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17th Triennial Conference 2014 Melbourne

LEATHER AND RELATED MATERIALS

Multi-technique analysis of gilt-leather wall coverings (16th–18th centuries)

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KEYWORDS: gilt leather, wall coverings, wall hangings, Renaissance, synchrotron FTIR, ion beam analysis, ATR-FTIR imaging, SEM/EDX

ABSTRACT

In this study, a multi-technique approach was developed to characterize the different components within gilt leather. A series of 40 gilt-leather hangings from different countries made between the 16th and 18th centuries were selected for analysis to learn about the manufacturing techniques and to better understand their degradation process. The analytical methodology had to take into account the complexity of the material with the presence of thin multilayers made of organic and inorganic compounds. Techniques that could provide non- or micro-destructive identification, high-sensitivity, and high spatial resolution were employed, that is: ion beam analysis, synchrotron infrared spectroscopy, scanning electron micros-

INTRODUCTION

Gilt leathers are luxurious decorations used all over Europe to embellish the interiors of the richest residences, dating predominantly from the 16th to the end of the 18th century (Fournet 2004). Despite the name, they are not defined by the presence of gold. According to a treatise by Fougeroux de Bondaroy (1762) and other technical sources (Schulze 2011), they were made by applying a silver foil to the leather, which had a gold-like appearance after being covered with a yellow varnish. It is still difficult today to date and classify polychrome gilt leathers because they are not signed, so, generally, assumptions are made by stylistic studies (Fournet 2006). A survey of the literature shows that few conservation projects have been complemented with analyses of these forms of decor (Van Soest 1989, Mannina et al. 2013, Tortora et al. 2014). Recently, 17th-century Italian and Flemish gilt-leather wall coverings at Moritzburg castle (Germany) were extensively studied and their materials analyzed by means of gas chromatography/mass spectrometry (GC/MS) and Fourier transform infrared spectroscopy (FTIR) (Schulze 2011). Schulze investigated the potential use of the two techniques, particularly for analyzing the "gold" varnish layer, but raised the need to develop a standard analytical approach.

This project aims to characterize the different components present within gilt leather made in various geographic areas between the 16th and 18th centuries to learn about the manufacturing technique, in particular the process specific to each workshop, and to better understand the degradation processes, especially silver tarnishing. A multi-technique approach was developed to be applied to a selection of decor from different countries and periods. The analytical methodology had to take into account the complexity of the material with the presence of multilayers made of organic and inorganic compounds, and the fact that chemical or technological markers may sometimes be in low concentration, either as traces or as a thin layer. Therefore, the aim was to find techniques that could provide non- or micro-destructive identification, high-sensitivity, and high spatial resolution. In this context, the research greatly benefited from the use of two large instruments: ion beam analysis (IBA) at the AGLAE (Accélérateur Grand Louvre d'Analyse Elémentaire) particle accelerator and synchrotron infrared micro-spectroscopy (SR-µ-FTIR). While IBA analyses were non-invasive, the analysis of the different organic layers present – that is, leather, glue, protective coating of the silver leaf,

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copy/energy-dispersive x-ray spectroscopy (SEM/ EDX), attenuated total reflectance spectroscopy-Fourier transform infrared spectroscopy (ATR-FTIR) imaging and pyrolysis-gas chromatography-mass spectrometry (Py-GCMS). The analytical methodology is discussed based on the results obtained on three selected gilt-leather artifacts, revealing some discrepancies and correlation in the appearance, chemical composition, layer thickness, and deterioration.



Figure 1 Image of gilt-leather sample AR11 from *Palazzo Chigi*, Ariccia (Italy)

Figure 2

Image of gilt-leather sample AR2 from *Palazzo Chigi*, Ariccia (Italy)

and "gold" varnish – required access to the stratigraphy of the material, therefore sampling was essential. Due to the nature of the materials and their sensitivity to solvents (particularly regarding the varnish), the sample preparation had to be thought through carefully so as to preserve the nature of the different components within each layer.

ARTIFACTS INVESTIGATED

A series of 40 gilt-leather hangings usually attributed to French, Italian, and Spanish makers from the 16th–18th centuries were selected in different European museums and institutions for analysis. Since the aim of this paper is to discuss the analytical methodology developed, the focus here is on the results obtained for the following three gilt-leather samples:

AR11, Palazzo Chigi, Ariccia (Italy)

The sample comes from a punched gilt-leather wall covering with a brocadestyle decoration *(a broccatelli),* made by the Italian leatherworker Agostino Nespola in 1674. It presents a reddish transparent layer applied on the gold varnish in the background areas. The silver leaf shows moderate tarnishing where covered by decorative layers and more marked tarnishing in the exposed areas. The "gold" varnish has a bright and light color and no particular sign of deterioration (Figure 1).

AR2, Palazzo Chigi, Ariccia (Italy)

This sample comes from an embossed gilt-leather wall covering with decorative motifs inspired by Dutch specimens, made at the end of the 17th century by an unknown leatherworker. The "gold" varnish has an intense auburn hue, and in the background areas it is covered by light-blue paint. Decorative details are overlaid with transparent green and red lacquer. The silver leaf has slightly darkened, and the gold varnish has become opacified (Figure 2).

CBD6, private collection (France)

CBD6 is older than the other two samples described above, and closer to the 16th-century Spanish production. This piece is probably the center part of a dismantled altar frontal that was later framed. A painted Madonna with Child is overlaid by punched decorations. The backgrounds, originally silvered and gold varnished, are now dark, probably because of over cleaning (back down to the leather surface). However, surviving gold areas, where the silver is not tarnished and the varnish is yellow, were found on the edges protected by the wooden frame (Figure 3).

METHODS OF STUDY

The surfaces of the gilt-leather coverings were examined under a stereomicroscope to evaluate their state of conservation and select representative areas for analysis and sampling.

IBA analyses were carried out directly on the objects non-invasively at the AGLAE in Paris. Particle induced x-ray emission (PIXE) provided elemental composition information using a 3-MeV proton beam. Rutherford backscattering spectroscopy (RBS), obtained under a 3-MeV alpha beam, enabled the stratigraphy of the analyzed area to be studied. The measurements were made in air with a beam current ranging from 1 to 2 nA.



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Figure 3 Image of gilt-leather sample CBD6 from a private collection (France)

Ultramicrotomy was used to access the leather stratigraphy by preparation of thin sections. A sample of a few mm² was taken for each leather hanging and sandwiched between two plastic pieces to provide rigidity during sectioning. Thin sections of 6–8 μ m in thickness were obtained in dry condition using an ultramicrotome equipped with a glass knife. Due to their high sensitivity to relative humidity fluctuations, they were stored at approximately 50% RH before and after analysis.

Optical microscopy images were collected from the sample surface and the thin sections using a Nikon Eclipse LV100 D microscope under normal and ultraviolet light.

Micro-FTIR spectroscopy was carried out on the SMIS beamline at the SOLEIL synchrotron on a Continuum XL microscope with a Nicolet 5700 spectrometer. The spectra were collected in transmission mode from the thin sections deposited on a ZnS window. Point spectra were collected using a beam size of $10 \times 10 \ \mu\text{m}^2$ and by averaging 64 scans at a spectral resolution of 4 cm⁻¹ over a spectral range of 4000–700 cm⁻¹.

Attenuated total reflectance spectroscopy-Fourier transform infrared spectroscopy (ATR-FTIR) imaging measurements were taken on a Bruker Hyperion infrared microscope equipped with an FPA detector coupled to a Vertex 70 spectrometer. Spectral images were collected over a spectral range of 900–4000 cm⁻¹ with a germanium ATR crystal.

Secondary electron microscopy with energy-dispersive x-ray microanalysis (SEM/EDX) was carried out on the thin sections (non-coated) with a Jeol scanning electron microscope JSM-5410LV equipped with an Oxford detector at 20 kV under controlled pressure conditions (12 Pa).

Pyrolysis-gas chromatography/mass spectrometry was performed on the varnish with a Shimadzu GCMS-QP2010Plus/Frontier Lab PY-2020iD system using thermally assisted methylation with TMAH (Py-TMAH-GC/MS). A sample of the varnish was analyzed according to the procedure developed for the analysis of historical varnishes (Echard et al. 2007).

The analytical technique specifics are given in Table 1.

Table 1

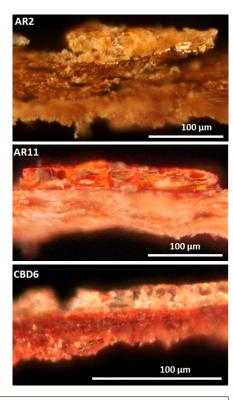
Specification of the analytical techniques applied to gilt leather (*samples that cannot be used for further analysis)

Analytical technique	Sample preparation	Spatial resolution / sensitivity	Information obtained	
IBA (RBS, PIXE)	no sampling	50 μm axial penetration depth: H ⁺ : 150 μm α: 20 μm	Depth measurements Composition and thickness of metal and alteration layers	
SR-µ-FTIR	thin section	10 µm	Compound class identification: leather (degradation, tannins) and varnish	
ATR-FTIR imaging	thin section*	3 μm	Compound class identification and spatial repartition: leather, varnish, and possibly glue	
SEM/EDX	bulk or thin section*	~ 100 nm	Elemental identification and repartition: pigment, drier, metal composition, and alteration	
Py-TMAH-GC/MS	sampling + derivatisation*	from 20 µg	Separation and molecular identification of varnish components	

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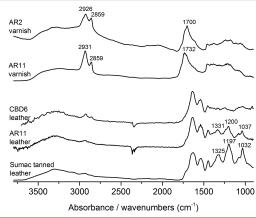


Figure 4

Optical microscopy images of the AR2, AR11 and CBD6 thin sections showing the different layers in the stratigraphy of gilt leather

Figure 5

SR-FTIR spectra of the varnish from samples AR2 and AR11 and ATR-FTIR spectra of the leather from samples CBD6 and AR11 and sumac-tanned leather

RESULTS AND DISCUSSION

General observation

The rendering of the "gold" varnish, which greatly affects the artifact's general appearance, depends on its color, thickness, and the presence of tarnish on the silver foil. Stratigraphy visible in the thin sections reveals the different layers: leather support, glue, silver foil, and "gold" varnish (Figure 4). Depending on their thickness, the glue and silver foil are often not visible by optical microscopy. Good cohesion of the layers was generally observed during preparation of the thin sections; however, in a few samples, flaking of the varnish and/or fragmentation of the leather support was encountered.

Leather

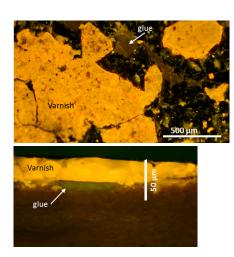
During sample sectioning, only the upper part of the leather was maintained in the stratigraphy; the lower part, less compact, was easily detached due to the absence of embedding. The infrared spectra of the leather support displayed some differences between samples in the region between 900 and 1800 cm⁻¹ (Figure 5). The bands centered at 1650 cm⁻¹ and 1530 cm⁻¹ are associated, respectively, with the Amide I and Amide II vibrations. Modifications in position, profile, and ratio of these bands can provide information on the conservation state of collagen (Gonzalez and Wess 2008). The spectral variations observed in the 1300–1400 cm⁻¹ and 1150–1250 cm⁻¹ regions may be related to the tanning materials used. The infrared spectra of CBD6 and AR2 measured in transmission show very similar profiles over the 900–1800 cm⁻¹ range; however they differ slightly from AR11 in the 1000–1400 cm⁻¹ region. This difference is enhanced by ATR measurement, probably because of the technique's higher beam penetration in the low-frequency range. As a result, the ATR-FTIR spectrum of the leather in AR11 displays strong bands at 1335 and 1200 and a double peak at 1086 and 1038 cm⁻¹, which are also observed in sumac extract and sumactanned leather spectra, suggesting a tanning process with hydrolysable gallotannin for AR11 (Figure 5). This result suggests that information on the tannin chemical class could possibly be obtained directly by ATR-FTIR without resorting to the extraction process currently in use (Falcão et al. 2013). SEM/EDX complemented the organic analysis, for example by revealing the diffuse presence of calcium in the layer of sample AR2, probably arising from residues of liming treatment.

Glue

The analysis of the glue layer between the leather and the silver foil was the most challenging, since it is generally not visible by optical microscopy as it probably impregnates the surface of the leather support. Nevertheless, pockets of glue a few microns thick are sometimes observed, as in sample AR11 (Figure 6), but it was too thin to be analyzed by synchrotron FTIR. In the continuation of this project, it is hoped that this limitation may be overcome by using ATR infrared imaging, possibly combined with data processing, to access a higher spatial resolution. To go further with the identification of the glue, expected to be protein-based, and to discriminate between gelatin and egg white, techniques such as liquid chromatography

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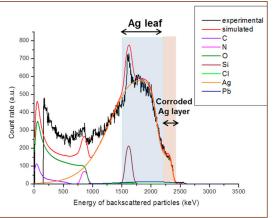


Figure 6

Optical microscopy images of sample AR11 under UV illumination: sample surface (top) and thin section (bottom)

Figure 7

RBS spectrum obtained with a 3-MeV alpha beam at the AGLAE. To fit the experimental spectrum, a corroded Ag layer had to be introduced at the surface of the silver leaf coupled to UV detection (LC-UV) or laser induced fluorescence (LIF) spectroscopy should be investigated (Nevin et al. 2006).

Silver foil

PIXE measurements of the silver leaf composition in most gilt-leather samples show a silver content of approximately 99% with traces of copper and sometimes gold. In a few samples, such as AR11, higher gold content (~0.12% as compared to ~0.05% or less in other samples) was observed, corresponding to a specific silver foil production. The RBS spectra simulation enabled the thickness of the silver leaf to be estimated (Feldman and Mayer 1986). In the corpus studied, the thickness of the original leaf ranges from 0.03 to 0.6 μ m, but most are centered around 0.1–0.2 μ m. The silver foils with values in the lower range likely suffered from alteration, which has caused a decrease of their original leaf thickness. The presence or absence of varnish over the silver surface could be determined, as well as the conservation state of the leaf, as shown in Figure 7. The RBS spectrum sometimes shows a layer of degradation of the silver leaf, corresponding to a maximum 10% of the original layer thickness and identified by PIXE as containing sulfur and chlorine.

The SEM/EDX measurements of the thin sections agree with the IBA analyses, also identifying mostly silver with minor amounts of copper in the silver leaf and the presence of chlorine and sometimes sulfur in the corroded layer (Figure 8). SEM images show that the silver foil has a thickness inferior to 1 µm, which is consistent with the values obtained from RBS. The technique provides complementary information regarding the localization of the element in the stratigraphy. In the elemental map of sample AR11, iron is correlated with the silver leaf. Considering that iron is not found in the composition of the metal foil, its presence might be linked to a layer above or underneath. Even if archival sources are unclear, it is known that glue, or possibly leather, could be colored in red. Iron could therefore come from a substance used in the glue before adhering the silver. Different types of deterioration of the silver foil are evidenced. In sample CBD6 (visually untarnished), the foil is uniform and well preserved, showing only the presence of chlorine co-localized with the silver in the elemental map. In both Ariccia samples (both visually tarnished), the foil is strongly disturbed by the formation of sulfide corrosion, either all along the interface of the leather support in AR2, or as a localized protrusion in AR11. The difference in the sulfide formation may be related to the cohesion of the different layers, since layers more cohesively bound may be less exposed to pollutants and tarnishing. In CBD6, the stratigraphy is well preserved and the different layers are compact, while in the Ariccia samples, flaking of the varnish and a higher friability of the leather support are encountered. Similar observations were made for the alteration of silver foils in medieval paintings (Salvado et al. 2011).

"Gold" varnish

The color of the varnish in the specimens studied ranges from light yellow to dark orange or red. In the thin sections, the thickness of the layer varies between 5 and 30 μ m, with differences in transparency and cohesion (Figure 4). Some heterogeneity is sometimes observed with the superposition

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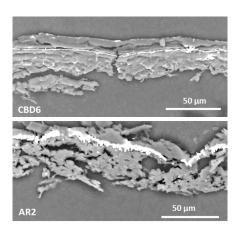


Figure 8 SEM images of CBD6 and AR2 thin sections

of layers (either a second varnish or an oxidized layer) and the presence of red coloring particles as in AR11 (Figure 6).

The spectra obtained by synchrotron FTIR on the varnish are similar to that of an oil-resin mixture (Figure 5). Differences are observed between gilt-leather samples in the position and profile of the peak at around 1700 cm⁻¹, associated with the carbonyl groups in oil and resin compounds, which may indicate some variation in the proportion of oil to resin. The gilt-leather samples AR2 and CBD6 display similar spectra for the varnish, which corresponds to an oil-resin mixture with major resin content. In contrast, the shift of the carbonyl vibration peak in AR11 towards higher frequency in the spectrum suggests the presence of a higher concentration of oil. The chemical image obtained by ATR-FTIR imaging on the CBD6 section shows that the varnish layer is homogeneous. The presence of a protein-based protective layer at the interface between the varnish and the silver foil could not be verified, and would possibly require the use of specific data processing. Far-infrared spectroscopy will also be tested for the varnish analysis, as it allows better discrimination of natural resins while preserving the sample (Prati et al. 2011).

The GC/MS technique provides a more precise characterization through separation and molecular identification of the different varnish constituents. Since this method is micro-destructive, it can only be applied as the last step. The Py-TMAH-GC/MS method was first tested for the identification of the varnish in two gilt leather samples because it has the advantage of limiting the risk of contamination and sample loss through the online derivatization. The chromatograms confirm the presence of a mixture of siccative oil, such as linseed oil, with a Pinaceae resin in the varnish samples and seem to suggest some variation in the resin/oil proportion, as for CBD6, where resin predominates. Chromatographic peaks assigned to linseed oil are methyl esters of palmitic and stearic acids, as well as the oxidative products, suberic, and azelaic acids. Methylated forms of three diterpenic acids belonging to Pinaceae resin were detected: dehydroabietic acid, 7-oxo-dehydroabietic acid, 15-OH-7-oxo-dehydroabietic acid. Other GC/MS protocols using different derivatization or injection modes should be further tested.

Finally, Raman micro-spectroscopy combined with ATR-FTIR imaging will be tested for the identification of the coloring particles observed in some varnishes.

CONCLUSION

The multi-technique approach developed for the analysis of gilt leather combines chemical identification, using elemental and molecular techniques, with spatial information from imaging techniques. Initially, two complementary techniques providing high brilliance and chemical sensitivity, ion beam and synchrotron FTIR, were chosen to investigate the corpus of gilt-leather decors. IBA allows multiple measurements to be carried out directly on the artifact and FTIR preserves samples for further analysis. However, limitations regarding spatial resolution, chemical specificity, and sensitivity, without mentioning access to these large instruments,

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MULTI-TECHNIQUE ANALYSIS OF GILT-LEATHER WALL COVERINGS (16TH–18TH CENTURIES) require the use of complementary analytical techniques. For that reason, SEM/EDX, ATR-FTIR imaging and Py-GC/MS have subsequently been tested to complement the initial analyses and are currently being applied on the gilt-leather corpus. In the continuation of the project, the analytical protocol will be optimized and new techniques will be investigated as, for instance, the imaging system recently developed at the AGLAE facility.

The comparison of the information obtained on the three selected giltleather samples, summarized in Table 2, validate the complementarity of the techniques and reveal some discrepancies and correlation in the appearance, chemical composition, layer thickness, and degradation. Such results are promising and it is expected that the information obtained from chemical analysis of the entire gilt-leather corpus, coupled with the stylistic study and the results of archival research, will give clues about the geographic origins of the artifacts and help to discriminate areas of production or even workshops. The information gained from the analyses will also help to improve current understanding of the condition and conservation of gilt-leather wall coverings, particularly those suffering from damage or deterioration.

Table 2

Summary of information obtained from the different analyses of the three gilt-leather samples (OM=optical microscopy)

	AR2	AR11	CBD6
General description	- Italian - end of 17 th century - varnish auburn opaque - slight silver tarnish	- Italian - dated 1674 - light varnish colour - moderate tarnishing	- Spanish - 16 th century - yellow varnish colour - no tarnishing
Leather	 brown colour presence of Ca 	- orange colour - hydrolysable gallo tannin	- brown colour
Glue	- not visible	- pockets of ~10 µm	- not visible
Silver	- visible in OM - composition : Ag, Cu - corrosion: Cl, S uniform	 not visible in OM composition : Ag, Cu, trace Au thin Fe layer under silver corrosion: Cl and S protrusion 	- not visible in OM - composition : Ag, Cu - corrosion: Cl uniform
Varnish	 - opaque, orange colour - thickness 20–40 μm - oil/resin mixture with major resin 	 translucent, dark orange red particle under UV thickness 25–30 µm oil/resin mixture 	 opaque, white colour thickness 13 µm oil/resin mixture with major resin

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