

A comparative interdisciplinary study of gilding techniques and materials in two Portuguese Baroque “talha dourada” complexes

I.C.A. Sandu, E. Murta, E. R. Neves, M.F.C. Pereira, A. V. Sandu, S. Kuckova, A. Maurício

Abstract

The gilded polychrome carved wood in Portugal, (*talha dourada*) gained its most rich expression during the Baroque epoch with the development of production techniques and the use of gold leaf. The present paper proposes a complementary and comparative study of two important complexes of *talha*: one from St. Alberto’s church, integrating the visiting circuit of the Museu Nacional de Arte Antiga (Lisbon), and the other from church of Vale de Figueira (Santarém district). Both complexes house *talha dourada*’s decoration from the 18th century, that were studied through an inter-disciplinary approach, in which analytical data (optical microscopy, fluorescent stain, XRF, SEM-EDX, micro-computerized tomography, FTIR, Matrix-Assisted Laser Desorption/Ionisation-Time of Flight Mass Spectrometry) complement historical data.

Keywords:

carved gilded wood, comparative study, gilding techniques, materials, analytical study.

Um Estudo Comparativo Interdisciplinar das Técnicas e Materiais de Douramento em dois conjuntos de talha dourada barrocos portugueses.

Resumo

A arte da *talha dourada* em Portugal ganhou a sua mais rica expressão durante a época barroca devido ao desenvolvimento das técnicas de produção e ao uso do ouro em folha. O presente trabalho propõe um estudo complementar e comparativo de dois complexos importantes da talha respectivamente em Lisboa, na Igreja de São Alberto, integrada no circuito de visitas do Museu Nacional de Arte Antiga, e no distrito de Santarém, na Igreja de Vale de Figueira. Os dois complexos albergam decoração em talha dourada do século XVIII, estudados através de uma abordagem inter-disciplinar, em que os dados analíticos (microscopia óptica, teste de coloração fluorescente, FRX, MEV-EDX, microtomografia computadorizada, FTIR, Matrix-Assisted Laser Desorption/Ionisation-Time of Flight Mass Spectrometry) complementam os dados históricos.

Palavras-chave:

“talha dourada”, estudo comparativo, técnica de douramento, materiais, estudo analítico.

Un Estudio Comparativo e Interdisciplinar de las Técnicas y Materiales de Dorado en dos Conjuntos de Talla Dorada del Barroco Portugués.

Resumen

El arte de la talla dorada en Portugal ganó su más rica expresión durante el período barroco a través del desarrollo de técnicas de producción y el uso de pan de oro. Este trabajo propone un estudio complementario y comparativo de dos grandes complejos de talla, el de la Iglesia de San Alberto, integra el circuito de visita del Museu Nacional de Arte Antiga en Lisboa, y el de la Iglesia de Vale Figueira, en Santarém. Los dos complejos albergan retablos dorados del siglo XVIII, que han sido estudiados a través de una metodología interdisciplinar, en la que los datos analíticos (microscopía óptica, tinción fluorescente, fluorescencia de rayos X, SEM-EDX, micro-tomografía computerizada, FTIR, MALDI-TOF-MS) complementan los datos históricos.

Palabras clave:

"talla dorada", estudio comparativo, técnica de dorado, materiales, estudio analítico.

Introduction

The cultural heritage of Portugal includes different typologies of decorative wooden gilded polychrome art-objects testifying a long evolution of gilding techniques namely from the late Renaissance period to Baroque epoch, roughly between 1500 and 1800. A decorative phenomenon, called "talha dourada", has been adopted and largely used, together with other artistic techniques such as "azulejos", in a form of a total work of art, as an expression of a national mark, religious faith and catechetical message vehicle [1-10]. Although subjected to intensive historical research [1-4], "talha dourada" decorations all over the Portuguese territory were not systematically investigated from the material and technical points of view, therefore many churches and museums house today altarpieces, retables and other wooden and gilded decorations, never studied. Only punctual information speaking of the conservation and restoration interventions performed on some national altarpieces with gilded decorations was reported [5-16]. Gilded decoration on wooden support has been extensively studied not only in Europe but also in other places, as in North and South America [17-21]. Therefore a comparative study with other similar objects and/or gilding layers would be necessary to increase the knowledge on these works of art.

Two important complexes of "talha" from two churches – the Chapel of Albertas in Lisbon, now part of the Museu Nacional de Arte Antiga (National Museum of Ancient Art) (Fig. 1) and the Church of Vale de Figueira nearby Santarém (Fig. 2), both carved and assembled during the 18th century, were studied through an inter-disciplinary and multi-scale approach.

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The church of Saint Albert, or of Albertas as it is normally called, was part of the extinct Discalced Carmelite Convent in Lisbon. Nowadays, it integrates the National Museum of Ancient Art (MNAA), being the only building that has survived from the former convent of St. Albert after the Lisbon’s earthquake in 1755 [11-12]. Its interior was rebuilt in a late Baroque style, (called in Portuguese “Joanino” style) during the reign of the King D. João V [1-2]. The wooden carved decoration in the lateral and main altarpieces followed a decorative program with characteristic pseudo-solomon columns, of “national” style, large windings of acanthus, phoenixes, vine leaves and grapefruits, typical of the late “Joanino” baroque style, already mixed with Rococo lambrequin and seashell motifs. This church houses one of the most beautiful and complex decoration conjugating large descriptive panels of “azulejos” and wooden carved “talha dourada” characteristic to the Portuguese baroque in a “total art” concept decoration. This ornamentation was subject to conservation, cleaning and consolidation interventions in 1982 and 1994 [12].

Another church, built during 17th-18th centuries, is S. Domingos church in Santarém (Vale de Figueira, VF), housing a smaller, but not less significant, complex of “talha dourada”. The main altarpiece as well as the side chapels altarpieces were made on the first quarter of the 18th century, and they are representative of the Portuguese “talha dourada” style, including Eucharistic throne, lateral mouldings and valances of the side doors. Before starting restoration interventions in the summer of 2009, several gilded samples were taken from this complex.

The conservation state of the gilded layers is generally good for both complexes, losses of cohesion and adhesion, in some areas and deposition of dirt and atmospheric particles are visible. The most urgent problems to be solved in the Saint Albert church are due to the lack of consistency of the plaster walls, which affects the altarpiece’s structural stability, and to the fluctuations of climatic parameters, such as relative humidity (RH) and temperature (T), registered inside the church throughout the year. The wooden ceiling and the left wall of the nave, in direct contact with the exterior, were subject to rainwater infiltration and environmental problems that caused constant detachments of gilded carved details [12].

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Figure 1. View and details of the complex of "talha dourada" from St. Albert church in Lisbon



Figure 2. View and detail of the complex of "talha dourada" from Vale de Figueira

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In the Saint Albert church all the carved, gilded and polychrome decorations, mainly in *Pinus silvestris*, sp. wood, were subject to conservation interventions on two occasions, in 1982 and in 1994. Consolidation interventions were performed using the wax-resin method (virgin wax and dammar resin in ratio of 7:2, hot melt with addition of an aromatic solvent). This treatment has the advantage to be easily implemented “in situ” and to be reactivated by the application of heat or by the use of organic solvents at any time. Another intervention campaign should be prepared and a methodology must be evaluated.

In the case of S. Domingos church, the restoration interventions in the summer of 2009 aimed to re-establish the integrity of the polychrome gilded decoration of the altar and to consolidate the original parts.

The study of these two “talha dourada” complexes aimed to:

- characterize the structure and manufacture technique of the gilding stratigraphy;
- identify the chemical composition of each layer constituting the gilded polychrome decoration (from the ground to the final finishing varnish or glaze);
- map the distribution of organic binders or varnishes in the stratigraphical structure of the polychrome composites;
- better understand the effects of a consolidation treatment using wax-resin (7:2) system applied in the case of MNAA gilded decoration.

Few analytical data on the stratigraphic structure and composition of layers in samples from the Church of Albertas were already available from a previous study [12].

The different analytical techniques proposed for this study create a novel complementary approach by using optical microscopy (OM) and scanning electron microscopy-energy dispersive X-ray spectrometry (SEM-EDX), X-ray fluorescence (micro-XRF) and X-ray micro-computerized tomography (micro-CT), Fourier Transformed Infrared micro-spectroscopy (micro-FTIR) and Matrix-Assisted Laser Desorption/Ionisation-Time of Flight Mass Spectrometry (MALDI-TOF-MS) [22-34] to fully characterize the gilded composites at different scales [26], describing main features of the analyzed samples from macroscopic to micro and molecular levels. Micro-CT [23-25] is a relatively recent technique useful for non-destructively studying the three-dimensional internal structure and texture of a polychrome sample, ranging from microscopic to macroscopic spatial scales. This can be performed after 3D reconstruction of its internal architecture enabling 2D and 3D interactive static and dynamic visualization by means of pictures and movies, respectively. An improved and faster approach to the identification of proteinaceous constituents in these samples can be done by complementing the cross-section observation under optical microscope upon the fluorescent staining [25-26] with the binder identification through MALDI-TOF-MS [30-32] and micro-FTIR [20, 22, 33-34].

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2. Sampling

To study the materiality of both gilded complexes, 7 samples from the “talha” in Lisbon (abbreviated MNAA) and 10 samples from the one in Santarém district (abbreviated VF) were taken in order to obtain cross-sections and to provide materials for spectroscopic, micro-analysis and tomographic reconstruction. The description of the micro-fragments and the analyses performed on each of them are given in the Table 1.

Table 1. Description of the samples and performed analyses

	Sample ID	Sampling area	Analyses
MUSEU NACIONAL ARTE ANTIGA	MNAA1	Nave, lateral altar decoration	MO, XRF, SEM-EDX, fluorescent stain, MALDI-TOF MS
	MNAA2	Nave, lateral altar decoration	MO, SEM-EDX, micro-CT, fluorescent stain
	MNAA3	Nave, lateral altar – left side decoration	MO, micro-CT
	MNAA4	Lateral Chapel of S. Teresa, altar decoration	MO, XRF, SEM-EDX, MALDI-TOF MS
	MNAA 5	Lateral Chapel of S. Teresa, altar decoration	MO, XRF, SEM-EDX, fluorescent stain, MALDI-TOF MS
	MNAA6	Lateral Chapel of S. Teresa	MO
	MNAA7	Nave, abgel – right side wall decoration	MO, SEM-EDX, micro-CT
CHURCH OF VALE DE FIGUEIRA	VF1	Decoration from the door of the central altar,	MO, XRF, SEM-EDX, micro-FTIR
	VF2	Upper part of the small right column – right side of the decoration of the central altar	MO
	VF3	Upper part of the base of the right column – right side of the decoration of the central altar (Tabernacle)	MO, XRF, SEM-EDX, micro-FTIR, micro-CT, fluorescent stain, MALDI-TOF MS
	VF4	Leaf of the chapitel of the right column – right side of the decoration of the central altar	MO, XRF
	VF5	Base of the right column – right side of the decoration of the central altar	MO
	VF6	Decoration of the lateral wall, left side from the altar (three-dimensional leaf decoration)	MO, XRF, micro-FTIR
	VF7	Decoration of the lateral wall, left side from the altar (flat decoration)	MO, XRF, SEM-EDX, microFTIR, micro-CT, stain MALDI-TOF MS
	VF8	Decorative column, “acantus” leaf from the upper part	MO, XRF, SEM-EDX, micro-FTIR, stain
	VF9	Decorative column, flat surface from the upper part	MO, XRF, micro-CT, MALDI-TOF MS
	VF10	Decorative column, back side	XRF, MALDI-TOF

3. Analytical protocol

The historical data was complemented with the results obtained from the analytical interdisciplinary approach, in which microscopy (OM and SEM-EDX), spectroscopy (micro-XRF; micro-FTIR), fluorescent staining tests and X-ray tomography were applied for better understanding the structure, composition and conservation state of the gilded polychrome composites. Together with the microscopic and spectroscopic analyses, the micro-CT tool provided compositional information and imaging of the structural features of polychrome layers within the samples for the assessment of the manufacturing techniques, materials and of the surface conservation state. The main advantage of this non-destructive imaging

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technique is the fact that it allows visualizing the entire volume (inside and at the surface) of a sample throughout, a great number (hundreds or thousands) of sections/slices. In particular, this visual information can be obtained slicing all the sample volume through three conveniently chosen orthogonal principal cut types: transaxial, coronal and sagittal corresponding respectively to ZZ', XX' and YY' observation directions.

The identification of organic constituents in these samples (binders, varnishes or other organic materials) was done by complementing the cross-section observation under optical microscope (without stain, before and after fluorescent stain) with MALDI-TOF-MS and micro-FTIR identification.

3.1. Optical microscopy and staining procedure on cross-sections

The cross-sections, obtained from micro-fragments of the gilded samples, were embedded in Polyester resin Mecaprex SS (Leica) and properly polished after the necessary curing time. Their observation was done using an Axioplan Zeiss 2 imaging binocular microscope (50x - 500x magnification), with both Visible and fluorescent light, and a Nikon DXM1200F digital camera. The filter blocks used for observing the fluorescence were f8 (G 365, FT 395 and LP 420) and f6 (BP 450-490, FT 510 and LP 515). Visible light observations (illumination position for dark field observation, abbreviated as f2) were performed in reflection geometry.

A non-covalent stain (Sypro Ruby, commercialized by Molecular Probes, USA) was used for fluorescence microscopic mapping of proteinaceous materials in cross-sections [28-29]. This fluorescent stain is used in the proteomics field and its advantages (nanogram sensitivity, high selectivity, low detection limit, a straightforward staining procedure and its availability as a ready-to-use solution) make it suitable for routine examination of paint samples [28-29]. A drop of the stain solution is applied directly on the surface of the cross-section and left for few seconds up to 1-2 minutes. The excess of solution is removed with absorbent paper and afterwards the sample is ready for observation under microscope, using a proper filter set (f6) with emission in green light (the positive staining has a bright orange color).

3.2. X-ray fluorescence analysis

Micro-XRF was done using a portable spectrometer (ArtTAX Pro), in the following experimental conditions: voltage 40 kV; current 600 mA; accumulation time 120 s; spatial resolution 70 mm. The measurement was performed both on micro-fragments and cross-sections.

3.3. Scanning Electron Microscopy – Energy Dispersive X-ray Spectrometry

A SEM – VEGA II LSH Scanning Electron Microscope (TESCAN - Czech Republic), coupled with an EDX – QUANTAX QX2 (ROENTEC – Germany) spectrometer was used. Quantax QX2 uses a detector of third generation Xflash, that does not need cooling with nitrogen and is 10 times faster than a traditional detector based on Si(Li). The EDX spectra have been

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acquired in the following conditions: 20KV voltage; 1×10^{-3} Pa; 5000nA; working distance 11-20 mm (16.6 mm for EDX), scanning speed: 200ns; magnification: 78x and 1000x. The cross-sections were covered with a fine layer of graphite using a specific "sputter coater".

3.4. Micro-computerized tomography

The micro-tomographical study was performed on 6 samples using a compact desktop high resolution Skyscan 1172 micro-tomograph. The analytical set-up consists of the combination of an X-ray shadow microscopic system and a computer with tomographic reconstruction and analysis software. The Skyscan 1172 contains an X-ray micro-focus tube with high-voltage power supply, a specimen stage with precision manipulator, a 2-D X-ray CCD-camera connected to the frame-grabber and a Dual Pentium computer with colour monitor.

In order to obtain high-resolution radiographs, milimetric samples are preferred. X-ray source and detector are fixed while the sample rotates around a stable vertical axis (ZZ'). Samples were scanned at a voltage of 100 kV with current intensity at 100 mA. A random movement of five with a five frame averaging was chosen to minimise noise. The basic physical parameter quantified in each pixel of a CT-shadow image (radiograph) is the linear attenuation coefficient. Beer's law relates the intensity (I) of X-ray photons passing through the object with thickness h, with the incoming intensity (I_0) and the attenuation coefficient (α) of the object.

The X-ray micro-tomograph settings and operation procedure were optimised to produce the best phase contrast images by reducing artefacts like beam hardening, ring, star and line artefacts as much as possible.

3.5. Fourier Transformed Infrared Micro-spectroscopy

A Nicolet Nexus spectrophotometer interfaced with a Continuum microscope with a MCT-A detector cooled by liquid nitrogen was used. The spatial resolution is 30 μm , the spectra being obtained with a resolution of 4 cm^{-1} and 128 scans, in transmission mode, in an interval between 4000 and 650 cm^{-1} , using a Thermo diamond anvil compression cell.

3.6. Matrix-Assisted Laser Desorption/Ionisation-Time of Flight Mass Spectrometry

Reagents - Trypsin (TPCK) from Promega Corporation, trifluoroacetic acid and 2,5-dihydroxybenzoic acid (DHB) both from Sigma, acetonitrile (p.a.) and ammonium hydrogen carbonate from Lachema Brno were used. The commercially available reverse phase ZipTip comes from Millipore Corporation, Bedford, MA, USA.

Specific cleavage with trypsin - Digestion of approximately 20 mg of each sample was carried out in 50 μL of solution containing 1 $\mu\text{g}/\mu\text{L}$ of sequencing grade trypsin (Promega) in 50

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mM NH₄HCO₃ at room temperature for 2 hours. The solution containing released peptides was desalted using ZipTips packed with reversed phase (C18) resin.

All mass spectra were acquired using a Bruker-DaltonicsBiflex IV MALDI-TOF mass spectrometer equipped with standard nitrogen laser (337 nm) in reflector mode. A 2 µl aliquot of the peptide mixture was mixed with 4 µl of DHB solution (16 mg DHB in 1 ml of a mixture of acetonitrile and 0.1% trifluoroacetic acid (1:2 v/v)). A 2-µl aliquot of the resulting solution was spotted on a stainless steel MALDI target plate and let to evaporate before analysis. At least 200 laser shots were collected for each spectrum and the data were analysed using the software package XMASS (Bruker) and the freely available open source tool mMass [30-32].

4. Results and discussion

In the Table 2, a summary of the results from the whole analytical protocol is presented.

Table 2. Results from the analyzed samples

Sample ID	Decorative part or element	Stratigraphical description (MO, Vis-UV and micro-CT)	Elements for each layer (XRF, SEM)	Inorganic component (micro-FTIR)	Organic component (micro-FTIR, MALDI-TOF-MS, fluorescent stain)	
MNA	1-3	Nave	GR	Ca, S, O, Sr	-	Animal (rabbit) glue
		OL	Si, Al, Fe, Mg, K, Ti			
		Gold leaf	Au (Ag-Cu)			
		Particulate deposit				
	4-6	Lateral Chapel of S. Teresa	GR	Ca, S, O, Sr	-	Animal (rabbit) glue
			OL	Si, Al, Fe, K, Mn, Ti		
			Gold leaf	Au (Ag-Cu)		
			Wax or other varnish?			
	7	Nave, entrance to the Chapel of the Lord of the Speech	GR	Ca, S, O	-	-
			OL	Si, Al, Fe, Mg		
			Gold leaf	Au (Ag-Cu)		
	VF	1-5	Main altarpiece	GR	Ca, S, O, Sr	Gypsum Clays
OL				Si, Al, Fe, Mg, K, Ti		
Gold leaf layers				Au (Ag-Cu)		
Wax surface layer (VF3)						
6-7		Lateral wall, decoration	GR	Ca, S, O, Sr	Gypsum Clays	Animal (rabbit) glue Waxy material (surface layer of VF7)
			Very thin OL	Si, Al, Fe, Mg, Ti		
			Gold leaf	Au (Ag-Cu)		
			Thick protective layer, not fluorescent under UV light (VF7)			
8-10		Decorative column, single element	GR	Ca, S, O, Sr	Gypsum Clays	Animal (rabbit) glue
			Two Red OL	Si, Al, Fe, Mg, K, Ti		
			Gold leaf (lamina)	Au (Ag-Cu)		
Abbreviations		MNA - <i>National Museum of Ancient Art</i> ; VF - <i>Vale de Figueira</i> ; GR - Thick white Ground; OL - Ochre Layer (bole?); Clays - Clay minerals				

4.1. Stratigraphical structure and characteristics of inorganic phases (OM, SEM-EDX, micro-CT)

Figure 3 gives the stratigraphical structure corresponding to various samples, at different magnifications. The MNAA's "talha" presents a uniform stratigraphic structure in all the 7 samples: from the bottom thick layer of ground (coherent and dense, without particular inclusions or infiltrations) to the ochre bole layer and the upper layer of gilding (Figure 3) made of an alloy-based lamina (Au/Ag/Cu). Residues of a wax-resin mixture and of atmosphere particulate are also present on the surface of the precious leaf. This data is consistent with the results presented elsewhere [12] on three samples from different areas (and belonging to different epochs) of the gilded decoration in this church. In this case the cross-section observation was used to assess the effectiveness of the wax-resin method [12]. In some cases the optical observation illustrates the impregnation of the whole sample (MNNA3 and 4, Figure 3) with the mixture used for consolidation, suggesting that in lacunar areas from the gilded decoration the mixture penetrated into the gilded composite, while in other fragments (MNAA1 and 2, Figure 3) remained on the surface of the leaf. Therefore the consolidation product assured protection and stability to the whole gilded composite.

For four samples taken from the VF complex, similar structure of layers to the "talha" from MNAA can be described: white thick ground with inclusions of variable size; layer of red-yellow ochre (bole?), gold leaf and waxy varnish (translucent in visible light).

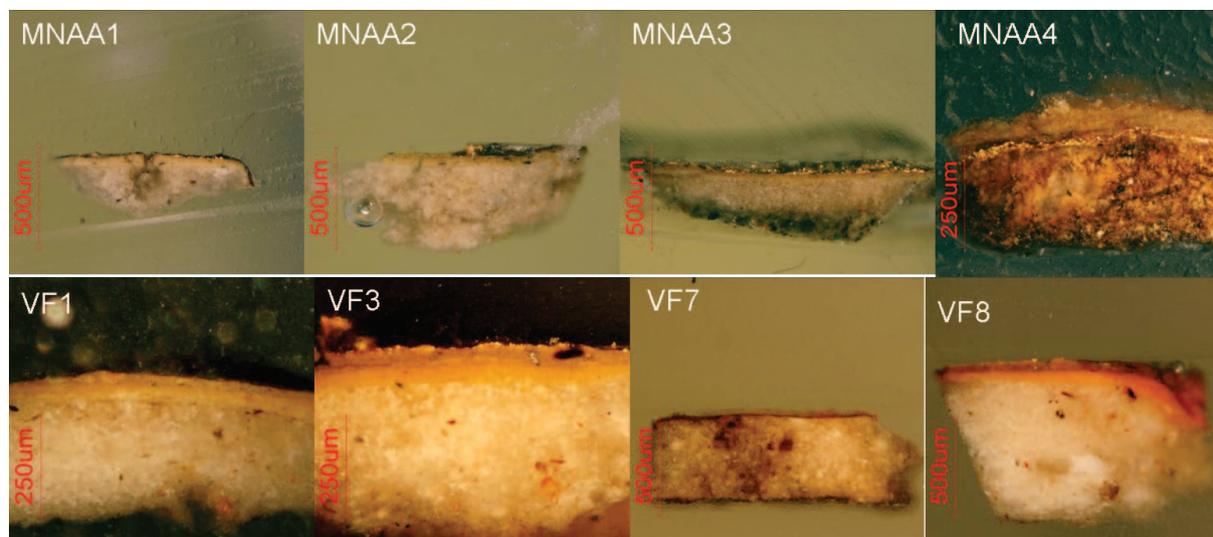


Figure 3. Selection of samples from MNAA's and VF's "talhas", observed and photographed under optical microscope, in the form of cross-sections

Figure 4 shows some examples of micro-CT slice images taken from the interior of 2 samples, VF7 and MNAA3. These images illustrate the three-dimensional structure of each sample in 3 directions (x, y, z) and the phase contrast inside the sample's architecture. The metal leaf is clearly distinguished from the non-metallic components (MNAA3) of the stratigraphical

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structure (different colours were used to better show this distinction). Holes or pores, structural discontinuity and irregular patterns, big inclusions and fractures inside the samples can be visualized with this technique and correlated with the compositional features.

In Figure 4, each color/grey scale image represents a section of the radiographic tomographic reconstruction of the inner structure of the sample: T – transaxial; S – sagittal; C – coronal. The red line in the RST image indicates the position of the transaxial section (T section). The resolution used for these images was: 4.27 $\mu\text{m}/\text{pixel}$ for MNAA3 and 2.85 $\mu\text{m}/\text{pixel}$ for VF7.

The sample from VF (VF7) displays a ground with heterogeneous porosity (big pores and also small, diffused ones) in which an interface is present between two different ground layers: the upper one, with high porosity, and the lower, thinner one, more compact.

In the case of the sample from MNAA (MNAA3), the various sections obtained through micro-CT scanning show a ground that has a relatively homogenous granulometry and sporadic porosity (big pores or holes can be detected within the structure) and with discontinuities due to fissures or small lacunae in the surface layers (indicating also a poor conservation state with a loss of cohesion between layers and also disaggregation of the particles inside the ground). This sample has an irregular shape and the T and C sections also show a separation surface within the ground layers. Small, bright inclusions (particles that generate very high beam attenuation) were also detected and were attributed to the presence of impurities. Those impurities could include iron oxides or other compounds having in their composition heavier chemical elements.

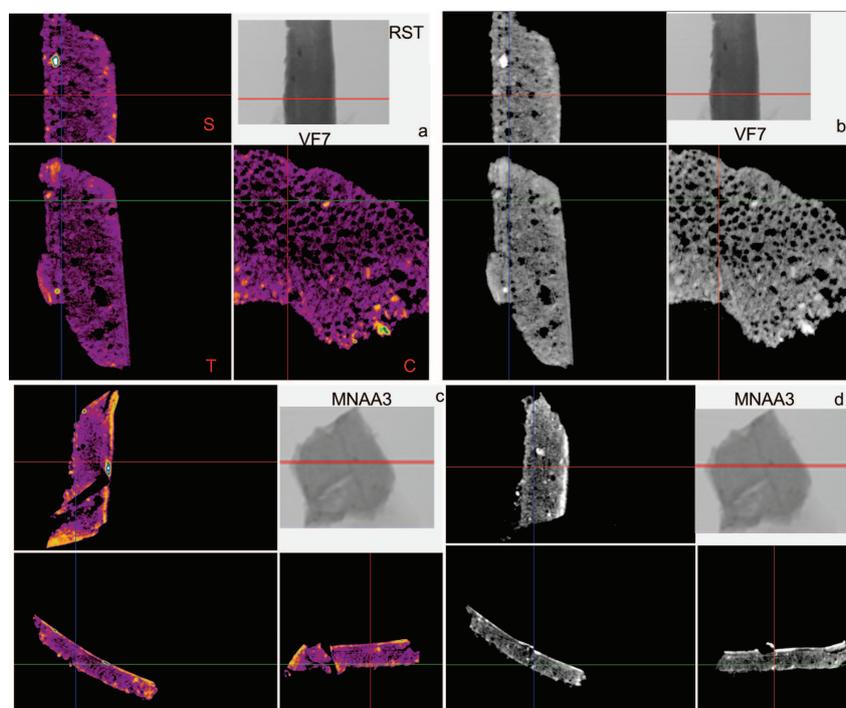


Figure 4. Micro-CT reconstructions of 2 samples: a) VF7 color images; b) VF7 grey scale images; c) MNAA3 color images, d) MNAA3 grey scale images.

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An interesting contribution given by microCT in this case is the possibility of distinguishing between the coarse layers of *gesso grosso* and finer *gesso matte*, which presence characterize the *water gilding technique* [35-37]. This dynamic observation can be complemented with the SEM imaging that also allows differentiating the distribution and orientation of gypsum grains within different layers (Figure 5, samples MNA4 and VF7-VF8). Not all the samples in this study allowed assessing the different morphology between the coarse *gesso grosso* and the fine *gesso matte*, but for some of them (as MNA7) it was possible to cross-reference the results of the two imaging techniques, SEM and microCT, thus showing where the *gesso grosso* vs. *gesso matte* distinction is more obvious (Figure 5). MicroCT images for MNA7 in Figure 5 have a resolution of 3.91 $\mu\text{m}/\text{pixel}$.

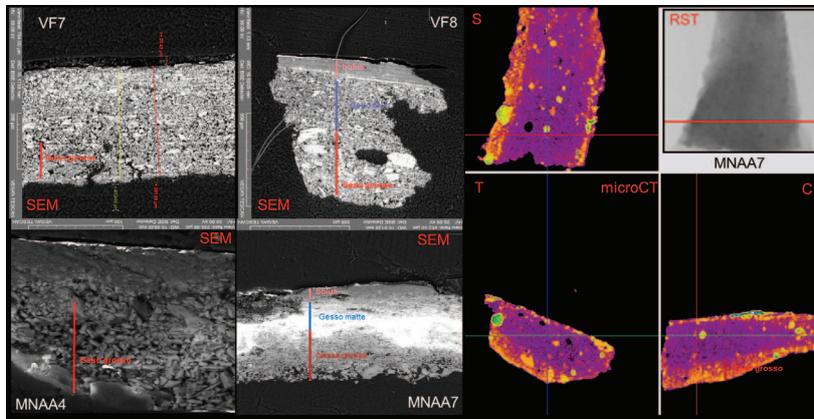


Figure 5. SEM and micro-CT imaging of samples showing the difference between *gesso grosso* and *gesso matte* layers

For the sample MNA4 (Figure 5) the SEM backscattered electrons image shows clearly different morphological and size dimension characteristics between the ground layer and the bole, that is much finer than the gesso (presenting long shaped crystals). The same distinction in shape and size between the grains of the ground and the upper ochre layer is also visible for VF samples (VF8-VF7 in Figure 5 and VF3 in Figure 6).

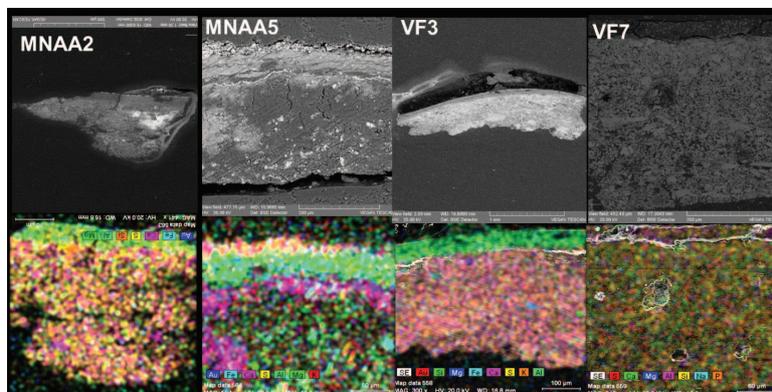


Figure 6. SEM-EDX imaging and mapping for 2 MNA samples and 2 VF samples.

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4.2. Compositional features (micro-XRF, SEM-EDX, micro-FTIR, MALDI-TOF-MS, fluorescent stain)

The elemental mapping performed by micro-XRF and SEM-EDX allowed distinguishing between the elements present in the ground layers and bole (Table 2) and also those characterizing the composition of the gold leaf (Table 3). The EDX analyses allowed to identify the presence of a gold-based alloy in all the samples (Table 3).

Table 3. Compositional identification of the gold leaf in 2 samples by SEM-EDX (weight and atomic % for each element in the metallic leaf)

Sample ID	Element	Series	[norm. wt.-%]	[norm. at.-%]	Error in %
VF8	Au	M-series	95,006	89,446	4,124
	Ag	L-series	3,352	5,762	0,173
	Cu	K-series	1,642	4,792	0,108
MNAA5	Au	M-series	86,644	74,761	4,259
	Ag	L-series	9,537	15,027	0,620
	Cu	K-series	3,819	10,213	0,428

Au/Ag/Cu leaf was detected in both cases, with a different atomic ratio between elements (Au:Ag:Cu). For the samples from MNAA (Table 3) the alloy Au:Ag:Cu = 7.5:1.5:1 has 86% (w%) of Au, corresponding to 21 carat gold. Samples from VF have a ratio of 19:1.2:1 (Table 3) and a massic composition of 95% Au, corresponding to an alloy of 23 carat gold. The use of highly gold-content alloys (87% Au) and of alloys made of Au/Ag/Cu was already reported from an extensive and comparative SEM-EDX research performed on several Portuguese polychrome reliquaries from Alcobaca, Açores, Coimbra, Aveiro, Tibães (16th century) [3, 26] and in other studies on retables from Coimbra (Cathedral’s main altarpiece, dated from 1502 to 1685) and Porto, dated from 17th-18th centuries [5-8] where 22-23 carat gold is reported (applied as water or mordant based techniques, the stratigraphy being characterized by the presence of ground, red bole layer and gold leaf). Our results confirm the extensive use of such alloys during 17th and 18th centuries up to 19th century in Portugal.

Although the analytical procedures can indicate the quantitative composition and the quality of the alloy, measuring the thickness of the gold leaf when laid in “talha dourada” structure, is extremely difficult. For MNAA samples the thickness of the leaf is approximately 5 mm and 10 mm for VF samples. Can this indicate that the gold beaters were more skilled and able laminating the thin gold leaf in a central town like Lisbon than in smaller communities like Santarém? It is known that the gold leaf quality and thickness depended not only of the astonishing malleability of the metal, but also of the subtle qualities and compressibility characteristics of the surface of the sheet material between which gold was confined during the beating. Although Natália Marinho Alves and other authors [4, 35] speaks of good quality of the gilding work, giving information about artists, materials and gilding technical

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procedures, there are a lot of technical data to be confirmed by extended studies (hopefully to be obtained within the Gilt-Teller project)[38].

The presence of elements such as Ca (Sr), O, S, is attributed to the ground made of calcium sulphates (e.g. gypsum). The micro-FTIR analysis of VF7 (Figure 7b) confirmed the presence of gypsum (3408 cm^{-1} – H_2O ; 1621 cm^{-1} – H-O-H; 1117.68 and 672 cm^{-1} - SO_4^{2-}) and probably clay minerals from the ochre upper layer (peaks detected in the interval $3700\text{-}3600\text{ cm}^{-1}$ attributed to the water; $970\text{-}1050$ and 915 cm^{-1} attributed to the Kaolinite-Illite group of phyllosilicates - Figure 7b)[34].

Kaolinite was detected together with gypsum in the composition of bole layers in the three samples coming from gilded decoration in the church of MNAA and analyzed in 2009 [12]. In our study all the bole layers analyzed display a high content of Al and Si, corresponding to silicate minerals. Other elements identified by XRF and SEM-EDX (such as Fe, Mg, K, Ti) are typical of the bole layers, attributed to the clay minerals and (Fe, Ti, Mn)-oxides.

Figures 8 and 9 illustrate the results of the microscopic mapping with the fluorescent stain on cross-sections and comparison with auto-fluorescence (images taken under UV light). The Sypro mapping (Figure 9) shows a diffused distribution of proteinaceous material in the ground and bole layers but in case of samples from MNAA (especially MNAA5) the stain's colour is altered because of the wax-resin impregnation. The varnish coating in the VF7 sample is not fluorescent under UV light and has similar appearance as the sample VF3 (Figure 7), in which the micro-FTIR identified the wax (2917.71 and 2849.56 cm^{-1} – CH str; 1736.46 cm^{-1} – C=O str; 1463.26 cm^{-1} – CH def; 1172.19 cm^{-1} – C-O; 720 cm^{-1} – C-C str) as surface layer (Figure 7a). The FTIR spectrum of VF7 (Figure 7b) identified only few peaks that could be attributed to an organic material (2923.40 and 2851.22 cm^{-1} for CH_2 stretching) [33-34], while the absorption band around 1540 cm^{-1} (1536.73 cm^{-1} - VF7 spectrum) could indicate the presence of a proteinaceous binder in the ground layers, successively confirmed by the results of MALDI-TOF-MS (Figure 10). The MALDI-TOF mass spectrometric analysis identified the presence of animal collagen (rabbit glue) in samples MNAA1, 4 and 5; VF3, 7, 9 and 10 (Figure 10). The peaks specific for the collagen in animal glues are labelled in the mass spectra. The peaks uniquely belonging to rabbit glue are in the following m/z values: 1261, 1263, 1442, 1709, and 1831.

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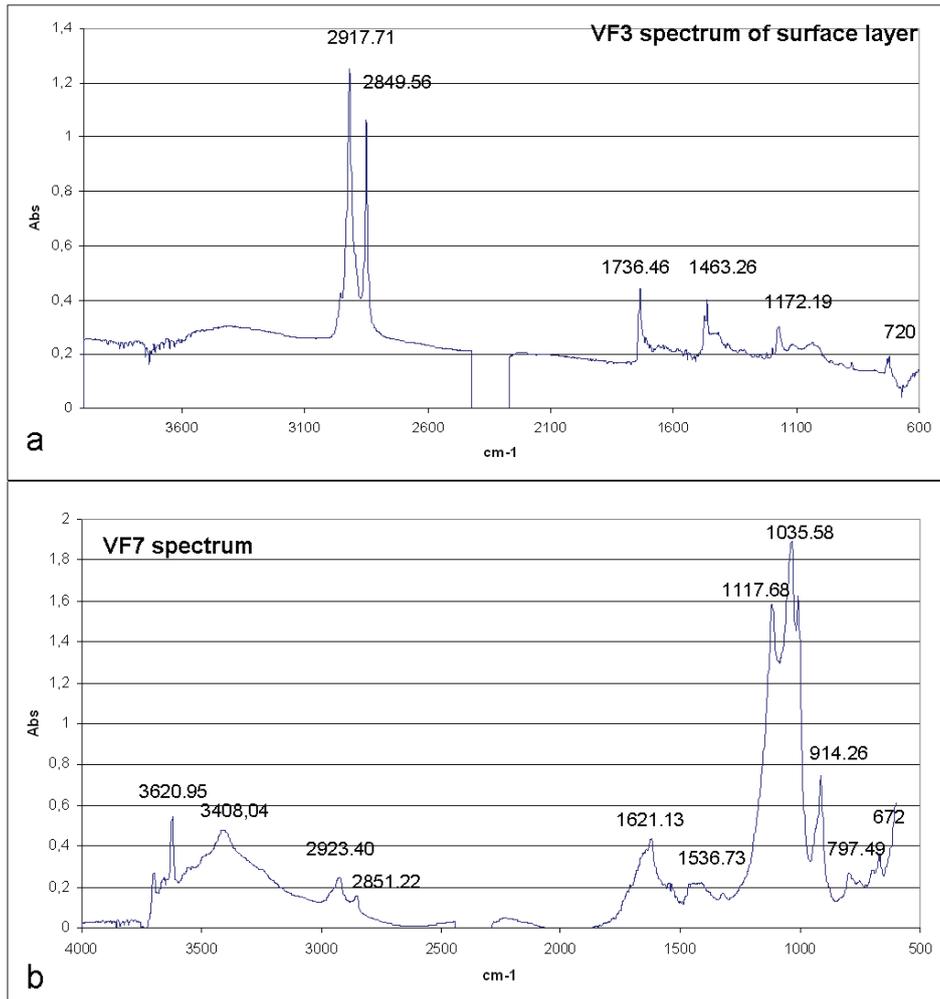


Figure 7. Micro-FTIR spectra of two samples from VF "talha":
a) protective surface layer from VF3; b) polychrome layers with residues of ground from VF7

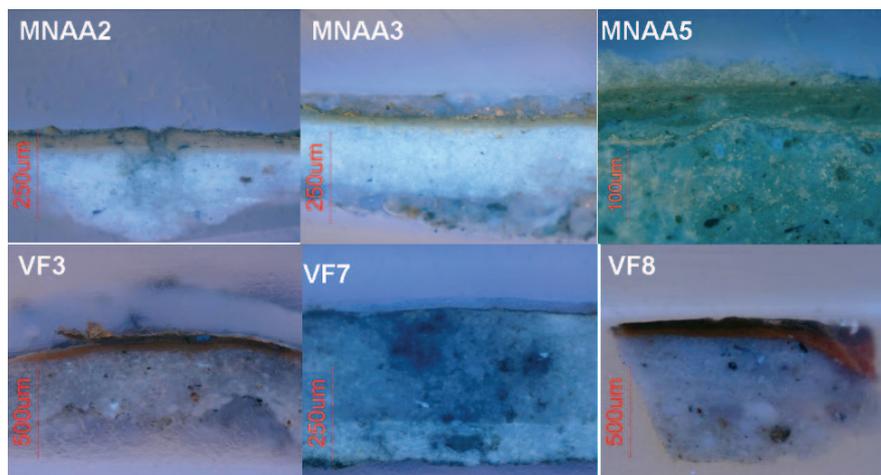


Figure 8. Fluorescent imaging (auto-fluorescence upon UV excitation)
of samples of "talha dourada" (3 from MNA A and 3 from VF)

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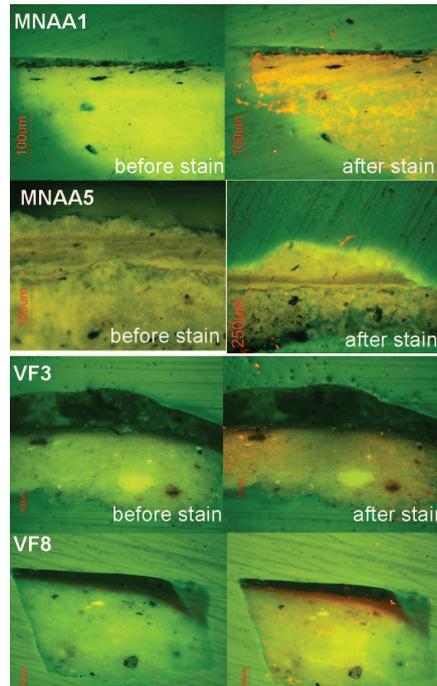


Figure 9. Fluorescent imaging (induced fluorescence upon Sypro staining) of samples of “talha dourada” (2 from MNA and 2 from VF)

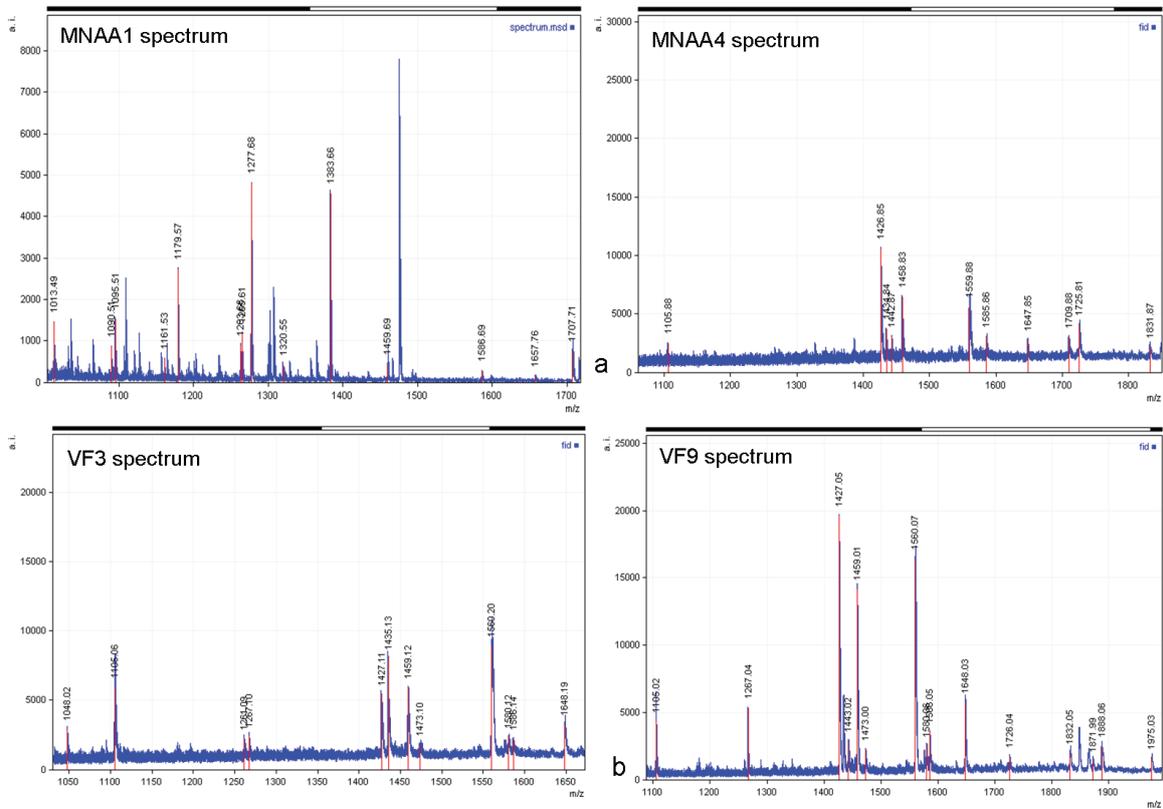


Figure 10. MALDI-TOF-MS spectra of: a) 2 samples from MNA “talha”; b) 2 samples of VF “talha”. The red peaks belong to the animal glue.

4.3. Conservation state and past interventions (OM, micro-CT)

In MNAA5 sample (Figures 3 and 8) the image of the ground has a dark and greasy aspect. This can be due to impregnation treatment residues with a wax-resin mixture, performed in the past [12]. The light yellow coloration in the layer subjacent to the metal lamina was probably due to the impregnation with an oily/resinous material. This was confirmed by sample analysis done on areas of water gilding technique but contaminated with an oily material not original but belonging to surface layers [12].

The various reconstructed slices obtained through micro-CT scanning, contributed to characterizing the MNAA3 sample's conservation state. These images display discontinuities in the entire composite structure, from the surface layer to the ground, due to fissures or small lacunae open in the surface layers. The micro-CT technique enables visualizing the porosity distribution and homogeneity in the samples. This is useful to be correlated to the characteristics of the materials and the application procedure of these materials. Normally the white ground preparation was applied in several layers, initially the *gesso grosso* (coarser and with vermicular shaped crystals), thoroughly sanded between applications and afterwards the *gesso matte*, much finer and less porous. The separating interface between layers, with half cut crystals, can be detected with the OM and micro-CT observation.

Some of the samples from VF (VF3 and VF7, Figures 3 and 8) have a protective thick layer of wax (with a grey fluorescence pattern), identified by micro-FTIR spectroscopy (Figure 7). The presence of the wax as a varnish can be also associated with some not qualified intervention of cleaning and re-varnishing in case of the sample VF4 (from decorative columns of the altar). The liturgical use of wax candles would lead to localized presence of the wax but the analyzed samples did not allow differentiating between a generalized and a localized presence of this coating material.

5. Conclusions

The proposed integrated multi-technique protocol to investigate the two gilded complexes enabled to ascertain the similarities and differences in the structure, materials and techniques of both "talha dourada" samples. In this respect, it was found that the stratigraphical structure of the gilding layers is quite similar: thick ground with *gesso grosso* and *gesso matte* differentiation, yellow-reddish layers of bole, metallic alloy leaf, varnish or particulate deposits or residues of the "wax-resin" treatments (for the complex in MNAA). The overall composition of ground and bole layers in the both complexes is very similar as well: gypsum and animal glue in the grounds; clay minerals and (Fe, Ti)-oxides mixed with rabbit glue in the bole. Water gilding technique can be thus identified, the gold leaf being burnished on the surface to increase the brilliant appearance by light reflection.

As far as the composition of the leaf is concerned, the analysis detected the presence of a relatively thick leaf (5 µm for MNAA and 10 µm for VF respectively) made of an alloy Au/Ag/

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Cu in different ratios, with a relatively high content of Au (21 carat gold for MNAA samples and 23 carat gold for VF samples) confirming data already reported in the literature about “gilding” materials on the territory of Portugal. The thickness of the leaf is slightly variable and this can point on a different quality of the gilding layer application between the church in Lisbon and the rural one or better skills to laminate the gold leaf (in this respect economic issues can be considered as reason for the use of a thicker leaf for the second case against the thinner leaf in the first case). The organic materials that were successfully identified are the animal glue in the preparative layers (ground and bole) and coatings based on tri-glyceridic materials (wax) in case of samples from Vale de Figueira church (it seems there was a local practice for varnishing the gold leaf with wax).

The micro-architecture investigated through micro-CT and SEM techniques showed different characteristics for the two complexes: MNAA samples display a ground with a relatively homogenous granulometry and sporadic porosity (big pores or holes can be detected within the structure) and with discontinuities due to fissures or small lacunae in the surface layers. The samples from VF (especially VF7) display a ground with heterogeneous porosity (big pores and also small, diffused ones), the gold leaf is continuous (sample VF3) and adherent to the substrate (bole layers). SEM images for both stratigraphical structures show clearly different morphological and size dimension characteristics between the ground layer and the bole, which are much thinner than the gypsum (presenting long shaped slender crystals).

The conservation state of the polychrome gilded layers is generally good, no major alterations or degradations could be observed on the surface or at the interfaces between layers in the stratigraphical structures. The conclusions of the restorer about the effectiveness of the wax-resin consolidation methodology applied on “talha” from MNAA, was that the surfaces are greatly favoured by this treatment, the preparation and gilded layers being nourished, consolidated and integrated, the final aspect becoming denser and with colour depth, and were confirmed by the analytical data.

The novel, complementary approach to the study of these two complexes showed a high potential for diagnosis and mapping enabling to better assess multi-scale and multi-level information on the compositional, micro-structural and conservative aspects of these important assets from the Portuguese cultural heritage.

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**A comparative interdisciplinary study of gilding techniques and materials
in two Portuguese Baroque “talha dourada” complexes**

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