
Revealing Degradation Patterns: Imaging Techniques for the Study of Metal Soap Formation on Painted Metal Objects

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Abstract

In attempting to document the degradation processes occurring on cultural heritage objects, imaging-based analytical techniques present many advantages, as they provide spatial and spectral information and allow the simultaneous investigation of the chemical and morphological characteristics of a sample. This study presents a protocol based on 2D chemical imaging – Fourier transform infrared microspectroscopy (μ -FTIR) and hyperspectral imaging (HSI) – aimed at monitoring the formation of metal soaps on model metal coupons. Oil-painted metal supports are in fact not immune to degradation due to metal soaps formation, a phenomenon that affects all oil-painted surfaces from the initial curing of the paint film. Copper and zinc sheets were coated with cold-pressed linseed oil and artificially aged for one

month in order to instigate the formation of metal soaps. Their reaction was then monitored by means of μ -FTIR. The chemical maps showed an increasing trend over time, elucidating some aspects and differences in the mechanism of formation of the organic salts for the two metal substrates. Additionally, the samples were analysed using two hyperspectral cameras, operating in the visible-near infrared and short-wave infrared spectral range. The appropriateness of the two cameras in the investigation of metal soaps, and the effect of the thickness of the coating on the data obtained, is discussed here.

Keywords

metal soaps, metal carboxylates, imaging, painted metals, metal conservation

Introduction

Metals, particularly copper, lead, iron and zinc, have historically been common supports for painted artworks due to their unique technical and aesthetic properties in comparison to canvas and wood (Pavlopoulou and Watkinson 2006, Terenzi et al. 2006). Some examples include oil-painted metal artworks popularised during the 16th and 17th centuries by old masters like Rembrandt and Rubens, and later in the 20th century by contemporary artists such as Frida Kahlo, Frank Stella and Alexander Calder. It is worth mentioning that the concept of oil-painted metals is not limited to polychrome metal objects but also encompasses metalwork artefacts covered with waxes, lacquers and oils as protective or finishing coatings (Hughes 1993).

Metal soaps are a class of water-insoluble organic salts (carboxylates) that form ubiquitously on artworks whenever a reactive metal ion is in close contact with fatty acids, which typically come from an oil binder (Hermans et al. 2016). Many scientific studies have been conducted in the past couple of decades to investigate the technical aspects of the formation of such compounds, from the initial chemical reaction (Baij et al. 2018; Casadio et al. 2019, 47; Garrappa et al. 2020) to the aggregation and migration mechanism of the crystalline species towards the surface of the painting (Herman 2016) and the subsequent implications for the preservation of the object (Gabrieli et al. 2017, Banti et al. 2018).

Even though it would be theoretically possible to transfer the knowledge derived from such studies to any oil-painted object, different substrates present unique physicochemical characteristics that must be taken into consideration (Campos et al. 2016). When dealing with the formation of metal soaps within a traditional pictorial film, the analytical focus is centred on the interaction between the metal ions of the mineral pigments and the binder in which they are embedded (Casadio et al. 2019). However, the presence of a metal support acts as a competing source of metal ions, in which case another type of interaction must be identified between the paint layer as a whole and the metal substrate. At a microscopic level, such organic-inorganic reactions occur between the fatty acids in the oil binder and the metal species present in the support, and they are influenced by both internal and external factors that affect the interface between the metal substrate and the oil paint. This adds a layer of complexity to the study of the oil-metal system.

Applying the most common analytical techniques used in the study of metal soaps found in paintings on canvas would imply sampling and embedding in a suitable resin as a cross-section (Kaszowska et al. 2013; Keune and Boon 2013; Hermans et al. 2018; Casadio et al. 2019, 195; Ortiz Miranda et al. 2020). In the case of painted metals, the paint alone would not give exhaustive information to shed light on the system's complexity, as the reaction of interest occurs at the interface between the metal and the paint layer (Pavlopoulou and Watkinson 2006). However, sampling the metal support together with the paint raises both technical and ethical concerns, as it can provoke irreversible damage.

The approach for a suitable elucidation of the degradation processes occurring on such objects must then involve the use of non-destructive techniques (NDT) for the investigation of the interface between the metal and the paint. A combination of well-chosen NDT can provide the in-depth information necessary to carry out conservation-restoration treatments (Cucci et al. 2019, Hennen Rodriguez et al. 2020).

For this study, Fourier transform infrared microspectroscopy (μ -FTIR) was used to investigate the painted metal model samples. This analytical technique can be used both to characterise metal soaps as well as generate chemical maps to understand the relative distribution of the carboxylates (Gabrieli et al. 2017), allowing comparisons between different regions of an artwork and an

understanding of the degradation pathways as the reactions progress. Specifically, artworks can be sequentially monitored by obtaining spectra of the same region of interest to generate a time series of chemical images.

Some considerations on the use of hyperspectral imaging (HSI) devices for the study of oil-painted metals are also presented. HSI techniques are a set of non-invasive, non-destructive techniques based on the acquisition of separate images at narrow intervals of wavelengths (< 10 nm), called bands, that can then be analysed as a function of the chemical composition of an object (Amigo 2020). Although HSI systems are not always readily available, they have proven successful in the field of cultural heritage to identify pigments or binders, or to monitor conservation treatments. Specifically, pigments have a strong characteristic signal in the visible and near-infrared range (VNIR, 400–1050 nm), whereas binders typically have a strong spectral signature in the short-wave infrared spectral range (SWIR, 1050–2500 nm) that makes their identification possible (Cucci et al. 2019, Amato et al. 2021).

The search for diagnostic signals of the presence of metal soaps in reflectance spectra acquired in the VNIR and SWIR spectral regions is described here. Such indicators could manifest themselves in changes in the shape or intensity of the signal that could imply a chemical variation due to the formation of metal soaps.

Materials and methods

Sample preparation

Model samples were prepared for artificial ageing in triplicates as $60 \times 60 \times 1$ mm hair-brushed copper and zinc sheets. The hair-brushed finishing – an industrial process which consists in utilising an abrasive material to create unidirectional lines giving a satin finish – was chosen to mimic the artists' common practice of scratching the surface of the metal prior to painting to increase the grip of the paint on the metallic support. The samples were degreased with absolute ethanol (VWR Chemicals) and coated with ~ 80 μ L of cold-pressed linseed oil (Kremer Pigmente). The linseed oil was applied on the metal surface with the aid of a rectangular painter's spatula.

Coating thickness evaluation

The low affinity of oil and metal for one another did not allow homogeneous coatings to be obtained, despite the

standardisation of the samples' preparation and coating procedure. The samples presented evident differences in coating thickness, a feature whose effect is intended to be taken into consideration in this study. The inhomogeneities of the oil coating were measured using the coating thickness gauge Surfifix by Phynix, resulting in ranges of thickness from 2 to 15 μm for the copper and from 3 to 25 μm for the zinc samples.

Accelerated ageing

The coated samples were cured overnight at 80 °C then artificially aged at 80 °C and 80% relative humidity in a laboratory oven for 30 days. The triplets were kept in a sealed box at constant relative humidity using a glycerol-water emulsion (50% w/w) (Forney and Brandi 1992) and monitored by means of μ -FTIR at spaced time intervals during 30 days. The samples were then left at room temperature and relative humidity for an additional 11 months. The levels of relative humidity and temperature were monitored using a PeakTech 5185 datalogger equipped with an immersion probe.

Fourier transform infrared microspectroscopy (μ -FTIR)

Fourier transform infrared spectra were collected using a Thermo Scientific Nicolet iN10 MX infrared microscope equipped with a motorised stage. For each sample, single scan spectra were acquired in reflectance mode (650–4000 cm^{-1}) with an aperture of 150 \times 150 μm^2 , step of 100 μm and at a resolution of 4 cm^{-1} on three different areas (7500 \times 1800 μm^2). Spectra elaboration was achieved with Omnic proprietary software and then visualised using the *ggplot2* package in the R free software environment for statistical computing and graphics (www.r-project.org/). Baseline, normalisation and atmospheric corrections were performed to remove residual signatures of atmospheric CO_2 and water in the spectra. Chemical heatmaps were generated in the spectral range of 1525–1650 cm^{-1} to cover the main vibrational bands corresponding to metal soaps ($\nu_a \text{COO}^-$). The results were comparable in all three areas analysed, hence only one of the regions of interest will be discussed in this work.

Hyperspectral imaging (HSI)

Twelve-month-aged samples were analysed with push-broom HySpex VNIR-1800 and SWIR-384 hyperspectral cameras by Norsk Elektro Optikk. The acquisition

geometry was standard at 45°/0° and the focus was set at 30 cm, with a field of view of 16°, yielding a pixel size of approximately 50 μm . The model samples and 99% white diffuse reflectance standard (Spectralon) were placed in-plane on a flat translational stage with mounted broadband spectral coverage tungsten-halogen lamps at 45° and the camera at 90°. Acquisitions were performed in the dark with the white standard playing the role of a wide spectral range Lambertian reflector with uniform response, to correct for the reflectance of the light source.

For the extraction of the spectra, an area of analysis of 1000 \times 1000 μm^2 (20 \times 20 px²) was chosen as a good compromise between signal quality (S/N ratio) and occurrence of representative regions of interest of comparable size within the sample. The bundled OEM software was used to implement dark current correction and calibrate the sensor in the pre-processing stage of the dataset. HypspecRadV2.0 software was used to convert the raw data into radiance pixel values. The processing of the data to obtain interpretable and comparable relative reflectance spectra was carried out using the ImageJ open-source software. After re-slicing of the datacube at the top avoiding interpolation, the data acquired both in the VNIR and SWIR ranges were corrected for illuminance, i.e. the dataset was divided by the radiance values of the Spectralon white diffuse reflectance standard, after stacking, re-slicing and adjusting its size to that of the samples (Babini et al. 2021).

Results and discussion

Fourier transform infrared microspectroscopy

According to previous studies (Hermans et al. 2019 and 2021), several coordination geometries of the most common zinc carboxylates can be detected by FTIR spectroscopy in the range of 1525–1650 cm^{-1} (Table 1).

By observing this spectral region, the formation of metal soaps at an early stage was monitored on the zinc and copper samples during a period of 30 days (Figure 1). It can be seen that the evolution over time of the intensity of the signal of the characteristic $\nu_a \text{COO}^-$ vibrational bands for the copper and zinc carboxylates increased.

Here, it was not possible to distinguish between different amorphous and crystalline forms of copper and zinc metal soaps, as only the signal of the general ionomer structure is visible (Hermans et al. 2016). However, from the qualitative monitoring using heatmaps, important

Table 1. Major asymmetric stretching vibrational bands for the different metal carboxylates and coordination geometries in the range of 1525–1650 cm^{-1}

		Zinc	Copper	Vibrational mode	References
Crystalline	Palmitate	1539–1536 s	1586 s	$\nu_{\text{as}}[\text{COO}^-]$	Izzo et al. 2021; Hermans et al. 2019 and 2021
	Stearate	1540–1539 s	1586 s		
	Oleate	1547 vs, 1527 vs	1588–1584 vs		
	Azelate	1556–1535 vs	1588 vs		
	2D coordination polymer	Type A	1524 s, 1545 s, 1590 w		
Type B		1536 s			
Amorphous	Oxo	1590 s		$\nu_{\text{as}}[\text{COO}^-]$	Izzo et al. 2021
	Chain	1544 s, 1565 w, 1630 s			

vs = very strong signal; s = strong signal; w = weak signal

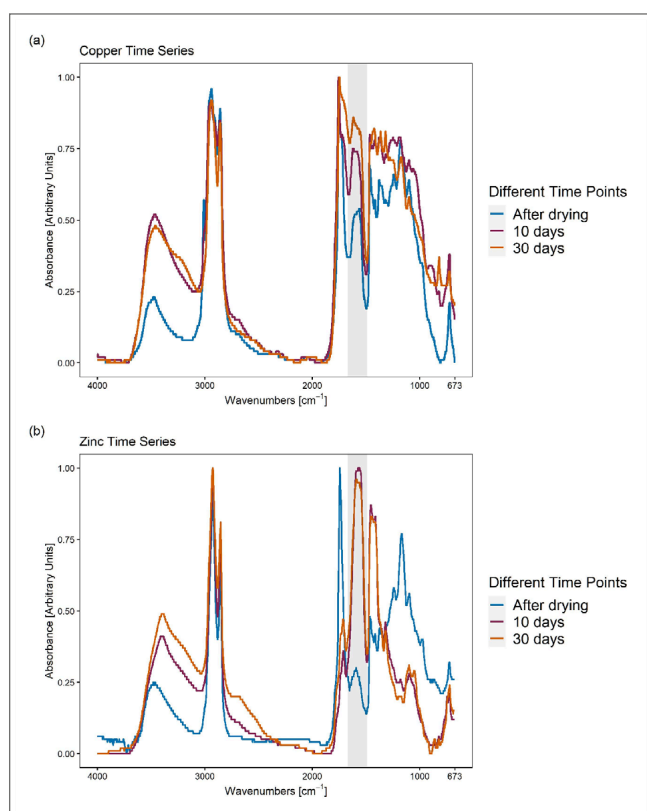


Figure 1. FTIR spectra for linseed oil-coated (a) copper and (b) zinc samples in the time intervals considered. The grey area indicates the vibrational bands related to the metal carboxylates

features were observed (Figure 2): in both substrates investigated, the intensity of the signal of the metal soaps increased in the time window considered, confirming what was observed by inspection of the point spectra. Additionally, differences in the reactivity of the two substrates could be highlighted, with zinc having higher reactivity compared to copper. This can be seen in the occurrence of red areas in the heatmaps (Figure 2c, 2d), and it is supported by previous knowledge of the reactivity of the two ions (Greenwood and Earnshaw 1997).

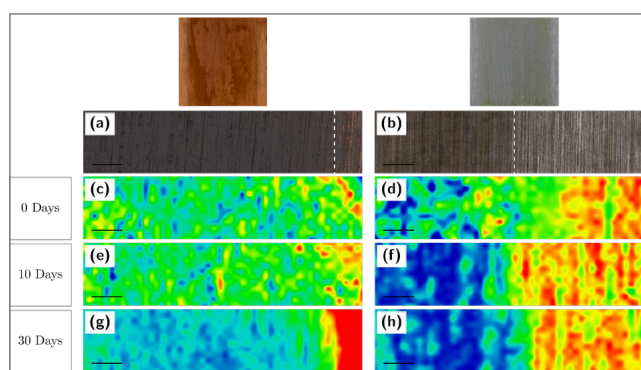


Figure 2. Copper and zinc coupons coated with linseed oil and respective analysed regions of interest (a, b). The dashed line separates areas showing higher (darker, $> 10 \mu\text{m}$) and lower (lighter, $< 10 \mu\text{m}$) coating thickness. The time series of μ -FTIR chemical images are obtained in the range of $1525\text{--}1650 \text{ cm}^{-1}$ after drying over night at 80°C (c, d), 10 days (e, f) and 30 days (g, h). Intensity range goes from red (maximum) to blue (minimum). The scale bar indicates $1000 \mu\text{m}$

It must be noted that the detection of metal soaps is also a function of the thickness of the oil, which could impede their detection when values are higher. For this reason, the metal soaps have a higher signal in thinner layers of the coating (Figure 2a, 2b) for both substrates.

Additionally, due to the lack of distinct features on the coated surfaces, the reproducibility of the selected areas of analysis was not always achieved when monitoring over time. In particular, the process of placing the samples on the stage manually was bound to introduce errors in consecutive analyses. This can be observed in the resulting heatmaps.

Hyperspectral imaging

From the analysis of the dataset, it was not possible to identify spectral features for the presence of metal soaps in the VNIR and SWIR spectral ranges, since these did not show characteristic signals in this range of wave-

lengths. Nevertheless, from the analysis of the spectra, relevant observations could be obtained, highlighting differences in the substrate and coating responses in the two ranges of wavelengths considered. In particular in the VNIR spectral range, it was possible to identify the characteristic reflectance spectra of the copper and zinc bare metals (Sárosi et al. 2010). As the thickness of the coating increased, the relative reflectance values, shape and position of the peaks of the metal support changed. For the copper substrate, the presence of the coating influenced the shape of the signal and its intensity, but it affected its position only marginally (Figure 3).

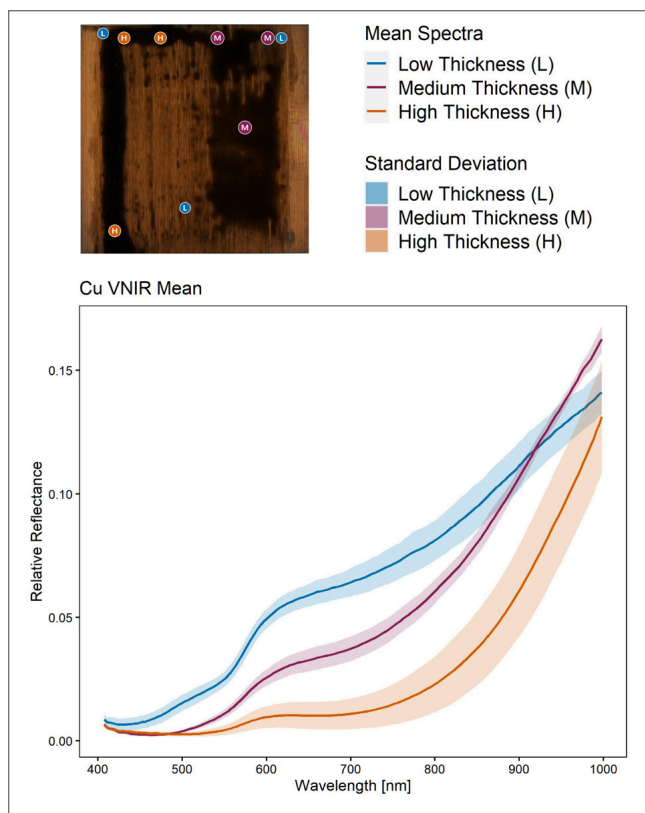


Figure 3. VNIR HSI analysis of a copper sample coated with a non-homogeneous layer of linseed oil. Average relative reflectance spectra obtained from three 20×20 px² areas for low (1–5 μm), medium (5–10 μm) and high (10–25 μm) coating thicknesses, and respective standard deviations, are shown

For the zinc, where the colour of the metal is significantly different from that of the coating, a shift in the main peak was visible, corresponding to the variation in colour of the coating from yellowish to reddish (bathochromic shift) as the thickness increases (Figure 4). This suggests that VNIR hyperspectral imaging could be used to determine the thickness of the oil coating indirectly.

In the SWIR range, where the oil binder has a strong signature signal, the characteristic spectrum of the

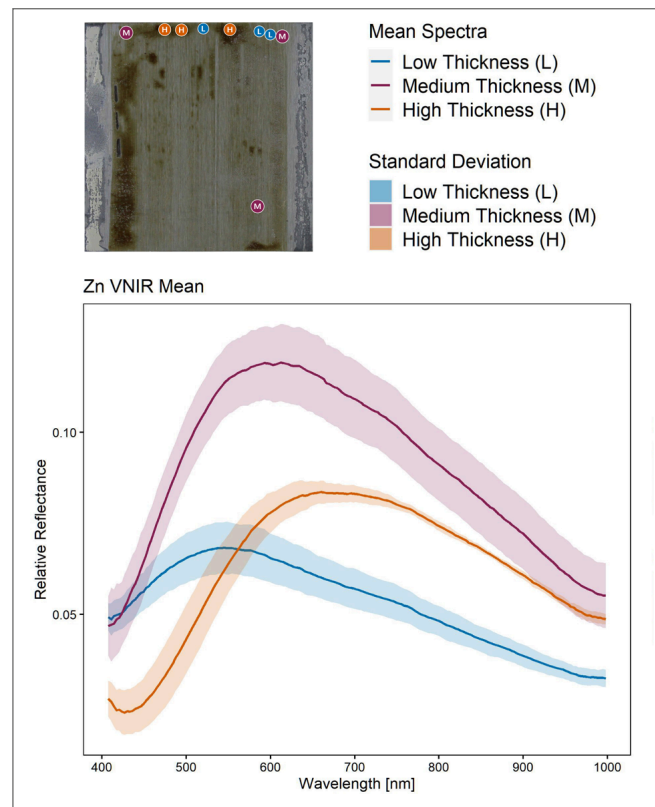


Figure 4. VNIR HSI analysis of a zinc sample coated with a non-homogeneous layer of linseed oil. Average relative reflectance spectra obtained from three 20×20 px² areas for low (1–5 μm), medium (5–10 μm) and high (10–25 μm) coating thicknesses, and respective standard deviations, are shown

drying oil was observed (Amato et al. 2021), with relative reflectance values increasing with the thickness of the oil coating (Figure 5).

The SWIR camera could therefore be a complementary tool to the VNIR camera and could be employed for a direct estimation of the thickness of the oil.

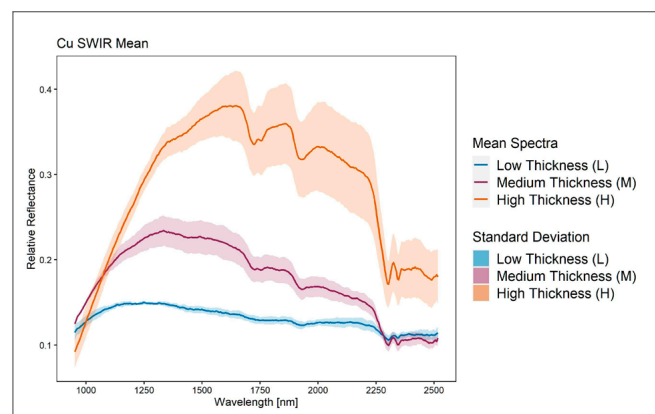


Figure 5. SWIR HSI analysis of the copper sample coated with a non-homogeneous layer of linseed oil (refer to Figure 3). Average relative reflectance spectra obtained from the three 20×20 px² areas for low (1–5 μm), medium (5–10 μm) and high (10–25 μm) coating thicknesses, and respective standard deviations, are shown

Conclusion

In this work, the question of whether the selected non-destructive imaging analytical techniques – namely μ -FTIR and HSI – are suitable for the study of metal soaps on painted metal objects was examined. In particular, the focus of the work concerned the ability of such analytical techniques to allow monitoring of the formation of metal soaps. Micro-FTIR proved to be determinant in both the detection and the monitoring of metal soaps on the copper and zinc model samples. In particular, it was shown that metal soaps can be detected immediately after the application of the oil coating. The intensity of the monitored νCOO^- vibrational bands corresponding to the different metal soap forms increased in the time frame considered. By utilising this technique, the differences in the reactivity of the two substrates could also be highlighted.

The detection of metal soaps using HSI cameras in the VNIR and SWIR spectral ranges was not successful due to the absence of identifiable spectral features in these regions. However, this technique raised questions with regard to the substrates and the thickness of the oil coating that could be extended to different applications in the field of cultural heritage, e.g. the study of unpigmented oil-coated technical objects. For the purpose of this paper, the focus was on qualitative monitoring, but this could readily be extended to a full statistical analysis.

In future research, the mid-wave infrared (MWIR) range should be explored for the HSI analysis of metal soaps, as MIR reflectance spectra are reported for these compounds.

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