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Case study

Characterization of the organic materials used in the painting of the vaulted ceiling at the Saadian Tomb of Mulay Ahmed Al-Mansour (Marrakech)

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ABSTRACT

The Saadian tombs from the era of sultan Ahmed al-Mansour (1574–1603) are beautifully decorated and have always been a major attraction for visitors to Marrakesh. The central mausoleum, named the Hall of Twelve Columns, encloses the tombs of Ahmed al-Mansour and his family. The hall has a huge vaulted ceiling, carved cedar doors, opening windows with wooden marquetry screen (Mashrabiya), and grey Italian marble columns. This paper presents the first attempt to identify the organic materials used by the Moroccan artisans. A GC/MS analytical procedure was used for the characterization of lipids, waxes, resins, pitch, tar, proteinaceous and saccharide materials in the same paint micro-sample. The analytical study identified the organic materials used in the polychrome and gilded decorations of the walls, ceiling and dome of the hall. Data showed that the polychrome decorations were painted using animal glue as a binder, and highlighted the treatment of the wall surface with linseed oil and the retouching of the paintings based on a saccharide binder. The use of a proteinaceous-resinous-oil mixture, applied on a proteinaceous preparation layer, for the gilded decorations revealed a very similar technique to that used at the time in Europe for mural paintings.

1. Research aims

The Saadian tombs in Marrakesh (1574–1603) are beautifully decorated with polychrome, gilded stuccos and decorative ornaments made from cedar wood. The loss of brightness and cohesion of the paintings has meant that the tomb has had to undergo a restoration campaign. An analytical investigation was thus undertaken in order to identify the organic materials present in the decoration, both as binders or restoration materials from previous campaigns. The painting technique of the Saadian tombs has never been studied even though their historic-artistic importance is of special relevance not only in terms of their value but also because of the lack of information regarding Hispano-Muslim painting techniques. Although some studies have been published on the painting materials used in the polychrome decorations of the Alhambra [1,2] and other buildings in Granada [3,4], and Cordoba [5], results have mainly related to the inorganic composition of pigments and preparation layers, while the organic materials were only identified in a few cases [2–4].

The aim of this study is to provide the first evidence regarding the organic materials used not only by the Saadian dynasty painters but also by the Moroccan artisans in general and to link this information to the Hispano-Muslim tradition.

2. Experimental

2.1. Introduction

The Saadian Tombs are part of a royal necropolis built by the Sultan Ahmed El Mansur el Dahbi (1549–1603). Sultan Ahmed el Mansur conquered Sudan in 1578 after beating the Portuguese army in the battle named the “battle of three kings” [6]. The necropolis is within the walls of the El Mansour mosque, at the northern entrance of the Kasbah of Marrakesh, the capital city of the Saadien dynasty (1554–1659). The Saadian tombs are one of the few remaining vestiges of the Saadian dynasty. The tombs were discovered and restored in 1917, and have been open to the public as an historical site since then. Due to their rich and beautiful decoration, the tombs are a major attraction for visitors to Marrakech.

From an artistic point of view, the stucco ornaments and ceramics, cedar ceilings and the elaborate carvings found in this cemetery are similar to ones in other buildings of the same epoch such as

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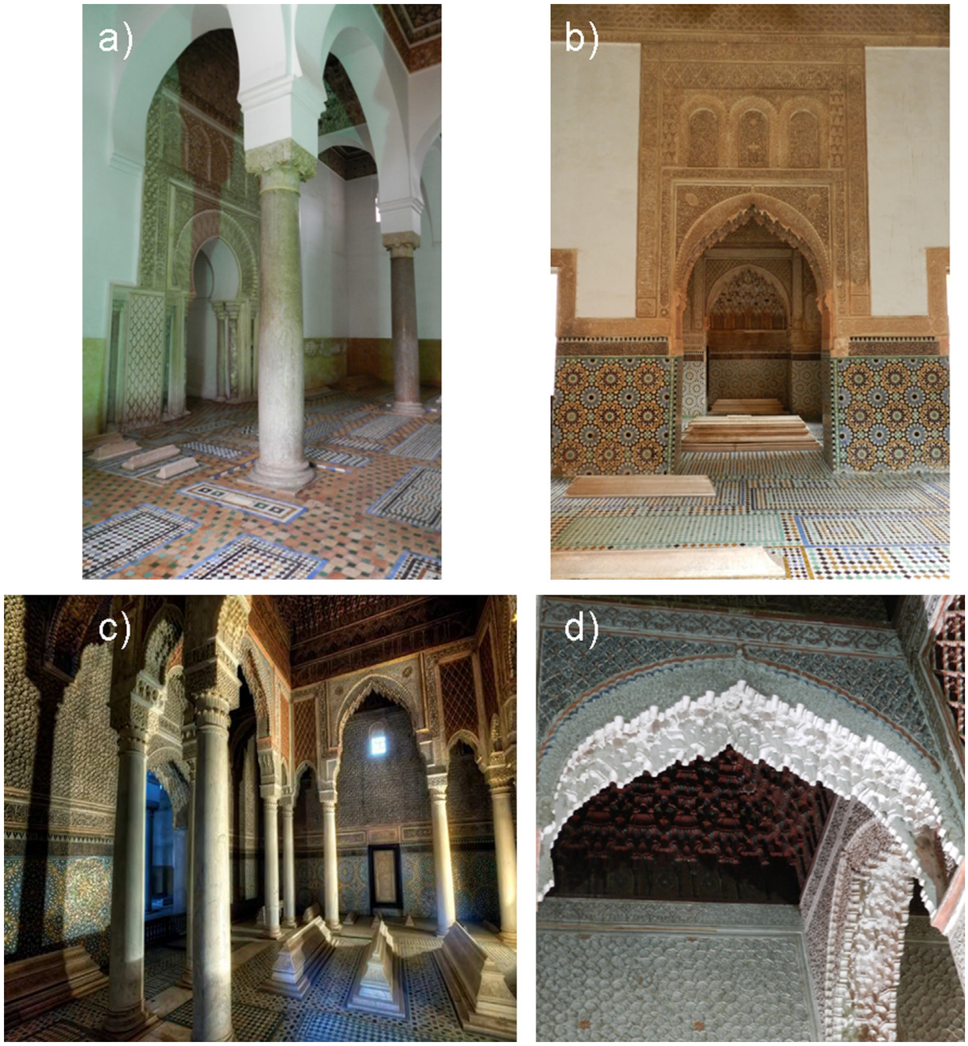


Fig. 1. Details of the beautiful decorations of the rooms of the Saadian Tombs: a) room 1; b) room 3; c) room 2; d) the dome that was selected for sampling.

the Ben Youssef Madrasa (Marrakech), one of the largest theological colleges in Morocco. These decorations seem to be also linked with the Hispano-Maghreb decorations at the Alhambra (Granada, Spain) [7–9].

However, to our knowledge no technical studies have ever been performed on the cultural heritage of painted works of art in Morocco. Moreover, studies on Hispano-Muslim polychrome decorations are scarce and have mainly focused on the identification of the pigments and plasters, paying almost no attention to the organic materials present in the paint layers [1–5].

The necropolis consists in an open-air cemetery with more than 100 tombs, two buildings that cluster around a courtyard, and a garden, flanked on the east and south by an inner wall with towers. The buildings host approximately seventy graves of family members of Sultan Ahmed El Mansur and their successors [10]. Some details of the decorations of the building rooms are shown in Fig. 1.

The first room (Fig. 1a) is decorated with polychrome stuccos and ornaments made of cedar wood [11], while the third room, called "the three niches", has marble columns and a dome with painted stalactites. The dome is covered with a series of cedar wood ceilings, which are also painted and gilded. This room contains the women's and concubine's tombs (Fig. 1b).

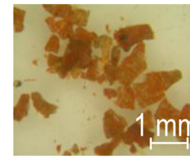
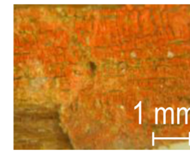
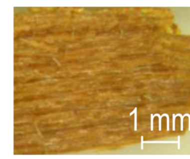
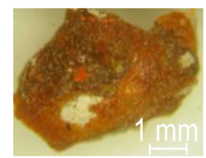
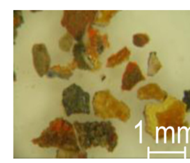
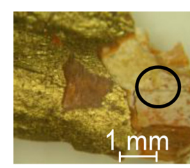
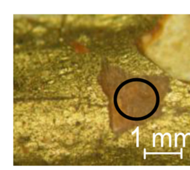
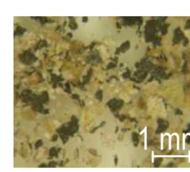
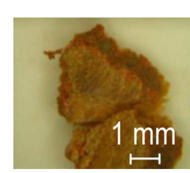
The second room or central hall (The Hall of the Twelve Columns) contains the graves of the Sultan Moulay Ahmed El Mansour, his son and grandson and features a richly decorated dome resting on 12 columns made of Carrara marble. The headstones, barely protruding from the tiled floor (Fig. 1c), are surrounded by white marble mouldings. The walls are covered with two meters of glazed tiles and an upper layer of elaborated carvings on plaster, showing inscriptions and geometric patterns. At the top of the walls, in contact with the ceilings, there is a framework made of wooden carvings. The cedar wood ceilings are painted and gilded. The lobed stalactite arches are made of red painted cedar wood (Fig. 1d).

Although the wooden ceilings have been subjected to ordinary cleaning maintenance in order to remove the dust, and the polychromes have been retouched and varnished, the site has never undergone a proper restoration and the painting technique of these beautiful decorations has not been investigated to date. The serious problems of detachment of the paint layers mean that an appropriate restoration plan needs to be defined.

This paper presents a GC/MS analytical study to investigate the painting technique and surface treatments used in the polychrome and gilded decorations at the Saadian tombs. Separation techniques have already been proved to be very suitable for the characterization of binding media such as complex mixtures of different organic materials [12–14].

A GC/MS analytical procedure was thus applied for the characterization of lipids, waxes, resins, pitch, tar, proteinaceous and saccharide material in the same paint micro-sample [15]. The Hall

Table 1
Description and pictures of the samples collected.

Provenience	Sample	Sub-Sample	Weight (mg)	Description	
Wall wood frame	W1	–	0.3	Red sample	
	W2	W2-R	0.3	Red paint	
		W2-W	0.1	Wood underneath the red paint	
	W3	–	13.4	Red sample	
	W4	–	1.1	Red and black paint layers	
Ceiling	R1	R1-A	0.2	Gilded sample from the ceiling –white preparation layer beneath the mordant of the gilding	
		R1-M	0.3	Gilding mordant	
Dome	C1	–	4.3	White and black sample	
	C2	–	1.5	Red sample	

The samples are labelled with ‘w’ for those collected from the wooden decoration of the upper part of the walls, ‘R’ for those from the gilded decoration of the ceiling, and ‘C’ for those from the wooden dome. Sub-samples separated in the laboratory and analysed individually are also indicated.

of Twelve Columns was chosen for the exploration of the organic material present in the tombs. Results from the study enabled the painting technique of the polychromies and gildings to be identified as well as the surface treatments and restoration materials in the site. This paper represents the first analysis of the painting technique of Moroccan artisans and a comparison is made with the Spanish and European tradition of painting technique and materials.

2.2. Materials and methodologies

2.2.1. Apparatus and conditions

A 6890 N GC System Gas Chromatograph (Agilent Technologies, Palo Alto, CA, USA), coupled with a 5973 Mass Selective Detector (Agilent Technologies, Palo Alto, CA, USA) single quadrupole mass spectrometer, equipped with a split/splitless injector was used. The mass spectrometer was operated in the electron impact (EI) positive mode (70 eV). It was used to analyse the lipid-resinous fraction of the samples with the combined analytical procedure. For details of the operating conditions and the analytical procedure see [15].

A 6890 N GC System Gas Chromatograph (Agilent Technologies, Palo Alto, CA, USA), coupled with a 5975 Mass Selective Detector (Agilent Technologies, Palo Alto, CA, USA) single quadrupole mass spectrometer, equipped with a PTV injector was used. The mass spectrometer was operated in the electron impact (EI) positive mode (70 eV). The MS transfer line temperature was 280 °C. The MS ion source temperature was kept at 230 °C, and the MS quadrupole temperature at 180 °C. This instrument was used to analyse the proteinaceous and saccharide fraction of the samples with the combined analytical procedure. For details see [15].

A microwave oven model MLS-1200 MEGA Milestone (FKV, Sorisole (BG,) Italy) was used. Acid hydrolysis conditions were as follows: power 250 W for 10 min; power 500 W for 30 min in the vapour phase with 30 mL of 6 N HCl at 160 °C for 40 min. Saponification conditions were as follows: power 200 W with 300íL of KOH_{ETOH} 10% wt at 80 °C for 60 min.

2.2.2. Samples

A 13-meter high platform was placed in the Hall of the Twelve Columns in order to sample the ceiling, the dome and the upper part of the walls (the wooden frame). A total of nine samples were collected using a scalpel. Almost all the samples analysed (Table 1) were less than 0.5 mg.

Sub-samples were taken in the laboratory from two of the samples. The red paint in sample W2 was scratched from the wood, paying attention to avoid contamination from the support. Sample W2-R, therefore, contained just the paint layer, while the wood sample (W2-W) was analysed separately in order to establish the organic materials present in the support. The red-brownish mordant and the preparation layer underneath the gilding sample (R1) were also analysed separately. The sampled layer in each sub-sample is shown in Table 1.

2.3. Experimental data and results

The samples were submitted to the analytical procedure based on GC/MS described in [15], and the results are summarised according to the identified materials (proteinaceous, lipid, resinous, saccharide).

2.3.1. Proteinaceous material

The protein content of all samples, calculated as the sum of identified amino acids, was found to be above the limit of 0.75 μg, that is the quantitation limit of the analytical procedure[1]. This indicates that quantitative analyses can be performed on the amino acidic content of all samples. Table 2 reports the relative amino acid contents of the samples, compared with those of the reference materials. The amino acid profile of all the samples showed the presence of hydroxyproline and a relatively high percentage content of glycine, consistent with the presence of animal glue.

[1] The detection and quantitation limits are determined on measures performed on analytical procedures blanks, as discussed in detail in the literature [15].

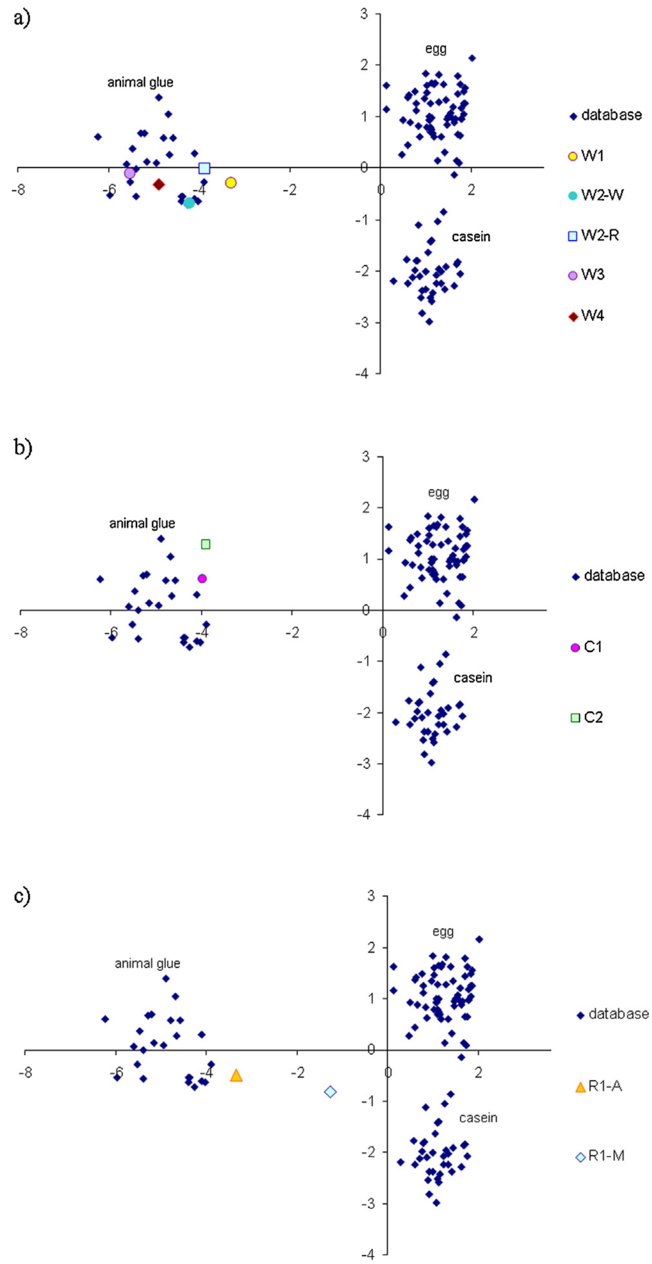


Fig. 2. PCA score plot of the relative amino acid content of the samples collected from the a) walls; b) dome and c) gilded ceiling.

The quantitative amino acid percentage content of the paint samples was subjected to a multivariate statistical analysis together with a data set of 121 reference samples of animal glue (24 samples), egg (63 samples) and casein (34 samples), using principal component analysis (PCA) [16]. The first two principal components account for 80% of the total variance of the data. The resulting PCA score plot (Fig. 2), showed that all the samples were located in the animal glue cluster or close to it apart from sample R1-M. In fact, sample R1-M (mordant layer from the gilding sample) showed a higher leucine and glutammic acid content (Table 2) than the rest of the samples. These percentages are not in agreement with characteristic animal glue values suggesting the presence of another material with a proteinaceous content in the sample. Although contamination cannot be ruled out, the use of proteinaceous materials (mainly egg white and animal glue) in the mordant layers has been extensively reported [17]. Not only do many ancient recipes report the mixture of different materials for the preparation of gilding

Table 2
Relative amino acid percentage contents of the samples collected from the walls, the dome and the gilded ceiling, compared with those of reference materials.

	ala	gly	val	leu	ile	ser	pro	phe	asp	glu	hyp
W1	8.0	24.9	3.5	6.4	2.5	3.9	7.2	4.9	10.9	21.5	6.3
W2-R	11.5	29.0	5.7	8.7	4.7	4.8	12.6	2.8	6.1	9.7	4.4
W2-W	10.2	24.5	3.9	6.1	2.8	4.7	14.7	3.3	8.5	15.7	5.7
W3	9.7	29.7	3.9	5.7	2.7	2.6	9.4	2.8	6.1	11.9	15.4
W4	10.4	26.7	4.1	6.3	3.0	2.7	10.0	3.1	6.5	15.0	12.1
R1-A	9.7	26.7	4.6	6.9	3.3	3.5	12.7	3.8	10.3	16.4	1.9
R1-M	7.5	17.0	6.1	10.8	5.7	5.2	12.4	5.3	10.5	18.2	1.3
C1	9.2	25.9	4.1	6.4	3.0	4.5	5.1	4.9	9.3	15.6	12.0
C2	9.8	23.5	4.7	7.3	3.4	13.1	5.7	3.2	6.7	11.6	11.0
Animal glue	12.3	29.4	3.9	4.7	2.5	3.8	12.4	2.8	6.6	9.9	7.7
Egg	7.7	4.8	7.7	11.0	6.7	10.3	5.7	6.4	12.6	15.0	0.0
Casein	5.0	3.0	7.6	11.9	6.6	5.8	11.5	5.9	8.5	22.2	0.0

mordant, but also the presence of animal glue through contamination from the lower layer (sample R1-A) should be considered.

2.3.2. Lipid and resinous material

Almost all the samples showed the presence of fatty acids above the quantitation limit of the procedure (2.2 μg)[1]. Samples W2-R and R1-A presented a lipid content between the detection (1.2 μg) and quantitation limits, indicating that although a lipid material is actually present, fatty acids cannot be quantified, due to the analytical blank contribution. Sample W2-W showed a content at a procedure blank level, indicating that fatty acids identified in the sample originate from the blank contamination. Fig. 3 shows the chromatograms of the lipid-resinous fraction of samples W4 and R1-M, which are representative of the profiles obtained.

Table 3 reports the values of the calculated parameters taken into account for the identification of the source of the lipid material as well as the summary of the materials identified from the lipid-resinous fraction.

The fatty acid profile as well as the characteristic parameters ($A/P > 1$: $\Sigma D > 40\%$) of almost all the samples point to the presence of a drying oil. The P/S ratio is in agreement with the

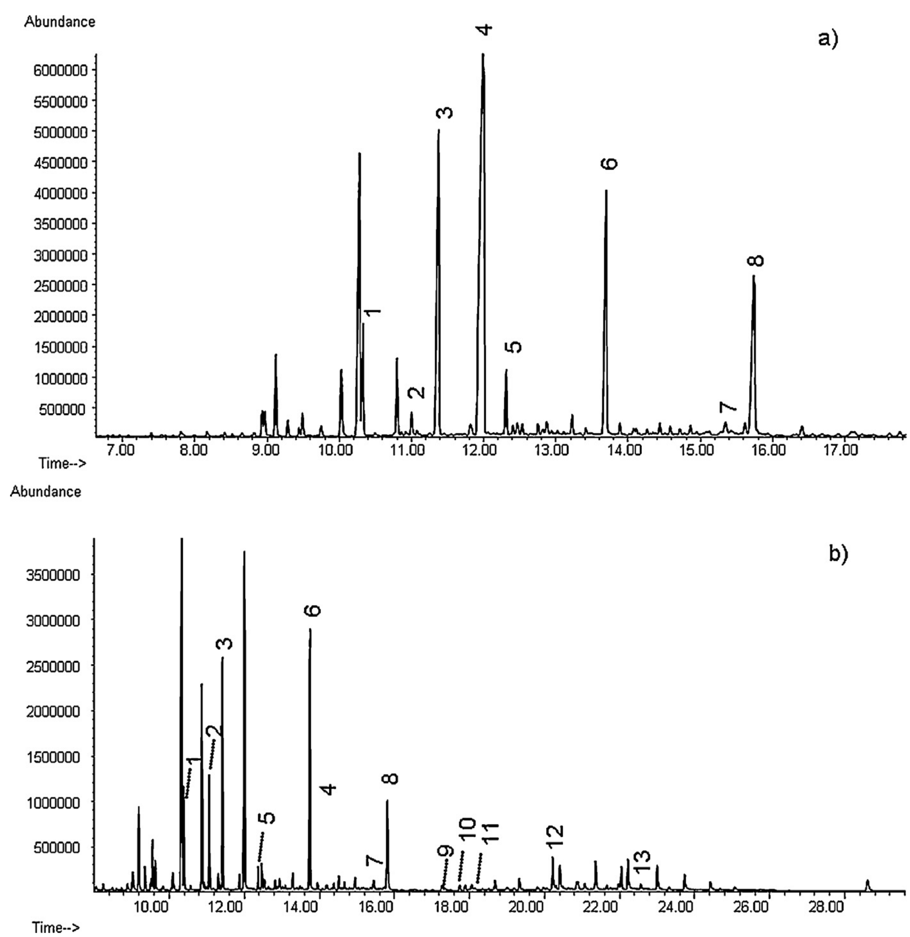


Fig. 3. Total ion chromatogram of the lipid-resinous fraction of samples: a) W4 and b) R1-M; peak labels in the figure correspond to the following compounds: 1: suberic acid; 2: vanillic acid; 3: azelaic acid; 4: 1,2 benzendicarboxylic acid, bis(2methylpropyl) ester, pollutant; 5: sebacic acid; 6: palmitic acid; 7: oleic acid; 8: stearic acid; 9: di-dehydroabietic acid; 10: dehydroabietic acid; 11: 15-hydroxyhexanedioic acid; 12: 7-oxo di-dehydroabietic acid; 13: tetracosanoic acid.

Table 3
Characteristic ratio values of the fatty acids and material identified from the analysis of the lipid-resinous fraction in the samples collected from the walls, the dome and the gilded ceiling, and identified materials.

	A/P	P/S	O/S	Σ Dic.%	Linseed oil	Pine resin	Beeswax	Vanillic acid
W1	1.0	1.5	0.3	44.8	Yes	No	No	Yes
W2-R	–	–	–	–	No	No	No	No
W2-W	–	–	–	–	No	No	No	Yes
W3	1.8	0.8	0.0	52.4	Yes	No	No	No
W4	1.9	0.9	0.1	51.3	Yes	No	No	Yes
R1-A	–	–	–	–	Trace	Trace	Trace	No
R1-M	0.7	2.0	0.1	40.2	Yes	Yes	Yes	No
C1	1.4	0.8	0.1	41.7	Yes	No	No	Yes
C2	1.3	0.7	0.1	39.2	Yes	No	No	Yes

A/P: azelaic over palmitic acid; P/S: palmitic over stearic acid; O/S: oleic over stearic acid; Σ Dic.%: sum of the dicarboxylic acids.

Table 4
Relative percentage content of monosaccharide.

	xyl	ara	ramn	fuc	galact ac	gluc ac	glu	man	galact
W1	7.1	1.6	1.3	1.5	0.0	1.4	37.4	13.2	36.5
W2-R	–	–	–	–	–	–	–	–	–
W2-W	66.3	1.9	0.7	0.0	0.0	0.3	9.3	14.5	7.0
W3	10.1	4.1	2.6	1.0	0.0	1.6	48.5	8.1	25.1
W4	55.0	6.3	0.0	0.0	0.0	0.0	0.0	17.8	20.8
R1-A	–	–	–	–	–	–	–	–	–
R1-M	–	–	–	–	–	–	–	–	–
C1	24.1	20.4	0.6	0.6	0.0	0.4	29.7	8.2	15.9
C2	60.1	1.4	1.4	0.0	0.0	1.0	11.0	17.6	7.4

Table 5
Summary of the materials identified in the samples from the Saadian tombs.

Description	Sub-sample	Proteinaceous material	Lipid material	Pine resin	Wax	Saccharide material
Wall wood framework	W1	Animal glue	Linseed oil	No	No	Yes
	W2-R	Animal glue	Contaminant	No	No	No
	W2-W	Animal glue	No	No	No	Wood contribution
	W3	Animal glue	Linseed oil	No	No	Yes
	W4	Animal glue	Linseed oil	No	No	Trace
Gilded ceiling	R1-A	Animal glue	Trace of lipid material	Trace	Trace	No
	R1-M	Animal glue + another source	Linseed oil	Yes	Yes	No
Dome	C1	Animal glue	Linseed oil	No	No	Yes
	C2	Animal glue	Linseed oil	No	No	Wood contribution

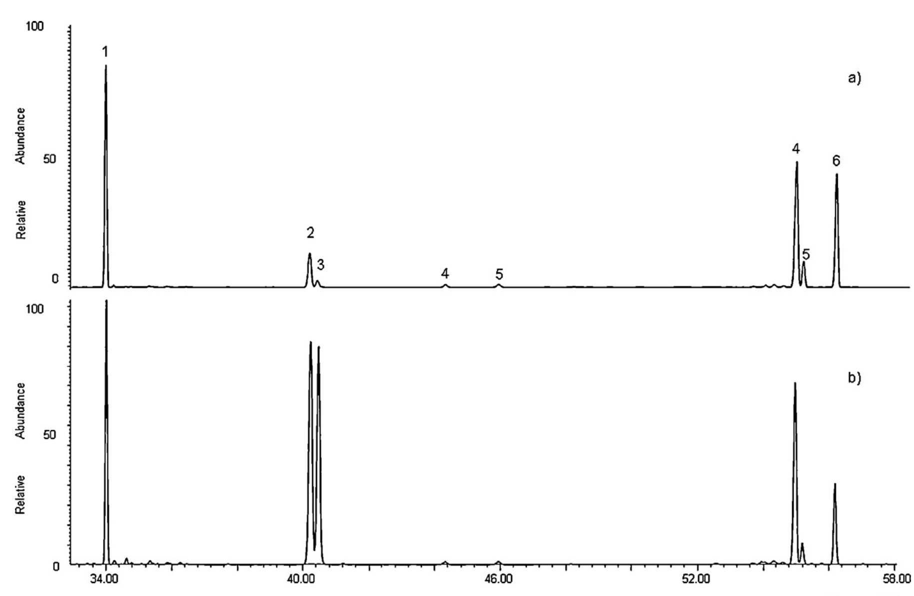


Fig. 4. Single ion chromatograms of the saccharide fraction of samples W1 a) and C1 b); peak labels in the figure correspond to the following compounds: 1: mannitol (I.S.); 2: xylose; 3: arabinose; 4: rhamnose; 5: fucose; 6: glucose; 7: mannose; 8: galactose.

characteristic parameters of linseed oil [14]. However, some samples showed particularities:

- sample R1-M parameters (A/P < 1; P/S > 2) do not fit with the expected values for a drying oil, though the azelaic and the dicarboxylic acid content points to its presence. The lack of agreement of the calculated parameters could be due to the beeswax that we found in the sample. Beeswax is a known source of palmitic acid [18] and was identified in the chromatogram on the basis of the presence of long chain linear saturated hydrocarbons, tetracosanoic acid, 15-hydroxyhexadecanoic acid and tetracosanol. Finally, the markers of an aged pine resin (di-dehydroabietic acid, dehydroabietic acid and 7-oxo di-dehydroabietic acid) [19] were identified in the sample chromatogram;
- samples R1-A and W2-R showed the presence of fatty acids between the detection and quantitation limits. Sample W2-R fatty acid profile points to environmental contamination, while sample R1-A profile, which shows a high relative percentage content of dicarboxyilic acid, suggests contamination from the drying oil identified in the layer above (sample R1-M).

The presence of vanillic acid (peak number 2 in Fig. 3), as a degradation compound of lignin [20], in the chromatograms of samples W2-W, W1, W4, C1, and C2, points to the contribution of the wood support from the sampling operation. In fact, sub-samples separated from the wood under the microscope in the laboratory before the analysis (W2-R, R1-A and R1-M) did not show the presence of vanillic acid. It should be noted that a substantial amount of vanillic acid was present in sample C2, in agreement with the saccharide fraction results discussed below.

2.3.3. Saccharide material

All the samples analysed showed saccharide material above the quantitation limit (2 μg)[1], apart from samples W2-R, R1-A and R1-M. The sugar profile of the samples is shown in Table 4, while Fig. 4 a) and b) show the chromatograms of samples W1 and C1, respectively, which are representative of the saccharide profiles obtained. Sample W2-W consists in a wood sample analysed in order to establish the composition of the wood used as a support.

The saccharide profile of sample C2 is in agreement with the profile of the wood sample, while the other samples show the contribution of a different saccharide material. All the samples showed a ratio xylose/arabinose higher than one. This ratio, which is lower than one in all the reference exuded gums from trees [21], highlights the contamination from the wood support [22]. This hypothesis is supported by the presence of vanillic acid, the degradation product of lignin. Recent data [23] have shown that contamination from the wood support as well as the presence of other materials, such as the proteinaceous ones previously identified in the samples, should be taken into account for the identification of saccharide sources in painting samples. Taking into account the decisional scheme [23] resulting from this study the source of the saccharide material is very likely to be a polysaccharide gum, and in particular tragacanth gum.

3. Conclusions

The GC/MS combined procedure led to the identification of several organic compounds in samples from the Saadian Tomb in Marrakesh. The results, summarized in Table 5, show the simultaneous presence of several types of organic materials in each micro-sample collected from the tomb, highlighting the importance of the combined GC-MS procedure used.

The gilding sample shows the most significant compositional difference from the other samples. The data from the paint samples from the wooden framework and the dome seem to indicate that the same painting technique was used in both areas, in fact the analysis mainly highlighted the widespread presence of animal glue and a drying oil, most probably linseed oil. The proteinaceous media was found both in the wood analysed (without painting layers) and on the painting layers (without the wood support). The presence of animal glue in the support could be the result of both the penetration of the binding media into the wood or the treatment of the wood before it was painted. A proteinaceous material was also identified in the Muslim paintings on plaster in Corral (Granada, Spain) [3] and in the polychrome woodwork in the Alhambra (Granada) [2], above all due to the preparation layer.

The absence of a drying oil in one of the paintings samples (W2-R) could suggest that the oil was not used as a binder but as a surface treatment of the paintings. However, to better support this hypothesis, and to understand the extent and reasons for the untreated part of the frame (W2-R), other samples from the same area need analysing. The absence of the drying oil in the wood sample (W2-W) prevents this material from being considered as an insulating layer before or between paint layers as suggested in the Alhambra woodwork [2].

Saccharide material was identified in some of the samples. Although in the plasterwork at “qubba Dar al-Manjara l-kubra” in Granada, as well as in both the dados from the Alhambra and the Royal palace of Santo Domingo (Granada) [4], the saccharide material was considered to be the binding media, its inconsistent presence in the Saadian samples points to its use as a restoration material.

Given the fact that the drying oil and saccharide material appear to be restoration materials, the fact that animal glue was also found in other studies on architectural decorations in south Spain, and that no other organic material was present, we believe that animal glue is the original binder. Despite this, we cannot exclude that animal glue could also derive from a consolidation campaign. Proteomic studies can identify the source of the animal glue [24]. If the binder and the eventual consolidating glue were of a different origin, then proteomics studies could be used in the future to verify this hypothesis.

As regards the gilding technique of the ceiling, animal glue was identified both in the mordant and the white preparation layer underneath. However, the mordant layer (sample R1-M) contained a different source of proteinaceous material, which could not be identified, as well as linseed oil, pine resin and beeswax. In this case, the linseed oil seems to come from the mordant composition and not from a superficial treatment, as far as the gold leaf was avoided when sub sampling. This result, suggests the use of a resinous-oil mixture in the mordant, in agreement with the European tradition of the time [17]. Pine resin was also found in the Alhambra woodwork mixed with oil [2].

These results provide a first attempt at establishing the materials used by the Moroccan painters in the 16th century. The materials identified in the Saadian tombs are in agreement with the scarce information in the literature regarding the Hispano-Muslim tradition dating back to the Nasrid period in Granada (1232–1492) [1–3] and add a fundamental piece of knowledge on the Muslim tradition of polychrome decorations.

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