What about discoloration in plastic artifacts? The use of Fiber Optic Reflectance Spectroscopy in the scope of conservation

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ABSTRACT

Fiber Optic Reflectance Spectroscopy (FORS) is a well-established technique for the study of traditional artworks. Nevertheless, its application for the analysis of modern and contemporary materials such as plastics is still a pioneering line of research. In this work, the application of FORS for the discoloration assessment of cultural heritage plastics is presented. The spectroscopic method successfully characterized the discoloration of a selection of naturally aged historical plastic objects in situ, and the results of yellowing and fading measurements are discussed. Even though further research studies are required to elucidate the potentialities and limitations of FORS in the investigation of cultural heritage plastics, this study paves the way for its application as a preliminary tool for the detection of incipient and severe discoloration in the plastic heritage caused by polymer and colorant degradation (e.g. yellowing, darkening and fading). The results suggested that FORS may be considered a reliable method for in-situ rapid characterization and monitoring of cultural heritage plastics degradation phenomena in museums in the scope of collection care.

KEYWORDS Historical plastic objects, Discoloration, FORS, In situ analysis, Colorimetry

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1. Introduction

Man-made polymers are increasingly used in modern and contemporary art and plastic artifacts are now common in heritage collections worldwide. Those usually include designer pieces, artworks and mass-produced objects. Many plastics have stability issues that limit the lifespan and, at present, their conservation is a crucial concern in heritage science.

Plastics are available in a wide range of formulations and are generally made by mixing a base polymer together with property and processing modifiers (additives) (Salamone, 1999). This process also includes the coloring, and colorants (i.e. dyes and pigments) are commonly used for the mass coloration of plastics (Charvat, 2004; Müller, 2003; Webber, 1979).

Therefore, color is one of the key elements that determine the visual appearance of a plastic object and the study of its alteration is a research priority as a wide range of polymer-based objects already displays color modifications (Quye and Williamson, 1999; Shashoua, 2008, Lavédrine *et al*., 2012). Among the different causes of deterioration, light is one of the most effective. Following exposure to light, polymers and/or colorants may undergo different degradation phenomena and their alteration, alongside with surface soiling, can be responsible for the plastic discoloration. The degradation of the polymer and its consequent discoloration (yellowing and darkening) has been widely studied (Hawkins,1984; Allen and Edge, 1992; Rabek, 1995), while little is known on the fading of colorants (Allen and McKellar, 1980). The effect of light usually result in changes of the original objects' appearance. Besides chemical changes visible through color variation, the absorption of light may also induce physical damage resulting in the plastic surface embrittlement and microcracking (Micheluz *et al*., 2021).

The detection of the earliest stages of discoloration can support the preservation of entire collections and help to define better conservation strategies. To this end, in situ and rapid methods of analysis would be an ideal tool for performing a preliminary investigation of the objects.

Fiber Optic Reflectance Spectroscopy (FORS) is a well-established technique in the study of objects surfaces and color analysis of traditional artworks on parchment, paper, textiles, stained glass/windows, wall, canvas, and panel paintings. The interests in the application of FORS in studying plastic-based objects is testified by the increasing number of publications on the subject (Cucci *et al*., 2013; Izzo *et al*.; 2019; Pintus *et al*., 2021). However, to the best of the authors' knowledge, it has never been applied to the study of plastic discoloration in the cultural heritage field.

This study intends to present preliminary results of analytical study carried out using FORS on discoloration in naturally aged historical plastic objects. These data made it possible to insight into the discoloration that develops from polymer and colorant degradation. Color measurements were also made to characterize the color variation.

2. Materials and methods

2.1. *Cases studies*

The selection of case studies includes a series of food containers with a red lid made of polyethylene (PE) (Fig. 1i, ii) and polystyrene (PS) (Fig. 1iii), and a naturally aged white telephone made of acrylonitrile butadiene styrene (ABS) (Fig. 1iv), probably produced in the second half of the 20th century.

The red plastic elements and telephone were specifically chosen because show evident discoloration, namely fading and yellowing, respectively. Due to the presence of Portuguese inscriptions such as 'Grão' (chickpea), 'Arroz' (rice), a Portuguese production can be attributed to the food containers.

Those objects were gathered from a private collection, which includes more than three hundred objects entirely made of plastic elements. The collection contains items from Portuguese plastic industry from the 1930s to 2000s and was studied as part of the research project "*The Triumph of Bakelite - Contributions for a History of Plastics in Portugal*" (Callapez, 2017), which contributed writing the history of the Portuguese plastic industry (França de Sá *et al*., 2020).

Discarding surface effects such as grime, dust, deposits on the surface, substance migration and absorption from adjacent surfaces, the observed discoloration can most likely be related to light-induced damage. Unfortunately, information on past storage and exhibition history of the red plastic elements and telephone are not available and contributions of relative humidity and temperature to the plastic degradation cannot be excluded.

2.2. *Fiber Optic Reflectance Spectroscopy (FORS)*

FORS measurements were recorded using a reflectance spectroanalizer MAYA 2000 Pro (Ocean Optics, USA), with single beam optical fibers QR200-12-MIXED (Ocean Optics, USA), equipped with a linear silicon CCD detector Hamamatsu and a halogen lamp HL-2000-HP (20 W output) (Ocean Optics, USA) in the 380-1000 nm range. The analyses were obtained with 8 ms integration time, 15 scans, 8 box width, and acquired at 45°/45° measurement geometry. Calibration of the spectrophotometer was

performed using a 99% Labsphere Spectralon diffuse reflectance standard.

2.3. *Color measurements*

Microflash mobile colorimeter DataColor (DataColor International, Switzerland) was employed for measuring the color of the red historical lids. The colorimeter is equipped with a Xenon lamp and the 1976 CIELAB color coordinates (L*, a*, b*) were calculated over an 8 mm diameter measuring area, considering the D65 standard illuminant and the 10° colorimetric observer (CIE 1964). The reflected specular component was excluded (SCE mode) in the measurements. The instrument was calibrated with a white (100% reflective) and black (0% reference) balance, in accordance with the DataColor calibration procedure. The white (porcelain) and black trap standards were provided by the manufacturer. The reported values are the average of three measurements, which proved to be sufficient to guarantee reproducibility. The color measurements of the naturally aged telephone were obtained from FORS spectra as the area of analysis was too small for the portable colorimeter. The 1976 CIELAB color coordinates (L*, a*, b*) where extracted from the Vis reflectance spectra (400-700nm) considering the spectral distribution of D65 standard illuminant and the color matching functions CIE 1964 standard colorimetric observer (10° standard observed) with 10 nm of step (Oleari, 2016).

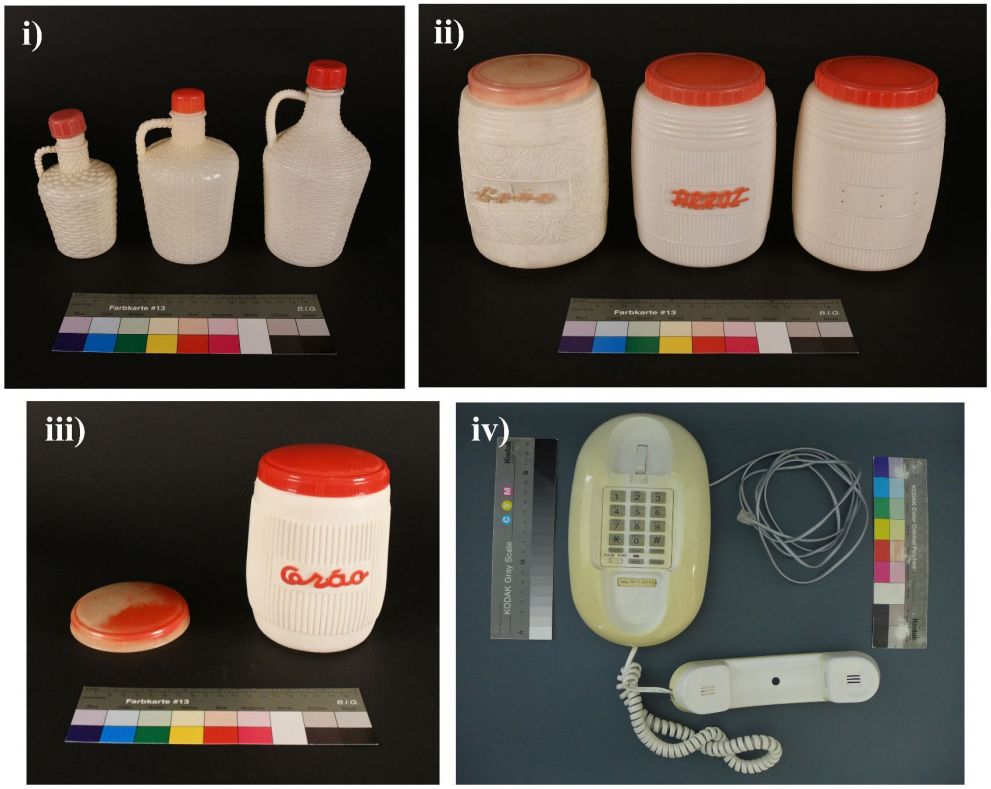

Fig. 1. The historical plastic objects under study, food containers with PE (i, ii) and PS red lids (iii), and ABS telephone (iv).

3. Results and discussion

In-depth material characterization of the red lids by means of infrared and Raman spectroscopy was recently conducted (Angelin *et al.*, 2021a) and the investigation revealed that Pigment Red 53 (5-Chloro-2-[(2-hydroxy-1-naphthalenyl)azo]-4-methyl-benzenesulfonic acid, $C_{17}H_{12}ClN_2O_4S^-$, C.I. n. 15585) is the main red colorant in mixture with Pigment White 6 (titanium dioxide TiO_2, C.I. n. 77891).

The synthetic organic pigment is found either faded (Fig. 2i, ii, iii) or well preserved (Fig. 2iii). Among the plastic samples, only lid **h** made of PS is not discolored (Fig. 2iii) which is characterized by the highest positive a* values and strong absorption in the blue-region (400-550 nm) due to π–π* and n–π* electronic transitions of its hydrazone/keto and azo/enol tautomeric forms, respectively (Hunger and Schmidt, 2018; Angelin *et al.*, 2021b). The s-shape profile of the spectrum was already observed for other modern red synthetic pigments based on *β*-naphthol (2-naphthol) - such as Pigment Red 1 (C.I. n. 12070), Pigment Red 3 (C.I. n. 12120), Pigment Red 48 (C.I. n. 15865), Pigment Red 49 (C.I. n. 15630), Pigment Red 57 (C.I. n. 15850) (Lewis, 1988; Feller, 2001). When fading occurs, significative

changes in the FORS spectra and in the colorimetric coordinates are observed. The absorption in the blue-region (400-550 nm) gradually disappears up to the completely loss of the s-shape (Fig. 2). This matches with the loss of the red color and whitening of the plastic elements as confirmed by the color measurements which show an increase of the L* coordinates and a decrease of positive a* values (Table 1). Fading of *β*-naphthol-based Pigment Red 48 in artificially UV-aged polypropylene (PP) films showed analogous spectral and color variations (de Freitas Brito Cavalcanti and Silveira Rabello, 2019). The final color of the red plastic lids is determined by the superimposition of the individual contribution of the Pigment Red 53 and Pigment White 6 as the polymeric matrixes does not absorb in the visible range (indeed plastics are nearly colorless) (Webber, 1979). As fading of the plastic lids becomes more severe, a shift of the inflection point towards lower wavelength is observed along with the appearance of an absorbance band between 400 and 550 nm (Fig. 2i, ii). Similar trends in reflectance measurements were already observed when parent organic pigments were mixed in different proportion with titanium dioxide pigment (Lewis, 1988). It would be likely related to the increase contribution of the white pigment at the expense of the red colorant.

While most of the lids show a homogenous discoloration, the lids **d** (Fig. 2ii) and **g** (Fig. 2iii) are instead characterized by a spotty pigmentation. For these two objects, significant differences in the FORS spectra and colorimetric coordinates between points of same plastic lid were observed. Where the color is almost completely faded (points 1 of lid **d** and **g**), an increase of the positive values of coordinate b* was detected likely due to the contribution of the polymer yellowing.

Sample	L* (σ)	a* (σ)	b* (σ)
a	44.41 (0.04)	25.63 (0.07)	8.68 (0.10)
b	41.13 (0.06)	35.13 (0.54)	13.71 (0.29)
c	40.71 (0.11)	41.23 (0.05)	17.38 (0.12)
d_1	60.76 (0.58)	5.50 (0.07)	7.49 (0.11)
d_2	47.91 (0.03)	23.89 (0.05)	11.23 (0.15)
e	41.69 (0.24)	29.23 (0.19)	11.03 (0.03)
f	41.98 (0.04)	36.06 (0.04)	13.21 (0.02)
g_1	57.72 (0.20)	4.17 (0.05)	15.26 (0.03)
g_2	34.40 (0.43)	40.94 (0.04)	22.93 (0.14)
h	37.20 (0.18)	48.77 (0.10)	26.16 (0.14)

Table 1. Average values and standard deviations (σ) of the naturally aged PE and PS red lids colorimetric coordinates.

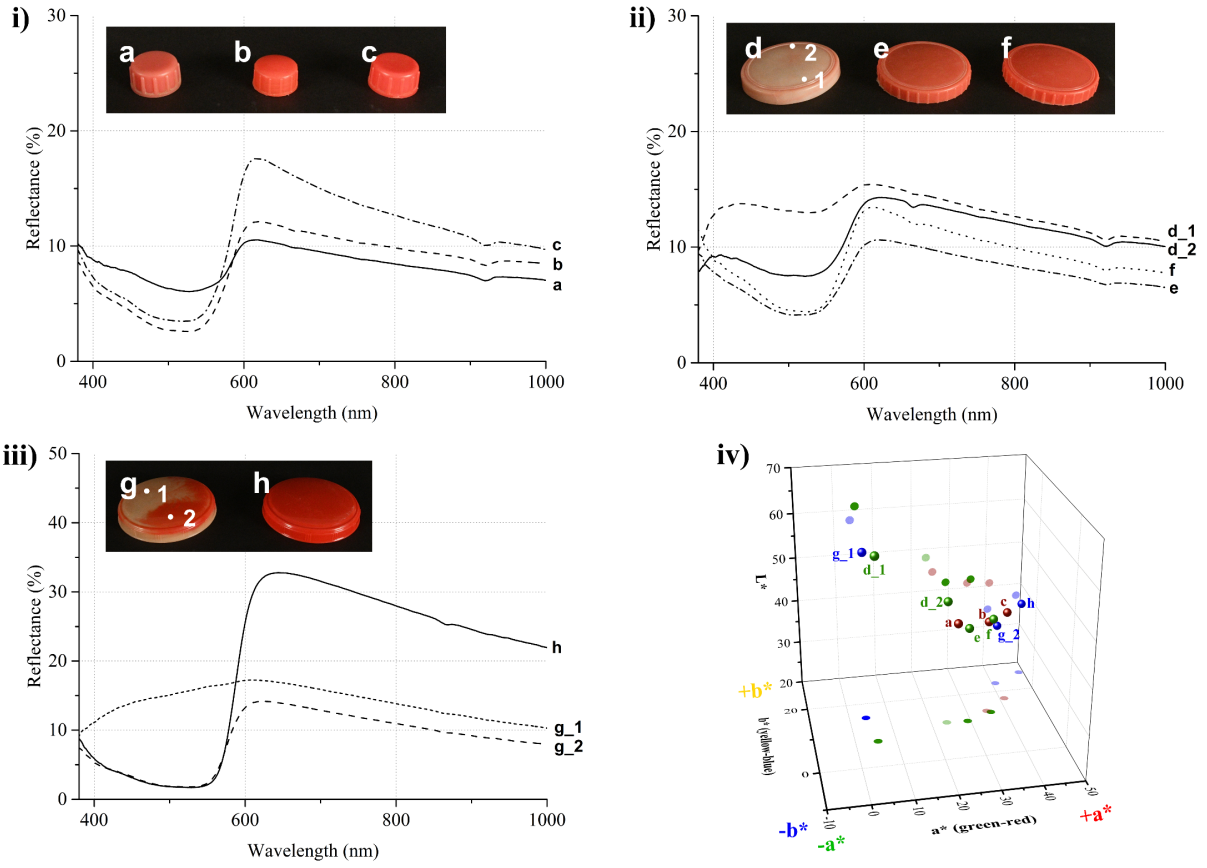

Fig. 2. Naturally aged PE (i, ii) and PS (iii) red lids FORS spectra and their L, a*, b* values in 3-dimensional CIELab76 Color Space (iv). Projections of the points along L* vertical axis and a* and b* perpendicular horizontal axes are also reported.*

The ABS telephone is characterized by areas with different degrees of yellowing. The bottom part of the handset, as well as its housing, are "pristine" white due to limited exposure to light (Fig. 1). The top of the handset and of the entire object are completely discolored (Fig. 3i, inset).

Interestingly, one can observe a gradient of yellowing along the side of the handset, which corresponds to areas more or less exposed to direct light. The portion of the handset that partially fits inside the telephone housing, and hence is less exposed to light, seems of the same color of the bottom (Fig. 3i, points 1 and 2), whilst the upper portion shows intense yellowing (Fig. 3i, points 3-5). The application of FORS enabled the detection of spectral differences among the analyzed points on the side of the handset (Fig. 3) and the base for housing was used as a reference for the original color. By comparing the reflectance spectra, it is possible to note that the slope of the rising edge between 400 and 450 nm progressively disappear from the whitest to the yellowest areas (points 1 and 5 respectively). This results in a shift towards higher wavelengths of the maximum of the reflectance, enhancing the yellow component. Similar reflectance drop on the first tens of nanometers as a result of yellowing was also observed after both artificial and natural light aging of ABS polymer plates (Signoret *et al.*, 2020). Although points 1 and 2 in the telephone transmitter appeared white at the naked eye, the reflectance spectra profile indicates an incipient yellowing, probably in an early stage. The formation of new chromophores as a consequence of the photo-oxidation of the ABS can be responsible for yellowing at the phone surface (Boldizar and Möller, 2003; Jouan and Gardette, 1992; Saviello *et al.*, 2014).

The colorimetric analysis showed a significant color variation between the analyzed points (Fig. 3ii). The exposed areas are characterized by higher positive b* values with progressively lower L* values, which indicates respectively yellowing and a slightly darkening of the plastic (Table 2).

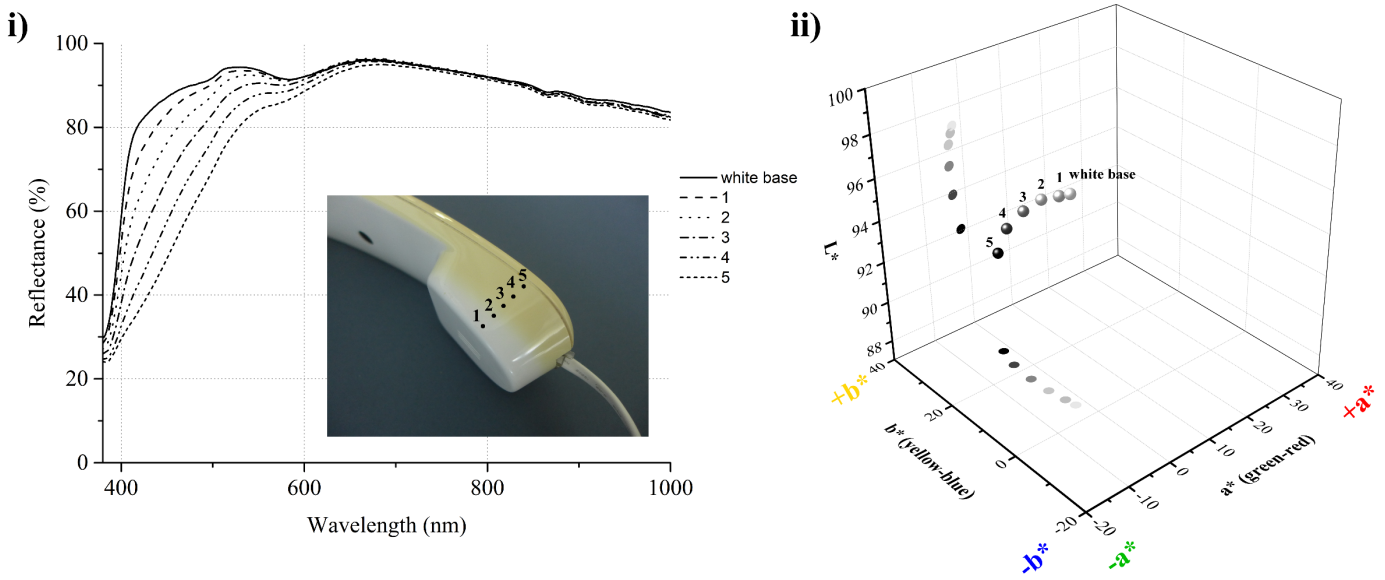

Fig. 3. FORS spectra of the naturally aged ABS telephone (i), and their L, a*, b* values in 3-dimensional CIELab76 Color Space (ii). Projections of the points along L* vertical axis and a* and b* perpendicular horizontal axes are also reported.*

Sample	L* (σ)	a* (σ)	b* (σ)
White base	97.08 (0.02)	-2.03 (0.10)	4.35 (0.07)
1	96.79 (0.01)	-2.48 (0.12)	6.93 (0.03)
2	96.28 (0.03)	-3.12 (0.20)	11.31 (0.05)
3	95.29 (0.03)	-3.34 (0.09)	16.56 (0.02)
4	93.88 (0.01)	-2.99 (0.15)	22.53 (0.02)
5	92.07 (0.02)	-1.69 (0.17)	27.66 (0.03)

Table 2. Average values and standard deviations (σ) of the naturally aged ABS telephone colorimetric coordinates.

4. Conclusion

This study proves the suitability of FORS for the characterization of discoloration in historical plastic objects. The fading of a selection of PE and PS red lids and yellowing of a naturally aged ABS telephone were successfully detected.

FORS demonstrated to be suitable in detecting different levels of fading severity, which correspond to different stages of degradation. In the case of organic pigments based on *β*-naphthol, the shift of the inflection points toward lower wavelengths could be used as an indicator of

the fading rate. The loss of the characteristic s-shape profile indicates the complete degradation of the organic pigment. Contribution of the yellowed PE matrix to the faded color of the plastic elements was observed at the highest /most severe stage of deterioration, where the red color was almost completely lost.

A degree of superficial yellowing was visible on the side of the telephone transmitter. In areas where the telephone results still preserved (and white), FORS revealed an incipient stage of yellowing. Thus, FORS measurements could be considered a useful preventive tool for detecting early degradation phenomena not detectable by simple visual inspection.

This work tackles the conservation problem of discoloration in plastics in collections. Only a few studies have already discussed the topic, and this work opens new avenues in the use of FORS for the assessment of discoloration in plastic heritage. Being FORS able to detect alterations also even before the chromatic effects become visually evident, its application can represent a preliminary step in a multi-technique protocol to investigate the discoloration of plastic objects in museum collections and a reliable analytical tool to monitor the progress of degradation. The detection of discoloration by FORS would inform effective conservation strategies and guide conservation scientists to further and more careful scientific investigation. Indeed, additional and complementary analytical methods can be selected to fully grasp the polymer degradation fully (e.g. Fourier transform infrared spectroscopy, FTIR) (Angelin *et al*., 2021c) and colorant alteration (e.g. pyrolysis gas chromatography/mass spectrometry (Py-GC/MS)) (Micheluz *et al*., 2021).

The interaction between ultraviolet-visible-near-infrared (UV-Vis-NIR) radiation and plastics poses new challenges due to the inherent complexity of the plastic material and lack of experimental protocol for the reflectance analysis. Vis reflectance spectra can be used to identify colorants and discoloration, as both polymers and additive packages should not absorbed in this spectral region. Still despite this, the use of the information collected in the UV and NIR portions for the identification and condition assessment of plastics should be clarified with further research. Plastics show a prevalent specular component in the reflected beam (due to their very smooth surfaces) and specific scattering processes (due to the semi-crystalline or amorphous structure of the plastic itself), hence, a full understanding of its application is still lacking. The geometry of acquisition can strongly influence the resulting reflectance spectra profile where diffusely reflected beams should be maximized in the detection.

5. Conflict of interest declaration

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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Eva Mariasole Angelin – is a researcher at the Conservation Science Department of the Deutsches Museum (Munich, Germany). Her research interests include the preservation of modern and contemporary cultural heritage, including color stability, characterization of the constitutive materials and degradation mechanisms. In July 2021, she defended her PhD thesis in Conservation Science at NOVA School of Science and Technology, NOVA University Lisbon (Portugal).

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Marcello Picollo – is a researcher at the Institute of Applied Physics “Nello Carrara” of the National Research Council of Italy (IFAC-CNR), Florence. He has been working on spectroscopic investigations of works of art since 1991; his main research focus is on artists’ material characterization using non-invasive spectroscopic and imaging techniques. Since 2009, he has been the coordinator of the IFAC Applied Spectroscopy Group on the research line “Integrated spectroscopic instrumentations and methodologies for the diagnosis and monitoring of Cultural Heritage objects and environment.”

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