



Study and characterization of mural paintings from XIX Century in a noble Venetian (Italy) palace

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ABSTRACT

The present paper describes the characterization carried out on the roof of a private palace located in the historical center of Venice (Italy). We focused our attention on the studies of original constituent materials (pigments and binders), in order to identify the pictorial technique of the artist and verify the stylistic attribution to Giuseppe Borsato, a Venetian painter operating between 18th and 19th century. Characterization of collected samples was carried on using both conventional and advanced analytical techniques, such as Optical Microscopy and Scanning Electron Microscopy coupled with Energy Dispersive X-Ray (SEM-EDS) microscopy, Fourier Transform Infrared Spectroscopy (FTIR) and High Performance Liquid Chromatography (HPLC) coupled with photodiode array detection (DAD) and Mass Spectrometry (MS). The finding of dating pigments (such as Cobalt blue and Cu-As based green) and of a complex binding's mixture compatible with the considered supposed period, allowed us to confirm the attribution hypothesis.

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1. Research aims

This paper presents a summary of the physical and chemical characterizations performed on the roof of a private palace located in the historical center of Venice (Italy). The study focused on a series of decorative panels painted with organic binders on *marmorino* plaster. During the whole study period a detailed characterization of original materials, in order to study the artist's painting technique and verify the attribution of ceiling made during the last restoration was performed. Another goal was to evaluate the potential of the High Performance Liquid Chromatography (HPLC) technique applied to artistic and cultural heritage through the characterization of collected organic paintings. The performed study was divided into two phases: the analysis of pigments conducted by scanning electronic and optical microscopy, and the analysis of binders conducted by means of FTIR as well as of HPLC coupled with UV-VIS and MS detectors. In more detail, these two techniques specifically permitted to lead to important results about the painter's technique and attribution.

2. Introduction

The study of an artist's painting technique is a quite complex process that must take into account various factors including the determination of original materials and the *modus operandi* of the

painter. A comprehensive study requires the combination of several analytical techniques and the support of a good knowledge of the artistic process. The selected case study is particularly interesting because the executive technique's reconstruction was aimed at verifying the stylistic attribution advanced during the last restoration, which occurred in 2007. The selected artwork is a painted ceiling belonging to a Late-Renaissance noble Venetian palace and attributed to Giuseppe Borsato (1770–1849). The ceiling, characterized by a marked neoclassical style, has been realized with organic binders on *marmorino* plaster and it is a splendid example of decorative Venetian art of that period. It is located in a bright large room on the first noble floor. The room walls are wholly covered with a pale yellow *marmorino* plaster embellished by a polychrome *marmorino* base-board and by floral decorations at the top. In these paintings the ceiling is shared out by an elegant neoclassical false architecture that represents a perfect frame for sixteen dancing female figures. These dancers, painted in neoclassical Pompeian style, differ in clothing, hairstyles and colors. The pictures are characterized by bright and almost iridescent colors, with a wide chromatic range including a great variety of red, blue and green shades.

3. Experimental

3.1. Standards

In order to find out specific binders used by the painter for the realization of the pictures, 18 commercially available binders have been preliminary investigated and characterized by different

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techniques. Standard of binders samples used come from Bresciani (Milan, Italy). The standard solutions were prepared by dissolving approx. 20 mg in 20 ml of an appropriate solvent. Table 1 contains information relating to the analyzed standards and solvents used in this study.

3.2. Sampling and sample preparation

In an analytic study, sampling should be performed before any restoration operation in order to identify information to degradation products of original materials and possible additions made during previous restorations. In this particular case it was not possible because, at the time of sampling, restoration of the ceiling was almost complete. However, since the purpose of this study was the characterization of the original materials, this was not an impediment. The samples for stratigraphic and pigment composition studies were collected using a scalpel blade n. 7.

Fig. 1 shows the complete mapping of the sampling points. These points were chosen on the base of minimum possible damage, as well as on representativeness and significance criteria, trying to gather as much information as possible about the variety of pigments used by the artist. To this aim, areas affected by glazing were prioritized in order to investigate more pigments in the same sample. Prior to the beginning of the embedding procedure, the samples were observed under optical microscope (OM) to verify the quality of the cutting. The observation was carried out by a stereo-microscope SZX9 by Olympus (Tokyo, Japan) with an adjustable system of optical fibers for a better illumination of the samples. This preliminary visual analysis showed the problem of the fragments small size that made it necessary to use a higher magnification. These small dimensions are due both to the particular hardness of the surface that gave samples in powdered form and also to the unrealistic possibility to produce paint loss in a recently restored opera. The embedding of the painting samples was carried on using a six silicone cells mold, a transparent epoxy resin (ca 400/L) by Conchem (Lodi, Italy) and the necessary equipment to mix it with the hardener. The cross sections prepared in this way were observed under OM and SEM-EDS. Samples for HPLC analysis and FTIR were taken from the external band adorned with floral decorations: this part was subjected to extraction with organic solvent.

Table 1
Scheme of the analyzed standards and the solvents used in the solutions.

	Binders	Solvent	Weigh (mg)	Volume (mL)	Conc. (mg/ml)
1	Casein	H ₂ O (pH 7.2) ^a		50	
2	Beeswax	CH ₂ Cl ₂ ^b	23	25	920
3	Rabbit glue	H ₂ O	23	25	920
4	Isinglass	H ₂ O	11	10	1100
5	Colophony	Ethanol	85	100	850
6	Manila copal	Ethanol	84	100	840
7	Dammar	Isopropanol	5	10	500
8	Arabic gum	H ₂ O	24	25	960
9	Shellac	Ethanol	6	5	1200
10	Milk	Isopropanol	29	20	1450
11	Fig latex	Isopropanol + CH ₂ Cl ₂	6	10	600
12	Mastic	Ethanol	16	20	800
13	Honey	H ₂ O	200	100	2000
14	Boiled linseed oil	Isopropanol	25	20	1250
15	Linseed oil	Isopropanol	31	25	1240
16	Dry linseed oil	Ethanol ^c	15		
17	Sandarac	Isopropanol	39	50	780
18	Egg yolk	H ₂ O	45	25	1800

^a Basic pH it was reached by adding NH₃.

^b The solution has been diluted for ten times with isopropanol.

^c After acid hydrolysis.

3.3. Extraction of organic binders

The extraction of organic binders was carried out by using two solvents with different polarity in order to cover the entire range of solubility. In this case acetone and dichloromethane, whose boiling points and solubility parameters (A, P, and H) report to the triangle of solvents and solubility are showed in Table 2, were chosen.

The powdered sample was treated with acetone and exposed to an ultrasonic-assisted extraction for 20 min with probe sonicator UP200S by Hielscher (Teltow, Germany) in order to disrupt the sample and completely solubilize the organic components. The mixture was then centrifuged for 15 min at 2500 rpm in order to separate the two phases. In all the samples, except n.5 and n.6, the solution was evaporated to dryness in a evaporator and reconstituted with isopropanol. The precipitate was subjected to a second extraction with dichloromethane. This procedure was repeated for all samples in order to obtain the corresponding test solutions. Table 3 summarizes the samples derived from different extraction and analytical techniques applied to each solution.

Solution 2c is the result of a second extraction from sample 2 obtained using ethanol after acid hydrolysis of the insoluble residue of dichloromethane extraction. This step guaranteed the solubilization of old siccative oil potentially present in the sample. The samples were placed in 12 ml vial, treated with 1.5 ml of concentrated HCl and manually shaken for a few seconds. The extraction of the organic phase was carried on after 48 h by adding 2.5 ml of hexane and vigorously shaking the vial. The hexane phase was then separated and two other extractions were performed again, each with 3.5 mL of hexane. After hexane partial evaporation, 1.5 ml of ethanol were added to the vial and the solution was further concentrated down to a final volume of 1 ml, in order to eliminate the non polar solvent, as previously reported [1].

3.4. Instruments and methods

The extraction of organics was performed with an ultrasonic device probe sonication as described above. The obtained extracts were concentrated down to 0.5 ml in a automatic evaporator Turbovap II Zymark (Darlington, MA, USA) and centrifuged for 10 min before injection. The water used for chromatographic separations and standards preparation was purified through the system Millipore MilliRo/MilliQ (Bedford, MA, USA) while organic solvents were "HPLC gradient grade" from ROMIL (Dublin, Ireland). The HPLC separation of the analytes was performed with an Agilent 1100 HPLC system (Avondale, PA, USA), equipped with Agilent 1100 vacuum degasser. The detectors used were a spectrophotometer UV-Vis 1100 Diode Array Detector (DAD) and a mass spectrometer Agilent MSD-Quadrupole 1100, coupled to the HPLC system via both electrospray ESI (ElectroSpray Ionization) and APCI (Atmospheric Pressure Chemical Ionization) interfaces. The chromatograms have been elaborated through the rev. A.09.01 Agilent ChemStation software. The HPLC columns used for chromatographic separation were Gemini C18 (150 cm × 2 mm, 5 μm) and Onyx monolithic C18 (25 cm × 4.6 mm, 2 μm), both by Phenomenex (Torrance, CA, USA), both protected by a guard column with the same stationary phases of the applied columns.

The analysis of samples 2(a,b), 3(a,b) and of all standards samples was performed by HPLC-MS. The Full-Scan mode with two different ionization conditions, APCI and ESI in both positive (+) and negative (-) mode was applied. The complexity of the considered artistic period not allowed to limit the testing field so, for screening proposes, a wide range of binders had to be searched for. The APCI (+) detection parameters derived from a previous study on terpenic resins [2]. In the other cases analysis were conducted with standard parameters in order to develop a general use method that could fit the largest possible number of compounds.

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