Soot removal from ancient Egyptian complex painted surfaces using a double network gel: Empirical tests on the ceiling of the sanctuary of Osiris in the temple of Seti I – Abydos

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Abstract

In this study, we evaluated the ease of removal of soot layers from ancient wall paintings by employing double network gels as a controllable cleaning method. The ceiling of the temple of Seti I (Abydos, Egypt) is covered with thick layers of soot; this is especially the case in the sanctuary of Osiris. These layers may have been accumulated during the occupation of the temple by Christians, fleeing the Romans in the first centuries A.D.. Soot particulates are one of the most common deposits to be removed during conservation-restoration activities of (Egyptian) wall paintings. They usually mask the painted reliefs and reduce the permeability of the painted surface. A Polyvinyl alcohol-borax/agarose (PVA-B/AG) double network gel was selected for this task since its properties were expected to be compatible with the cleaning treatment requirements. The gel is characterized by its flexibility, permitting to take the shape of the reliefs, while also having self-healing properties, featuring shape stability and an appropriate capacity to retain liquid. The gel was loaded with several cleaning reagents that proved to be effective for soot removal. Two sets of soot removal tests were conducted with these gel composites. The cleaned surfaces were examined in situ with the naked eye and with a digital microscope in order to select the best gel composite. The gel composite, loaded with a solution of 5% ammonia, 0.3% ammonium carbonate, and 0.3% EDTA yielded the most satisfactory results and allowed to safely remove a crust of thick soot particles from the surface. Thus, during a third phase of the study, it was used successfully to clean a larger area of the ceiling.

1. Introduction

Accumulation of soot layers on the ceilings and walls of ancient Egyptian tombs and temples is a very common phenomenon. In the Roman period, numerous isolated tombs and temples, dating back to the Pharaonic period, were inhabited by Christian hermits. When they suffered from persecution and economic pressure by the Romans, these sites served as shelters for them and consequently some of these pagan sites were turned into churches [1, 2]. As a result of that occupation, the wall paintings of these Pharaonic sites became begrimed with thick soot layers produced by the domestic activities of the new inhabitants (cooking, lighting, heating, etc…). In addition to the adsorption of various pollutants, these soot layers obscure the wall paintings and reduce the permeability of their surface; [3, 4].

Numerous reagents have been used for the cleaning of soot and smoke particulates from artwork surfaces. They include solvents, salts, and surfactants [5, 6]. The most commonly used reagents are as follows: a) solvents such as ammonia, ethanol, white spirit, acetone, trichloroethylene, and ethyl acetate, b) surfactants such as orvus, vulpex, and triton X-100, and c) salts such as sodium bicarbonate, ammonium carbonate, sodium carbonate, tri-ammonium citrate, and ethylenediaminetetraacetic acid (EDTA). Traditionally, they are used individually or in mixtures and applied by cotton swabs or poultices [5–9]. As an example, in a recent study, Zn nanoparticles mixed with vulpex was successfully used to remove soot, patches of waxes and blood from the wall paintings in the temple of Isis in Luxor, Egypt [3]. In another study concerning conservation-restoration of the mortuary temple of Ramses III in Luxor, enzymes were used to remove soot deposits, blood of bats and a deteriorated varnish of animal glue applied as a protective coating in a previous conservation treatments [10].

In our study, cleaning tests were performed on the ceiling of the Osiris sanctuary in the temple of Seti I at Abydos, Egypt. Seti I dedicated one of the seven sanctuaries in this temple to the god Osiris. Osiris is one of the most important and popular gods in ancient Egyptian history. This importance is derived mainly from two circumstances. The first one is that he suffered a dramatic death: either by drowning or by being murdered by his brother Seth. According to the myth, his corpse was dismembered and then reconstituted and mummified by his wife Isis and his sister Nephthys. Isis, after becoming impregnated by the mummified Osiris, gave birth to a son Horus who was secretly raised by her to protect him from his uncle, and upon reaching maturity, Horus managed to triumph over Seth and was crowned king of Egypt. This myth is significant as it ensured Egyptians that they could survive after death. Secondly, Osiris was popular due to the fact that, after the victory of his son, he was installed as the ruler of the netherworld where every deceased Egyptian had to travel through for regeneration after death. The cult of Osiris was connected to Abydos (situated in Thinite province – upper Egypt) in addition to Busiris (situated in the east – central Delta) [11–14].

The sanctuary of Osiris is the most interesting one in the temple and is located in the middle of a series of seven sanctuaries devoted to different deities; see Fig. 1. All the sanctuaries contain false doors on their west side except for the Osiris sanctuary that has a real door. The latter leads to a complex of small rooms dedicated to the celebration of the Mysteries of Osiris.

The scope of this work is to evaluate the use of gels, as a controllable and safe cleaning method, for removing thick soot layers from the ceiling of the sanctuary of Osiris without provoking damage to the sensitive paint layers underneath (see Fig. 2). In previous studies, we developed a polyvinyl alcohol-borax/agarose (PVA-B/AG) double network gel and studied its properties. It was tested to remove deteriorated consolidant layers from wall paintings of the same sanctuary and it was able to successfully remove the consolidant without damaging the paint layers [15]. In addition, we investigated the different characteristics of the PVA-B/AG double network gel. According to these investigations, the gel is characterized by the following features: a) it is a flexible gel with the ability to adapt its shape to complex surfaces such as the case of ancient Egyptian painted reliefs, b) it features good shape stability when applied on painted surface for extended contact times, and c) it can be loaded with a wide range of reagents such as mixtures of polar and non-polar solvents, chelating agents, and surfactants, d) and it has a good liquid retention, a relevant property in sites at elevated temperature and at (very) low levels of relative humidity [16].

Based on the aforementioned features, the PVA-B/AG double network gel was deemed suitable for cleaning tasks in the sanctuary of Osiris. For this reason, the gel was loaded with various cleaning reagents, already documented in literature to have the ability to remove soot particles. The prepared gel composites were applied to the ceiling of the Osiris sanctuary during two cleaning tests and the results were assessed visually and microscopically. In view of the difficulty of reaching the cleaning locations (curved ceiling ca. 4 m above floor height), the option was taken not to use other/more sophisticated (spectroscopic) characterization equipment on site to assess the difference before and after treatment. The most effective gel composite was adopted to treat a larger area of the surface as a final demonstration of its efficiency.

2. Methods

2.1. Materials

Polyvinyl alcohol *(PVA)* (98.0-98.8% hydrolyzed, M.W. 146,000-186,000) and Ethanol 96% (technical) were acquired from Acros Organics. Disodium tetraborate decahydrate (borax, ACS, ISO reagent), and Titriplex® III for analysis (EDTA, ethylenediaminetetraacetic acid, disodium salt dihydrate) were obtained from Merck. Agarose *(AG)* (molecular biology grade, low EEO/Multipurpose), dimethylformamide *(DMF)* (> = 99%) and trichloroethylene were supplied by Fisher scientific. Acetone ≥ 99% (technical) was purchased from VWR chemicals. Sodium dodecyl sulfate *(SDS)* (purity 98%) and ammonium carbonate were obtained from Sigma-Aldrich. Ammonia solution 33% (pure reagent for analysis, M.W. 17.03) was acquired from ADWIC. Ethomeen® C25 was obtained from Kremer pigmente. Triton X-100 was acquired from Talas. Vulpex liquid soap was purchased from Diffher & Johnson.

2.2. Gel preparation

PVA-B/AG double network gel composites were prepared according to the procedures explained elsewhere [15]. The gel concentrations used for the first and second tests were 3% PVA, 1% agarose and 0.6% borax.

2.3. Strategy of cleaning tests

The evaluation was divided into three cleaning tests in order to determine the most efficient cleaning reagents that can be adopted for the removal of the soot layers. In the first and second tests, small gel patches loaded with various cleaning reagents were applied on small areas; the cleaning results were evaluated in order to determine the most effective one. Finally, the most efficient gel composite was employed to clean a larger area of the ceiling in the Osiris sanctuary.

3. Results And Discussions

3.1. First cleaning test

Six cleaning reagents were prepared based on literature data; the concentrations of these reagents were adjusted to be compatible with the gel. For instance, the total concentrations of the salts (e.g. EDTA, ammonium carbonate, and SDS) was kept below 1% since high concentrations of these salts cause excessive syneresis and cause the consistency of the gel to be much stiffer [16–18]. The cleaning reagents used are presented in Table 1. Gel composites GC2, GC3, GC4, and GC5 showed minor syneresis due to the inclusion of EDTA, ammonium carbonate, and SDS; the syneresis effect was higher in GC3 than that in gel composites GC2, GC4, and GC5.

Table 1
The six 3/1% PVA-B/AG gel composites loaded with the selected reagents.

Gel composites (GC)	Reagents	Concentrations in water (%)
GC1	Ammonia	5
	Acetone	5
	Triton X-100	0.5
GC2	Ammonia	5
	Ammonium carbonate	0.3
	EDTA	0.3
GC3	Ammonium carbonate	0.5
	EDTA	0.5
GC4	Trition X-100	1
	EDTA	0.5
GC5	Acetone	5
	Ethanol	1
	SDS	0.7
GC6	Ammonia	20
	DMF	10

The six gel patches were applied directly on the blackened surface of the ceiling of the Osiris sanctuary and gently pressed to adapt their shape to the structure of surface (see Fig. 3). Figures 4 (a & b) show photographs of the ceiling before and after treatment. The diameter of the circular gel patches was ca. 5 cm and had a thickness of ca. 3 mm. They were covered with polyethylene film to reduce the evaporation of the liquids. The color of the gel composites changed after several minutes of application due to the reaction with the soot deposits (see Fig. 3). After approximately 45 minutes, the gel pieces were gently peeled-off and then the exposed paint surface was treated with dry cotton swabs by gentle rolling. The temperature and relative humidity were 35° C and 41% respectively during the treatment. Figures 4 (c-h) illustrate the final results of the soot removal by the six gel composites. Visually and microscopically, GC1, GC2, and GC3 allowed to remove the thick soot layer and reveal the original surface underneath. On the other hand, GC6 did not result in any noticeable change on the treated spot. In none of the cases, the paint underneath the soot was affected by the cleaning agents. However, it was difficult to determine which was the best cleaning result due to the heterogeneity of the original surface: in some areas, the paint layers became detached prior to their soiling with soot particles. Thus, it was decided to perform more tests on other areas of the ceiling where the paint layer was still intact.

3.2. Second cleaning test

This test was performed on two areas of the North side of vaulted ceiling of the Osiris sanctuary; one red painted oval area and a nearby rectangular area, originally painted blue. Both areas were fully covered with soot (see Fig. 5). GC1 and GC2, already used during the first test, were again employed in this test in addition to three other gel composites (see Table 2). The five gel patches, each with a diameter of ca. 4 cm and a thickness of ca. 2 mm, were applied on the two different areas using the same protocol as described above. The temperature and the relative humidity were 33° C and 40% respectively.

Table 2
The five 3/1% PVA-B/AG gel composites employed during the second soot cleaning test.

Gel composites (GC)	Reagents	Concentrations in water (%)
GC1	Ammonia	5
	Acetone	5
	Triton X-100	0.5
GC2	Ammonia	5
	Ammonium carbonate	0.3
	EDTA	0.3
GC3	Ethomeen C25	1
GC4	Trichloroethylene	5
	Vulpex	1
GC5	Ethomeen C25	5

This test offered more clear results than the previous one. By means of visual examination, it became obvious that GC2 allowed to homogeneously remove most of the soot/dirt layers from the two treated painted areas. It also succeeded in softening the deposits, so that they could easily be removed afterwards by rubbing the surface with dry cotton swabs. Using a digital optical microscope, it is clear that GC2 also effectively removed the soot from the cracks of the paint layer while the other gel composites did so in a less efficient manner. As shown in Figs. 6 and 7, some deposits are still present on the surface and within the cracks. Thus, we can conclude that GC2 is the most efficient gel composite for the removal of soot layers in this specific context. It is worth mentioning that GC5 (loaded with 5% ethomeen C25) was less efficient than GC3 (loaded with 1% ethomeen C25) and also left behind an oily stain on the treated surface.

3.3. Third cleaning test

In this test, a large GC2-loaded gel patch was applied on an area of the ceiling of around 18 × 10 cm^2. The concentration of PVA and agarose was adjusted to be 4% and 1% respectively for the following reasons: a) improve the workability of the gel, b) increase its shape stability and c) enhance the liquid retention to suit the elevated temperature in the archaeological site and to permit reuse of the gel. The gel was assembled piece by piece on the surface to form a large single gel slab, by taking advantage of its self-healing properties (see Figs. 8 (a-c)). Once applied on the surface, the gel was covered with a polyethylene sheet and after ca. 45 minutes, was peeled-off, as indicated in Fig. 8 (d). During this test, the temperature was 35.5° C and the relative humidity was 30.5%. It was obvious that the gel has adapted to the reliefs on the ceiling and removed part of the deposited soot while softening the rest. Thus, the softened residual deposits could be easily removed with dry cotton swabs. As such, similar results could be drawn during the second test regarding the suitability of the CG2-loaded gel for soot deposits removal.

The same gel slab was then reused two times on nearby locations; the results are summarized in Figs. 8 (e & f). Unfortunately, during peeling-off the gel, it left residues behind on the surface. This indicates that the mechanical properties of the gel significantly declined, likely due to the loss of an important part of its cleaning liquid load. The highly porous and dry surface is able to absorb these liquids without any problem. Moreover, the cleaning efficiency noticeably decreased in the third cleaning trial (see Fig. 9).

4. Conclusions

In this study we evaluated the possibility of cleaning the soot-soiled ceiling of the sanctuary of Osiris in the temple of Seti I at Abydos, Egypt by means of a PVA-B/AG double network gel patches. For this reason, the gel was loaded with several cleaning reagents. Most of the PVA-B/AG gel composites tested, allowed to remove the soot layers to different degrees in the first and second cleaning tests. In none of the cases, the underlying paint layers were affected. Based on visual and microscopical observations, the gel loaded with 5% ammonia, 0.3% ammonium carbonate, and 0.3% EDTA allowed to remove most of the soot/dirt particulates from the surface as well as from the cracks of the paint layers without causing any noticeable damage. In a third test, the above-mentioned gel composite was used to clean larger area of the ceiling of the sanctuary (ca. 18 × 10 cm^2). In addition, we evaluated the possibilities of reuse of the same gel slab for extra two times. The results were satisfactory during the first two treatments while the results of the third repeat treatment were less so. However, after the second and third surface treatments with the same gel slab, it adhered to the surface and left residues during peeling-off. This suggests a decline of the mechanical properties of the gel that can be ascribed to the excess absorption of part of its liquid load by the porous surface.

Regarding the PVA-B/AG double network gel, it could be successfully loaded with variable types of cleaning agents ranging from polar/non-polar organic solvents, salts, and surfactants. These reagents can be loaded individually or in combinations. However, the concentration of some of these reagents should be adjusted to be compatible with the gel such as when salts and non-polar solvents are loaded and to avoid excessive syneresis to the gel [15, 17, 18]. The PVA-B/AG double network gel proved to be flexible enough to adapt to the structure of the complex surface of the reliefs. By regulating the concentrations of the PVA and agarose in the double network, it was possible to create gels with flexibility and liquid retention suitable for the cleaning needs of the case study. The self-healing character of the gel allowed for an effortless application of the gel to the surface in small adjacent pieces that healed within a few minutes to form as single large slab. The reuse of the gel depends on the porosity of the surface to be cleaned. When the porosity of the surface is low, the gel would not lose too much of its liquids which makes it suitable for another application. In case of strongly absorbing (porous) surfaces, reuse of the gel is not recommended.

The results presented in this work can serve as a model for future soot removal that would be conducted in the other sanctuaries of the temple. Furthermore, they can be applicable to other cases; however, conservators-restores should always implement pre-tests in advance to regulate the proper gel formulation and contact time that suit the wall painting to be treated.

Abbreviations

PVA-B
Polyvinyl alcohol-borax
AG
Agarose
EDTA
Ethylenediaminetetraacetic acid
SDS
Sodium dodecyl sulfate
DMF
Dimethylformamide

Declarations

Availability of data and materials

All the data are available within the manuscript.

Competing interests

The authors declare that they have no competing interests.

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Authors' contributions

EA designed the strategy for gel cleaning tests and prepared the gels as well as he wrote the first draft of the manuscript. AM carried out the field tests. KJ and JC contributed to the strategy of the research as well as writing and revising the manuscript. All authors read and approved the final version of the manuscript.

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Figures

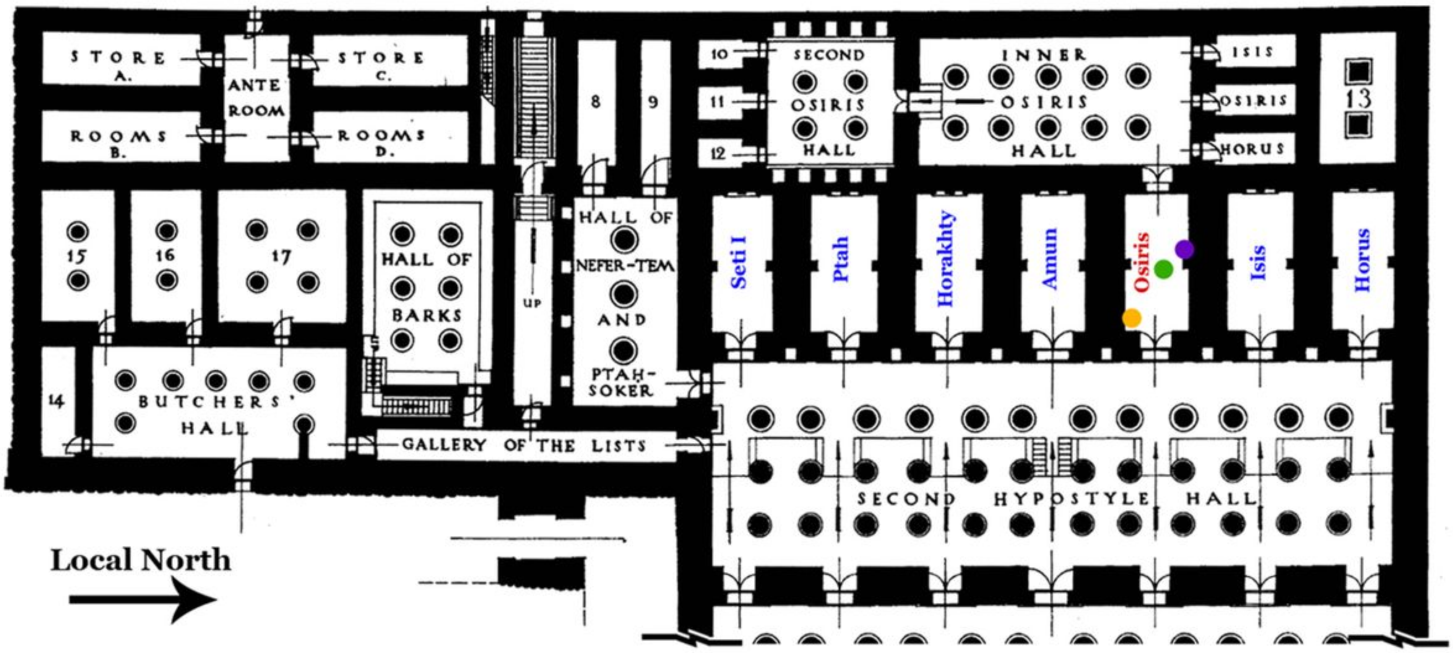

Figure 1

Part of the plan of the temple illustrating the seven sanctuaries and the complex of smaller rooms behind the Osiris sanctuary; adapted from [19]. The color dots refer to the three areas where the gel cleaning tests were conducted (Green dot: 1st test, Purple dot: 2nd test, Yellow dot: 3rd test)

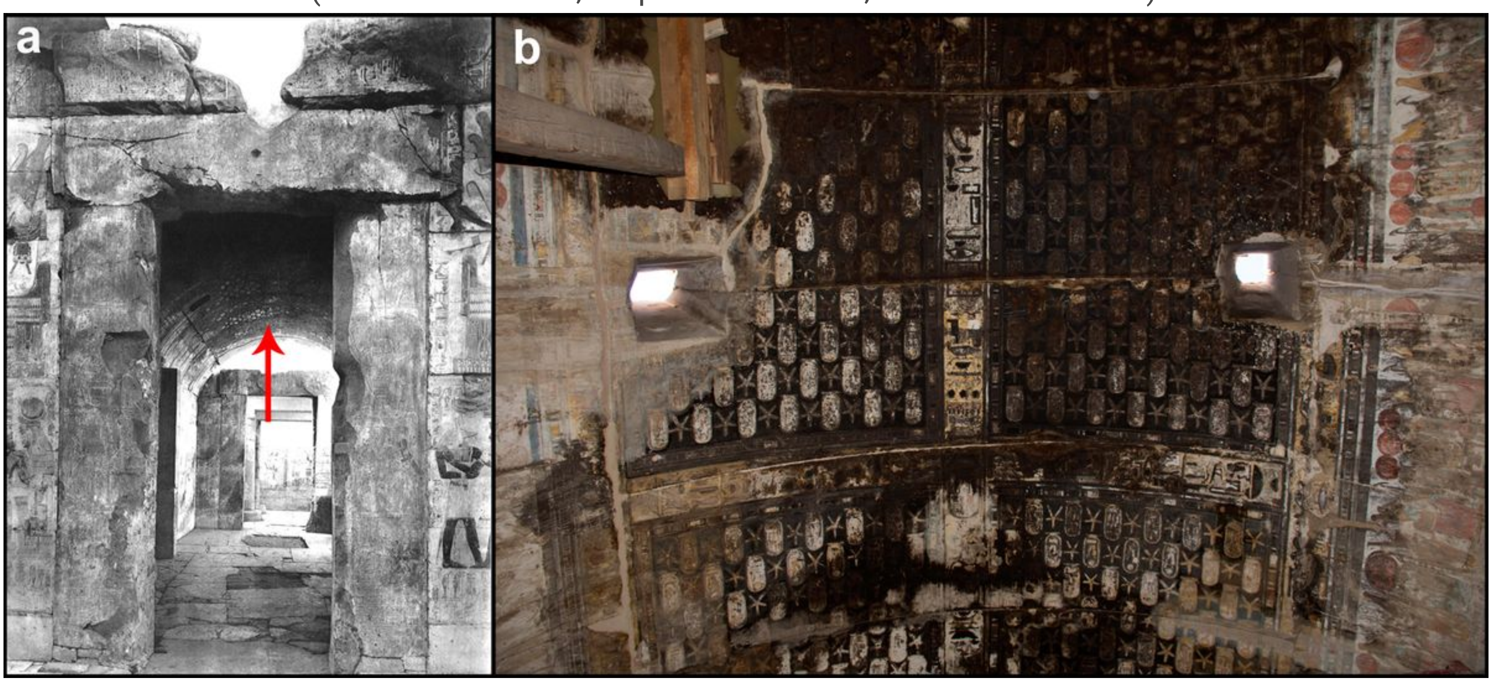

Figure 2

a) Black and white photograph from 1930s of the Osiris sanctuary (looking to the West), showing the vaulted ceiling covered with black soot layers (red arrow) [19]. b) Photograph of the painted ceiling of the Osiris sanctuary, largely covered in soot.

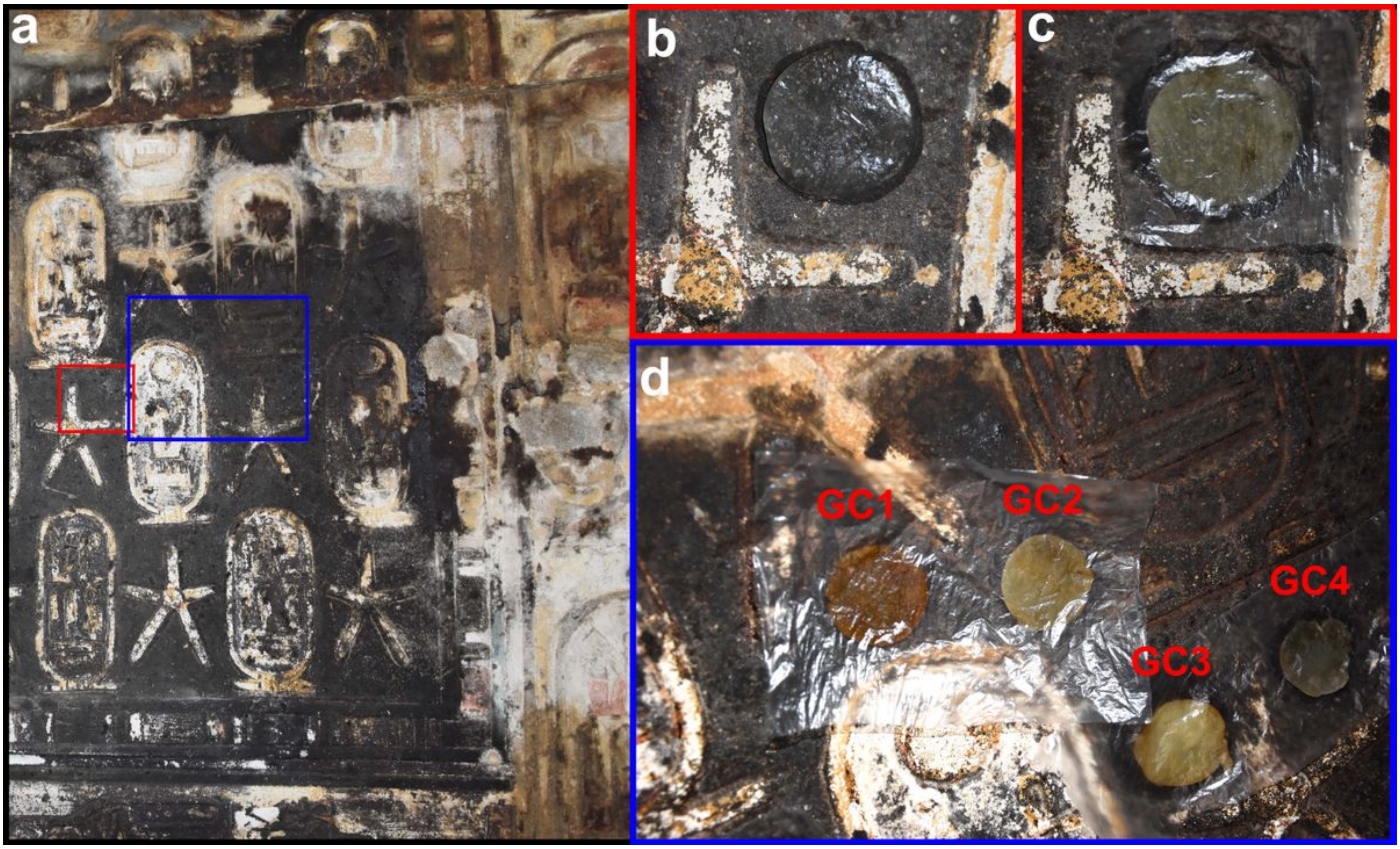

Figure 3

Color changes in the gel patches after application for 30 minutes. a) View of the surface selected for treatment. b) Transparent appearance of gel patch (GC5, see red rectangle in (a)) few seconds after application c) Changes to the color of the same gel patch after 30 minutes of application. d) Color of GC1, GC2, GC3, and GC4 patches (inside blue rectangle in (a)) after 30 minutes of application (all gel patches are covered with polyethylene foil).

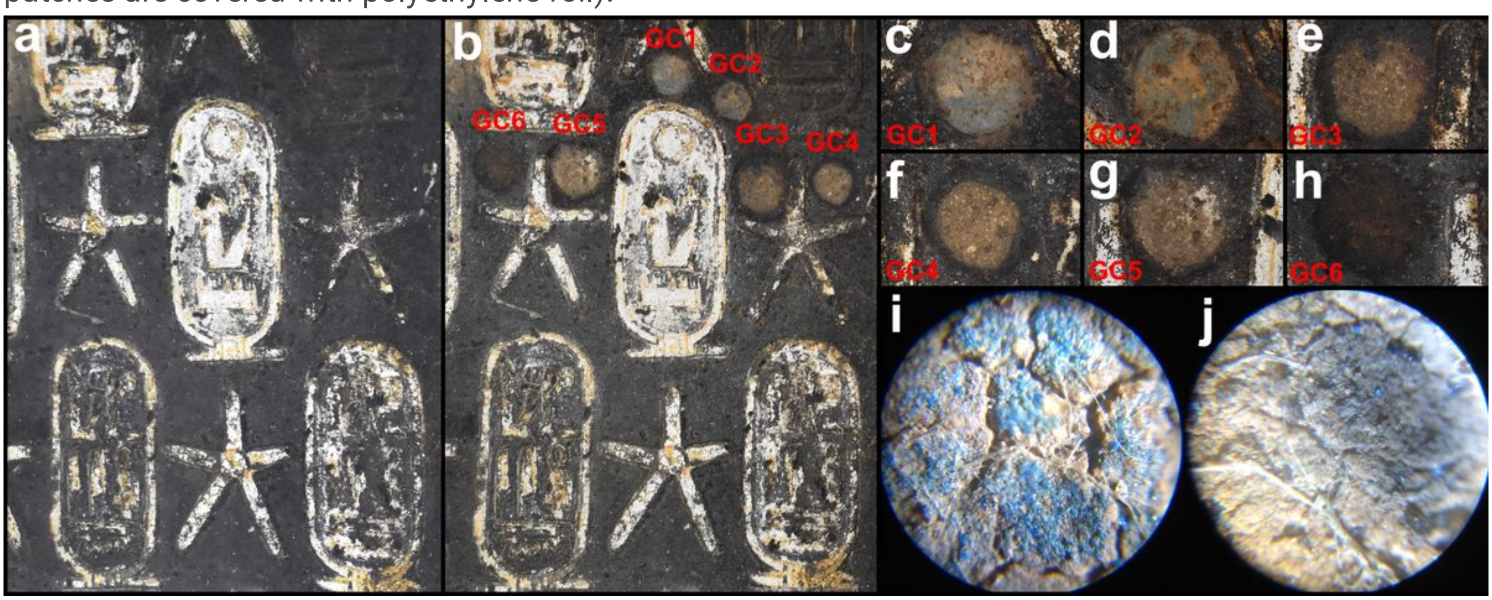

Figure 4

a) View of the selected area prior to cleaning. b) The same view after treatment with 6 circular gel patches. c-h) Close up view of the treated spots with the six cleaning composites. i) Optical microscopy (OM) image of GC2 treated spot showing the blue paint layer free from soot deposits on the surface and in the cracks. j) OM image of GC5-treated spot, still showing the presence of soot particles on the surface after the treatment.

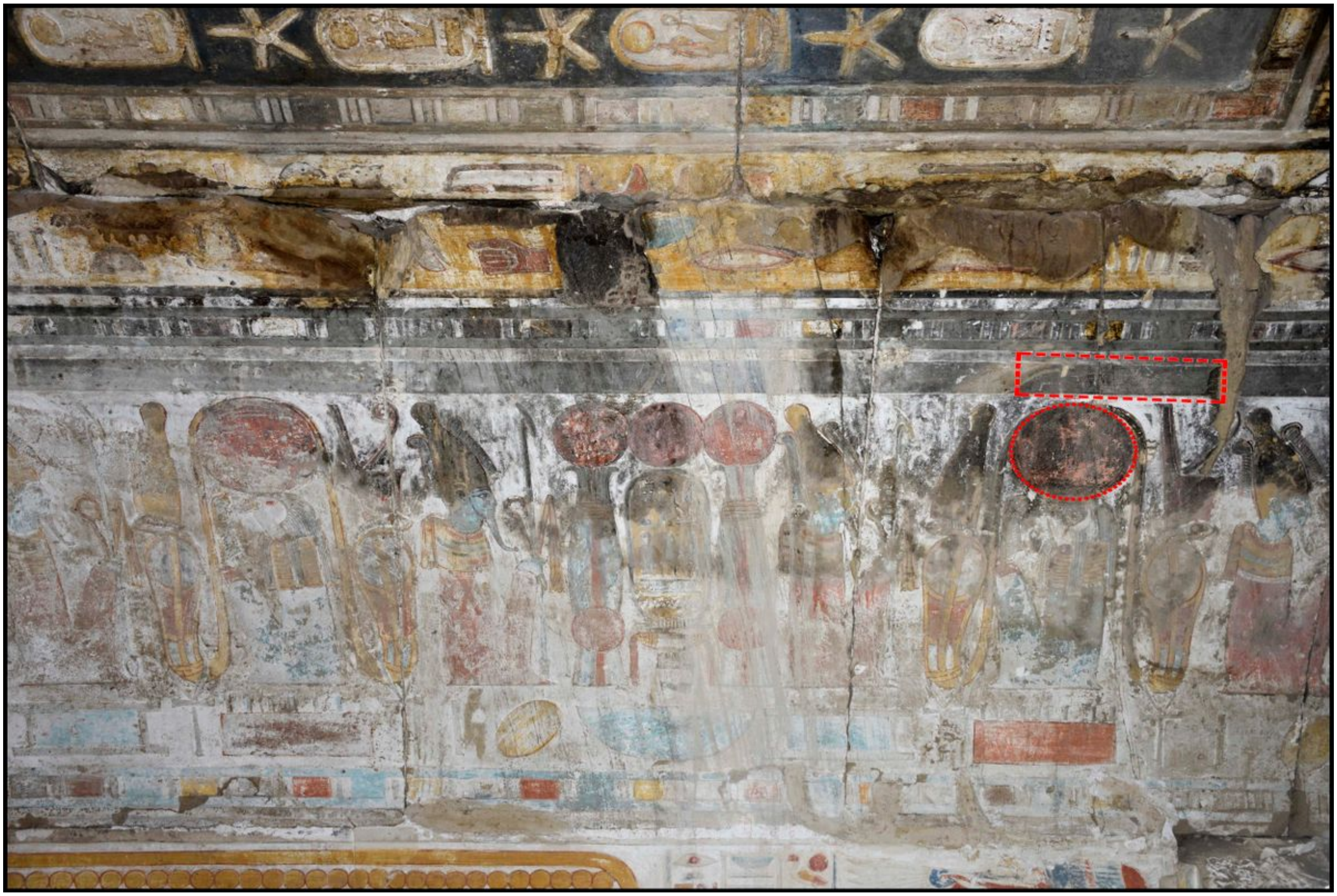

Figure 5

Selected areas for the second test located on the side of the vaulted ceiling, North wall.

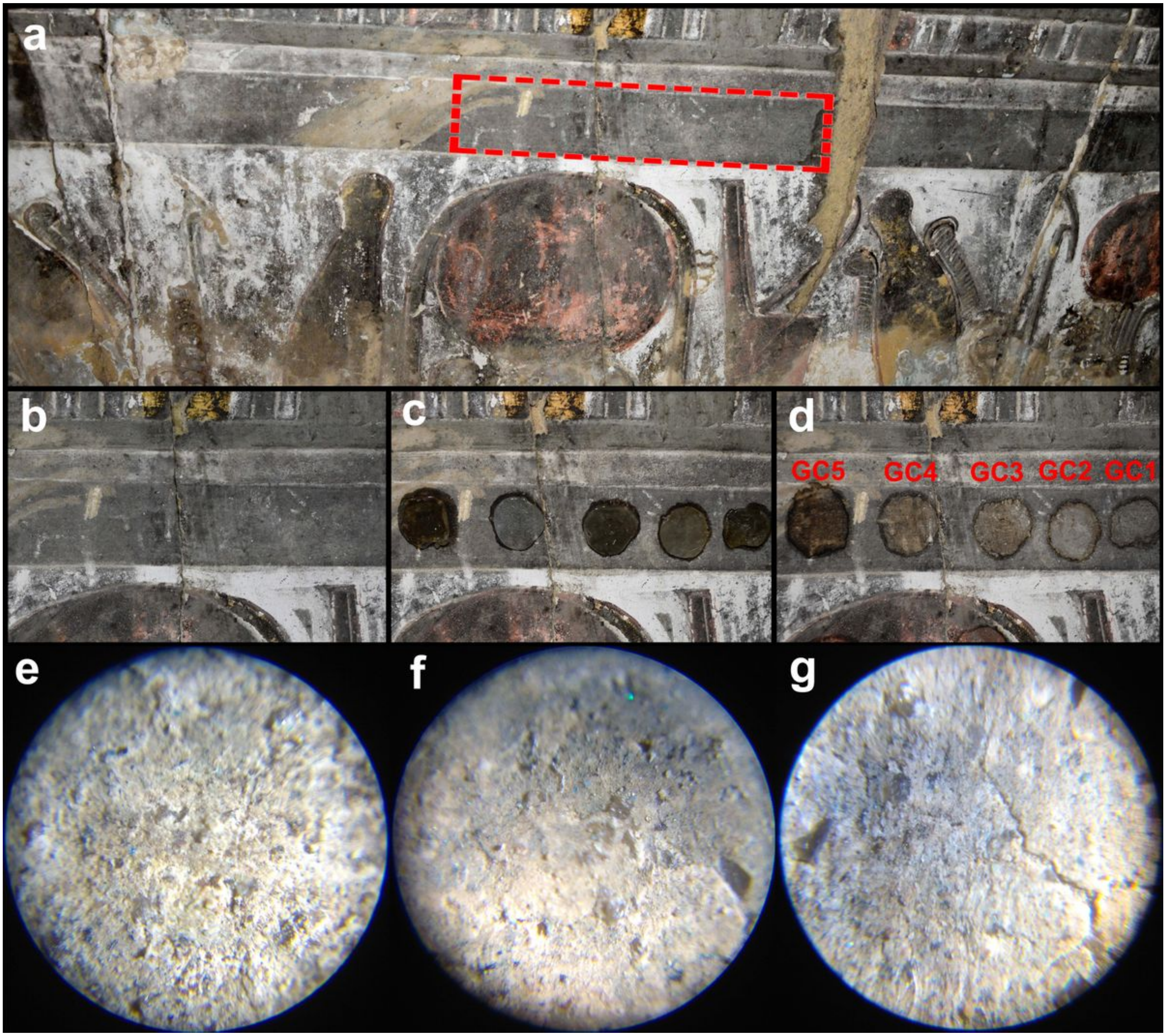

Figure 6

a) General view of the selected area for the test, b) before treatment, c) during application of the five gel composites, and d) after treatment. e) Spot treated with GC2 under the microscope shows no soot deposits while f) and g) show residues of soot particles on the spots treated with GC4 and GC5 respectively. All microscopic images acquired at a magnification of 120 x.

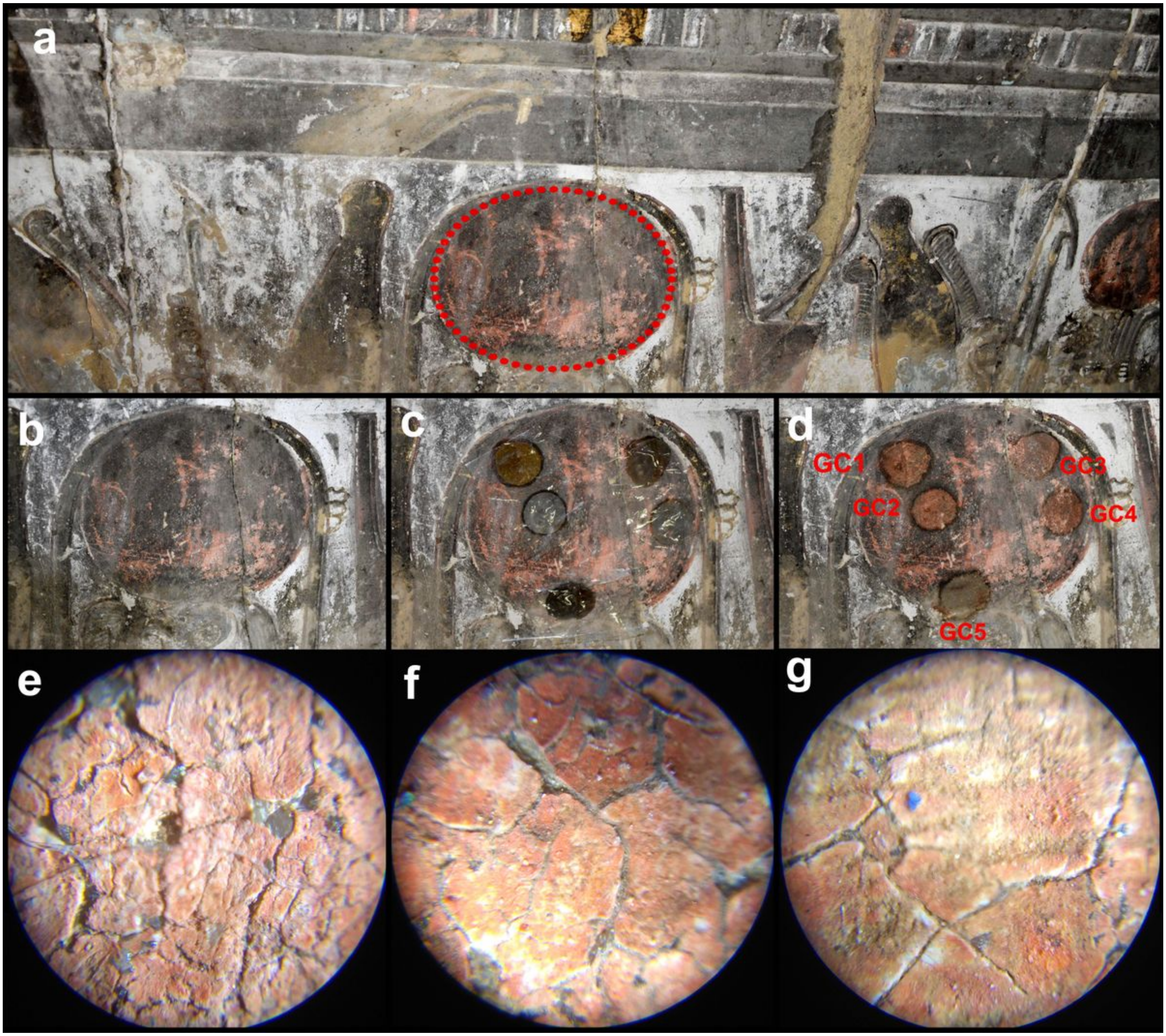

Figure 7

a) General view of the selected area for the test, b) before treatment, c) during application of the five gel composites, and d) after treatment. e) Spot treated with GC2 under the microscope shows no soot deposits while f) and g) show residues of soot particles on the spots treated with GC4 and GC5 respectively. All microscopic images acquired at a magnification of 120 x.

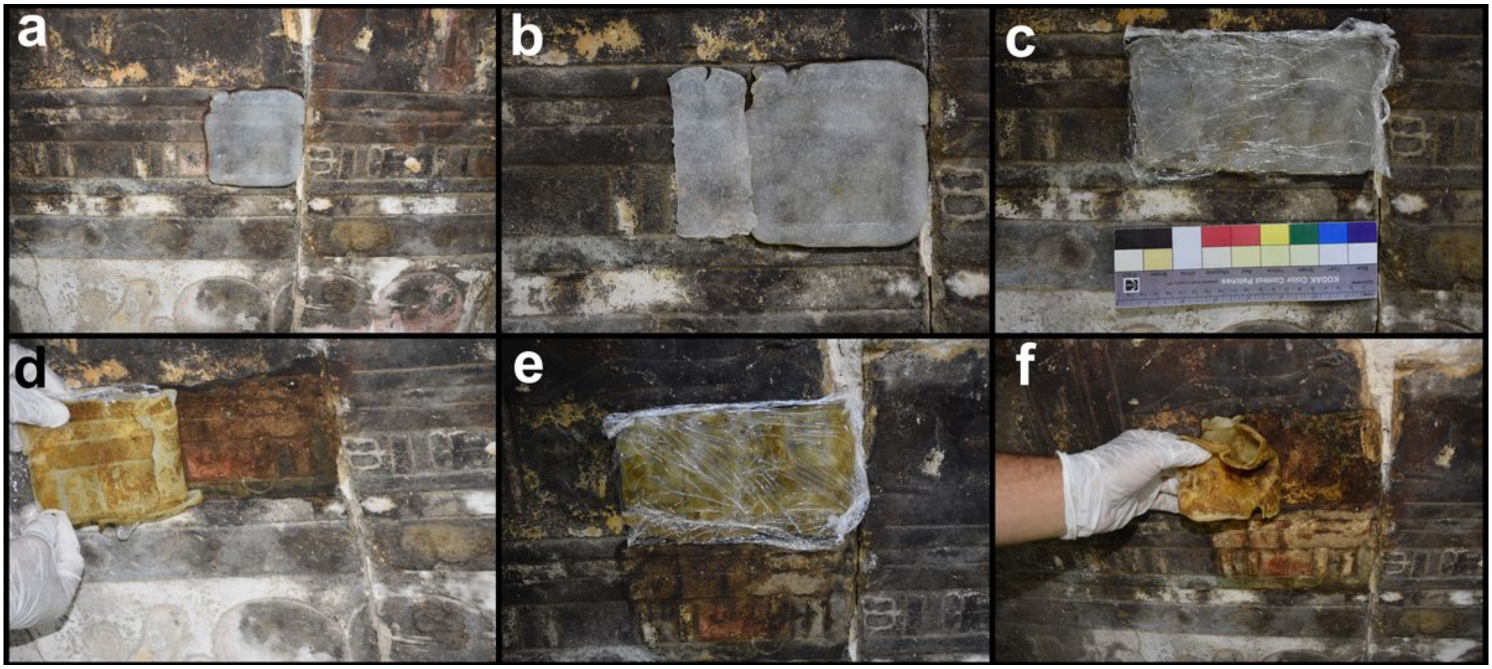

Figure 8

a), b) and c) illustrating the assembling of the PVA-B/AG gel (loaded with 5% ammonia, 0.3% ammonium carbonate, 0.3% EDTA) and covered with a plastic sheet. d) Removing the gel after ca. 45 minutes. The gel self-healed into a single gel slab e) Applying the other side of the same gel slab for the second time application. f) Removing the gel slab after the second application. It is noticeable that the adhered to the surface due the decline of its mechanical properties.

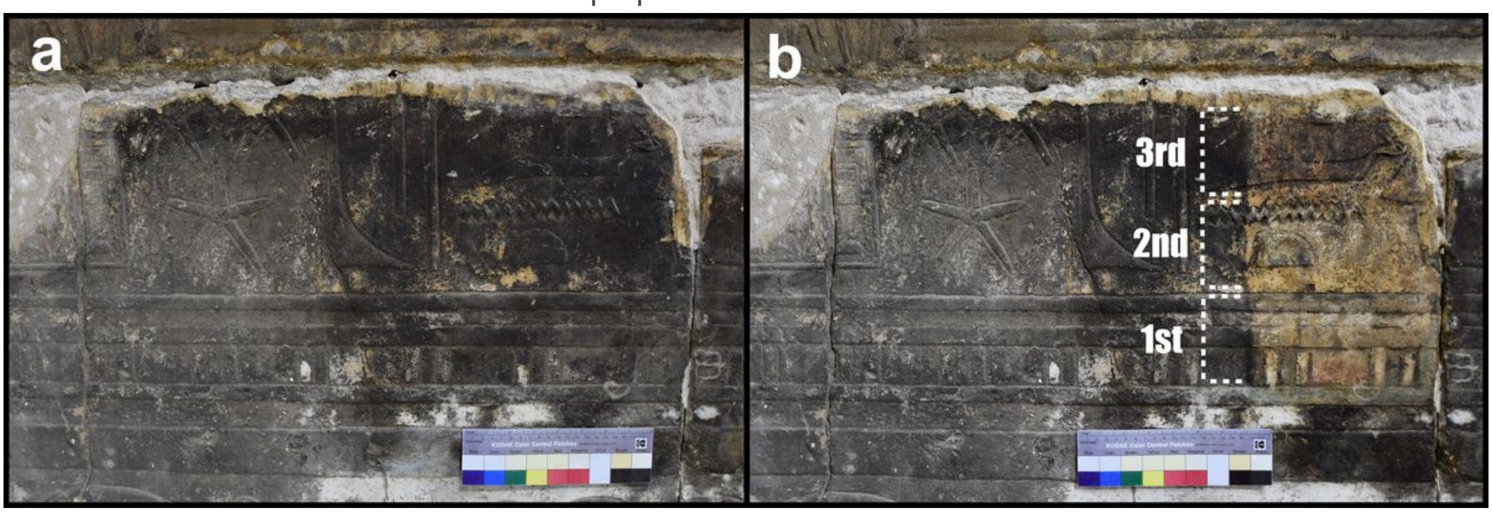

Figure 9

View of the selected area for the third test before the cleaning. b) The same view after the first, second, and third treatment with the same gel slab. It is clear that the third treatment did not show as satisfying results as the first two treatments.

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