

## TWO RECIPES FROM PORTUGUESE TRADITION OF GILDING ON WOODEN SUPPORT BETWEEN LABORATORY REPRODUCTION AND ANALYTICAL INVESTIGATION

Irina Crina Anca SANDU<sup>1\*</sup>, Francesca PABA<sup>2</sup>, Elsa MURTA<sup>3</sup>, Manuel Francisco COSTA PEREIRA<sup>4</sup>, Luis DIAS<sup>1</sup>, José MIRÃO<sup>1</sup>, António Estevão GRANDE CANDEIAS<sup>1</sup>

<sup>1</sup> Evora University/HERCULES Laboratory/Palacio de Vimioso, Largo Marques de Marialva, 8, 7000-809 Ev ora, Portugal

<sup>2</sup> NOVA University/FCT, Caparica Campus, 2829-516 Caparica, Portugal

<sup>3</sup> Laboratório José Figueiredo (LJF), Direcção Geral do Património Cultural (DGPC), Rua das Janelas Verdes, Lisboa, Portugal

<sup>4</sup> Centre for Natural Resources and Environment (CERENA), Instituto Superior Técnico, Universidade de Lisboa, 1049-001 Lisboa, Portugal

### Abstract

*This paper has the main purpose to compile and highlight the first data obtained from experimental studies on documented reconstructions of gilded composites performed within a research project on gilding materials and techniques in Portugal ([www.gilt-teller.pt](http://www.gilt-teller.pt)) funded by FCT. Two water gilding recipes were appropriately chosen from the treatises written by the Portuguese Filipe Nunes (1615) and José Lopes Baptista de Almada (1749) as being representative for Baroque époque. Based on these recipes, the production of raw materials - "gesso grosso", "gesso fino", bole, animal glue (from lamb and goat skins) - was made as faithful as possible. Their application was then performed in laboratory following the indications given by these authors or by treatises of previous époques (e.g. the Bolognese treatise for thawing leather), on plane and curved wooden supports (pine and oak species) using three types of leaf: gold (Au/Ag/Cu) of 22 and 23.75 karat respectively and silver. After the completion of gilding, the samples' surfaces were divided into areas and on each different finishing layers (wax, animal glue size) and decorations (punching, "esgrafitado", "estofado") were applied. An analytical campaign using optical microscopy (OM) and scanning electron microscopy (SEM) on surfaces and cross sections, X-ray diffraction (XRD), micro-computerized tomography (microCT) and colorimetry (CIEL\*a\*b\*) was undertaken in order to characterize the gilded composites and to assess the faithfulness of the reproductions in the laboratory. Correlations between the information given by the recipes and the composition and stratigraphical patterns of the reconstructions can be established. Furthermore, the study aims to highlight the difficulties encountered in analyzing real samples and comparing the results with those from reproductions as the number and types of layered materials are not always reproducible. A critical approach is needed and criteria for faithful reproduction of ancient recipes are suggested.*

**Keywords:** Water gilding; Recipes; Reconstructions; Treatises; Cross sections

### Introduction

This paper presents the results of an experimental study undertaken during a research project, entitled *GILT-Teller: an interdisciplinary multi-scale study of gilding techniques and materials in Portugal, 1500-1800 (PTDC/EAT-EAT/116700/2010)* funded by the Portuguese Foundation for Science and Technology [1]. The reproduction in laboratory of documented reconstructions of gilding recipes was one of the tasks developed within this project aimed to

\* Corresponding author: [irinasandu@uevora.pt](mailto:irinasandu@uevora.pt)

compare the model composites thus obtained with real samples taken from altarpieces and sculptures from the Portuguese territory during the project's development. Among an important number of historical and contractual documents consulted in archives and specialty libraries in Portugal [2-3], two treatises were chosen for the selection of the recipes of gilding [4]. The treatise written by Filipe Nunes, was published for the first time in 1615 [5], while the one written by José Lopes de Baptista Almada, was published in 1749 [6] (Figure 1). Two recipes were chosen from each treatise as being representative for the gilding tradition in Baroque époque both in water and mordant gilding techniques.



Fig. 1. The cover pages of the two treatises

The results to be presented here will only deal with reconstructions on plane wooden supports of the water gilding recipes from the two authors N.B (Nunes, burnished) and A.B.(Almada, burnished) (Figure 2), and their comparison with real samples from altarpieces and sculptures studied in the project.

Ninety six model samples were done using two different wooden species (a soft one, pine - P and a hard wood, oak - C), two different skin glues (goat - CC and sheep - CB), three types of commercial leaf (gold, Au, of 22 and 23.75 karat and silver, Ag). After the completion of gilding, the samples' surfaces were divided into areas and on each different finishing layers (wax, animal size) and decorations (punching, "esgrafitado", "estofado") were applied (Figure 3a and 3b).

For each composite a duplicate was done, labeled as "a" for natural ageing and "b" for artificial ageing (e.g. 1P-CC-NB-Au22a and 2P-CC-NB-Au22b).

The preparation and application of animal glues, gesso grounds and bole layers was explained in a previous publication [4], therefore the present one will give an overview on the model samples and the procedures followed and will further show to which extent the reconstructions are comparable with real samples with gilding layers. The Gilt-Teller project disposes of a database of samples (more than 450) and analytical results from three different typologies of artifacts with gilding layers: altarpieces, polychrome sculptures and choir stalls dated between 1500 and 1800. Therefore few examples of real samples from the database were used and compared to the similar ones from the model composites.

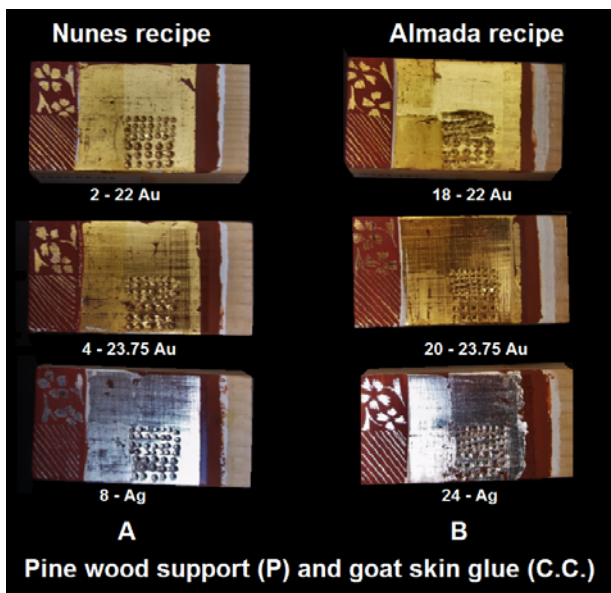


Fig. 2. Two sets of model samples: A) gold and silver leaf composites made after the recipes from Nunes treatise; B) gold and silver leaf composites made after the recipes from Almada’s treatise

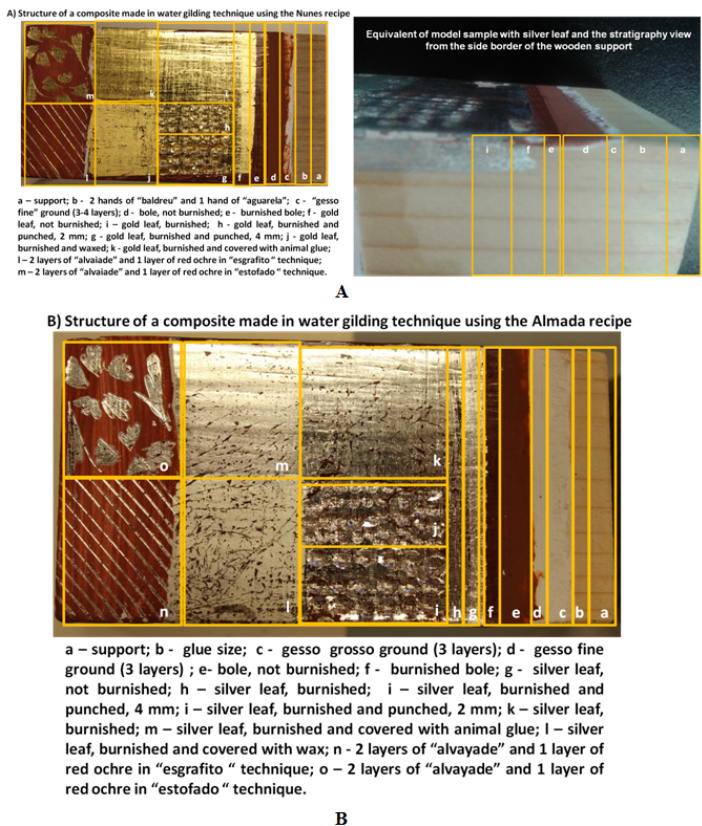


Fig. 3. General scheme of the reconstructions: A) gold leaf of 22 karats applied according the recipe by Nunes; B) silver leaf applied according the recipe by Almada

## Experimental

A multi-technique analytical campaign using optical microscopy (OM) and scanning electron microscopy (SEM) on surfaces and cross sections, X-ray diffraction (XRD), micro-computerized tomography (microCT) and colorimetry (CIE L\*a\*b\*) was undertaken in order to characterize the gilded composites and to assess the faithfulness of the reproductions in the laboratory. The analytical investigations carried out on the model samples could be a valuable aid in the study of real historical samples as for them we know exactly what we are analyzing from the point of view of the materials, way of applications and number of layers according to the chosen recipes. Thus, the main aspects to be considered for this study were:

- observation of samples under stereomicroscope (SM) to assess the importance of a complete sample for stratigraphic analysis and the rational choice of sampling positions;
- surface and cross section observation under optical microscope (OM, Vis and UV) and SEM for characterization of heterogeneity, particle size and shape of crystals/particles in grounds and bole layers, the observation of the layers (the number of layers and differentiation between *gesso grosso* and *gesso fino*, burnished and not burnished bole, burnished and not burnished leaf), the distinction between the number of layers applied in materials of the same composition (gesso and bole), the comparison of the thickness between model and real samples (preparation, bole and gold leaf);
- observation and characterization of decorative techniques such as *estofado*, *esgrafitado* or *punçonado* using microscopy and microCT;
- observation and comparison of the fluorescence pattern of surface layers under optical UV microscopy, and comparative characterization of surfaces state between model and real samples;
- characterization of interfaces between support and gesso ground, between gesso and bole and between bole and leaf using OM and SEM BSE observation at different magnifications on surfaces and cross-sections.

### Materials and procedures of application

Figures 4 and 5 show the main materials used for creating the laboratory reconstructions according the two water gilding recipes and the way they were applied. The website of the project also contains a fully documented video (<http://www.gilt-teller.pt/index.php/site/historicalReconstructions>) on the making process of the reconstruction from the preparation of the raw materials (skins, gesses, bole etc.) to the final application of surface finishing decorations (*estofado* and *esgrafitado* or punching, *punçonado*, with two different sized punches – 2 and 4 mm) and varnishes.

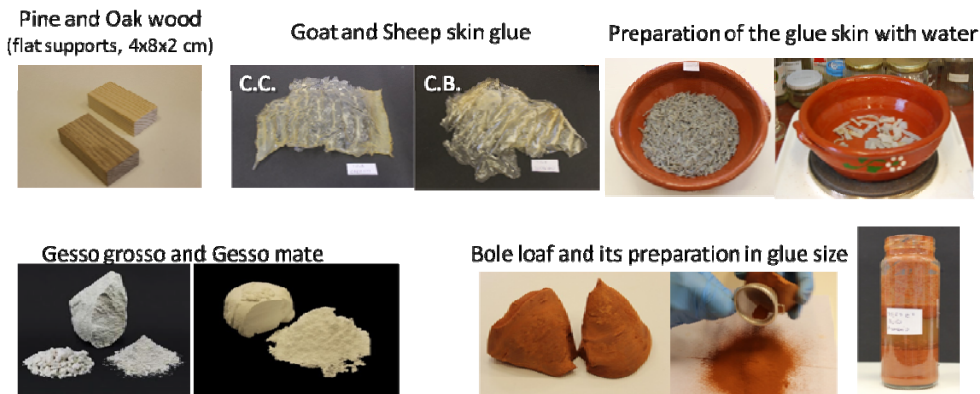
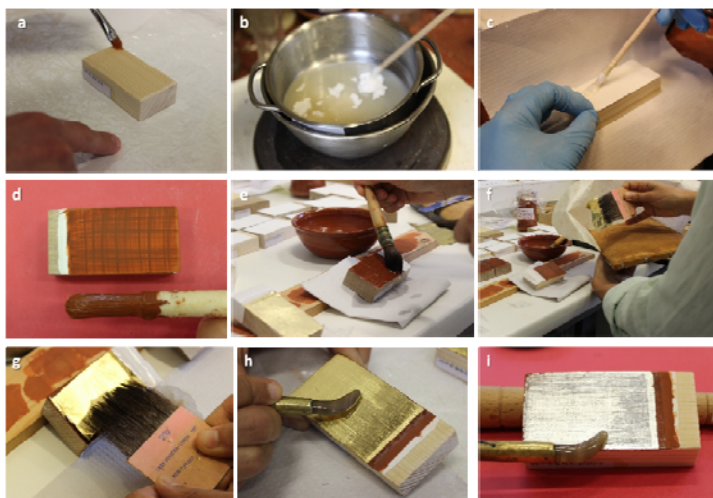


Fig. 4. Main materials used for the gilded and silver composites



**Fig. 5.** Main steps of the laboratory reconstruction of gilded and silvered composites: a) sizing of the support with glue skin size; b) mixing the glue size and gesso for the ground; c) ground application; d) bole application in perpendicular layers; e) water activation of the glue from the bole layer; f) manipulation of the leaf prior application on the bole; g) application of the leaf over the bole; h) burnishing of the gold leaf; i) burnishing of the silver leaf.

### *Experimental conditions*

Cross-sections were obtained using an Epofix embedding resin with hardener, dry polished with successively finer grades of Micro-mesh abrasive cloths (500, 800, 1200, 2400 and 4000 mesh). A felt was used for the final polishing. For the better optical microscopy observation a drop of glycerol was applied and then a glass lid over the surface of the cross-section.

Optical microscopy (OM) images were taken from the surfaces and cross-sections at different magnifications (from  $100\times$  to  $500\times$ ) using a LEICA DM2500M stereozoom microscope, coupled to a digital camera. Visual light observations (illumination position for dark field observation, position 1) were performed in reflection geometry and for the fluorescence observation an UV block filters was used (position 3).

A BRUKER X'PERT-PRO diffractometer was used in the following experimental conditions: Cu source ( $1.54\text{\AA}$ ), generator Settings - 35 mA and 40 kV, scanning  $5-80^\circ 2\theta$ , 0.05 step size  $^\circ 2\theta$  and scan step time 50 s. X'PERT PLUS software with a PDF4 data base have been used to analyze the diffraction patterns.

The micro-tomographical study was performed using an high resolution Skyscan 1172 (now Bruker) microtomograph and a computer with tomographic reconstruction (NRecon) and visualization (CTvox) 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. Small samples were scanned with constant power (10 W) and variable voltage (máx 100 kV) and current intensity (máx 100  $\mu\text{A}$ ) in order to enhance phase's contrast. 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 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 (I) of the object.

A HITACHI S3700N interfaced with a QUANTAX EDS microanalysis system was also used to obtain the backscattering images, point analysis and chemical mapping data. The QUANTAX system was equipped with a BRUKER AXS 5010XFlash® Silicon Drift Detector

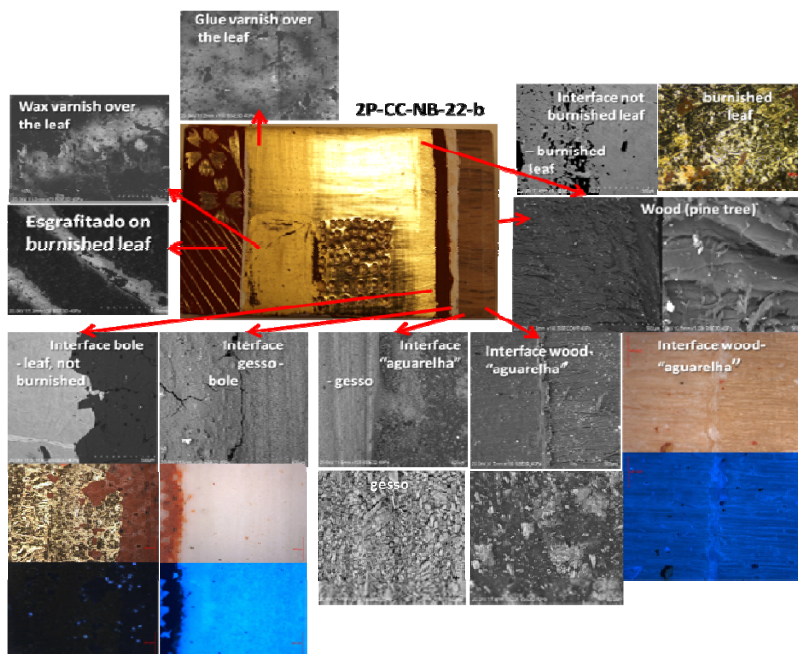
(129 eV Spectral Resolution at FWHM/Mn  $K\alpha$ ). The operating conditions for EDS analysis were as follows: backscattered electron mode (BSEM), 20 kV accelerating voltage, 10 mm working distance. The cross-sections were previously coated with graphite while the surface analysis was carried out directly on model samples, having a fine carbon bead crossing over the width of the surface.

The colorimetry was performed with a Datacolor Check II Plus colorimeter with head opening of 3 mm and illumination geometry D65/10°, for 3 consecutive measurements the average values of  $L^*$ ,  $a^*$ ,  $b^*$ , C, h being reported.

### Results and Discussion

When using fragments from model samples is much easier to take a complete stratigraphic sequence (from support to surface layers) than for a real sample. The incomplete sequence that we often encounter in real samples means a loss of information on probable layers inside the stratigraphic pattern, such as presence or not of glue sizing over support, thickness and types of gesso grounds (when more than one type was used). Therefore many of the samples we analyzed in this paper include wooden support (as whole section or as a fragment) but this is not a common situation found in real samples. Another advantage the model samples present against the real ones is the fact that we can retrieve a larger number from the same areas and larger also in size.

The multi scale observation of the surfaces for each layer of materials applied according the 2 recipes combined the color imaging using optical microscopy under visible and UV light with grey scale imaging of SEM-EDS at different magnifications. Figures 6 and 7 show an example for the gilded composite made according the Nunes recipe of water gilding with gold leaf of 22 karats. The figures show optical images differentiating the appearance and fluorescence answer of a leaf when burnished versus un-burnished surface and of the leaf with glue and wax coatings versus the un-coated leaf.



**Fig. 6.** Details of a model sample made according the water gilding recipe from Nunes treatise with interfaces between various layers of the composite observed under SEM and OM - Vis and UV

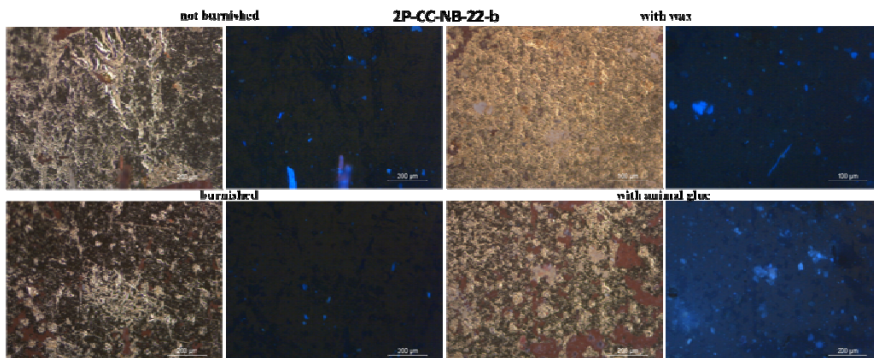


Fig. 7. Details of the surfaces from the gilded sample from Fig 6 observed under optical microscope, Vis and UV radiation, at 100x

The burnishing treatment over the surface created some roughness and losses of the leaf, showing the layers of red bole and sometimes the white ground beneath it. Therefore the fluorescence that sometimes can be observed punctually for the burnished leaf is due to the underlying substrate (bole mainly) containing a certain amount of glue. The leaf itself is not fluorescent but when coated with animal glue, that is known to be an organic auto-fluorescent material under UV, will acquire a certain degree of bluish fluorescence color. The wax also has no or only a limited fluorescence but its presence can be detected in many real cases as a coating or as a residue from consolidation treatments (the so called wax-resin method) or from dropping of the candles used to illuminate the churches [7-8].

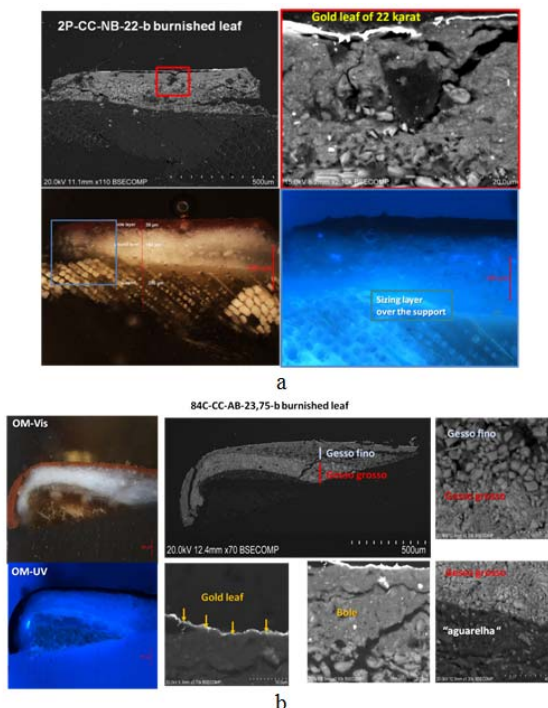
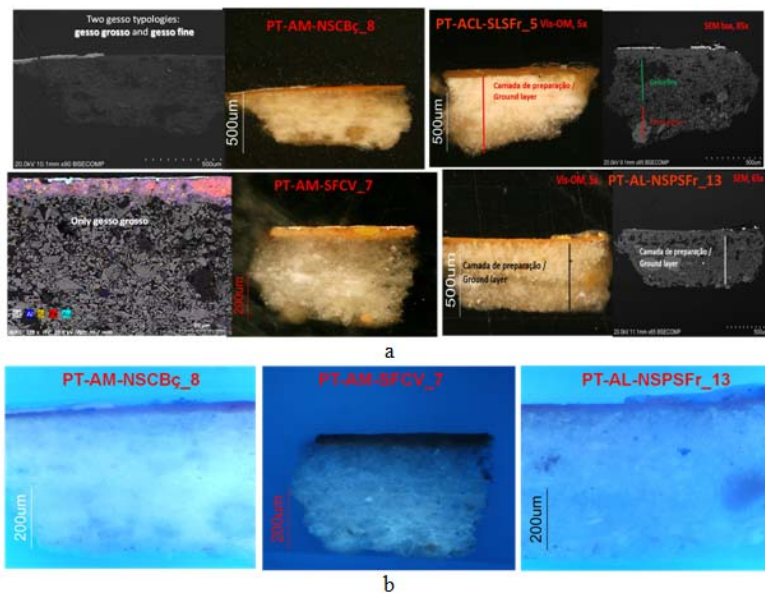


Fig. 8. Comparison between the stratigraphical structure and sequence of layers for two model samples made with a) Nunes recipe of water gilding and 22 karat gold leaf; b) Almada recipe of water gilding and 23.75 gold leaf

As sizing layer or in mixture with a little bit of gesso to be applied as *aguarelha* over the wooden support, the glue can also be visualized due to its fluorescence in a cross-section, such it is the case of Figure 8. The same figure also shows the stratigraphy for the Almada's recipe of water gilded composite, where the difference between *gesso grosso* and *gesso fino* layers is obvious in the SEM BSE images. The thicknesses of gilding layers are comparable: the 3-4 layers of *gesso matte* in the Nunes recipe have between 75 and 200  $\mu\text{m}$ , while the 3 layers *gesso grosso* + 3 layers *gesso matte* in the Almada recipe are around 150-200  $\mu\text{m}$ . The bole layers have also similar thickness (30-40  $\mu\text{m}$ ) in both types of composites, being made of 4 layers each. The gold leaf is not easy to measure very precisely but for burnished areas around 1-3  $\mu\text{m}$  could be assessed by OM (at 100-200x). The SEM measurement at higher magnifications (32.000x) showed a smaller thickness of around 90-100 nm, therefore the SEM result can be considered more accurate than the optical microscope assessment.

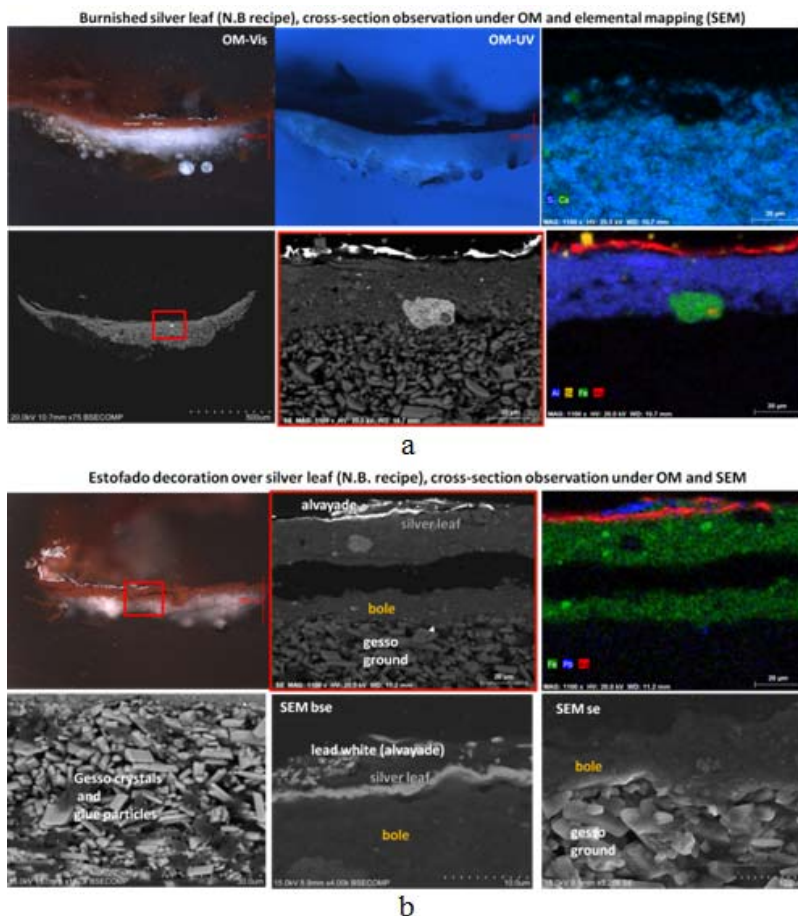
In the case of real samples (Figure 9) one or two types of gesso can also be found, but the thickness of the whole ground is higher than for the composites (around 400-500 and even up to 1000  $\mu\text{m}$ ) suggesting more applications than the 4 or 6 as the recipes recommend (probably twice the number of hands, 8 or even 10-12) [9-10]. The bole layers seem to have a more similar thickness in real samples than for the model ones. It is not possible though to distinguish between the several applications of gesso in the ground or of the bole hands. Sometimes the OM-Vis images seem to reveal such a distinction between layers but the SEM image of the same cross-section will only indicate such a differentiation only if the size and shape of the grains are visibly different (Figures 8 and 9). At higher magnifications under SEM BSE the interfaces between ground and bole and between bole and leaf can be better observed and there is no visible distinctions between the goat or sheep skin glues, as both tend to form small agglomerates around the gesso crystals (Figure 9). Both glues as sizing layers or in mixture with gesso display a bluish fluorescence under UV which is common to animal skin glues [11-12] as shown in Figure 10 (cross sections a and b). The color and intensity of this fluorescence is similar to the one observed in cross-sections of real samples (Figures 9, and 10).



**Fig. 9.** Gesso grounds in: a) 4 real samples (*gesso grosso* and *gesso fine* and only one type of gesso) observed under visible light at the OM and under bse at SEM; b) 3 of the 4 samples, observed under UV



The SEM-EDS (Figures 10 and 11) and XRD results (Figure 11) on grounds from the model samples show a slightly different composition from a gesso type to another, generally gypsum was identified for *gesso fino* and bassanite (hemi-hydrated sulphate) for *gesso grosso*, coarser than the previous gesso. The long acicular shape of the *gesso fino crystals* is confirmed by the SEM images and the identification of its composition (gypsum or di-hydrated calcium sulphate) together with bassanite by XRD on powdered samples (Figure 11).

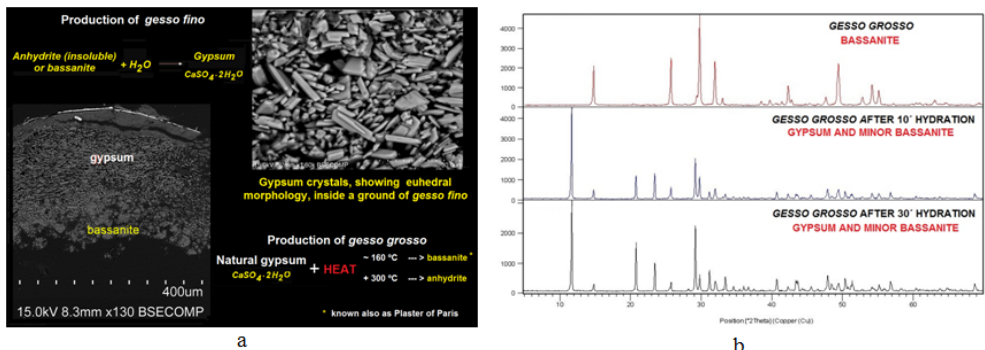


**Fig. 10.** Two fragments from the water gilded composite with silver leaf made according Nunes recipe, comparison of stratigraphic pattern and sequence of layers for: burnished leaf sample and b) for an *estofado* decoration sample.

Apparently the procedure used for the preparation and application of *gesso grosso* is not reproducing faithfully what the generalized practice assess, because after several months after its application in mixture with glue an incomplete hydration occurred, leading to the formation of a hemi-hydrated state, bassanite.

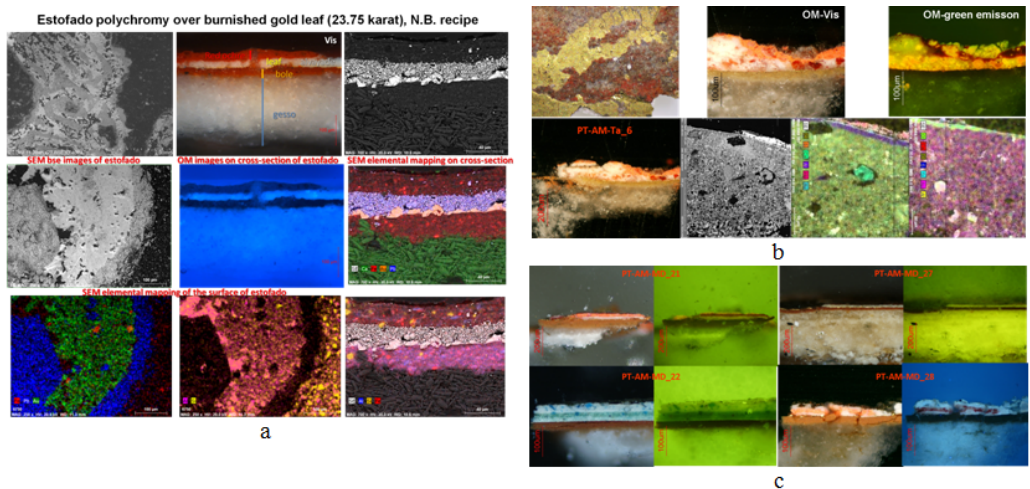
The *gesso grosso* would have been expected to be anhydrite as the process of obtaining it from raw materials [4] indicates a complete transformation of the natural gypsum during heating at around 300 °C to insoluble anhydrite (Figure 11). Bassanite, the hemi-hydrated version of the calcium sulphate, was identified instead, meaning that during the process of mixture and heating at 160°C with glue this material will still hydrate in the presence of water. The product was kept in laboratory conditions during several months and then hydrated during 30 minutes and a great part of it turned into gypsum. The assay of hydration for only 10

minutes shows intermediary results (Figure 11b). When mixed with glue the process of hydration is not as effective (therefore this could be the explanation for the occurrence of bassanite in real cases also) as for gesso powder and water, this one occurring very rapidly.



**Fig. 11.** SEM and XRD results on gesses in model samples: a) processes leading to *gesso grosso* and *gesso fino* and the appearance of the two types of gesso in cross-section, b) diffractograms of *gesso grosso* after different intervals of hydration

The analyses performed on real samples revealed a different situation, as *gesso grosso* is generally made of coarse anhydrite while the *gesso fino* is gypsum. But there are also few cases where the hemi-hydrated compound was identified, as the scientific literature reports the presence of bassanite in mixture with anhydrite in the *gesso* layers from paintings and sculptures [13].



**Fig. 12.** Comparison of “estofado” decorations for: a) model sample executed with the recipe of Nunes, observed on the surface and also in cross-section; b) real sample from the altarpiece of Tancos church (Santarem) in cross-section; c) several real samples with polychromy from the garments in the altarpiece from Miranda de Douro Cathedral in cross-sections

The real samples with polychromy over the gold leaf display a large variety of sequences in layers and compositions, but some of them are quite similar to the sequence and pattern of layers in the composite made according the Nunes or Almada recipes of *estofado* or *esgrafitado* over burnished leaf (Figure 12). The use of lead white (*alvayade* in the original recipe) is extensive as white layer but also in mixture with other pigments for fleshs and garments of Saints and other characters incorporated in the structure of the altarpieces or as

individual statues [7-10]. Nevertheless, it is not common to find in the real samples two layers of lead white as the recipe recommends.

The 3D microstructure of the model samples, the distribution and dimensional parameters of the ground, bole and lead bearing layers in the *estofado* or *esgrafito* areas were also investigated using microCT [14]. The gold or silver layers are almost “transparent” due their reduced thickness. Mechanical features related with the applied gilding techniques are very expressive in the 3D images.

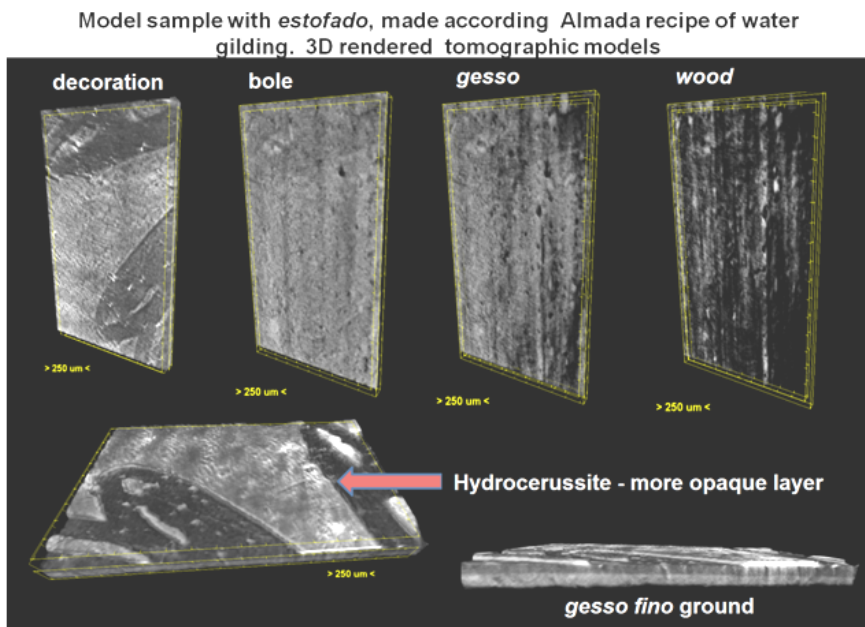


Fig. 13. Tomographic 3D rendering of an area with *estofado* from model sample made according the recipe of Almada (burnished gold leaf).

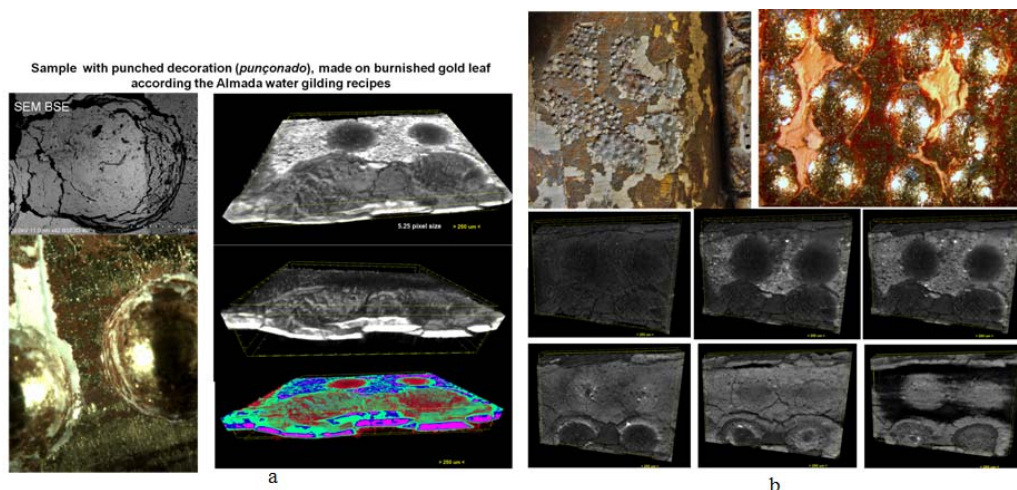


Fig.14. 3D microCT rendering of punched work (*punçonado*) made on: a) model sample according the recipe by Almada for water gilding technique; b) real sample with punched decoration from St. Joseph Sculpture (16<sup>th</sup> century, Aveiro Museum)

The micro-tomographic technique proved useful in creating 3D rendering models of model samples, sequencing in slices the layers made of different materials, as it is the case of an *estofado* (Figure 13) or a punched work area (Figure 14). In the later case, the comparison with a real surface from historical samples with punched marks (a polychrome sculpture of Saint Joseph from Aveiro Museum is given as example) [15] shows similarities although some dimensional differences were observed in the followed protocol.

Table 1 and Figure 15 give the colorimetry data for different surfaces for three sets of model samples: 2-4-8 (Nunes recipe using gold and silver leaf and goat glue over pine wood), 18-20-24 (Almada recipe using gold and silver leaf and goat glue over pine wood) and 82-84-88 (Almada recipe using gold and silver leaf and goat glue, over oak wood).

It is obvious that for the same parameter, leaf and surface, the numbers range around the same value. Values for the sets made using the Almada recipe (two types of gesso, same type of glue and leaves) seem to be similar or very close to the set made using the Nunes recipe (only one type of gesso, same type of glue and leaves). Generally, the distinction can be made between the gold and silver leaf (e.g. higher values for L\* in the case of silver leaf than for gold leaf; lower values for a\*, b\* and C\* for silver leaf than for gold leaf), although the “h” (hue) does not change so much between a leaf and another (there should nevertheless be noticed that silver has slightly higher “h” values than the other types of leaf).

**Table 1.** Colorimetric parameters (L\*, a\*, b\*, C\* and h) for the model samples surfaces

Sample ID	L*	a*	b*	C*	h
2_fb	65,63	6,46	28,76	29,48	77,34
18_fb	58,16	5,82	27,42	28,03	78,02
82_fb	61,70	6,32	28,98	29,66	77,70
4_fb	62,12	5,82	28,26	28,85	78,37
20_fb	55,53	6,77	24,60	25,51	74,62
84_fb	42,88	5,77	23,12	23,83	75,98
8_fb	100,94	1,16	5,78	5,89	78,68
24_fb	98,97	0,78	6,16	6,21	82,77
88_fb	83,88	1,03	6,47	6,55	80,93
2_g	66,90	7,96	35,94	36,81	77,51
18_g	68,42	8,35	35,21	36,19	76,67
82_g	66,03	8,35	36,26	37,21	77,03
4_g	64,40	7,99	34,53	35,44	76,98
20_g	62,15	8,31	29,99	31,12	74,50
84_g	60,11	8,43	31,29	32,41	74,93
8_g	98,32	0,88	5,00	5,07	80,01
24_g	95,59	1,51	7,19	7,53	78,14
88_g	86,47	0,96	5,65	5,73	80,39
2_w	78,75	9,65	39,16	40,33	76,15
18_w	77,86	9,50	37,85	39,02	75,91
82_w	79,86	9,22	38,17	39,27	76,42
4_w	74,98	8,64	37,12	38,11	76,90
20_w	77,90	9,25	37,68	38,80	76,21
84_w	74,09	9,39	34,35	35,61	74,72
8_w	96,67	0,71	4,83	4,88	81,65
24_w	93,71	1,22	6,59	6,70	79,51
88_w	91,61	1,04	7,14	7,22	81,71

**Legend:** fb = burnished leaf, without coating;  
g = animal glue coating; w = wax coating

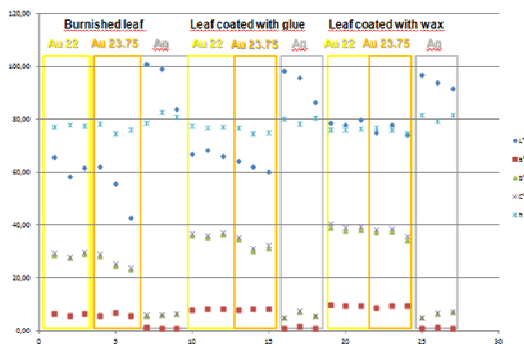


Fig. 15. Colorimetric parameters according to the type of leaf (if gold or silver and its purity) and the surface finishing (only burnished but not coated, coated with glue and coated with wax)

## Conclusions

From the study of model samples and their comparison with real samples from altarpieces and sculptures with gilding layers, the following conclusions can be drawn:

- in both cases the difference is discernible between the layers of *gesso grosso* and *gesso fino* (*gesso matte*);

- the compositional analysis showed the presence of bassanite in the *gesso grosso* meaning that during the preparation and application with animal glue a hydration process occurred, while the gypsum was identified for *gesso fino* layers; this situation is more rarely encountered in real cases, where anhydrite is the main component for *gesso grosso* (although bassanite can be also found in few cases), this can be justified with the low temperature (160°C) of the oven;

- the number of applications (*hands*) cannot be distinguished in the layers of the same material (such as *gesso* and *bole* applications);

- in general, the ground layer in real cases is always of greater thickness especially regarding the thickness of the preparation of altarpieces;

- the thickness of the *bole* is comparable with the *bole* present in the real cases, but its thickness can be very variable (5-40 microns) and the color can also vary (from yellow ochre to red and orange) in real samples;

- the thickness of the gold leaf is comparable with real cases, varying from 1 to 3 microns when measured with OM, while SEM allows a more accurate measurement, around 90-100 nm of thickness;

- the colorimetric parameters of the leaves show a good distinction between the gold and silver ones and also between different types of surface finishings (generally the burnished, not coated leaf has lower values for all parameters,  $L^*$ ,  $a^*$ ,  $b^*$ ,  $C^*$ ,  $h$ );

- for the technique of *estofado* it is rare to encounter the two layers of lead white described in the recipe but similar patterns and sequence of layers can be found in polychrome sculptures (e.g garments);

- the comparison between model samples and real ones in the 3D rendering made with microCT allowed the validation of the experimental protocols and could help in the prediction

of decaying processes in historical objects. Some limitations concerning this methodology are related to the major differences of the X-Ray attenuation in the materials used in the polychrome layering. The wooden tissues are relatively transparent and some layers are quite opaque. In this way, rendering 3D visualization of the complete sample is a difficult task. Apart this limitation, micro-CT can be a starting point for further studies of technical and technological procedures, relevant to conservation of art objects, since it is a non destructive methodology.

The study should be completed after artificial ageing which will be performed in order to have a better understanding on how the materials used for the laboratory reproductions age in accelerated conditions and if this type of ageing can be compared with the natural ageing.

This research was also meant to illustrate to which extent the model sample composites are useful for a comprehensive study of real samples and for retrieving information on their materials, techniques and eventually faithfulness in following the recipe recommendations.

The main advantage of model samples is that the number, size and completeness in terms of layers sequence and integrity are bigger than in the case of real samples. The main drawback of them is the fact that not always the degree of reproducibility of the recipe can match the real situations, where deviations from the treatises recommendations or different rules can be found and applied.

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