Transparent Varnishes on Copper Alloys Dating from the 19th Century: Characterisation and Identification Strategies

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Abstract

Many varnished copper-based artefacts dating from the 19th century can be found in museum collections. Although conservators deal on a daily basis with these surface finishes, few studies on this topic have been conducted during the last few decades on European objects. A more thorough understanding of these historical coatings would be helpful to establish accurate diagnoses and to contextualise the results with respect to the historical written sources. This paper presents the first results of the LacCa (Lacquered Copper Alloys) project currently being undertaken by an international multidisciplinary team. LacCa aims to develop a methodology that conservators can apply to identify and preserve these coatings. To this end, both historical objects and ancient varnish recipes have been studied. In a first step, the identification and characterisation protocol was validated using mock-ups, the results of which are presented in this paper.

Introduction

A significant number of copper-based artefacts in museum collections were originally covered by a transparent, often tinted, varnish. This technology was applied to various categories of objects such as scientific instruments and decorative bronze items, including horological and liturgical objects. The varnish had a primary role of protecting the metallic surface from corrosion. However, for the last two categories, the purpose was also aesthetic, often applied as a low-cost method for imitating gold.

These historical surface finishes present several difficulties for conservators. When gold-imitating varnishes are well preserved, they can be difficult to distinguish from authentic gilding by visual inspection. This is especially true if the varnished parts cannot be dismantled to reveal characteristic traces on the reverse. Furthermore, there does not appear to be any convenient published conservation methodology despite the fact that these finishes are common on objects in museum collections.

This paper presents the first results of the LacCA (Lacquered Copper Alloys) project, a study aimed at identifying the composition of the varnishes and examining their morphology by researching both historical objects and ancient varnish recipes from the 19th century. The results will help identify and create diagnosis criteria for conservators based on simple and affordable methods. A methodology was developed using mock-up coupons. The aim was to validate an identification and characterisation protocol for varnished copper alloys. The focus was on different portable and non-invasive techniques, used within the framework of on-site campaigns in Swiss and French museums on historical artefacts.

State of the art

Only one systematic analysis campaign on varnished copper-alloy objects has been published during the last few decades (Lanterna and Giatti 2014). The analyses were performed by Fourier transform infrared (FTIR) spectroscopy on a corpus of 16 scientific instruments from the Museo Galilei and Fondazione Scienza e Tecnica (Italy), dating mostly from the 19th century. Binders including shellac, sandarac and mastic were detected.

In addition, only a few case studies concerning gold varnishes applied to copper alloys in decorative arts are available. Long (2000) studied the varnished brass parts of a pair of Argand lamps (1839–48). The lamps were examined under UV radiation, and a natural resin ('garnet lac') was detected by FTIR.

Thomson mentioned the case of a Grecian couch with varnished copper-alloy elements (ca. 1820–40) which were analysed by x-ray fluorescence (XRF) spectroscopy and examined under ultraviolet (UV) radiation. In addition, she included in the article a short review of varnishing manuals, the majority from English or American written sources and dating from the 18th and 19th centuries (Thomson 1991). A more extensive review of gold varnish recipes is available, but it covers several centuries and different kinds of organic and inorganic substrates (Wellmer 1999).

A parameter that appears to be important and potentially influential in the final result of the coating is the varnish thickness. However, none of the studies previously cited considered this parameter and no data have been found in the literature.

In addition to analytical laboratory techniques, such as the previously cited FTIR, other techniques were applied to identify the varnishes. Of particular interest for the LacCA project are techniques easily accessible to conservators. UV examination and electric resistance tests are mentioned in literature for identifying varnishes on copper alloys and distinguishing them from genuine gilding. Thomson (1991) mentions weak fluorescence on the brassware of the Grecian couch under UV light. In particular, examination under UVA radiation has the advantage of being an affordable and quick way to detect characteristic fluorescence from organic coatings (Cosentino 2015). Unfortunately, no published reference data specifically for copper-alloy substrates were found. This is an issue, as the varnishes might be very thin and

the reflective metallic surface might influence the type and colour of UV fluorescence.

Rogge and Lough (2016) showed that UVA-induced visible fluorescence and false-colour reflected UVA images of tintype varnishes are not sufficient to identify the different varnish materials. The varnishes all emitted a visible fluorescence but were applied on a white metal and not on copper alloys.

Other techniques – like solubility tests or other chemical tests – have been cited (Thomson 1991, Long 2000) but have the disadvantage of being destructive. This is an issue on varnished metallic substrates, as a partial varnish removal might induce local anodic corrosion.

Experimental

Three series of mock-up samples were prepared with different varnishes selected from 60 recipes from 23 ancient manuals, dating from 1803 to 1893 (Table 1). As spirit varnishes are over-represented (72%) and shellac is widely used as at least one binder, it was decided to prepare the varnishes summarised in Table 1. As it was recommended for gold varnishes to avoid interaction with added colourants (Lami and Tharel 1881–91), bleached dewaxed shellac was used for varnish C and D. For comparison with varnish A (thickness and UV imaging), a recipe (B) with coloured shellac was also prepared (Table 2). Mirror-polished and satin-finished coupons, made from CuZn37 alloy (60×60 mm), a common brass alloy, were employed. Prior to varnishing, coupons were degreased with ethanol. An additional aluminium sample was used to compare imaging produced on a white metal. Non-fluorescent glass substrates were used as a reference for UV imaging.

A first series of coupons was produced to determine the range of measurable thickness of the varnishes. Manual dip coating and brush application were chosen because they are mentioned in 19th-century literature (Tingry 1803, Dessaignes 1861). On decorative elements, the surface geometry may be complex, with a thinner layer on the reliefs. Therefore, it was chosen to simulate the thinnest possible varnish by applying only one layer of a simple shellac varnish (A), although several layers might be necessary to replicate ancient recipes (Nosban 1843, Londe 1893). In the case of dip coating, no indication about the removal speed was available in literature so the coupons were immersed vertically and

Table 1. Type of produced (brass) series and characterisation techniques

Series	Code	Substrate finish	Application technique	Varnish recipe	Spectroscopic ellipsometry	Confocal microscopy	Eddy current	UV	FTIR
Series 1	Smd_A-Al aluminium	Satin	manual dip coating	A				×	
Series 1	Smd_A	Satin	manual dip coating	A	×			×	
Series 1	Mmd_A	Mirror-polish	manual dip coating	A	×			×	
Series 1	Mb_A	Mirror-polish	brushing	A	×			×	
Series 1	Sb_A	Satin	brushing	A		×		×	
Series 2	Smd_B	Satin	manual dip coating	B				×	
Series 2	Sb_C	Satin	brushing	C		×			
Series 2	Mb_C	Mirror-polish	brushing	C		×			
Series 2	Sb_D	Satin	brushing	D		×			×
Series 2	Mb_D	Mirror-polish	brushing	D		×			
Series 3	Mid_A2	Mirror-polish	industrial dip coating	A	×		×	×	×
Series 3	Mid_C2	Mirror-polish	industrial dip coating	C	×		×		
Series 3	Mid_D2	Mirror-polish	industrial dip coating	D	×		×		×
Series 3	Mid_A11	Mirror-polish	industrial dip coating	A		×	×	×	
Series 3	Mid_C11	Mirror-polish	industrial dip coating	C		×	×		
Series 3	Mid_D11	Mirror-polish	industrial dip coating	D		×	×		
Series 3	CMid_A (cylinder) 9–10 µm	Mirror-polish	industrial dip coating	A		×	×		

Table 2. Recipes and varnish viscosities

Recipe		Varnish A Blonde shellac (based on Riffault et al. 1862)	Varnish B Orange shellac (based on Londe 1893)	Varnish C Vernis Anglais (Valicourt 1872)	Varnish D Vernis mutatif 'gold varnish' (Tingry 1803) (ounces converted to grams)
Ingredients	Shellac	125 g*	225 g		30.57 g**
	Sandarac			250 g	61.14 g
	Elemi			'big as a hazelnut'	61.14 g
	Mastic			30 g	
	Camphor			'big as a walnut'	
	Turmeric				22.92 g
	Saffron				636.9 mg
	Gummi-gutta				22.92 g
	Dragon's blood				30.57 g
	Alcohol	2 kg	1000 mL	1 kg 'wine spirit'	611.43 g 'pure alcohol'
	Turpentine			30 g	
	Crushed glass				91.71 g
Measured viscosity (cP) according to Bingham model		1.81	2.08	3.34	4.40

* In the original recipe, shellac in flakes was used.

** In the original recipe, seed lac was used.

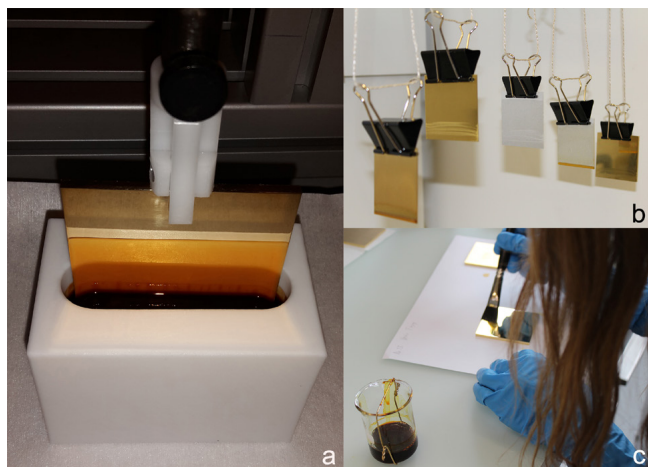removed quickly to be hung up and left to dry at room temperature (Figure 1b).

Figure 1. Varnishing of the samples: (a) automated dip coating, (b) manual dip coating and (c) brushing

The brush application (Figure 1c) was performed according to the procedure indicated in a varnish manual (Nosban 1843): 'Heated in a way it doesn't burn the upper side of the hand…' Prior to varnishing, heating of the substrate in an oven at around 30°C proved to be convenient. This makes it possible to correlate a precise temperature to more general recommendations in the ancient literature, allowing to eliminate blanching or retraction of the varnish which may occur during drying, especially for varnish D. Prior to varnishing, the excess was removed from the brush and the varnish applied in juxtaposed layers using a 10 mm large squirrel-hair brush.

Series 2 was prepared in the same way with more complex recipes: varnish C and varnish D (the latter is mentioned in five French manuals dating from different periods). These were prepared with one of the recommended water bath procedures (Tingry 1803) at around 50°C and the alcohol grade adapted to fit the historical period. All varnishes were filtered using a vacuum pump.

In order to validate the thickness measurement as well as the FTIR protocol, series 3 was produced using an automated dip coating after adjusting the viscosity of the varnish solution. According to the preliminary measurements performed on the first two series, a thickness of around 2–3 μm was selected for all recipes. As thicker varnishes can sometimes be observed on scientific objects, 11–12 μm thicknesses were also produced. In addition to the flat coupons, one brass cylinder (diameter 50 mm) was coated with a 9 μm layer.

A multi-analytical approach was used for characterising the three series of coupons. Cutting-edge laboratory techniques, such as spectroscopic ellipsometry, confocal microscopy and FTIR, were used together with techniques more easily accessible to conservators, such as UV imaging and eddy current (Table 1).

Thickness measurements

Depending on the thickness range, spectroscopic ellipsometry and confocal microscopy were used. Reference zones were determined in order to perform comparative measurements with an eddy-current probe and validate the on-site measurement protocol. Ellipsometry can measure thicknesses smaller than 2 μm locally on polished substrates and was done on a straight line from the top to the bottom of the coupon (perpendicular to the brush application). Mapping with confocal microscopy was used for thicknesses greater than 2 μm.

For series 3, homogeneous zones were located on the samples with a mask and comparative measurements performed with an eddy-current probe (Phynix Surface Pro S gauge and dual FN 1.5 probe). The probe was applied freehand five times at a 90° angle after the recommended calibration process using a brass substrate with an identical geometry and an 8 μm calibration foil. According to the manufacturer, the gauge has a range of 0–200 μm with an accuracy of ± 0.7 μm or 1%.

Infrared spectroscopy in specular reflectance mode

Infrared analyses were performed by specular reflectance (SRefl) using an Alpha portable spectrometer (Bruker) with an R-Alpha module. This measurement configuration permits non-invasive, contactless measurement, but requires a reflective surface in order to obtain meaningful signatures. The samples were placed in front of the spectrometer head at 15 mm working distance. Focusing and selection of areas was performed with an internal video camera. The analysed areas had a diameter of approximately 5 mm.

UV imaging

An unfiltered Canon CN 750 camera with a 60 mm macro lens, a Baader UV/IR cut and an X-Nite CC1 filter were used. The images were taken against a black cardboard background. Two UV lamps[1] (Dominique Dutscher, main emission peak at 365 nm) were mounted

on custom-made stands at 45° and at a distance of 13 cm. The reproducibility of the imaging conditions was ensured by locating all elements on the cardboard and measuring the tilt angle and working distance of the lamps (Figure 2a).
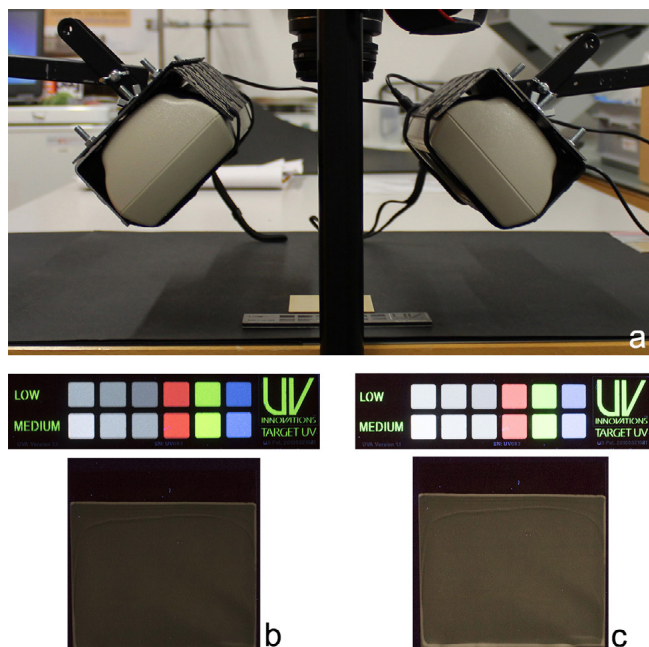

Figure 2. (a) Setting, (b) after image processing and (c) after readjusting exposure time on sample 11

An image of the UV Innovations™ target was taken separately for each series to avoid any reflection of the fluorescent target on the metallic surface. The exposure time (2 seconds) was adapted to the lowest fluorescence level of the target. Image processing was performed later with Photoshop. As the fluorescence level of some samples appeared to be too low for the target (leading to systematic under-exposure of the samples or systematic over-exposure of the target), exposure was increased after the usual image processing by +2. In the last step,

the colour sampling tool was used for each coupon to obtain average L*a*b* values from three zones and to compare the colorimetric data (Figure 2b).

Results and discussion

Thickness measurements

The results of the thickness measurements are summarised in Tables 3 and 4.

Table 3. Series 1 and 2: Thickness measurements and results

Manual dip coating/brushing				
	Varnish recipe	Code	Thickness (µm) Average (full sample)	
			Spectroscopic ellipsometry	Confocal microscopy
Series 1	A	Smd_A	-	< 1.6
		Mmd_A	1.30	-
		Mb_A	1.0	-
		Sb_A	-	< 1.6
Series 2	C	Sb_C	-	< 1.6
		Mb_C	1.8	-
	D	Sb_D	-	2.8
		Mb_D	2.87	-

Due to the low average thickness of the varnishes on series 1 (< 1–2 µm), the majority of the samples were measured using spectroscopic ellipsometry. Satin-finished coupons could only be measured, to a limited extent, by confocal microscopy, since the varnish was too thin on the edges. This means that on satin-finished surfaces, a thicker layer of varnish may be necessary to provide sufficient protection.

Results confirmed that the more viscous the varnish, for varnishes A, C and D (Table 2), the thicker the layer when using the same application technique.

Table 4. Series 3: Thickness measurements and results

Industrial dip coating					
	Varnish recipe	Code	Thickness (µm)/average in reference zone		
			Spectroscopic ellipsometry	Confocal microscopy	Eddy current
Series 3	A	Mid_A2	2.52 ± 0.1	-	2.10 ± 0.3
		Mid_A11	-	11.7 ± 0.6	11.20 ± 0.6
		Mid_A (cylinder)	-	9.47 ± 0.2	9.0 ± 1.2
	C	Mid_C2	2.67 ± 0.1	-	2.3 ± 0.5
		Mid_C11	-	10.3 ± 0.4	9.5 ± 0.8
	D	Mid_D2	3.17 ± 0.3	-	2.5 ± 0.6
		Mid_D11	-	11.7 ± 0.7	11.70 ± 0.2

Both application techniques induced a similar thickness on identical substrates. On the brushed coupons, the partial overlapping of two layers gave maximum values of around 2 µm.

The comparative tests showed that the eddy-current measurements performed manually (freehand without a stand) with adequate calibration ensured the precise measurement of the selected varnishes and metal substrates, especially for flat surfaces. The test performed on the cylinder was also conclusive, but the possibilities on smaller convex forms might be limited.

UV imaging

As shown by UV imaging and the similar a* b* values (Figures 3 and 4) in comparison to the reference brass satin sample, the fluorescence of bleached shellac can be very limited when the coating is extremely thin (< 2–3 µm). As expected, increasing the thickness of the same varnish (A) generates stronger fluorescence, which is evidenced by higher L* and b* values. The bleaching process of shellac can decrease the quantity of fluorophores initially present (Sutherland 2010). As a result, varnish A is much more difficult to detect than the orange reference shellac (B) at a comparable thickness. Varnishes C and D generate a more visible chromatic response even at a lower thickness, which can be explained by the presence of more fluorescent ingredients (organic colourants).

Based on the results presented in Figures 3 and 4, the fluorescence colour obtained on brass was more similar
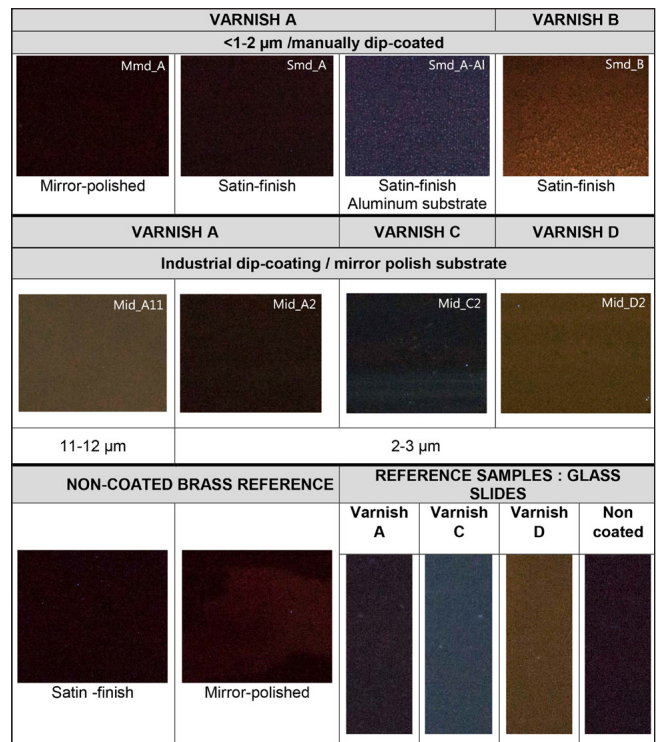

Figure 3. Results of the UV imaging

to the reference colour on the glass slides (ΔE 3.34) in comparison to the aluminium substrate (ΔE 17.80).

Fluorescence was only slightly influenced by the treatment of the metal. The polished sample was more reflective and appeared reddish under the lamp, which was confirmed by the colorimetric values tending towards red on the green-red axis. However, ΔE is low (3.98) for the samples coated with varnish A whatever the metal treatment. Additional comparative tests conducted with varnish D (naturally more fluorescent) on two different metal finishes also did not show any difference.
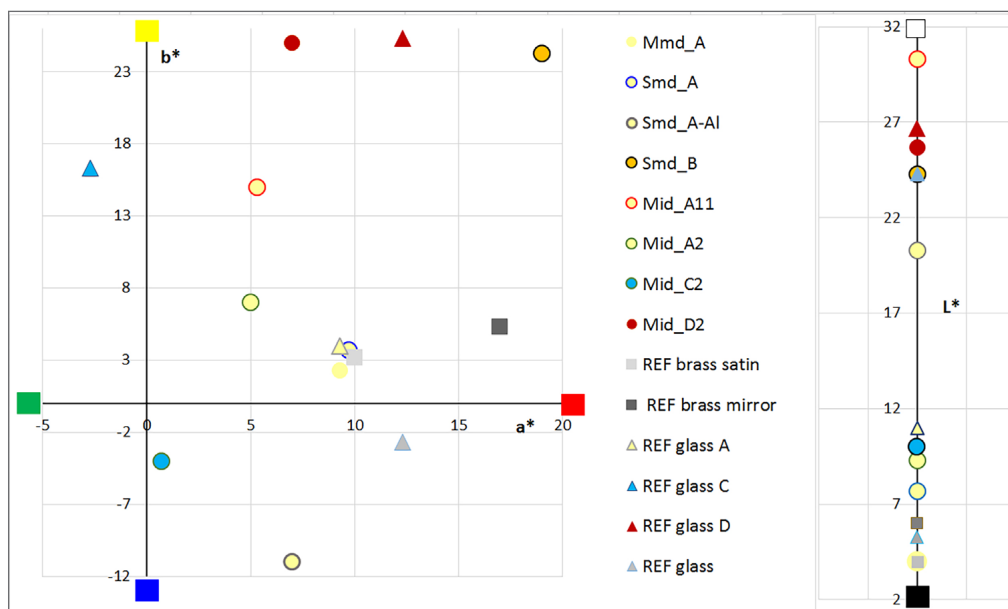

Figure 4. Colorimetric data for the UV imaging

FTIR

SRefl-IR signatures obtained for some of the samples in Table 1 were compared with reference spectra from their ingredients. Measurements were repeated for each sample but no significant differences were noted between each analysis. As showed in Figure 5 for varnish A (Mid_A2) and shellac IR signatures, all spectral features of shellac are recognisable in the varnish spectrum, confirming the ability to characterise single component varnishes.
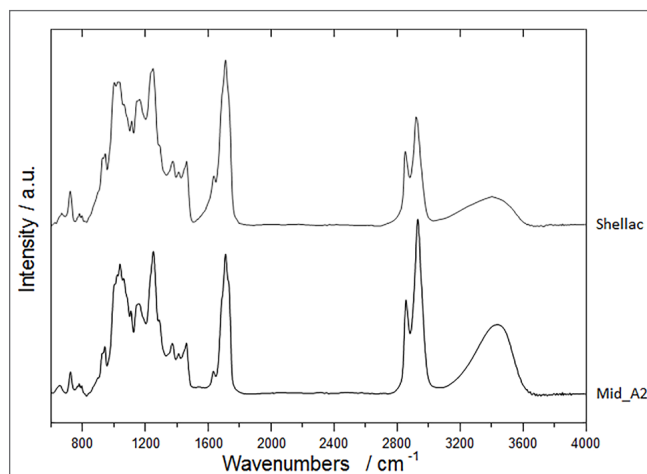

Figure 5. Representative SRefl-IR spectrum from the varnish A sample compared with the reference spectrum for shellac, used for varnish A (baselines have been subtracted).

The reliability of SRefl-IR measurements on metal surfaces appears to be independent of metal finish. This is highlighted in Figure 6 by the similarity of spectra between samples Sb_D and Mid_D2. Therefore, the finish of the object's metal does not have a significant influence on the effectiveness of IR reflection analysis. In addition, it can be noticed that results are satisfying despite the thinness of the varnishes (2–3 μm).

Figure 6 presents the spectra of the components of varnish D. The common features between compounds and varnishes in the SRefl-IR spectra can be observed. The main bands of the principal ingredients, such as elemi and sandarac, are distinguishable in the varnish spectra (bands at 1034, 1375 and 1460 cm^{-1} for elemi, and 888 and 1690 cm^{-1} for sandarac). The main bands of the compounds present in minor proportions, such as shellac and dragon's blood, with the presence of bands at 1242 and 1613 cm^{-1}, respectively, were also identified. Despite the small amounts involved (a few dozen grams), the main bands of gummi-gutta and turmeric were also identifiable in the varnish spectra, at 1633 and 1518 cm^{-1}, thanks to the absence of other interfering bands in these

spectral regions. Finally, the presence of saffron was not detectable in the SRefl-IR spectra of samples Sb_D and Mid_D2. This was doubtless because of the very low proportion of saffron in the recipe (less than 1 gram).
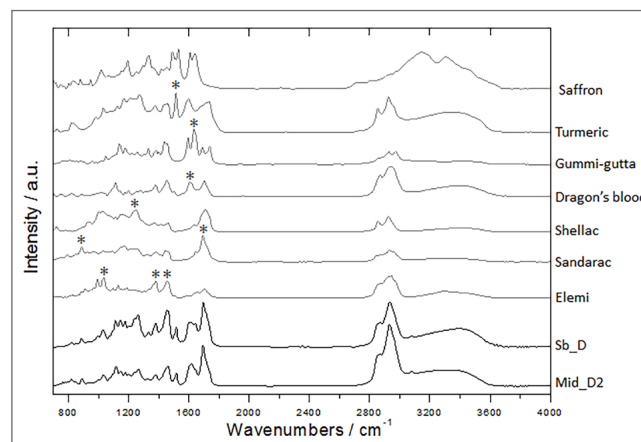

Figure 6. Representative SRefl-IR spectra obtained for varnish D on mirror-polished and satin-finished brass compared with the reference spectra of the ingredients used for varnish D (baselines have been subtracted). The bands cited in the text are marked by *

Complementary analyses were carried out on the same samples (Mid_A2 and Mid_D2) by gas chromatography coupled to mass spectrometry (GC-MS). The results obtained accorded with the recipes. Indeed, for varnish D, the compounds detected could be attributed to elemi, sandarac and dragon's blood. However, these compounds may also have come from a variety of other resins. Therefore, it would be very difficult to attribute them properly without the original recipe. At that stage, no gain in sensitivity was obtained by GC-MS, but this will be further investigated on artificially aged samples.

Conclusion

The tests allowed the chosen analytical techniques (eddy current, UV imaging and FTIR) to be validated for on-site characterisation of varnished copper alloys. The experiment was conducted on mock-ups allowing possible varnish thickness parameters, particularly possible minima, to be taken into account. Eddy-current measurements proved to be an adequate non-invasive method to measure films even as thin as 2 μm on flat surfaces. However, the accuracy of similar measurements on more complex surface geometries needs further investigation.

UV imaging of the samples confirms that this simple tool is useful and efficient to detect the presence of varnish on copper alloy. The resulting colour seems to

be less influenced by brass substrate than white metal. Nevertheless, the varnish thickness may play a crucial role in limiting detection and generates very weak fluorescence. In this case, the imaging protocol needs to be adapted. FTIR analysis shows that the components of both simple and more complex varnish mixtures can be identified in a conclusive way regardless of the type of metal finish.

This paper presents the preliminary results of the LacCA project, which will include analytical campaigns on real varnished objects from four collections (Musée des Arts Décoratifs, Paris; Lausanne Historical Museum, International Museum of Horology, La-Chaux-de-Fonds; and Musée des Arts et Métiers, Paris). Furthermore, the parameters and effects of the substrate preparation and composition will be investigated to determine the effect of the alloy composition and the chemical etching of the surface prior to varnishing. Finally, the varnished samples will be aged artificially in order to mimic the state of historical varnished objects.

Acknowledgements

Notes

1 Window size: 150 × 50 mm.

Materials list

Brass coupons
Kaufmann & Fils
www.kaufmann-fils.ch/

Shellac flakes, orange, no. 60410
Shellac flakes, decolourised, wax-free, no. 60450
Sandarac, no. 60100
Gum elemi, no. 62050
Mastic, no. 60050
Camphor, no. 78710
Turmeric, no. 37220
Saffron, no. 37110

Gummi-gutta, no. 37050
Dragon's blood, no. 37000
Alcohol, no. 70800
Turpentine, no. 62000
Kremer Pigmente
www.kremer-pigmente.com/de

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Authors

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Sigrid Mirabaud has a PhD in analytical chemistry from the University of Lille. Until 2019, she was the head of the research laboratory at the department of conservators at the Institut national du patrimoine and is currently a resident scholar at the Institut national d'histoire de l'art.

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Laura Brambilla has a PhD in chemical sciences from the Università degli Studi di Milano, Italy. Since April 2013, she has worked in the R&D team at HE-Arc CR, first as a scientific collaborator and from 2017 as a professor. She is currently project leader for different projects and one of the organisers of the ICOM-CC Metal 2019 conference.