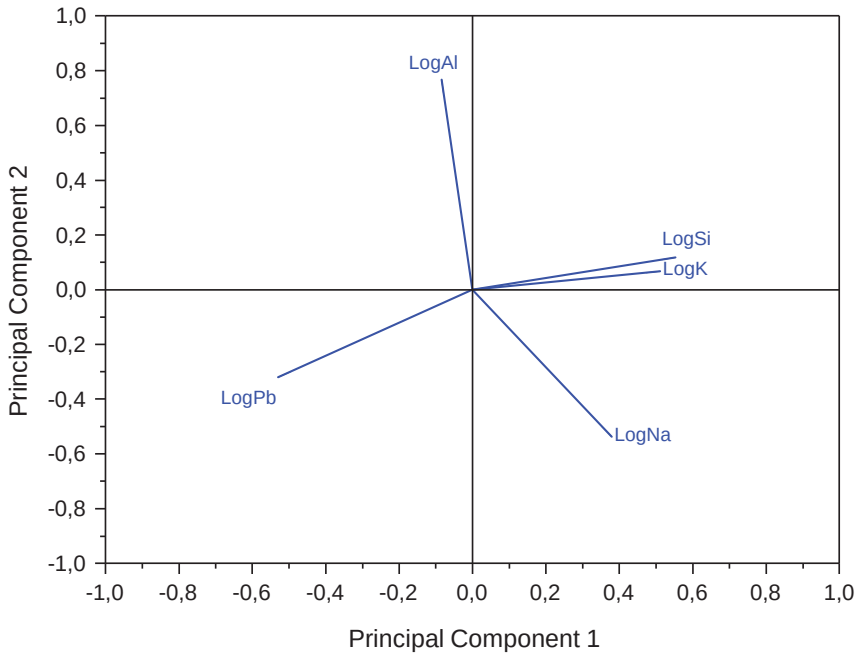


Figure 15. Loadings plot of the Principal Component Analysis of the glazes.

The score plot in Figure 14 is very clear and shows that: i) none of the glazes is similar to that of the Albuquerque coat-of-arms; ii) the glaze of two of the tiles with the stilt marks (Bac140/03 and Bac140/07 - yellow cluster) is only similar, in the group, to Hispano-Moresque glazes, while that of Bac140/06 (green cluster) is closer to them than to the others and mostly separated by its lower content in lead; iii) finally, the other four glazes can be clustered with the João de Góis panels in *Igreja da Graça* and the panel *Nossa Senhora da Vida*, as well as with the *Rape of Europa*, because in the projection of Figure 15 they fall within a small area (red cluster), exactly inside a triangle defined by the average compositions of those three panels which represent a chronologic period of 10 to 15 years.

The stilt marks on the tiles from which Bac140/03, -/06 and -/07 were collected suggest a Hispano-Moresque technology as used in Seville. As far as we know, to avoid such unsightly marks and increase the number of tiles that could be fired together, Flemish potters have always fired tiles upright, over two cylinders of rolled clay (Figure 16), occasionally causing the running of dense paint as is often seen in the patterned tiles and figurative panels of Bacalhôa.

Figure 16. A fragment of tile that was never applied and still has adhered one of the supporting cylinders for firing upright (image: *Museu do Palácio da Bacalhôa* c. Associação de Colecções | The Berardo Collection).

4.2. The biscuits

The interpretation of the composition of biscuits is very different from the interpretation of glaze compositions, because clays are natural products and not the result of a man-made recipe. Also, they are naturally variable, even if extracted from the same pit, because the layers result from tens of thousands, sometimes more than a million years of natural deposition and transformation, and the composition of a layer representing such a long period may vary considerably as the extraction attains deeper sublayers of clay.

The result of a comparative PCA analysis of the biscuits of all the samples studied with the same tiles and panels previously identified is presented in Figure 17 through a plot in the plane of the two first principal components (PC1 and PC2). PC1 explains 59 % of the variation and is controlled in the positive sense mostly by the contents in aluminium (Al), silicon (Si), potassium (K) and iron (Fe), and in the opposite sense mostly by the contents in magnesium (Mg) and calcium (Ca). PC2 explains 20 % of the variation and is controlled in the positive sense mostly by the content in magnesium and iron, and in the opposite sense by the contents in sodium (Na) and silicon (Si), as seen in Figure 18, where the loadings plot is represented as a vector graph. The planar projection of Figure 17 accommodates graphically ca. 79 % of the compositional variation of the biscuits of all test items included in the PCA analysis.

The score plot of the PCA in Figure 17 shows that: i) the biscuit of sample Bac140/02 is very similar to that of the Albuquerque coat-of-arms panel (green cluster); ii) samples Bac140/03, -/06 and -/07 fall together (blue cluster) in the midst of the Hispano-Moresque tiles; iii) Bac140/01, -/04 and -/05 are dispersed in the first and fourth quadrants where the panels attributed to the circle of João de Góis are set too- they could reasonably be clustered together because they stand alone thanks to their low contents in calcium and magnesium and relatively high in aluminium and potassium (red cluster represented in three separate groups).

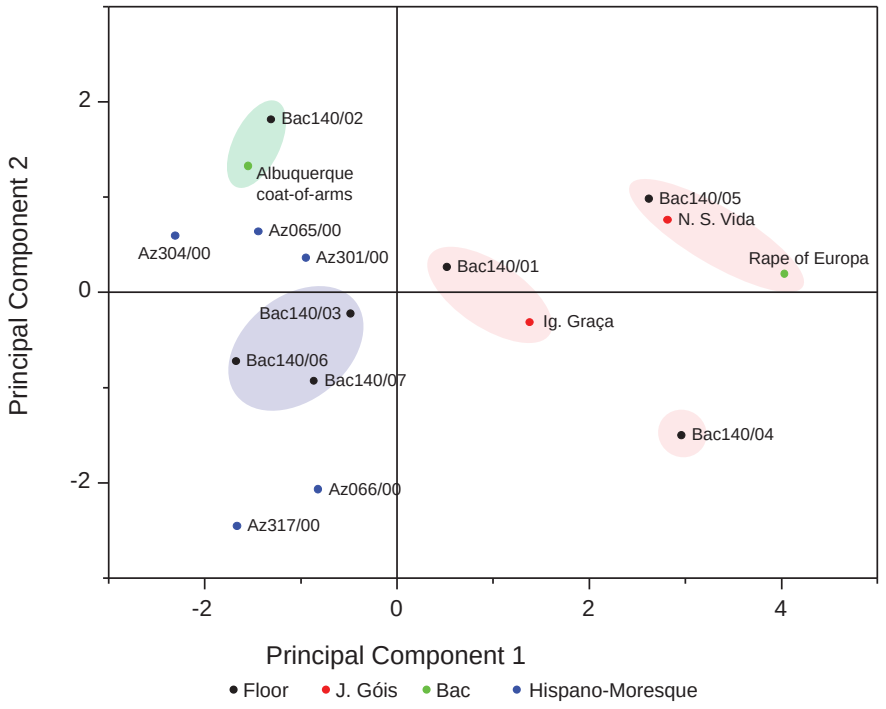


Figure 17. Score plot of the Principal Component Analysis of the biscuits compared to those of known types.

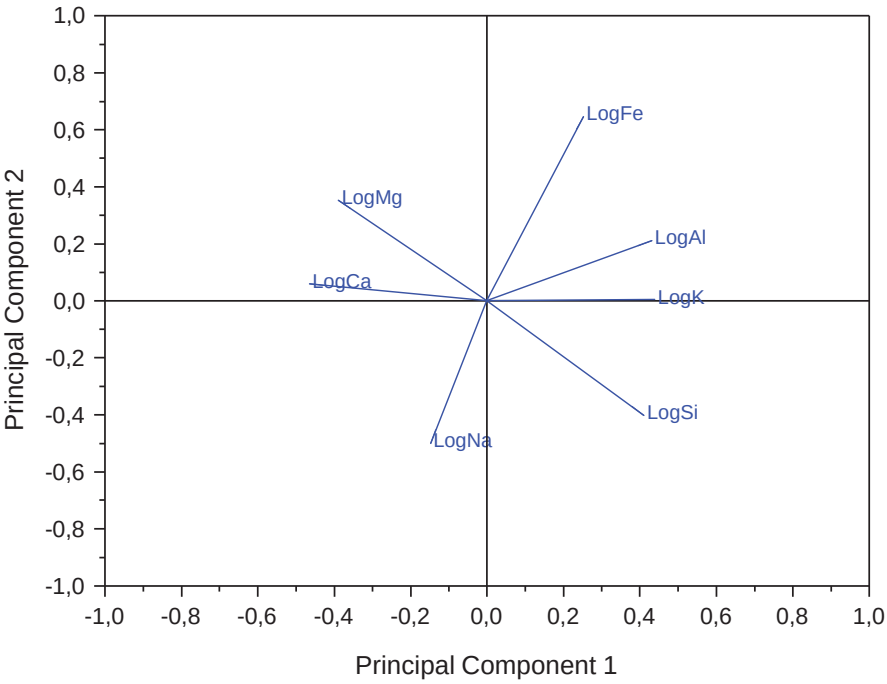


Figure 18. Loadings plot of the Principal Component Analysis of the biscuits.

The rhomboid-shaped hollows of euhedral dolomite crystals that characterize the biscuits of imported tiles, as well as the biscuits that were presumably imported unglazed [7; 12] are also present in the biscuit of Bac140/02 (Figure 10). Therefore, although the tile was very likely glazed in Lisbon as shows the PCA of the glazes in Figure 14, the biscuit belongs to the set that was likely imported (or else was made with imported clay) and because of that it clustered in the biscuit PCA of Figure 17 with the Albuquerque coat-of-arms panel.

As for the biscuits of Bac140/04 and -/05, they share the characteristics of the clay used by the circle of João de Góis [8]. Bac140/01 deviates from them, particularly in the high content in calcium, resulting in a cream-coloured biscuit, while the clays used in Bac140/04 and -/05 result in terracotta-coloured biscuits (Figure 7). We have already encountered biscuits similar to that of Bac140/01, with relatively high contents in calcium, in the production of João de Góis, precisely in the tiles that bear his monogram at *Igreja da Graça* [11], explaining their proximity in the PCA plot of Figure 17. A further point to note in the comparison is that the regular and aggregated framboidal crystallizations of pyrite, as found particularly in Bac140/01 (Figure 9), also occur in the biscuits of tiles by the workshop of João de Góis at *Igreja da Graça* (Figure 19) and elsewhere. Although such crystallizations are known in several clays, they are not a particularly common feature.

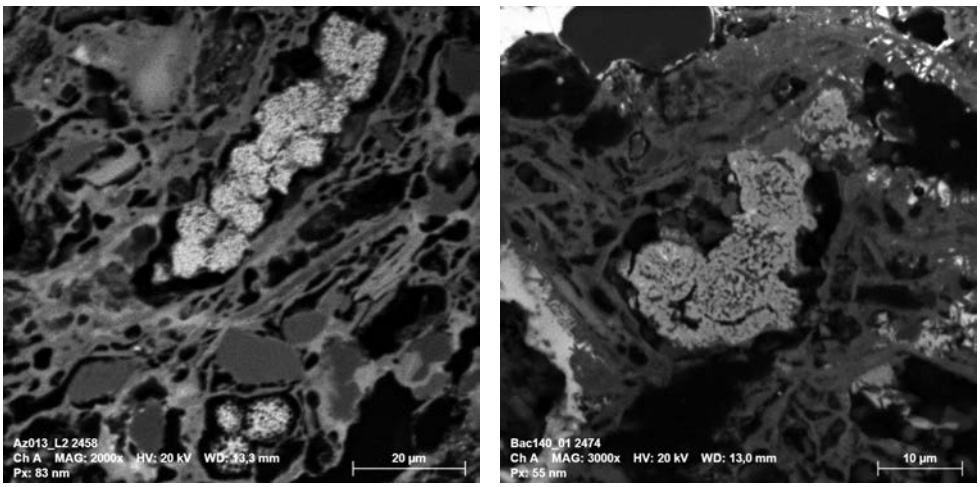


Figure 19. Framboidal pyrite in the tiles of *Igreja da Graça* signed by João de Góis (Az013/L1- left side) and the Bacalhôa oratory floor (Bac140/01 - right side) (images: LNEC).

5. CONCLUSIONS

The only tiled floor remaining at the *Palácio da Bacalhôa* graces a former oratory and is a masterpiece of which no existing peers are presently known to us. Seven tiles were sampled, five of them from the centre of the floor, where more colours and hues are present. Therefore, taking into consideration the variability seen in the floor itself, the conclusions are drawn from a relatively small number of pieces. Still, the results form a coherent image and we believe that, notwithstanding the eventual occurrence of individual elements that may have been renovated, as suggested by tiles of unusual shades of blue found here and there, the conclusions are generally applicable to the whole floor.

Tiles of at least two different geographic provenances were identified. The tiles marked "1" in Figure 20a, one of which corresponds to sample Bac140/03, have stilt marks, as have tiles 140/06 and 140/07 in Figure 8, and the composition of their glazes is unknown to us from Portugal at the time when the floor was presumably laid. The results suggest that they were probably manufactured in Seville, Spain. But while the biscuits of all three tiles are similar, two different glazes were identified in them, only one of which (that of Bac140/03 and 140/07) is fully compatible with the glazes of mid-16th century Hispano-Moresque tiles that we researched in the past. The other, even though still compatible with a 16th century chronology, may be from a different workshop or from the same workshop but at a later time, when the technology had evolved towards a reduction in the use of lead which was an expensive raw material.

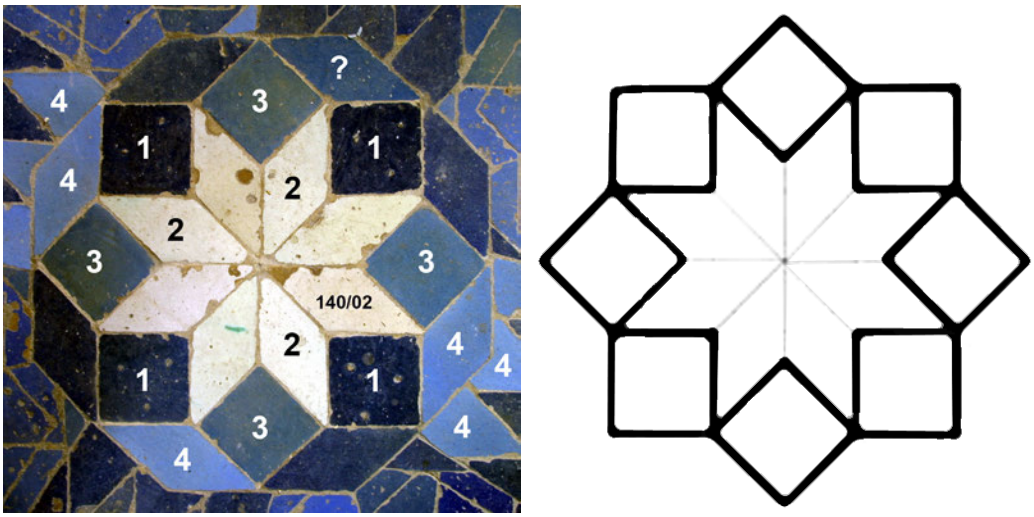


Figure 20. Left side (20a) - The chromatic elements of the central star of the oratory floor; Right side (20b) - The geometric design based on Islamic eight-pointed star patterns.

All the other tiles studied have glazes morphologically and compositionally compatible with the 16th century productions of the Lisbon workshop of João de Góis where azulejos were already being manufactured in 1561 [14]. One of these tiles (marked 140/02 in the image of Figure 20) was very likely glazed over a foreign biscuit, part of a large set imported around 1565 [7; 15]. However, results point to the use of Portuguese clay for the remaining biscuits (Bac140/01, -/04 and -/05). One of them is glazed in white and its biscuit is richer in calcium than the rest, a composition similar to tiles from the panels of *Igreja da Graça* signed by João de Góis [11] in which the same aggregations of framboidal pyrite were also found. The panels of *Igreja da Graça* may be dated through their inferable connection with the burial there of Afonso de Albuquerque in May 1566 [11]. The clays used in the tiles from which samples 140/04 and 140/05 were collected are similar to those used e.g. in the panel *Nossa Senhora da Vida*, which may be dated to the second half of the 1570s up to 1581 [16].

The different provenances and chronologies point to the widespread use of remainders-tiles that were manufactured by several workshops at different times, maybe for several different purposes, and kept in deposit in the palace for future use. When was the floor laid? The conclusion that a medley of tiles of several provenances and chronologies was

used, only allows stating 1565 as an *ex post* date. A likely date would set the floor in the 1570s because of the presumed chronologies of the tiles made in Lisbon and also because around 1565, when the lining of Bacalhôa with majolica tiles was proceeding at full pace and, presumably, on a "cost no object" basis [1; 15], the decision would very likely have been to purchase new tiles of homogeneous colours, instead of using remainders with such a variety of hues, often colliding with what might be expected from a symmetrical design, as in the case of the pieces marked "4" in Figure 20a with which one of the rhombi (marked "?") is at odds.

In the *Museu do Palácio da Bacalhôa* are kept various scratched and worn blue tiles indicating that more such floors once existed in the estate. In the oratory the floor tiles are not worn as should befit a span of around 450 years, however the private use of a space well inside the house adjacent to a bedchamber and the likely use of a carpet from the door to the altar, explain the absence of visible wear. On the other side, the tiles are obviously decayed, particularly those nearer to the walls from where harmful moisture passes on to the floor, and some of these have clearly been renovated sometime in the past. Also, the white tiles at the entrance are broken (Figure 2) suggesting that they may be older than the rest, maybe the only remainders of an earlier floor.

The design of the floor is unique but its star elements, based on the common Islamic eight-pointed star patterns (Figure 20b), follow the 16th century Mudéjar decorative style, with an unusual prevalence of rhombic shapes that makes it all the more interesting. Since many of the tiles are presumably of local manufacture, they must have been cut on site to set shapes and dimensions, rather than imported ready to assemble. Unfortunately, the author of the exquisite design and the master tiler or tilers who carried out the decorative scheme in the *alicatado* manner with such skill will remain unknown, unless documentary evidence is one day found.

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