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Archaeometric Study of the Mural Paintings by Saturnino Gatti and Workshop in the Church of San Panfilo, Tornimparte (AQ): The Study of Organic Materials in Original and Restored Areas

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Abstract: In the context of the archaeometrical study of Saturnino Gatti's wall paintings, a significant aspect concerned the study of the organic component to understand both the original binders used in the original areas and the products used for pictorial reintegration and restoration of the painted surfaces. Thanks to the results obtained from various non-invasive and multi-band imaging techniques, it was possible to define Gatti's original painting technique and identify the materials subsequently applied in significant samples. To this end, molecular analyses based on mass spectrometry were carried out. Different procedures in gas chromatography–mass spectrometry (GC-MS) and in pyrolysis coupled with gas chromatography–mass spectrometry (Py-GC-MS) were adopted. The analyses revealed a variety of organic materials on the mural paintings, most of which are from past restoration interventions and have synthetic origin. The overspread presence of paraffin is likely due to the application of a mineral wax-based coating/consolidant. In particular, the execution technique encompassed the use of tempera-based paints, while retouched areas were characterised by the presence of oil-based resins.

Keywords: proteins; lipids; paraffin; synthetic materials; paint binders

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1. Introduction

This paper contributes to the Special Issue "Results of the II National Research project of AIAr: archaeometric study of the frescoes by Saturnino Gatti and workshop at the church of San Panfilo in Tornimparte (AQ, Italy)", in which the scientific results of the Second National Research Project conducted by members of the Italian Association of Archaeometry (AIAr) are discussed and collected. The cycle of frescoes is undoubtedly the masterpiece of the painter Saturnino Gatti. The wall paintings are framed by an architectural score, which creates illusionistic effects, running along the perimeter of the apse. A beautifully painted vault crowns the whole, and the sequence of images gives a beautiful 'cinemascope' effect.

The colourful scenes, subject to periodic restorations, are the subject of numerous studies, as highlighted by the vast literature.

Preliminary investigations of the painting technique, as well as the restoration materials of the pictorial cycle, were carried out using spectroscopic analysis and mainly non-invasive techniques, such as multiband imaging and single-spot techniques already optimized for works of art in cultural heritage [1–3].

The multispectral investigations carried out prior to the sampling campaign highlighted a diffuse presence of retouching, conservation and consolidation materials. Moreover, evident detachment and exfoliation phenomena were observed, together with a

strong efflorescence. Chemical characterizations of the soluble salts by means of ion chromatography and attenuated total reflectance Fourier-transform infrared spectroscopy have been performed within the AIAr project [4]. Although the Renaissance pictorial cycle is of national interest, the pictorial technique has not yet been defined. Furthermore, the characterization of the consolidant and protective materials is necessary for the restoration, which aims to recover the legibility of the work, undermined by the degradation of the surface materials. For these purposes, within the research project, it was decided that twelve samples would be collected for chromatographic analyses. Currently, several works have demonstrated the capability of characterizing natural organic materials of different natures, such as lipid, protein, resinous and waxy materials with gas chromatography techniques coupled with mass spectrometry [5–10] and pyrolysis [11]. The pyrolysis analysis is also essential for the investigation of synthetic products used in restoration through the study of markers produced by the pyrolysis [12–14].

Despite the heavy and numerous restoration interventions, some areas were found to be of particular interest for the investigation of the paintings, such as residues of original gilding decorations, or original areas without overpainting or where aged coating films were less abundant.

2. Materials and Methods

2.1. Samples

The experimental sections are divided into two subsections: one dedicated at the identification of the original binding media and aged coatings, and the other to study the materials from the lacunae, as well as retouched and restored areas. Samples from the original paintings were collected mainly from the upper part of the vault and Area 1—Panel E (Figures 1a and 2), while for the consolidation/restoration ones they were collected mainly from Area 4—Panels A (Figure 1b), D and E (Figure 3). The bad state of conservation and severe legibility problems due to degraded pictorial varnishes, retouching and stuccos are clearly visible.

Figure 1. Visible image of Area 1—Panel E (**a**), and Area 4—Panel A (**b**).

Figure 2. Sampling points of Area 1—Panel E and upper part of the vault for the characterisation of the original painting materials.

Figure 3. Sampling points of Area 4—Panels A, D and E for the characterisation of restoration materials.

2.1.1. Original Binding Media and Aged Coatings

Six samples were collected and analysed from the upper part of the vault and panel E (Figure 1a). Figure 2 shows the sampling points, and a brief sample description and the aim of studying the issues of the investigated areas are reported in Table 1. The areas, even if they were not as affected by thick overpainting and consolidants compared to the other investigated areas, still presented a film of degraded protective layers. Samples collected from these areas were analysed using gas chromatography–mass spectrometry (GC-MS), using an analytical procedure that enables the identification of natural organic materials based on lipids, proteins and terpenoids [15].

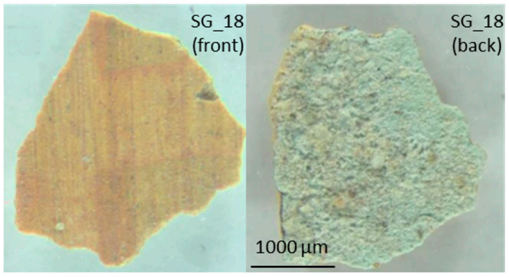
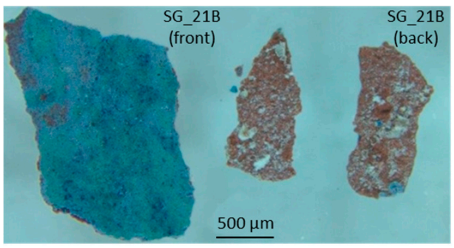
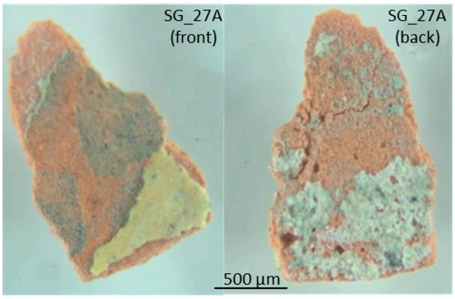
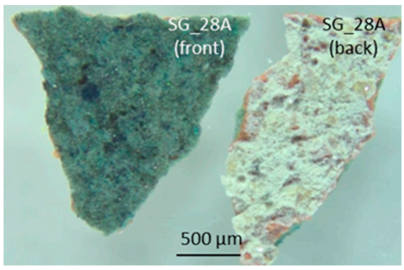
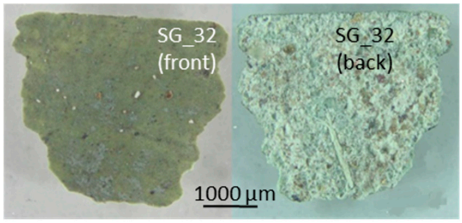
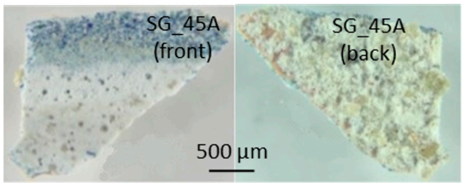
Table 1. Description of the samples collected for the identification of original binders and degraded superficial varnishes or coatings.

Sample	Area	Description	Aim of the Investigations
SG_30	Panel E	Fragments of white plaster + greyish pictorial finish taken from an area which under UV appears characterized by an uneven yellowish response. Macroscopically, the surface appears chromatically altered (with a spotted effect).	Identification of the degraded protective
SG_34	Panel E	Fragments of white plaster + grayish paint finish taken from an area that shows no fluorescence response under UV. Macroscopically, the surface appears glossy.	Identification of altered protective and/or other consolidating treatments undergone in the past
SG_37c	Vault, top part	Gray pictorial spread on a layer of degraded plaster, taken from a degraded area due to detachments and lifting of the surface layers.	Identification of organic binders and protective consolidants
SG_41C	Vault, top part	Purplish pictorial spread on a layer of degraded plaster, taken from a degraded area with lifting of the surface layers.	Identification of organic binders and protective consolidants
SG_43	Vault, top part	Relief layer of material of an organic nature selectively taken from the pictorial surface. These are tablets to simulate the golden decorations of the halos and robes of the Almighty (or relief base for the application of gold leaf), now detached.	Understanding of painting technique
SG_44	Panel E	Traces of a layer of preparation for the application of gold leaf for the creation of rays of the risen Christ. The surface of the layer taken is raised and tenaciously adheres to the plaster. Given the small number of traces still present, the sampling has been reduced to minimal quantities.	Understanding of the gold leaf application technique

2.1.2. Lacunae, Retouched and Restored Areas

Six samples were collected from Panels A, D and E, and the sampling points are shown in Figure 3. Table 2 reports a short description and visible images of the samples. Given the complexity of the pictorial fragments, which showed the co-presence of layers attributable to both original (or relatively ancient) work and subsequent restoration interventions, samples were divided into two aliquots. The first aliquot was analysed using GC-MS for the identification of lipid, terpenoid and waxy fractions, by means of an analytical procedure based on one-spot hydrolysis and transesterification [16,17]. The other aliquot was analysed with Py-GC-MS to verify the presence of proteinaceous and polysaccharide material [18] and detect synthetic polymers. In particular, analytical pyrolysis was carried out in combination with tetramethylammonium hydroxide (TMAH) to favour the detection of some organic binders by means of thermally assisted hydrolysis and methylation [19,20].

Table 2. Description and images of the samples collected for the identification of organic materials from lacunae, retouched and restored areas.

Sample	Area	Description	Image
SG_18A	Panel A	Yellow-orange pictorial layer and whitish preparation/primer, taken along an existing gap.	
SG_21B	Panel A	Blue pictorial layer on a red-brown layer (the so-called 'morellone') and underlying plaster fragments.	
SG_27A	Panel D	Yellow-blue pictorial application and white preparation/primer.	
SG_28A	Panel D	Dark green pictorial layer and fragments of plaster. The area also has pictorial integrations with glazes.	
SG_32	Panel A	Green pictorial layer and white preparation/primer, taken along the edge of a gap.	
SG_45A	Panel E	White layer ('lumeggiatura') (highlighting) applied on the underlying blue pictorial surface.	

2.2. Analytical Instrumentation

2.2.1. Instrumentation for the Analysis of Original Painting Materials

A 6890 gas chromatograph coupled with a 5975 single quadrupole mass-selective mass spectrometer (Agilent Technologies, Palo Alto, CA, USA) was used. The analytical procedure, the instrumental conditions and analytical parameters of which are reported elsewhere [15], allows the combined analysis of protein, lipid–resinous and wax content in samples. Samples were subjected to a multistep chemical pretreatment which involved several steps of extraction, hydrolysis, purification from inorganic species and silylation before GC-MS analysis.

2.2.2. Instrumentation for the Investigation of Restoration/Consolidation Treatment

- A Trace GC 1300 system equipped with an ISQ 7000 MS detector was used (ThermoFisher Scientific, Waltham, MI, USA) for the analysis of approximately 80–100 μg of sample. The transesterification reaction and the complete methodology are described elsewhere [16–18]. The data interpretation was performed using NIST and MS Search 1.7 libraries and ad hoc databases created by the authors. The Chromeleon 7 software was used for the data acquisition and processing.
- Thermally Assisted Hydrolysis and Methylation (THM)–Single Shot Pyrolysis–Gas Chromatography/Mass Spectrometry (TMH–SS-Py–GC/MS) was performed on small aliquots of samples (around 30–80 μg) in eco-cup pyrolysis crucibles. The samples were then treated with 3 μL of tetramethylammonium hydroxide (TMAH), 25%, in methanol.
- A PY-3030D pyrolizer (Frontier Lab, Koriyama, Japan), connected to a Trace 1310 gas chromatograph (ThermoFisher Scientific, Waltham, MA, USA) with an ISQ7000 mass spectrometer (ThermoFisher Scientific, Waltham, MA, USA), was used. The analytical parameters and software for collecting, processing and interpreting the mass spectral data are reported in the literature [19,20].

3. Results and Discussion

3.1. Original Binding Media, Aged Coatings and Consolidants

The presence of residues of synthetic origin (see following paragraph) caused an interference in the analysis of amino acids in sample SG_34; therefore, it was not possible to establish whether or not proteinaceous material was present. For all other samples, GC-MS analyses evidenced the presence of low amounts of proteinaceous material, which resulted above the limit of detection (LOD, 0.3 μg of the eleven amino acids quantified).

In sample SG_43, amino acids were present above the quantification limit (LOQ) of the analytical technique (0.6 μg in total). The relative amino acid content was thus subjected to multivariate statistical analysis according to the principal component method (PCA) together with a database of reference samples containing animal glue, egg and casein [15]. The resulting score plot is shown in Figure 4, which indicates that sample 43 contains egg. This result is of considerable importance because the sample had been taken from an area of the top part of the vault, selectively scraping the pictorial surface: we believe egg is an original binder used by the artist. The remaining samples present amino acids below the LOQ, and it is therefore not possible to identify the proteinaceous binder. Animal glue is absent, though, thus ruling out the presence of animal glue [21,22].

Beeswax is identified by the presence of relatively high amounts of palmitic acid and tetracosanoic acid and (ω-1) hydroxy even-numbered fatty acids (with the most abundant 15 hydroxyhexadecanoic acid) (Figure 5), long-chain linear alcohols with a number of even carbon atoms (Figure S1), α-(ω-1)diols and linear aliphatic hydrocarbons with an odd number of carbon atoms (Figure 6) [7,23]. On these bases, beeswax was clearly detected in all samples, with the exception of sample SG_44, and was particularly abundant in samples SG_37c and SG_43. Waxes were used as material for the consolidation and protection of wall paintings, as reported in many technical manuals. This is the case in Secco-Suardi's technical manual dated 1866, where restorers were advised to use mineral materials, such as paraffin, for the consolidation of wall paintings and painted facades [24–26]. The use of

paraffin as a consolidating agent is evidenced by the restoration of the Pisa Cemetery in 1858 [23,27] and numerous restoration reports, including that of Venturini Papari for the Roman paintings in Pompeii [28].

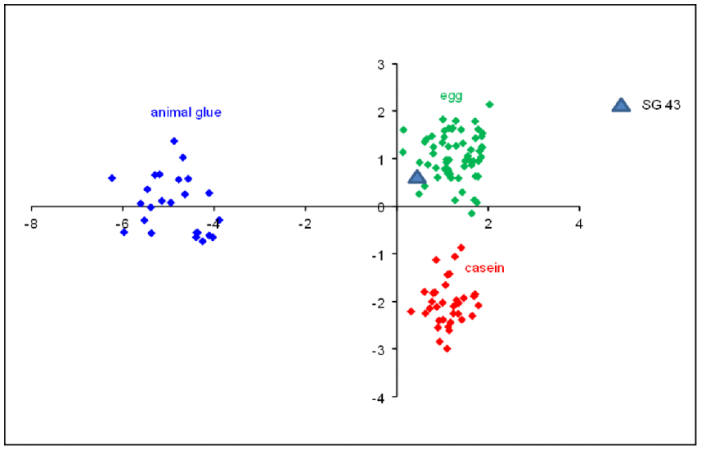


Figure 4. PCA score plot related to the percent amino acid profile of sample SG 43 (Vault) together with the database of profiles of reference samples containing animal glue, egg and casein.

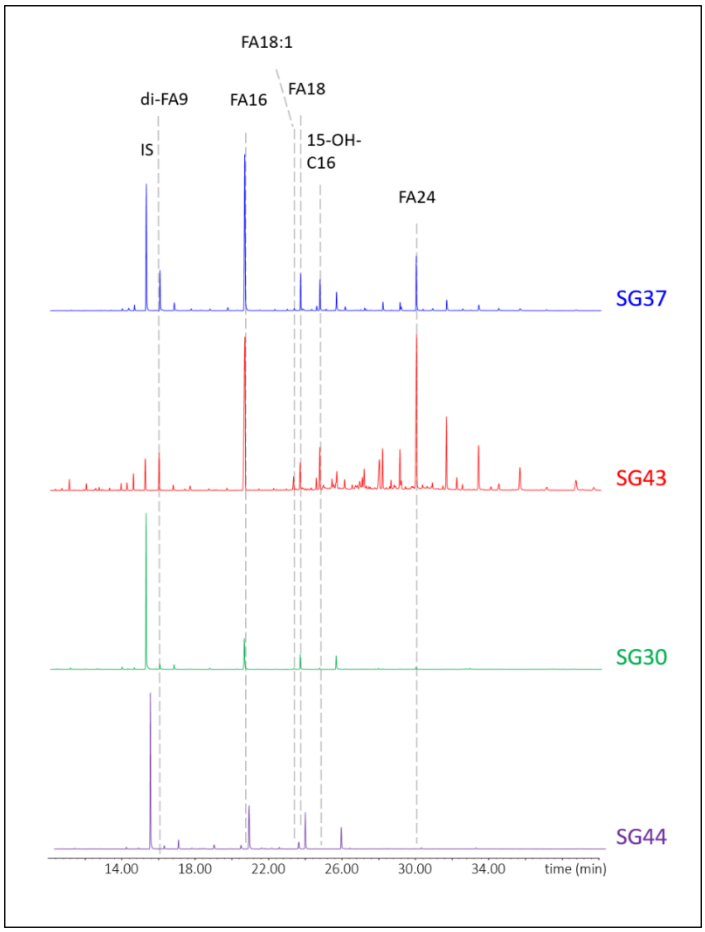


Figure 5. Extracted ion chromatogram of fragment ion m/z 129, characteristic of trimethylsilyl (TMS) esters. IS: internal standard: tridecanoic acid–TMS; di-FA9: azelaic acid–TMS; FA16: palmitic acid–TMS; FA18:1: oleic acid–TMS; FA18: stearic acid–TMS; 15-OHC-16: 15-hydroxy hexadecanoic acid–TMS; FA24: tetracosanoic acid–TMS.

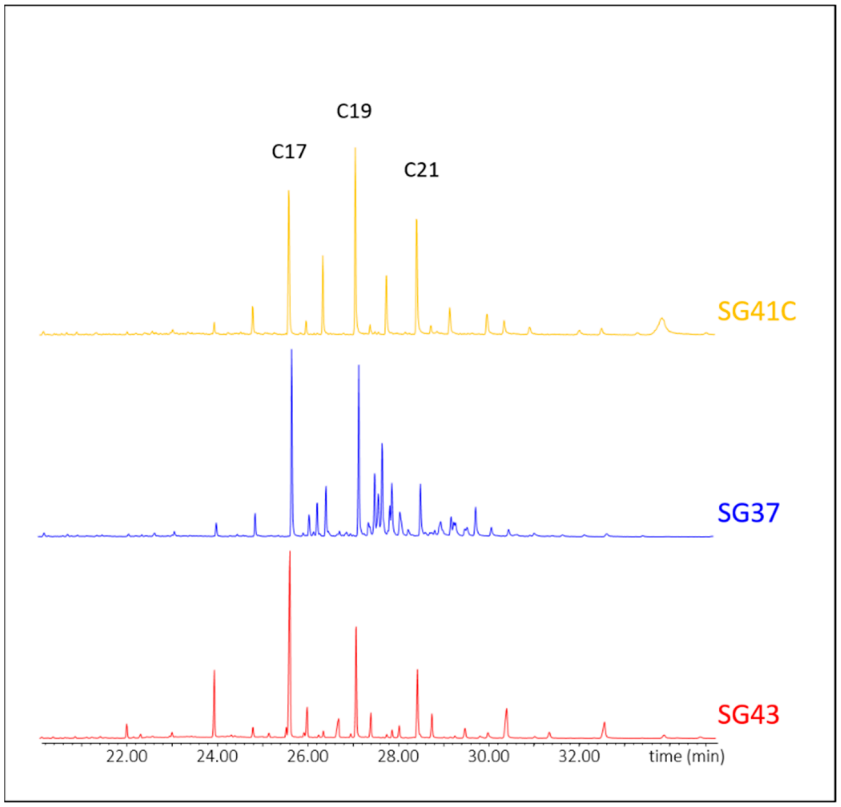


Figure 6. Extracted ion chromatogram of the fragment ion m/z 85, which is abundant in the mass spectra of linear aliphatic hydrocarbons. Cn indicates a linear aliphatic hydrocarbon with n carbon atoms.

Therefore, the use of paraffin and mineral waxes is also plausible in the case of Saturnino Gatti's wall paintings.

Beeswax makes the detection of glycerolipidic material difficult, as it substantially alters its chromatographic profile. The presence of relatively high amounts of azelaic acid, an oxidation product of polyunsaturated acids typical of glycerolipids of vegetable origin, points to the presence of an oxidized drying oil [29]. Traces were detected in both samples SG_30 and SG_44, but the chromatographic profile does not allow the determination of its nature, while relatively higher amounts were detected in samples SG_37c and SG_43 (SG_43 being a sample collected from the relief base for the application of gold leaf from the vault).

The analysis of the lipid–resinous fraction also evidenced the presence of a Pinaceae resin given the identification in the chromatogram of trimethylsilyl esters of dehydro-abietic acid and 7-oxo-dehydroabietic acid [21]. These are clearly visible in the chromatogram of the SG_43 sample (Figure S2), but detectable in the chromatograms of all the samples, except for sample SG_41C, due to an analytical interference which compromised the silylation reaction.

3.2. Lacunae, Retouched and Restored Areas

An extensive presence of paraffin was found in all the samples taken from Panels A, D and E, characterized by a series of linear alkanes with an number of carbon atoms higher than 25, and with a "Gaussian" shape [23,30] (Figure 7). Interestingly, paraffin was also found in samples taken from Panel E for the characterisation of the original binder, although it showed a different chromatographic profile and was particularly abundant in samples SG_30 and SG_44 (Figure 8). Paraffin was detected in all samples from Panel A, with the exception of sample SG_43.

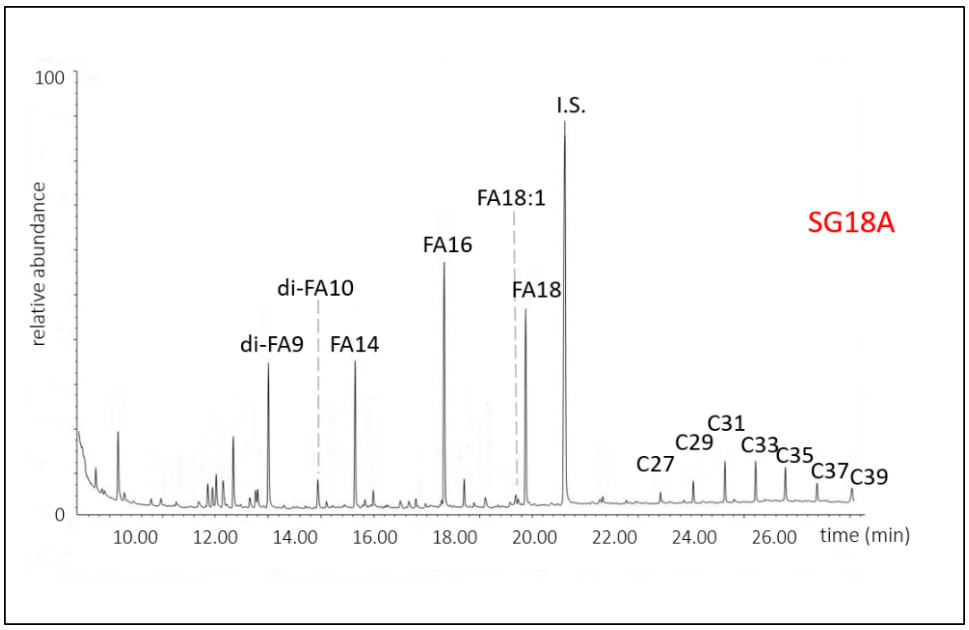


Figure 7. Total Ion Current (TIC) Chromatogram of sample SG_18A after methyl ester derivatization (ME). IS: internal standard: nonadecanoic acid–ME; di-FA9: azelaic acid–ME; di-FA10: sebacic acid–ME; FA14: myristic acid–ME; FA16: palmitic acid–ME; FA18:1: oleic acid–ME; FA18: stearic acid–ME; Cn linear aliphatic hydrocarbon with n atoms of carbon.

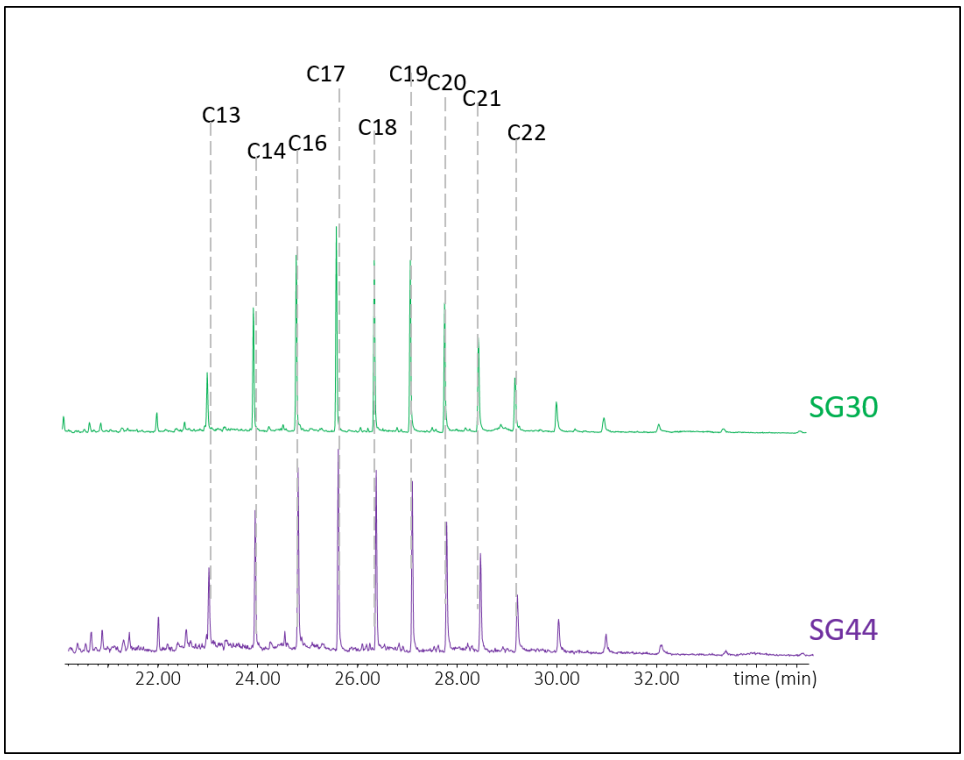


Figure 8. Chromatographic profiles of the ion extracted with m/z 85 for samples SG 44 and SG 30. Cn indicates a linear aliphatic hydrocarbon with n atoms of carbon.

A glycerolipid material was detected in samples SG_18A, SG_27A, SG_28A and SG_32, based on the identification of saturated monocarboxylic fatty acids (palmitic and stearic) and dicarboxylic acids (sebacic, azelaic and suberic acids) (Figure 6). The chromatographic profile, and in particular the relatively high content of dicarboxylic acids, indicates the presence of a siccative oil [29]. Moreover, SG_18A and SG_32 still present oleic acid, a

monounsaturated fatty acid generally still present in not completely mature oil paint films. Its presence suggests that the drying oil found in the samples is most likely a later material rather than an original one used by Saturnino Gatti.

Myristic acid, a saturated fatty acid naturally present in many vegetable lipid materials, is ubiquitous. Its relative abundance in the chromatographic profile strongly suggests that it is not derived from the drying oil, but it could be present as an additive (such as a surfactant or emulsifier).

A plant resin of the Pinaceae family was identified in samples SG_27 (both yellow and orange) and SG_32, based on the detection of abietic acid and its oxidation products [21] (dehydroabietic acid, 15-oxo-dehydroabietic acid and 7-oxo-dehydro-abietic acid). This terpenoid material, not traditionally used as a binder in wall paintings, could be present as an additive of the binder used for retouching or in oil-resinous coatings. The presence of abietic acid also emphasises that the painting is relatively fresh and therefore not of the same period as the creation of the painting cycle.

Synthetic polymers were also found. In particular, an oil-modified alkyd-based resin was identified in samples SG_28A and SG_45A, based on the detection of orthophthalic acid, benzoic acid, glycerol and a drying oil [12]. An acrylic-styrene resin was identified in sample SG_21B, thanks to the main presence of styrene, butyl acrylate, n-butyl methacrylate and α-methylstyrene [31,32].

These synthetic materials are likely present as binders of the paints used for the reintegration, being amongst the most used contemporary paints.

For the identification of possible proteinaceous and polysaccharidic fractions remaining from the original binder, the samples were also subjected to analysis with Py-GC-MS in Double Shot mode (350–650 °C) as described in the literature [33]. The analyses were not conclusive, however, as the typical markers of the proteins present in casein or egg-based paints were not detected. This aspect could be linked to natural aging and cross-linking processes, as well as to the interference due to the inorganic component of the samples and the many materials used in restoration and painting reintegration.

In conclusion, it can be stated that the samples under investigation appear to have been taken from areas that were significantly, if not completely, repainted. Therefore, it was not possible to identify the original organic binder, while the composition of the different paints used for reintegration was clarified.

4. Conclusions

The analysis of organic materials revealed the presence of a variety of natural and synthetic materials, indicating numerous interventions during the years.

It is difficult to establish the binding media of the original painting layers, due to extensive contaminations and co/presence of many sources of organic materials, both of natural and synthetic origin. Despite this, the presence of proteinaceous material, most likely egg, was observed in the samples coming from the vault, suggesting the use by Saturnino Gatti of an egg tempera, at least in some areas.

A siccative oil was detected and clearly identified in several samples collected near some lacunae together with synthetic resins, suggesting that this is a restoration material. Additionally, terpenic materials and alkyd resins were detected near/close to lacunae and retouched areas, and they could be associated with the use of the so-called «colori da ritocco» [27], which are paints specifically designed to be used for retouching paintings in restoration interventions.

Beeswax and more than one type of paraffin wax were extensively detected in the sections of the wall paintings analysed.

In conclusion, the results obtained from the organic analyses provided a great deal of information regarding the materials presumably used in the creation of the pictorial cycle, as well as in the various phases of reworking and restoration that took place over the years (testifying also the importance of the pictorial cycle and the desire to preserve it over time).

Together with the results obtained from the various analytical techniques presented in this Special Issue, the knowledge relating to this unique pictorial cycle has been expanded, and will certainly be put to the best use for its conservation and valorisation.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/app13127153/s1.

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