# TRAVELLING BENEATH THE GOLD SURFACE – PART I: STUDY AND CHARACTERIZATION OF LABORATORY RECONSTRUCTIONS OF PORTUGUESE SEVENTEENTH AND EIGHTEENTH CENTURIES GROUND AND BOLE LAYERS

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# Travelling Beneath the Gold Surface – Part I: study and characterization of laboratory reconstructions of Portuguese seventeenth and eighteenth centuries ground and bole layers

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**ABSTRACT**

*This paper is the first part from an experimental study on documented reconstructions of gilded composites performed within a research project on gilding materials and techniques in Portugal between 1500 and 1800 (PTDC/EAT-EAT/116700/2010). This study deals with the various aspects related with the choice and preparation of raw materials used to produce gesso and bole layers, the imprecise terminology that the historical recipes and treatises provide, the different steps to be followed in the laboratory experiments and their validation with analytical techniques. The results refer solely to the water gilding technique reconstructions on flat wooden samples, focusing on the preparation and application of ground and bole layers. This is important to show how the practical work performed in the laboratory can be complemented with analytical evidences obtained from different analytical techniques such as optical microscopy, scanning electron microscopy and X-ray diffraction. This can be a starting point for further studies of technical and technological procedures, relevant to conservation studies. Correlating the terminology and literature information with the practical data and critically approaching the different sources, both historical and analytical, is the basic rule for the correct reproduction of ancient recipes with modern materials and techniques.*

## 1. Introduction

Gilding technical procedures and materials were described since early times in the arts and crafts literature and they were used quite extensively until today in the decorative arts that involved carving and sculpting in different materials such as wood, ceramic, glass, and metal [1].

Between history and conservation practice, the art technological sources field proposes many recipes, reconstruction procedures and the criteria that should guide the laboratorial work [2]. In most cases, analytical expertise is required to assess whether the reconstructions are faithful to the original, and if the indicated parameters in terms of materials, procedures and steps to be followed were respected [2-3].

The main questions that often arise when someone wishes to put into practice the ancient gilding recipes are whether the materials we have access to in modern times have the same quality, properties and behavior as the ancient ones, and to which extent we are able to really interpret and reproduce the old recipes when terms and procedures are not always clearly explained. Are we sure to use the same glue and gesso as the ancient craftsmen to prepare the gesso grounds? Is the animal glue (and which kind: sheep, goat or rabbit?) we will use of the same quality as the ancient one? Is the *gesso grosso* or *gesso mate* properly prepared and the layers clearly distinguishable in the stratigraphic composite? Which kind of leaf should we use considering that literature confirms the general use of 23 and 24 karats of ternary alloys of gold with other metals (silver

and copper)? Which kind of surface (protective and/or decorative) treatment is more appropriate: varnishing with shellac or other type of varnish; *fosco* (mate) effect obtained by the application of an animal glue layer, or *sgrafito* or *estofado* decoration using tempera paint?

Without aiming to answer all these questions, this paper presents the initial results of experimental procedures conducted during the research project *GILT-Teller: um estudo interdisciplinar multi-escala das técnicas e dos materiais de douramento em Portugal, 1500-1800* (PTDC/EAT-EAT/116700/2010) funded by the Portuguese Foundation for Science and Technology [4].

One of the tasks developed within this project is to characterize with documented reconstructions gilded wood carved altarpieces and sculptures from the Portuguese baroque epoch. Among an important number of historical and contractual documents consulted in parish archives and in regional and the National Library in Portugal [5] two treatises, one written by Philippe Nunes (1615) [6] and other by José Lopes de Baptista Almada (1749) [7], were chosen for their gilding recipes (Table I). Two recipes were chosen from each treatise as being representative from the 17$^{th}$ and 18$^{th}$ centuries, both in water and mordant gilding techniques.

The reproduction in the laboratory of these recipes was focused on obtaining the raw materials for the gesso grounds, bole and animal glue layers and on their application on wooden samples. Digital photography and video recording documented the procedures.

The main criteria for the choice of wood species was the shape and number of reconstructions, the number of recipes to be reproduced and the more relevant literature for the timespan considered as the most productive and representative for the Portuguese gilded wood carved decoration (*talha dourada*). The final choices were pine and oak wood. The other criterion was the availability, behavior and characteristics of the raw materials for the aimed reconstructions and for the further accelerated ageing. The choice was gesso and two different animal glues. The added value of this study was the behavior information of the different gilding layers/materials when applied on flat and 3D surfaces. The results here presented refer only to the water gilding technique reconstructions on flat wooden surface samples (Figure 1), focusing mainly on the ground and bole layers preparation and its application.

It should be underlined that all procedures are very subjective and depend on the artist experience. Although subjected to previous known recipes, it is the experience and sensibility of the artist that controls all procedure of glue, *gesso grosso* and *fino* making and the exact ratio of solubility of glue in the water or of gypsum on the diluted glue.

## 2. Recipes and Materials

### *2.1. The Recipes and Their Terminology*

Table I presents the two water gilding recipes recommended by Philippe Nunes and José Lopes Baptista de Almada.

The *gesso* terminology is quite confusing because many ancient publications use it as a generic term for *grounds* and do not indicate its precise composition, whether is an anhydrite, a dihydrated calcium sulfate (gypsum), hemihydrated calcium sulfate (known also as *plaster/gesso of Paris*, made of bassanite) or calcium carbonate. In the latter case, we found the Portuguese term *cré*,

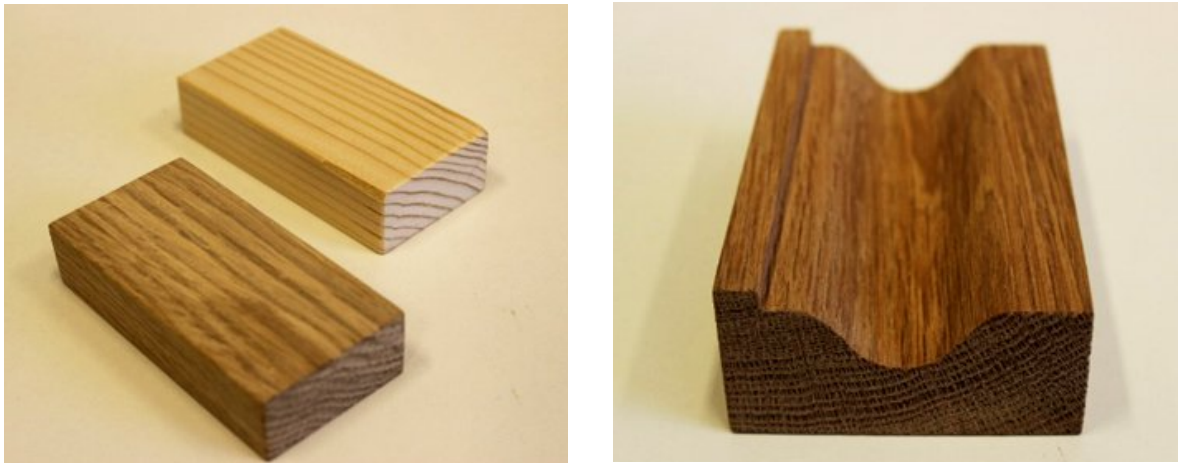

*Figure 1. Wooden supports typologies: flat (left) 4x8x2 cm and three-dimensional (right) 8x12x4 cm.*

which is different from gesso. The term *gesso-cré*, although without reference to its composition, was found in the account book *Livros de contas do Mosteiro de Tibães*, dated after 1760, from a Monastery in northern Portugal that kept most of its coeval documentation [8]. What is obvious from the literature is that in the southern European countries the tradition of plaster panels or wooden sculptures made use of gypsum/anhydrite while the northern countries made use of calcium carbonate ground [9]. The analytical results obtained from a series of Baroque altarpieces in Portugal showed that sometimes calcium carbonate can be found mixed with the gypsum in the ground layer [10]. Another interesting data on the composition of the gesso grounds is the identification of *celestite* (strontium sulfate) in Portuguese retables [11] and the fact that the gypsum source was traced back to the mines of Óbidos (Leiria) and Soure (Coimbra). Other provenances for the gesso used in Portugal are also mentioned such as Morocco, Spain and France [12-13].

The distinction between *gesso grosso* and *gesso fino*, *mate* or *sottile* is common to different countries and periods both for painting and sculpture making [14-16]. Translations sometimes introduced errors in denominating the materials or layers.

The analytical research made by several authors [9-10, 17-19] confirmed that *gesso grosso* is mainly made of anhydrite. Other studies show that a mixture of anhydrite with hemihydrate or even dihydrate sulfate can be found in grounds from paintings and sculptures [20]. The use of analytical techniques is required as to provide a precise composition of the different layers of *gesso*. Optical microscopy and scanning electron microscopy coupled with energy dispersive spectrometry (SEM-EDS), which is able to map the elements on each layer of a stratigraphy, are often used in these studies but it is not enough to assess which of the types of *gesso* is present. Figure 2 shows a ground made of calcium, sulfur and oxygen which means that a calcium sulfate is present but other techniques, such as X-ray diffraction or micro-Raman, are needed to assess if anhydrite, hemihydrate or dihydrate calcium sulfate.

The *gesso fino* denomination is linked to the material characteristics and its production, much more expensive than *gesso grosso*, is mainly constituted of fine lamellar crystals of dihydrated calcium sulfate. The *gesso grosso*, made of coarse, heterogeneous crystals, is applied on several layers on top of the wood to reduce the irregularities of the support and is sanded with sandpaper sheets

*Table I. The two water gilding and alum thawing recipes in their original language and translated into English.*

| Author Year | Short description according the original text | Main steps to be reproduced |
|---|---|---|
| **Philippe Nunes, 1615** | **Pera assentar ouro em pedra, pao e vidro, e couro**<br><br>Pera asentar ouro em pedra, se ha de guardar a ordem seguinte. Primeiramente se ha de **imprimar**, e depois de seca a imprimadura se lhe ha de pôr o mordente e como estiver em cezão, dourar.<br>O pao se doura de dous modos: a hum delles chamão **ouro mate**, como he o que fica assima dito, e que assi serve também no pao como na pedra, e o outro se chama ouro burnido. O ouro mate se acenta sobre o pao aparelhado como dizemos na pintura até ser imprimada, e depois se lhe põe o mordente, e quando está já quasi seco se lhe acenta o ouro com algodão. E se quiserdes fazer hum ouro muito fermoso que pareça ouro burnido, fazey que o mordente seja pulimento de Ocre claro, ou escuro, e depois de estar muito polido e lizo (que nisto está sayr o ouro bom) depois de enxuto lhe acentay o ouro que ficará muito fermoso, e tão bom como se fora burnido.<br>O **ouro burnido** se faz assi. Depois de estar o pao encolado lhe day uma mão de gesso comum, e seja ao modo de lavadura delgado, e se na cola lhe botardes huma cabeça de alhos serve para que não falte, depois lhe day tres ou quatro mãos de gesso mate, o qual se faz assi. Tomase o gesso comum, e depois de moydo e peneirado se bota em huma panella chea de agoa clara, e cada dia se lhe muda e se bate duas ou três vezes, e aos dez dias fica gesso mate então o tiray e sequay, e uzay delle. Depois de dardes estas mãos que digo, lhe dareis duas de **bollo comum**, e depois outras duas de **bollo fino**, e sejão todas estas mãos dadas com cola quente, depois de enxuto quando quereis dourar molhareis muito bem, e sobre o molhado com agoa clara acentay o ouro, e depois de seco burni com o bornidor, que se faz de **pederneira** muito lizo e ficará o ouro muito fermoso. (...) | - 2 hands of "baldreu" glue (to be added with a head of garlic);<br>- 1 hand of "aguarelha" (diluted animal size);<br>- 3 or 4 layers of *gesso mate* (fine) (without *gesso grosso*);<br>- 2 layers of common bole;<br>- 2 layers of fine bole;<br>- apply the gold leaf in water gilding. |
| **José Lopes de Baptista Almada, 1749** | ***Do modo de dourar brunido em madeyra***<br>Em primeyro lugar se tomará huma vazilha, ou panella nova de barro, e se encherá de retalhos de pelles de luvas, e logo se tirarão para fora, e se lançarão em hum alguidar, no qual se hão de lavar em tres agoas, tirando-lhe huma, e lançando-lhe outra: estando muyto bem lavadas, se tornarão a lançar dentro da mesma panella, e se encherá de agoa, pondo-se a ferver, até se desfazerem todos os retalhos, mexendo-os para isso muyto bem, e depois tirada para fóra, se coará por hum peneyro em huma vazilha limpa, onde se deyxará até que se coalhe. Desta colha coalhada, depois de fria, se tirará a que estiver por cima, e se guardará em huma vazilha para a tempera do bolo armenio, e a seguinte, antes de chegar ao fundo, em outra vazilha tambem para se fazer o gesso mate, de que abayxo trataremos; e da ultima se fará o ***Gesso grosso***. Lançando huma pouca em huma panella (conforme a obra que se houver de dourar) e se porá ao fogo a aquecer muyto bem, e então se lhe lançará hum pouco de gesso, desorte que a agoa, ou colla fique muyto delgada, e com huma brocha, molhada nesta colla quente, se dará huma mão sobre a madeira, que se quizer dourar, toda por igual, e depois de estar muyto bem enxuta, se porá outra vez a panella ao lume, e se lhe lançará mais gesso, desorte que fique mays grossa a agoa, ou colla, que a primeyra, e estando quente, della com a mesma brocha se dará segunda mão sobre a pimeyra. Estando enxuta a segunda, se lhe dará terceyra na mesma fórma, pondo terceyra vez a panella ao lume, misturando-lhe mais gesso, ou colla, se for necessario, advertindo que estas collas hão de dar-se sobre a madeyra de tal sorte estendidas, e puxadas com a brocha, que não fiquem mais grossas em humas partes do que em outras; porque sendo assim ficarão encobrindo o mais miudo do entalhado, ou outra qualquer cousa. Dadas estas sobreditas mãos de colla, se continuará com o **Gesso mate**. Preparando-o na forma seguinte. Tomar-se-ha huma pouca de segunda colla, que acima mandamos guardar, e posta em huma panella aos lume a aquecer, [mas pouco] se lhe irão lançando as pedras de gesso mate, e dentro della desfazendo-as com a mão, (porque com esta se desfazem melhor que com a espatola, ou qualquer outra cousa) e depois de estarem as sobreditas pedras desfeytas, e em consistencia da segunda mão do gesso grosso, se coará por hum peneyro em hum alguidar, onde se deyxará esfriar, e depois se cortará em talhadas com huma faca, das quaes se lançarãoas que forem necessarias em huma panellinha nova, e se porá ao fogo até estarem derretidas, e então com huma brocha se dará huma mão na obra, sobre a terceyra do gesso grosso, e depois de enxuta muyto bem, se lhe dará segunda, e terceyra, e dada esta, depois de enxuta, e muyto bem secca, se alizará com huma pelle de lixa, e então se entrará a fazer a *Tempera do bolo*... | **"preparation" of glue (sizing of the wooden support)**<br><br>- 3 layers of *gesso grosso* (1st one of "aguada");<br>- 3 layers of *gesso fino*;<br>- 4 layers of Armenian bole;<br>- apply the watergilding technique. |

*(cont.)*

| | | |
|---|---|---|
| **Unkown author, *ca* 1400** | ***Concia p(er) una pelle***<br><br>*Havve alume de rocho i(n) polvere onc(e) 2. doi ova b(e)n(e) dibatutj poi tollj uno bono pugno de farina cio e el fiore et tanto sale q(uan)to bastasse p(er) i(n)salare una libra de carne et tanto olio q(uan)to condisse una menestra et una bona foglieta daq(ua) calda et mecti i(n) la dic(t)a aq(ua) p(ri)ma lo alu mj b(e)n(e) s(u)btilj poi la farina et poi el sale et miscola b(e)n(e) poi mectice lio va et lolio et mista b(e)n(e) et qua(n)do laq(ua) e calda mecti dentro la pelle depilata et manegiala b(e)n(e) et strocila b(e)n(e) traendola et remitendola i(n) la dic(ta) aqua calda poi p(re)mila et remene la i(n) la dic(ta) aq(ua) calda poi lassala stare p(er) una nocte o 4. hore alma(n)cho poi la tra fora senza astirarla et polla asciugare et remenala b(e)n(e) a la stroppa poi la pumicia da luno lato et laltro e de fact(o).* | - two ounces of alum in powder;<br>- two eggs;<br>- a handfull of flour;<br>- salt as much as the neces-sary to salt a Libra of meat and the quantity of olive oil enough to spice a soup;<br>- half liter of boiled and hot water;<br>- alum very well grinded;<br>- flour;<br>- mix well and add the eggs and oil. When the water is hot immerge the skins without the fur, mix well and then remove them and put into hot water. The skins should stay immersed at least 1 night or four hours. At the end remove it and leave them dry on a stick. Polish using pumice stone on both sides. |

of different grading between each application. The *gesso fino*, made of finer and more homogenous crystals, is applied over the layers of *gesso grosso*, and is also sandpapered between each application. This smoother layers improve the surface over which the bole and gold leaf has to be applied [9, 18, 21]. Figure 3 shows an example of ground layers made of *gesso grosso/ gesso fino* sequence.

The bole (or bollus) is the red or yellow ochre layer applied over the ground and sometimes burnished with agate stone, to receive the gold leaf. Originally, the ancient gilders made use of Armenian Bole, named as such according to its provenience although more recently local iron oxide mixed with argillous/clay minerals (iron, aluminum and silicon are usually detected in the bole layers, Figure 2) were used to obtain the bole, even though the ancient denomination was maintained, as it is the case of the recipe given by Almada. Dark bole is also mentioned, mainly for applying the silver leaf.

The animal glue terminology is also imprecise and confusing regarding the raw material used to prepare the sizing for the panel and the binder solution for the gesso ground. Parchment and glove leather glues were used since ancient times until their production ceased in the 20$^{th}$ century. Nunes (1615) recommend the exclusive use of gloves leather for sizing, priming and gilding [6]. Many other European treatises mention the term "skin glue" obtained from thawed animal skins (goat, sheep/lamb, cow or pig), parchment or "glove clippings" [8, 22-23]. It is very probable that scraps of different thawed skins, used to make gloves, were recycled for this purpose. Nowadays, it is common to consider that animal skin is obtained from rabbit skin or parchment but these cannot be considered the only probable raw materials for the production of animal glue in ancient times.

A recent research [8] shows the different terminology used by 17$^{th}$-18$^{th}$ century documents (treatises and work contracts or daily account books) in Portugal for different types of glues and qualities. The cheapest glue was probably made by carpenters and cabinet makers from cheaper cuts, cartilage, bone and nerves, while the higher quality glue was named "gloves glue" or

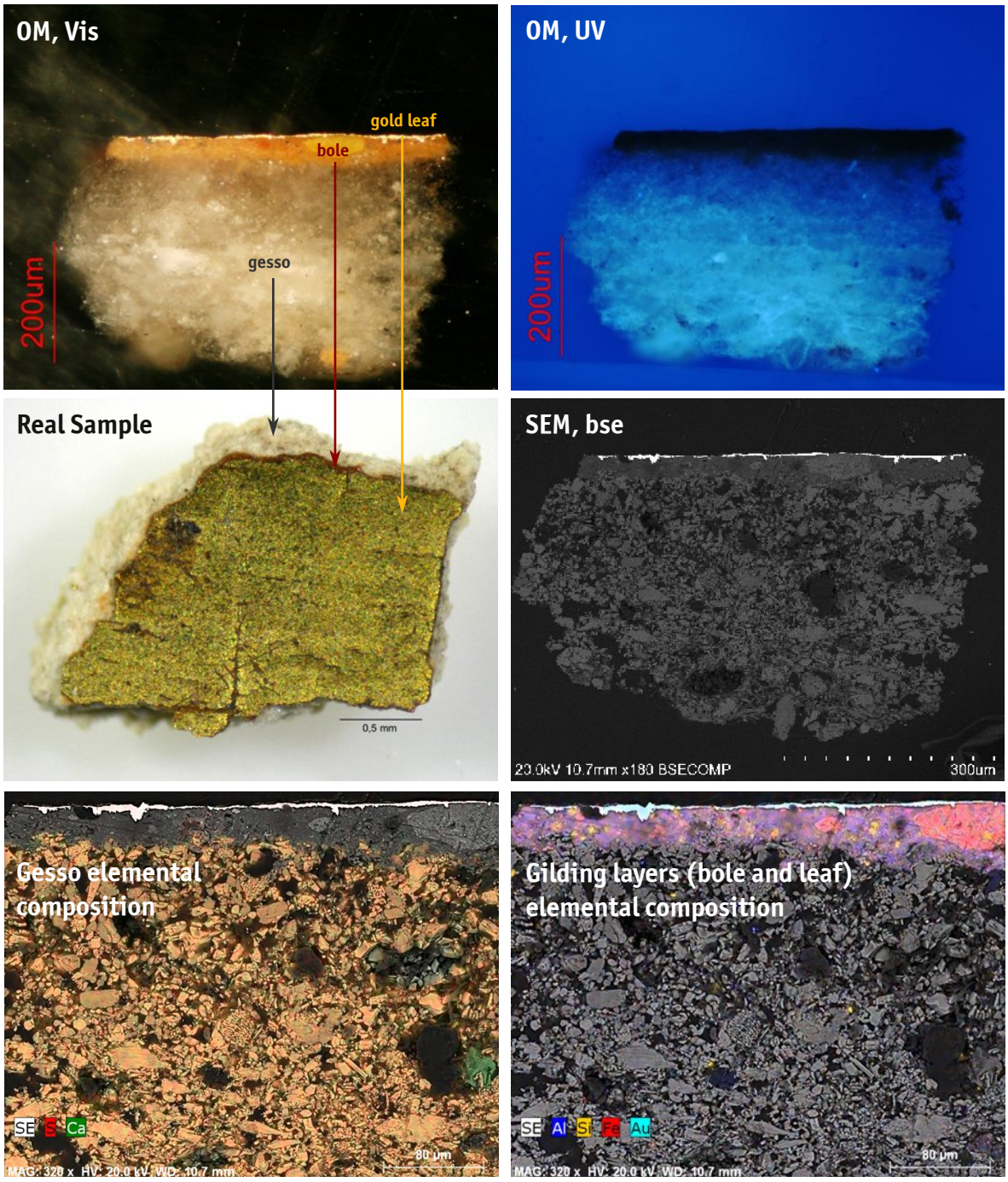


*Figure 2. Optical microscopy and scanning electron microscopy on a cross-section from a real sample from the main altarpiece of the S. Francis Church in Castelo de Vide, Portalegre.*

"parchment glue". Clippings (*retalhos*, meaning leftovers discarded by glove makers) and gloves glue (*colla de luvas*) were identified as base of a binder material for gilded altarpieces, polychrome statues and carved ornaments. The facts regarding the use of animal skins that are relevant for the reconstructions are: the use of young animal skins; process of tanning the skin; the need to let the animal skin boil in water during the glue preparation (probably the alteration of the adhesive properties of glues subjected to temperatures above 60º C was not well known) and the possibility

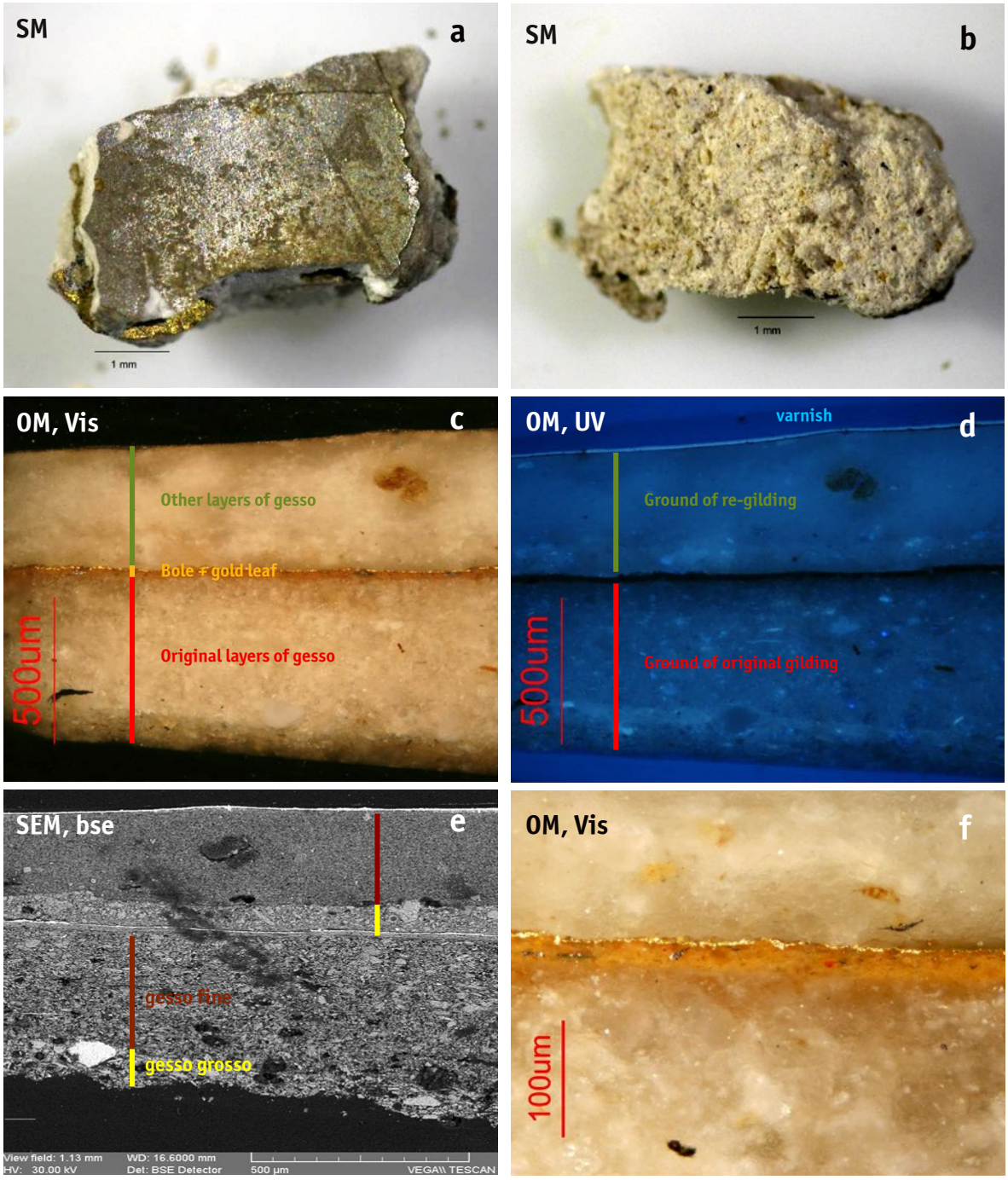


*Figure 3. Optical microscopy and scanning electron microscopy on a cross-section from a real sample from Bucelas church (Loures, Lisbon) showing two phases of gilding. Image f shows the detail of the gilding layers at the interface between the two grounds.*

to strain the glue before use [8]. The use of garlic is also mentioned as component of the mixture made of gesso and animal glue [6] or added to the glue size and then called "alhada" [24]. This practice was common to other gilding traditions and not only in Portugal [25].

*Ouro burnido* (burnished gold) and *ouro mate/ fosco* (mate gold) is another distinction made by the two treatises regarding the type of gilding technique and the surface treatment of the leaf after its application [26]. Only the gold leaf applied by water gilding technique can be burnished, the

mordant technique does not allow this treatment. The published researches on the purity of ancient gold leaf mentions pure gold (24 karats) and *ouro de lei*, *ouro subido*, or *ouro fino* (between 20 and 24 karats); values of 23.25 and 22 karat are quite common, 16 karats leaf is also in use after the second half of the 18$^{th}$ century [1, 27-31]. A recent study [10] shows the use of silver leaf with a varnish (the most common the shellac, or *goma-laca*) in order to imitate the appearance of the real gold leaf. Figure 3 gives the case of a real sample from the main altarpiece of Bucelas church (Loures, Lisbon, 18$^{th}$ century) where two phases of gilding were identified, the more ancient (original) having a gold leaf applied over bole and gesso layers, while the more recent is silver leaf with a thin layer of varnish with a bluish fluorescence.

### 2.2. Raw Materials: Gesso Powders and Alum Thawed Animal Skins

#### *2.2.1. The Gesso Reproduction*

Considering the difficult interpretation of the historical documents on the making of *gesso grosso*, we approached the process using analytical characterization to prove the composition of three typologies of commercially available gesso. To reproduce technical recipes described by Nunes and Almada, gypsum products were analysed in order to understand its composition and compare its quality with the ground layers from the cases we are studying. The procedure consisted in monitoring: the operational conditions and the materials' behavior of raw materials' times and temperature of burning the gypsum; the quantity of the material used and the grain size; and the weight loss after the burning process.

To analyze the commercial products X-Ray fluorescence (XRF) (Philips 1480 X-Ray spectrometer, 4 internal routine programs) was used. To confirm the data we also used a XRD Difractometer PANALYTICAL X´PERT PRO with the following parameters: Cu Kα radiation, I= 35mA, V= 45kV Scan 5-70° 2θ, step 0.033, t= 100s, I= 35mA, V= 45kV.

The analysed materials, namely two rocks, were brought from Óbidos. Their crystals were differentiated as fine gypsum and very fine gypsum according to their grain size. Figure 4 shows the XRD of the fine gypsum and very fine gypsum composition.

The initial results showed that the fine gypsum is pure di-hydrated calcium sulfate while the very fine gypsum is made of insoluble anhydrite and a little percentage of dolomite. Only the upper one was considered adequate as raw material for our experiments. The fine gypsum was fragmented in small pieces and then divided in four groups of the same weight (38 g) heated in the oven (Nabertherm L 9/11/SKM Model) in variable conditions of temperature and exposure (Table II).

The resulting powder was immersed in water for four days to obtain dihydrated gesso (*gesso mate*) and after each hydration step a further XRD analysis was performed (Figure 5). The experiments were useful to answer several of our questions: which is the temperature when the transformation of gypsum into hemihydrated gesso happens; which is the range of temperature where the hemihydrated gesso and anhydrite are obtained; which is the temperature where only anhydrite is obtained and if is still soluble in water at 600°C or more temperature is necessary.

However, despite the clear appearance of the samples from fine gypsum selected for this study, it was found that there were impurities that contaminated the gypsum's final product. Only by visual identification we cannot be sure if a fine

*Table II. Parameters of the hydration procedure for the four groups of fine gypsum.*

| Fine gypsum | Initial Temperature (°C) | Final Temperature (°C) | Time (h) | Initial Weight (gr) | Final Weight (gr) | XRD Result | Still able to be hydrated |
|---|---|---|---|---|---|---|---|
| **Group 1** | 130 | 160 | 1h15 | 38 | 37 | Hemi-hydrated gesso | Yes |
| **Group 2** | 130 | 160 | 2 | 38 | 35,6 | Hemi-hydrated gesso | Yes |
| **Group 3** | 300 | 25 | 8 | 38 | * | Hemi-hydrated – soluble anhydrite | Yes |
| **Group 4** | 500 | 600 | 1h30 | 38 | 25,5 | Soluble anhydrite | Yes |

** Tampered result.*

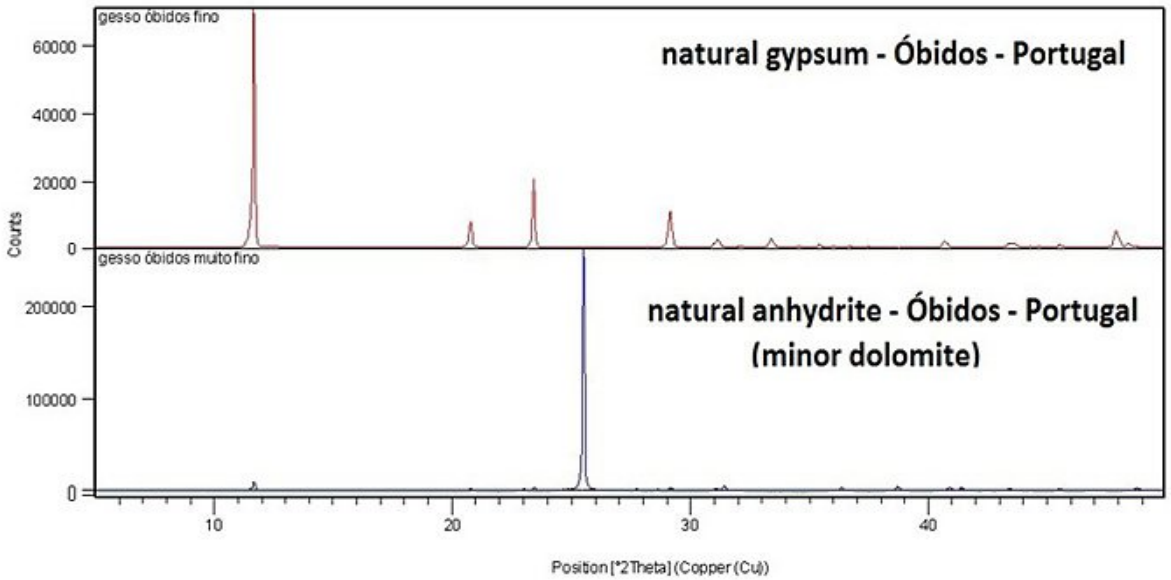


*Figure 4. XRD difractograms of fine gypsum (natural gypsum) and very fine gypsum (natural anhydrite).*

grained gypsum rock is pure or not. The first piece of rock analyzed indicated only the presence of gypsum but a second one, from the same location and having the same appearance, presents a very complex composition with impurities. Indeed, the XRD results from the second sample indicated the presence of anhydrite, carbonates and minor pyrite in the starting material. After the small fragments were heated at 160° C many harder particles of carbonate and anhydrite were found. The carbonate particles also presented a darker color. Therefore, that material was considered not adequate for obtaining the pure gesso grosso and gesso mate. Considering the difficulty of recognizing the purity of the raw materials, selenite type, a very pure transparent variety of gypsum, pure fibrous crystals originating from Morocco, were then used to prepare the gesso mate for our model samples (Figure 6) and not the gypsum originally from Óbidos.

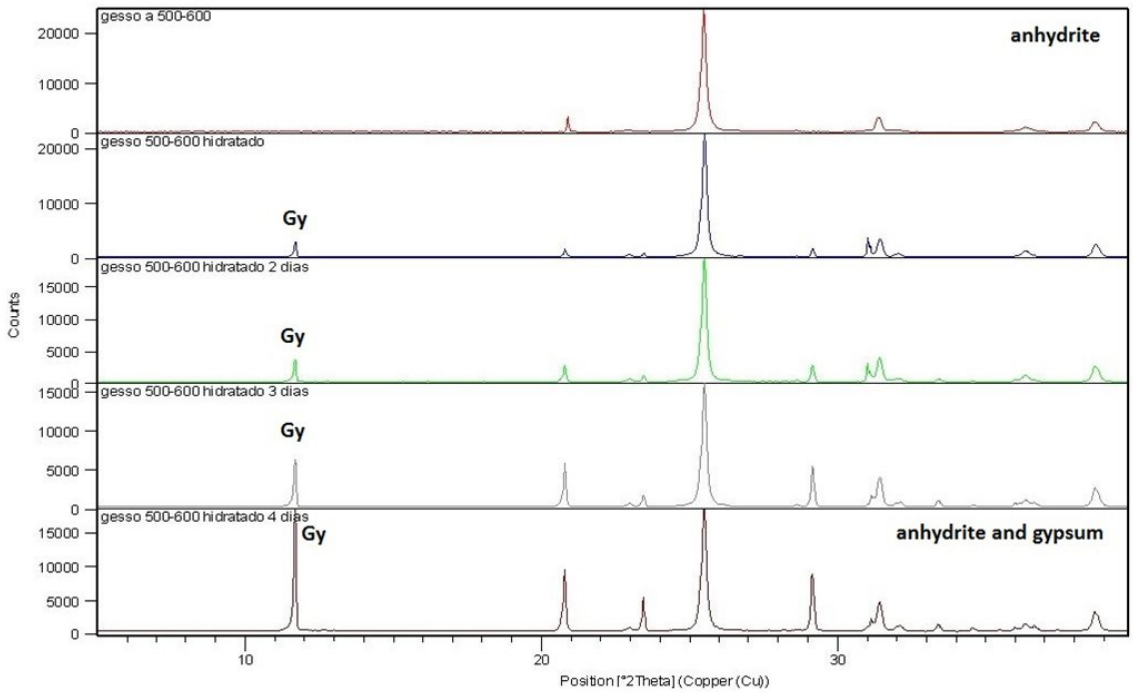


*Figure 5. XRD diffractograms of hydrated anhydrite after four days where an increase of the peaks intensity of gypsum (Gy) can be observed.*

The Moroccan gesso was characterized by XRD and resulted to be very pure. The probability of the gesso being imported in ancient times from other countries like Morocco is also confirmed by literature [11] which states that at the time there were very few gesso mines in Portugal and therefore foreign sources had to be found to supply the necessary quantity for retabular production.

### *2.2.2. Animal Glue Production*

The thawing procedure of animal skin is very old and aims to render the leather more resistant to the atmospheric and biological agents. The thawing materials were more or less the same during the history: calcium hydroxide, vegetal tannins, animal fats and alum.

In the absence of Portuguese recipes, we decided to follow an Italian manuscript known as *Manuscritto Bolognese* (ms 2861, Table I). Although referenced by many authors, this particular recipe from the Italian Bologna province was first translated to English by M. Merrifield in 1849 [23]. It refers to the historical use of materials in Italian oil painting over a broad period of time. This translation was not always well succeeded because the Bologna vulgar dialect can sometimes be of difficult understanding so they all lack a complete description of all the necessary materials. Professor Pietro Baraldi, from the Università di Modena e Reggio Emilia, made a very useful translation and critical review of the recipe [32].

Our thawing experience procedure in fresh animal skins from young lamb (sheep) and goat was made manually and with tools according to the recipe. The fresh skins were salted with NaCl to prevent the rapid process of deterioration of tissues (putrefaction). The salt penetrates into the skin, which still has around 65% of water, and produces its partial elimination by an osmotic effect. After

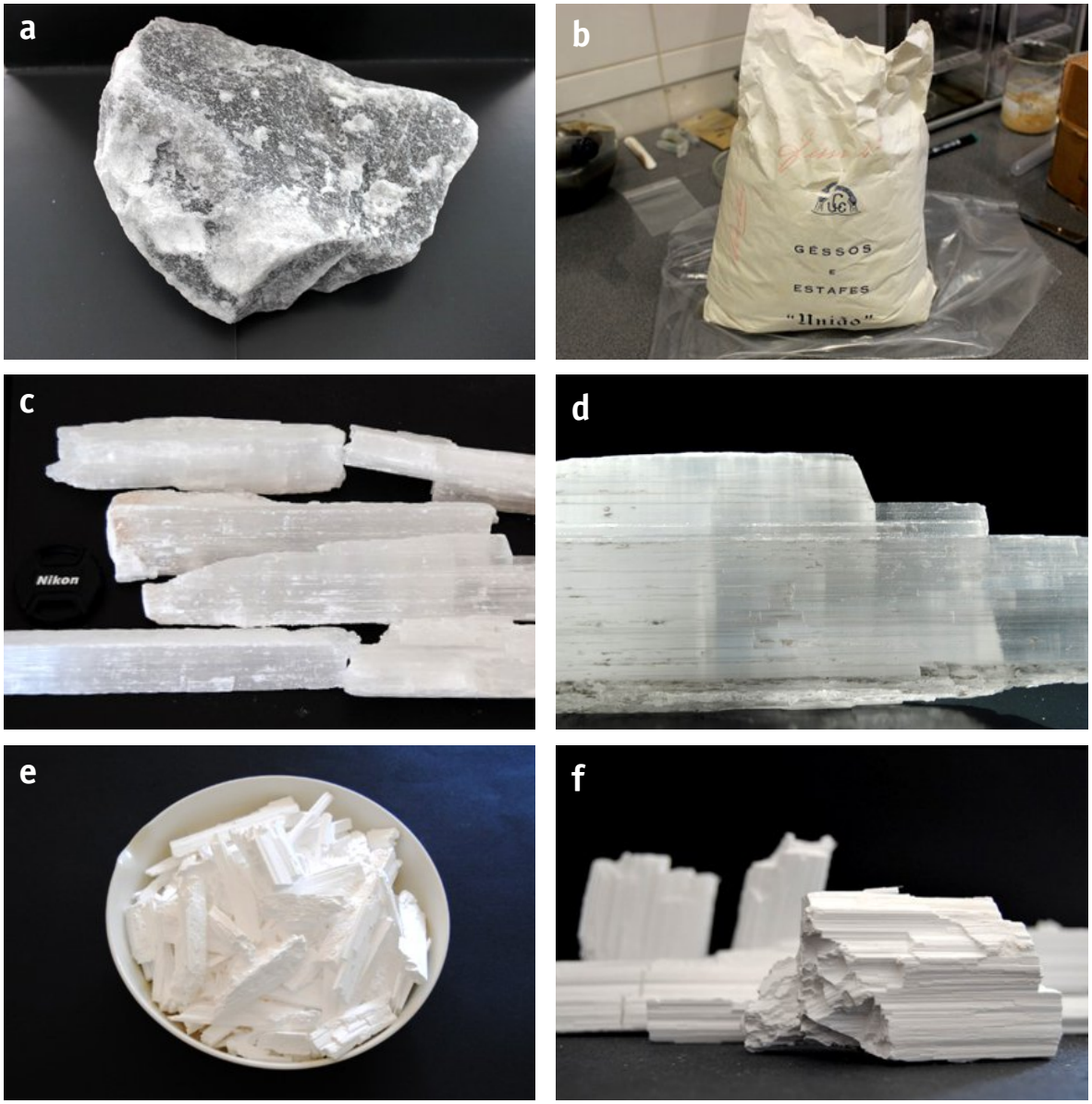


*Figure 6. The gesso: a) gypsite rock; b) Portuguese commercial products (gesso from Óbidos); c-d) Morocco's gesso crystals before; and e-f) after burning in the oven.*

abundant washing in water, the skin is re-hydrated and the salt and dirt removed. The skins are then exposed to open air in vertical position to lose the excess water.

The following process is the elimination of the fur by the application on the skins inside for three hours of a saturated solution of sodium sulphide ($Na_2S$) and calcium hydroxide, $Ca(OH)_2$, obtained from hydrating the calcium oxide, known as *pedra de cal*. The chemical reactions that occur during this process are complex but essentially are based on the solubilisation of the keratin by breaking the disulphide bond (-S-S-) contained in the cistine molecule. The presence of calcium hydroxide stabilizes the pH of the washing bath around 12.5, optimal for the depilation and for the reduction action of sulphide and sulfhydrate. The chemical reagents penetrate into the skin until the follicular level and attack the radix


*Figure 7. Main steps of the thawing procedure of two animal skins: a) sheep (white) and goat (black) skins; b) chemical reagents used for depilating the skins (sodium sulphide ($Na_2S$) and calcium hydroxide, $Ca(OH)_2$); c) preparation of the mixture for the depilation; d) immersion of the skins into the mixture; e) mechanical removal of the hair; f) ingredients of the Bolognese manuscript recipe; g) mixing the products for the thawing process; and h) heating the mixture before the treatment of the skins..*

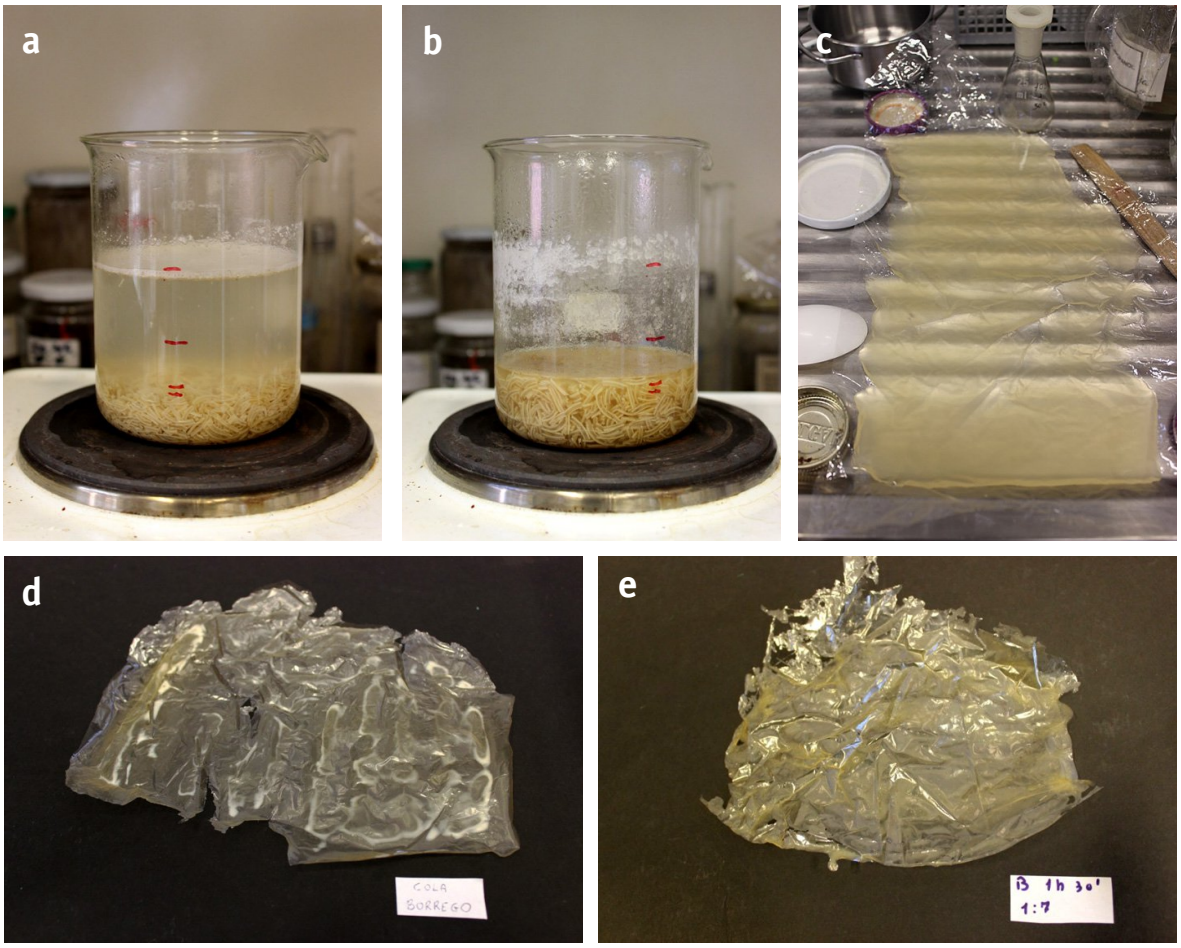


*Figure 8. Main steps to obtain sheep skin glue: a) hydrated small fragments of sheep skin to be dissolved on a heating plate; b) monitoring of the water level; c) drying the glue solution on a melinex sheet; d) dried foil of sheep gelatine; and e) dried foil of sheep gelatine, ratio 1:7 in water boiled for 1h30.*

of the fur that is then removed mechanically with a big blade, especially made for this task.

The depilated skins were left overnight in water and washed to remove all the residual fur. The following day the removal of the residual tissues that connect the skin to the bones was performed mechanically. The final process was the exposure to the air of the skins after stretching on wooden sticks in order to eliminate the water for complete drying. The skins were then ready to be thawed using potash alum according to the Bolognese Manuscript recipe.

Figure 7 shows the steps of the thawing procedure executed at the LIFER leather factory located in the village of Nossa Senhora de Machede, Évora with the help of the Mr. Lidório Fernandes.

The preliminary tests led to: a protocol for both the skins as well as temperature and time for boiling the skins in water in order to obtain a gelatinous composition; the drying technique; and the procedure to obtain the sizing material (glue) from the animal gelatin.

The dried thawed skin was then cut in small fragments and left stand in water overnight. Once hydrated, it was put into a Becker with water in ratio of 1:7 (1 of skin and 7 of water) and then heated on a heating plate. After different trials to understand which is the ideal water temperature,

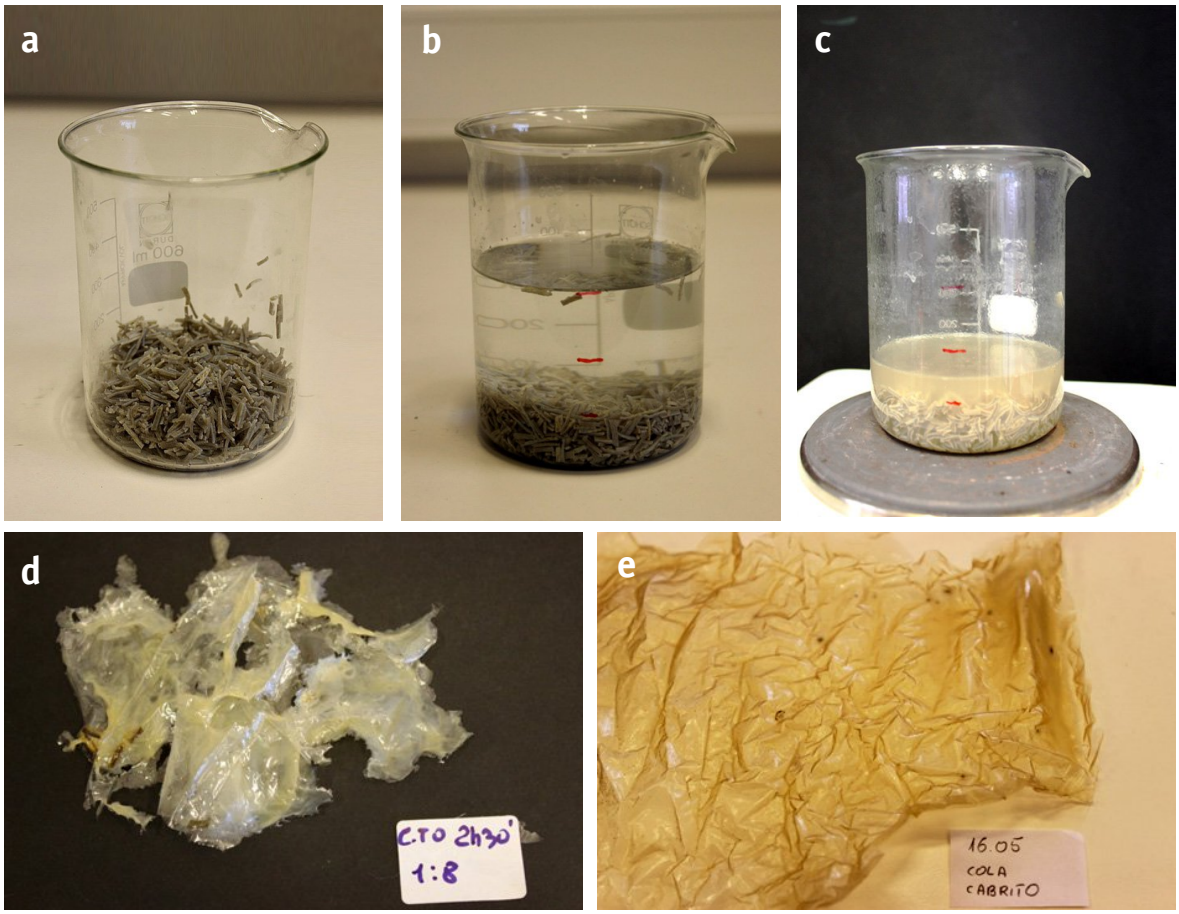


*Figure 9. Main steps to obtain goat skin glue: a) small fragments of tanned goat skin; b) hydrate small fragments of goat skin; c) dissolved on a heating plate and monitoring the water level; d) experiment of dried foil of goat gelatine in a 1:8 ratio in water for 2h30; and e) dried foil of goat gelatine.*

considering that the terminology in the treatises speak of "boiling water", this means reaching 100°C, it became obvious that the gelatin can be obtained by maintaining the heating temperature around 60-70°C. The procedure is considered finished when, after one day of heating, one third of the initial product is obtained. A careful monitoring of the water level is therefore recommended.

During heating, the skin fragments were not completely transformed into gelatin by which filtration using a sieve was necessary to separate the residues from the final product. The gelatin was then applied on a melinex sheet in a thin layer that once dried formed a rigid and transparent film that could easily be broken in small pieces or scales. This product can be conserved in a dried environment and re-used by hydration to obtain the liquid animal glue/size.

Figures 8 and 9 shows the main steps to obtain the glue solutions or gelatin from the alum thawed animal skins. A slight difference in color and appearance is observed between the sheep and goat glues, both during the preparation in water solution and as film after the drying of the solution on a melinex sheet.

### *2.3. Application of Animal Glue Sizing and Gesso Grounds*

Figure 10 illustrates the application on the wooden model sample of a layer of sizing, prepared from the animal glues and water in ratio of 1:7.


*Figure 10. Main steps to the application of ground layers: a) Heating in "warm water bath" the goat gelatine; b) keeping the gelatine solution warm; c) first application of animal size with brush in flat sample; d) first application of animal size with brush in three-dimensional sample; e) mixing gesso with the gelatine; and f) application of first layer of ground on the flat sample.*

According to the two Portuguese recipes, the gesso grounds must be obtained by mixing gesso powder with the binder, animal glue solutions obtained from the gelatin originated from thawed animal skins. Some authors mention the use of a piece of garlic boiled together with the gesso mixture that in the eighteen century was called *alhada* [24]. The astringent properties of garlic and the various sulfur compounds favor the adhesion of the several layers of *gesso grosso* and subsequently the ones of *gesso fino* that is applied on top and sanded with abrasive papers between the different applications.

In this phase, several trials were done to understand which would be the proper quantity of water needed to obtain the glue hide, with good adhesive and cohesive properties for the gesso powder. The dried film scales were put overnight in water; after the hydration the excess water was eliminated and the resulting gelatin was heated in "warm water bath" at around 60 °C,

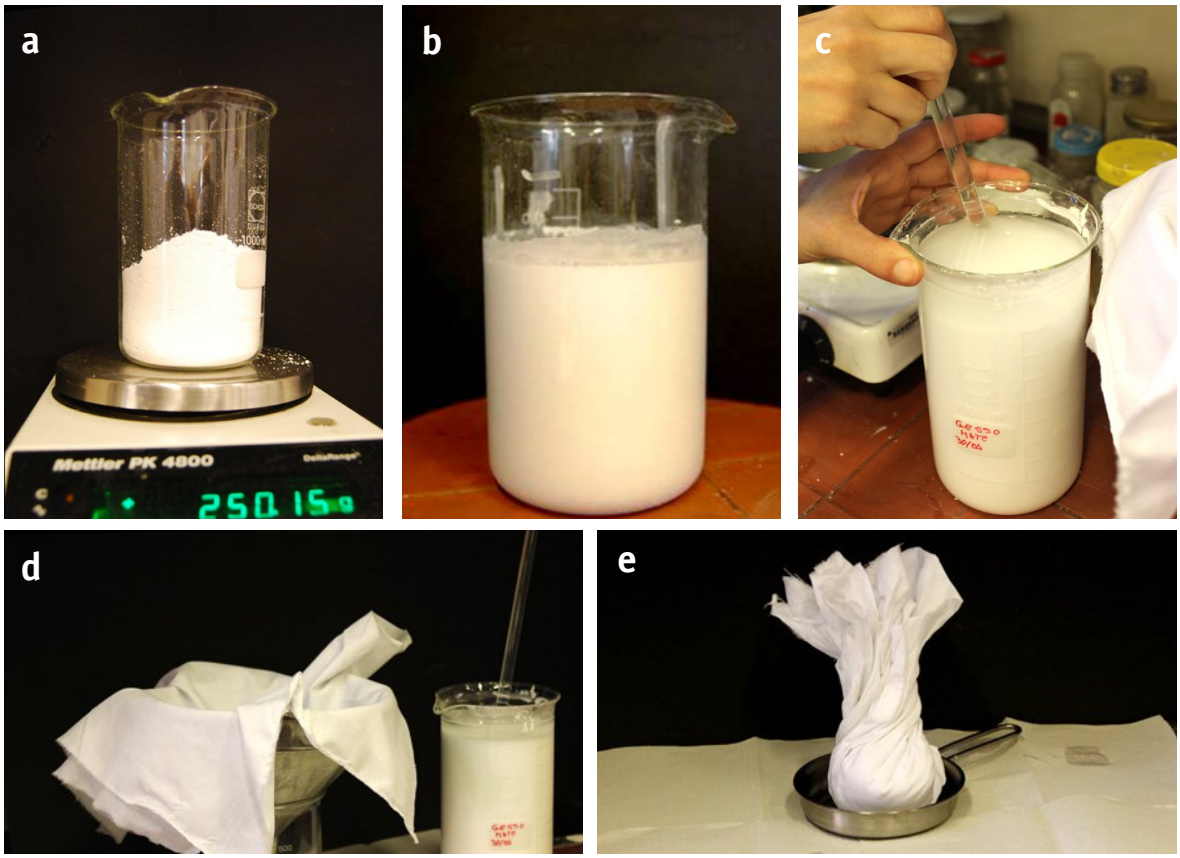


*Figure 11. Main steps to prepare gesso mate from gesso grosso: a) measuring 150 gr of gesso powder; b) hydration with continuous changes of water; c) stir once a day preventing gypsum from setting; d) pouring the hydrated gesso into a cloth; and e) the water is squeezed off to obtain a loaf of gesso.*

in order not to alter its properties. Then, the necessary quantity of gesso was added considering the density of the mixture, its color and viscosity. This is the part where experience and sensibility is more required because the result depends much from both the material and the artist's hand. It is necessary to obtain a homogenous mixture, with a good hiding power and easily sanded, without lumps.

The preparation of the *gesso mate* from the *gesso grosso* (previously calcinated gypsum) includes the hydration with continuous changes of water and shaking of the suspension during several days to prevent the gypsum setting. This procedure assures a good quality of *gesso fino* particles, dispersed and separated into water. Then, the slaked gesso is poured into a cloth and the water squeezed off, a loaf being formed from the damp thixotropic *gesso fino*. This loaf will be mixed with the animal glue solution and will form a liquid mixture to be applied by brush.

Figure 11 shows the main steps for the obtaining of gesso fino by hydrating the gesso grosso and removing the excess water and the application of different layers of ground (Figure 12) followed by sanding the surface with an abrasive paper.

### *2.4 The Bole Layers*

For the preparation of the bole we used the loaves made of a red argillous material, immersed for five days into water. In the first days the mixture was agitated to promote the disaggregation of the mineral and its hydration. Once a suspension


*Figure 12. Main steps to prepare ground and its application: a) sieving the loaf of gesso; b) grind the gesso in mortar to obtain gesso fine powder; c) mixing it with animal glue solution in a "warm water bath"; d) application with brush of several gesso fino layers; e) sanding the surface with an abrasive paper; and f) final sanding with fine abrasive paper.*

without lumps was obtained, the argillous material was left to sediment on the bottom of the bucket and the excess water was eliminated. For obtaining the bole paste, 100 g of the sedimented material was mixed with 40 g of animal glue in a aqueous solution of 10 %. The number of layers varies according to the recipe and the application of the bole and glue mixture was made overlapping vertical and horizontal layers.

Figure 13 illustrates the main steps of bole obtained from powdering the natural mineral material and mixing it with animal glue solution (10% w/w) followed by its application procedure in perpendicular layers and by final polishing with a burnisher.

## 3. Conclusions

Although limited to the description of ground and bole layers preparation and application, the present paper shows the complexity of the documented reconstructions criteria and procedures for gilding techniques. These preliminary results of the task developed within the Gilt-Teller project are important to show how we can complement the practical work done in the laboratory with analytical evidences obtained by using complementary analytical tools and can be a precious source for further studies on gilding recipes, materials and techniques. Correlating the terminology and literature information with the practical data and critically approaching the different sources (historical and analytical) is the basic rule for correct reproduction of ancient recipes with nowadays means and materials.

According to our results, the quality control of the materials used is one of the most important aspects of the whole process of reconstruction. This control takes into account the rational choice of the most pure lots, obtained from the raw materials of high purity or from certified foreign

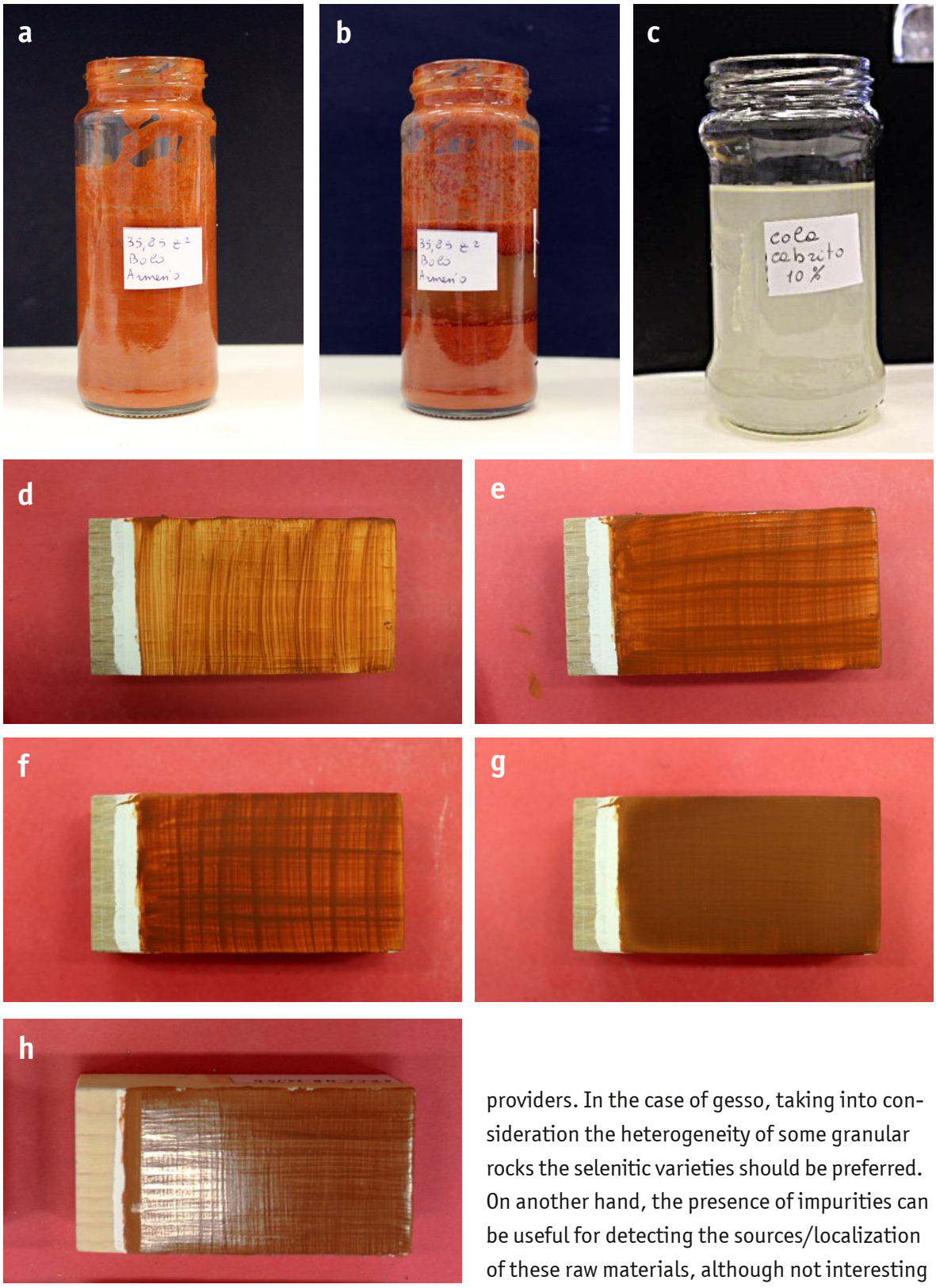


*Figure 13. Preparation of red argillous material: a) loaf of bole immersed in water; b) material was left to sediment on the bottom of the bucket; c) lamb glue solution dissolved 10% (w/w) in water; d) first application of bole in the upwards position; e) transversal application of second layer; f) succeeding applications of bole layers in perpendicular layers; g) four layers of bole; and h) final polishing with a burnisher.*

providers. In the case of gesso, taking into consideration the heterogeneity of some granular rocks the selenitic varieties should be preferred. On another hand, the presence of impurities can be useful for detecting the sources/localization of these raw materials, although not interesting to the present work.

The second part of this study will bring into attention the procedure for gold leaf application and

surface treatments and the analytical results on the reconstructions for the whole gilded composite.

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## 5. References

[1] I.C.A. Sandu, L.U. Afonso, E. Murta, M.H. de Sá, *Gilding Techniques in Religious Art Between East and West, 14th-18th Centuries*, International Journal of Conservation Science 1(1), March 2010, pp. 47-62

[2] S. Kroustallis, J. Townsend, E. Cenalmor Bruquetas, A. Stinjman, M. San Andrés Moya (eds.), *Art Technology - Sources and Methods 2*, Archetype Publications, London, 2008

[3] I.C.A. Sandu, M. Helena de Sá, M. Costa Pereira, *Ancient "gilded" art-objects from the European cultural heritage: a review on different scales of characterization*, Surface and Interface Analysis 43(8), 2011, pp. 1134-1151, doi: 10.1002/sia.3740

[4] I.C.A. Sandu, *GILT-Teller: An interdisciplinary multiscale study of gilding techniques and materials in Portugal, 1500-1800*, VIIII Jornadas de Arte e Ciência - Conservação e Restauro de Artes Decorativas de Aplicação Arquitectónica, 29th September 2012, CITAR-UCP, Porto, pp. 127-142, 2013

[5] L. Jorge, P. Monteiro, *From treaty to gilding practice: documentation on techniques, materials and decorative processes in the context of Portuguese gilded wood carving (talha dourada)*, ICOM-CC SPAD Working Group Interim Meeting on Polychrome Sculpture, Tomar, 28th-29th May

[6] P. Nunes, *Arte Poética, e da Pintura e Symmetria, com Princípios de Perspectiva*, edição facsimilada de 1615 com estudo introdutório de Leotina Ventura, Editora Paisagem, Porto, 1982, p. 126

[7] J. Lopes de Baptista Almada, *Prendas da Adolescencia, ou adolescencia prendada com as prendas, artes, e curiosidades mais uteis, deliciosas, e estimadas em todo o mundo*, Lisboa, Off. de Francisco da Silva, 1749

[8] A. Le Gac, *Preliminary research on glues made of thawed leather, parchment and rabbit skin*, in E. Jablonska and T. Kozielec (eds.), Parchment and leather heritage: Conservation-Restoration, Nicolaus Copernicus University, Torun, 2012, pp. 31-58

[9] I. Cardoso, *Gesso layers in Portuguese Baroque altarpieces: materials, practices and durability*, Doctoral thesis, Institute of Archaeology, University College London, London, 2010

[10] I.C.A Sandu, E. Murta, S. Ferreira, M. Costa Pereira, A. Candeias, J. Mirão, C. Miguel, F. Paba, *More than gold – an interdisciplinary, complementary study of gilding materials and techniques in baroque altarpieces from Portugal*, ECR - Estudos de Conservação e Restauro, CITAR-UCP, Porto, 2015, submitted

[11] J. Coroado, V. Antunes, V. Serrão, M.J. Oliveira, L. Dias, A. Candeias, J. Mirão, L. Carvalho, A.I. Seruya, *Presença de Celestite em Retábulos Portugueses*, in: V. Serrão, V. Antunes, and A.I. Seruya (ed.), *As Preparações na Pintura Portuguesa. Séculos XV e XVI*, Faculdade de Letras da Universidade de Lisboa, 2013, pp. 75-84

[12] J. R. G. Costa, *O Gesso em Portugal*, Estudos, Notas e Trabalhos do Serviço de Fomento Mineiro 28, 1986, pp. 93-117

[13] J. Velho, C. Gomes, C. Romariz, *Minerais Industriais: Geologia, Propriedades, Tratamentos, Aplicações, Especificações, Produções e Mercados*, edição de autor, 1998

[14] A. I. Seruya (dir.), *Policromia: A Escultura Policromada Religiosa dos Séc XVII e XVIII – Estudo comparativo de técnicas, alterações e conservação em Portugal, Espanha e Bélgica*, Actas do Congresso Internacional, Lisboa, 29 – 31 de Outubro de 2002, Instituto Português de Conservação e Restauro, 2004

[15] D. Bigelow, *Gilded wood: conservation and history*, Sound View Press, Madison, Connecticut, 1991

[16] M. Matteini, A. Moles, *Tecniche della pittura antica: le preparazioni del supporto*, Kermes 4, Nardini Editore, Firenze, 1989

[17] I. Cardoso, *As camadas preparatórias em retábulos e esculturas douradas e policromadas portuguesas*, in: V. Serrão, V. Antunes, A. Isabel Seruya (eds.), *As Preparações na Pintura Portuguesa. Séculos XV e XVI*, Lisboa, Faculdade de Letras da Universidade de Lisboa, 2013, p.167-198

[18] S.S. Gomez, *Las preparaciones de yeso en la pintura sobre tabla de la escuela española*, Doctoral Thesis, Departamento de Pintura, Facultad de Bellas Artes, Universidad Complutense de Madrid, Madrid, 2005

[19] S. Santos Gómez, M. San Andrés Moya, J.L. Baldonedo Ródriguez, O. Conejo Sastre, M.I. Báez Aglio and A. Rodriguez Muñoz, *Contribution to the study of grounds for panel painting of the Spanish School in the fifteenth and sixteenth centuries*, in: A. Roy, P. Smith, and D. Bomford (eds.), *Painting techniques, history, materials and studio practice: contributions to the Dublin Congress*, 7-11 September 1998, International Institute for Conservation of Historic and Artistic Works, London, pp. 115-119

[20] M. Stols-Witlox, *Grounds 1400-1900*, in: J. H. Stoner and R. Rushfield (eds.), *Conservation of Easel Paintings*, Routledge, 2013, pp. 160-169

[21] S. Santos Gomez, M. San Andrés Moya, A. Rodriguez, *Reconstruction of documented preparation methods for gesso grosso and gesso sottile in Spanish School panel paintings*, in: in S. Kroustallis, J.H. Townsend, E. Cenalmor Bruquetas, A. Stijman and M. San Andres Moya (eds.), *Art Technological Research: towards a new discipline*, Archetype, London, 2008, pp. 178-181

[22] M. Stols-Witlox, *Sizing layers for oil painting in western European sources (1500-1900): historical recipes and reconstructions*, in: S. Kroustallis, J.H. Townsend, E. Cenalmor Bruquetas, A. Stijman and M. San Andres Moya (eds.), *Art Technological Research: towards a new discipline*, Archetype, London, 2008, pp. 147-165

[23] L. Carlyle, *The Artist's Assistant. Oil Painting Instruction Manuals and Handbooks in Britain 1800–1900 with Reference to Selected Eighteenth-Century Sources*, Archetype, London, 2001, p. 16

[24] N.M.F. Alves, *A arte da talha no Porto na época barroca: artistas e clientela, materiais e técnica*,

Arquivo Histórico, Câmara Municipal do Porto 1989, p. 202

[25] I. Bonaduce, M.P. Colombini and S. Diring, *Identification of garlic in old gilding by gas chromatography-mass spectrometry*, Journal of Chromatography A 1107 (1-2), 2006, pp. 226-232, doi: 10.1016/j.chroma.2005.12.053

[26] C. Cession, *The surface layers of baroque gilding: examination, conservation, restoration*, in: Cleaning, retouching and coatings: Contributions to the 1990 IIC Congress Brussels, 1990, pp. 33-35

[27] I. Sandu, E. Murta, E. Neves, A. Pereira, A.V. Sandu, S. Kuckova, A. Maurício, *A comparative, multi-scale and interdisciplinary study of gilding techniques and materials in two Portuguese Baroque "talha dourada" complexes*, ECR - Estudos de Conservação e Restauro 4, CITAR-UCP, Porto, 2012, pp. 47-71

[28] C. Barata, A.J. Cruz, F.T. Rocha, *Sobre os materiais utilizados na talha da epoca barroca do noroeste de Portugal - primeiros resultados*, in: G. Vasconcelos e Sousa and E. Vieira (eds.), I Encontro Luso-Brasileiro de Conservação e Restauro, CITAR, Porto, 2012, pp. 40-50

[29] A. Bidarra, J. Coroado, F. Rocha, *Fingerprinting gold leaf from Portuguese Baroque altarpieces*, Microscopy and Microanalysis 17 (S2), 2011, pp. 1778-1779, doi: 10.1017/S1431927611009767

[30] A. Bidarra, J. Coroado, F. Rocha, *Contributos para o estudo da folha de ouro de retábulos Barrocos por microscopia óptica e electrónica*, Revista Geconservación/conservação 1, 2010, pp. 183-191

[31] A. Le Gac, A.I. Seruya, M. Lefftz, A. Alarcão, *The main Altarpiece of the Old Cathedral of Coimbra (Portugal). Characterization of gold alloys used for gilding from 1500 to 1900*, ArchéoSciences 33, 2009, pp. 423-432

[32] *Manoscritto Bolognese. Secreti per colori: note sul manuscrito (ms. 2861) della Biblioteca Universitaria*, http://www.bub.unibo.it/it-IT/Biblioteca-digitale/Contributi/Manoscritto-bolognese.aspx?LN=it-IT&idC=61817

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