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Evaluation of the Efficiency of Polymethyl Methacrylate/ Hydroxypropyl Methacrylate (MMA/HPMA) Copolymer for the Conservation of Mural Paintings

Mahmoud Sayed Korany, Wael Sabry Mohamed

Abstract: This study investigates the mural paintings in tomb TT81 of the 18th Dynasty, belonging to Ineni, in the Theban Necropolis, a UNESCO World Heritage Site in Egypt. It examines the composition, structure, and techniques of the mural layers, as well as main deterioration patterns and contributing factors. Cross-sectional examination, stereomicroscopy, and scanning electron microscopy assessed stratigraphy, paint layer details, and microstructure. X-ray diffraction identified pigments and fillers, and ATR-FTIR determined the paint medium. The efficiency of Polymethyl Methacrylate/Hydroxypropyl Methacrylate (MMA/HPMA) copolymer in consolidating murals under sudden microclimatic changes was evaluated. Samples treated with 1%, 2%, and 3% copolymer were analysed using microscopy, colorimetry, and water contact angle measurements. Results revealed animal glue tempera with Egyptian blue, green, red and yellow ochre, and carbon black. Previous deterioration included coloration changes, cracking, flaking, vandalism, and prior resin–oil restorations. The 2%–3% copolymer effectively consolidated layers, improved water resistance, and minimized visual alterations.

Keywords: Theban Necropolis, scanning electron microscopy, tempera, microclimate, flash floods, consolidation

Evaluación de la eficiencia del copolímero de metacrilato de metilo/hidroxipropil metacrilato (MMA/HPMA) para la conservación de pinturas murales

Resumen: Este estudio investiga las pinturas murales de la tumba TT81 de la Dinastía XVIII, perteneciente a Ineni, situada en la Necrópolis Tebana, sitio inscrito en la Lista del Patrimonio Mundial de la UNESCO en Egipto. Se examinaron la composición, estructura y técnicas de las capas murales, así como los principales patrones de deterioro y los factores que contribuyen a ellos. El examen de secciones transversales, la estereomicroscopía y la microscopía electrónica de barrido permitieron evaluar la estratigrafía, los detalles de la capa pictórica y la microestructura. La difracción de rayos X identificó los pigmentos y cargas, mientras que el ATR-FTIR determinó el medio pictórico. Se evaluó la eficacia del copolímero de metacrilato de metilo/hidroxipropil metacrilato (MMA/HPMA) en la consolidación de murales sometidos a cambios microclimáticos bruscos. Las muestras tratadas con copolímero al 1 %, 2 % y 3 % fueron analizadas mediante microscopía, colorimetría y medición del ángulo de contacto con el agua. Los resultados revelaron el uso de témpera con cola animal y pigmentos como el azul egipcio, ocre verde, rojo y amarillo, y negro de carbón. El deterioro previo incluía cambios de coloración, agrietamiento, descamación, vandalismo y restauraciones antiguas con resina y aceite. El copolímero al 2 % –3 % consolidó eficazmente las capas, mejoró la resistencia al agua y minimizó las alteraciones visuales.

Palabras clave: Necrópolis Tebana, microscopía electrónica de barrido, témpera, microclima, inundaciones repentinas, consolidación

Avaliação da eficiência do copolímero de polimetilmetacrilato/hidroxipropilmetacrilato (MMA/ HPMA) para a conservação de pinturas murais

Resumo: Este estudo investiga as pinturas murais da tumba TT81 da 18.ª Dinastia, pertencente a Ineni, na necrópole tebana, classificada como Património Mundial da UNESCO, no Egito. Analisa-se a composição, a estrutura e as técnicas das camadas murais, bem como os principais padrões de deterioração e os fatores que para eles contribuem. A estratigrafia, os detalhes da camada pictórica e a microestrutura foram avaliados através de observação em corte estratigráfico, estereomicroscopia e microscopia eletrónica de varrimento. A difração de raios X permitiu identificar pigmentos e cargas, e a ATR-FTIR foi utilizada para determinar o aglutinante pictórico. Foi avaliada a eficiência do copolímero de polimetilmetacrilato/hidroxipropilmetacrilato (MMA/HPMA) na consolidação de pinturas murais sujeitas a alterações microclimáticas súbitas. Amostras tratadas com 1%, 2% e 3% de copolímero foram analisadas por microscopia, colorimetria e medições do ângulo de contacto com a água. Os resultados revelaram uma têmpera de cola animal com azul egípcio, ocres verde, vermelho e amarelo, e negro de fumo. A deterioração prévia incluía alterações de cor, fissuração, destacamentos, vandalismo e intervenções anteriores com resina–óleo. O copolímero a 2%–3% consolidou eficazmente as camadas, melhorou a resistência à água e minimizou as alterações visuais.

Palavras-chave: necrópole tebana, microscopia eletrónica de varrimento, têmpera, microclima, cheias repentinas, consolidação

Introduction

Luxor, or Ancient Thebes, and its necropolis are a UNESCO World Heritage Site, containing numerous temples and tombs. Theban tombs, especially in Sheikh Abd el-Qurna, exhibit exceptional artistry and reveal the social, religious, and political contexts of ancient Egypt through their mural paintings and inscriptions (Moussa *et al.* 2009).

These murals face serious threats from environmental conditions, including temperature and humidity fluctuations (Marey, 2009), flash floods (Demas and Agnew 2012), wind-driven suspended particles and sand (Mikayama *et al.* 2015), biological deterioration, human impact, and the geological structure of the tombs. High temperatures and fluctuations damage plaster and paint layers, causing cracking, peeling, flaking, delamination, pigment fading, superficial erosion, and accelerated biological growth (Moussa 2007). Flash floods threaten structural stability, particularly in tombs carved into the shale of the Esna Formation, causing swelling, shrinkage, and rock deterioration (Wüst & McLane 2000; Hemeda 2021). Humidity promotes fungal and mold growth, the lixiviation of the materials and the dangerous salts effects. Human activities—including vandalism, tourism, and incompatible restoration—further exacerbate deterioration (Abd El-Tawab & Abu El-Hassan 2021). Previous restorations often used materials incompatible with the original compositions, such as insoluble nylon coatings that are difficult to remove and may harbor bacteria (Moussa *et al.* 2021).

Conservation of these murals, particularly from the Eighteenth Dynasty, is essential. Natural and synthetic adhesives commonly used include gelatine, rabbit skin glue, isinglass, Klucel G, BEVA 371, Evacon R, Mowilith 20, and Paraloid B-72 (Poulis *et al.* 2022). Acrylic polymers such as Paraloid B-72, and Lascaux Hydroground 750 have been used to protect wall paintings (Refaat *et al.* 2020). Other consolidants—polyvinyl acetate, polyvinyl alcohol, polyacrylates, silicone–acrylate copolymers, gelatine, Primal AC33, Ketone Resin N, Laropal A81, and calcium alkoxides—address flaking, powdering, surface disruption, and paint loss, also it has been used the ethyl silicate for the consolidation and the hydrophobic protective effect (Su et al., 2018; Farmakalidis et al., 2016; Becherini *et al.* 2018). Nanocomposite polymers based on TEOS and SiO_2–Al_2O_3 have been tested as superhydrophobic protective films (Marey Mahmoud 2022).

Polymethyl Methacrylate/Hydroxypropyl Methacrylate (MMA/HPMA) is a copolymer combining the stability of PMMA with the adhesion and flexibility of HPMA, suitable for consolidating mural paintings (Abdel-Maksoud *et al.* 2024). PMMA is transparent, moisture-resistant, and UV-stable, preserving optical properties while resisting heat and flash floods (Ali *et al.* 2015; Kavda *et al.* 2023; Adaikalam *et al.* 2024; Ali 2021; Prasad & Lal 2023). HPMA's hydroxyl groups enhance adhesion via hydrogen bonding and strengthen mechanical properties, allowing penetration of microcracks (Briggs & Jialanella 2010; Trujillo-Lemon *et al.* 2007). Similar acrylic systems have successfully conserved other artifacts (Abdel-Kareem & Nasr 2010; Hassan & Mohamed 2017; Abdel-Maksoud *et al.* 2024).

Despite the wide use of acrylic-based polymers in different conservation fields, their application in mural painting conservation remains controversial, particularly due to issues related to compatibility, permeability, and long-term behavior. Therefore, the present study does not propose MMA/HPMA as a general protective or preventive treatment for mural paintings. Instead, it aims to experimentally evaluate, under controlled laboratory conditions, the consolidating and adhesive efficiency of the MMA/HPMA copolymer when applied at low concentrations, with particular focus on its penetration capacity, crack-filling ability, and short-term response to environmental stress. The study explicitly recognizes the need to distinguish between laboratory-based performance and potential in situ application, which must remain strictly limited and carefully controlled.

The case study is the tomb TT81 in Sheikh Abd el-Qurna, part of Ancient Thebes and its UNESCO World Heritage necropolis. TT81, dating to the 18th Dynasty, belongs to Ineni, an architect and official under Amenhotep I–Thutmose III. The tomb has a T-shaped layout with a transverse hall leading to a pillared hall, a central axial passage, and an inner chamber with a shrine of four statues. Walls feature inscriptions and scenes highlighting Ineni's achievements, royal projects, and religious devotion, including a small chapel for ritual activities to ensure his safe passage to the afterlife (Porter & Moss 1970; Dziobek 1992).

Materials and Methods:

—*Experimental Setup*

• *Preparation of Mural Painting Samples*

Four mural painting mock-up samples with the same stratigraphic structure and composition as the archaeological samples were prepared. Each sample consists of a coarse preparatory ground layer, followed by a fine preparatory ground layer, then a whitewash layer, and finally a paint layer composed of hematite red pigment mixed with animal glue. Then the mock-ups were kept at room temperature for 21 days (Marey Mahmoud 2022). The first sample serves as the control, while the other three samples were treated with MMA/HPMA at concentrations of 1%, 2%, and 3%.

• *Accelerated Ageing of Mural Painting Samples*

The artificial ageing of the mural painting mock-up samples was performed in an oven (OCT.40, Chem-Tech Egypt) at the Oil Paintings Laboratory, Faculty of Archaeology, Luxor University. Samples were exposed to temperatures of 50–70 °C and relative humidity of 40–60% (Alemam 2021). Temperature and humidity were periodically raised and lowered to simulate seasonal, and daily variations. Each cycle comprised 8 h of exposure followed by 4 h at room temperature (Marey Mahmoud 2022), repeated 10 times (Ramadan *et al.* 2024). MMA/HPMA was applied by brushing at room temperature and pressure, repeated three times with 30 min intervals. Treated samples were cured for 15 days (Darwish 2013).

The application protocol adopted in this study is intended solely for experimental evaluation and does not represent a standardized conservation treatment. MMA/HPMA was applied by soft brushing under ambient laboratory conditions, following light surface pre-humectation to enhance penetration and reduce surface accumulation. The consolidant was applied in three successive applications with controlled intervals, allowing partial solvent evaporation between layers. This methodology was selected to simulate localized consolidation scenarios rather than large-scale or preventive treatments.

• *Preparation of Polymethyl methacrylate / Hydroxypropyl methacrylate (MMA/HPMA) by Emulsion Polymerization*

In a 250 mL round-bottom flask, poly (MMA-co-HPMA) was prepared by emulsion copolymerization of methyl methacrylate (MMA) and hydroxypropyl methacrylate (HPMA) at a 90:10 weight ratio. The emulsion contained 10% monomer mixture with sodium dodecyl sulfate (SDS) as emulsifier. The mixture was stirred for 30 min at room temperature, then heated to 70 °C. The initiator (PPS) was added, and stirring continued for 5 h. All components—monomer mixture, emulsifier, and initiator—were dissolved in the aqueous phase prior to polymerization.

— *Analytical and Investigative Techniques for Mural Painting Samples and Evaluation of MMA/HPMA Conservation Efficiency*

Various analytical and investigative techniques were employed to identify the components, structure, and techniques of the mural painting layers (Ali *et al.* 2021), and to assess key factors contributing to their deterioration. Since the samples were not subjected to prior restoration or conservation treatments, their preservation state remains unaltered, reflecting authentic characteristics (Cortea *et al.* 2024). These techniques were also applied to evaluate the efficiency of MMA/HPMA in conserving the mural painting samples.

• *Stereomicroscope Investigation*

A bench-top research zoom stereomicroscope (Discovery V.20) with reflected light and a Zeiss Axiocam ERC5S digital camera, at the Conservation Department, Faculty of Archaeology, Luxor University, Egypt, was used to examine cross-sections of mural painting samples at various magnifications. It also enabled detailed observation of the paint layer surface (Mohie & Korany 2025), including aged experimental samples before and after MMA/HPMA treatment. The examination assessed changes in surface texture, consolidant penetration, and its effectiveness in stabilizing the paint layer and filling microcracks.

• *Scanning Electron Microscope (SEM) Investigation*

A SEM Model JSM 5400 LV, equipped with an EDX Link ISIS detector from Oxford Instruments under high vacuum, at the Scanning Electron Microscope Unit, Assiut University, Egypt, was used to examine the microstructure and surface morphology of the mural painting samples. This enabled detailed investigation of pigment particles, layer adhesion, and deterioration features, providing insights into the samples' structural integrity and composition. SEM analysis was also applied to aged experimental samples before and after MMA/HPMA treatment to assess the material's penetration, its effect on pigment cohesion, and its role in filling intergranular voids and microcracks in the paint layers.

• *X-ray diffraction (XRD) Analysis*

X-Ray Diffraction equipment, Model PW/1480 with a Fe-Filter, operating at 40 kV and 30 mA with a scanning speed of 0.02/sec, at XRD Lab., Faculty of Archaeology, Cairo University, Egypt, was used to identify the pigment materials in the paint layer, the whitewash, and the filler materials in the ground layer.

• *Attenuated Total Reflection Fourier transform infrared (ATR-FTIR) spectroscopy Analysis*

Attenuated Total Reflection (ATR)-FTIR spectra were obtained using an ALPHA II FTIR spectrometer (Bruker Optik GmbH, Germany) in the range 4000–400 cm^{-1} with a resolution of 4 cm^{-1} at the National Research Centre, Dokki, Cairo, Egypt. This analysis was employed to identify the paint medium or binding materials in the paint layers. FTIR spectroscopy can characterize both organic (e.g., binders) and inorganic (e.g., pigments) materials. It measures the fraction of infrared light absorbed at specific wavelengths, producing spectra that reflect molecular vibrational modes. Each spectrum acts as a chemical fingerprint, enabling the identification of minerals and providing insights into their molecular structure (Jiménez-Desmond & Pozo-Antonio 2025).

• *Transmission Electron Microscope (TEM) Investigation*

A JEM-1230 electron microscope (JEOL Ltd., Japan), set to 200 kV, at the National Research Centre, Dokki, Cairo, Egypt, was used to characterize the shape and size of the prepared MMA/HPMA.

• *Colorimetric Measurements*

A portable colorimeter (FRU-WR10, Shenzhen Wave Optoelectronics Technology Co., Ltd., China) was used at the Conservation Department, Faculty of Archaeology, Luxor University, Egypt. Operating within the CIELAB colour system, the device measures colour differences (ΔE) using the formula: $\Delta E^*_{ab} = \sqrt{(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2}$, where L* represents lightness, a* the red–green coordinate, and b* the yellow–blue coordinate (Baiza *et al.* 2021; Becherini *et al.* 2018). Colour changes in aged experimental samples were recorded before and after MMA/HPMA treatment. L*, a*, and b* values were measured at three random locations per sample, avoiding cracks. Measurements were also taken on an untreated control sample, comparable in pigment, binding medium, and painting technique, to ensure accurate comparison (Becherini *et al.* 2018).

• *Contact Angle Measurements*

Contact angle measurements were performed using a goniometer (OCA 15EC, DataPhysics Instruments GmbH, Germany) operating at 24 V DC with a maximum power of 70 W. A high-resolution IDS camera captured droplet images on sample surfaces, enabling precise angle measurements to assess the wettability of aged experimental samples before and after MMA/HPMA treatment at 1%, 2%, and 3% concentrations. Measurements were carried out at the Textile Laboratory, Centre of Excellence for Innovative Textile Products and Technology, National Research Centre, Cairo, Egypt.

Results and Discussion:

— *The Case Study Mural Painting Structure*

• *Cross-Section*

Examination of cross-sectional samples from the case study mural painting—including fragments that had fallen near the wall, about 2 mm away—revealed a stratigraphy consisting of the tomb wall (either carved rock or constructed sections), a coarse preparatory plaster layer, a finer plaster layer, and a very thin whitewash layer, topped by a single pigment layer [Figures 1a–1e]. In some samples, the artist applied a red pigment layer followed by an additional green layer [Figure 1f].

• *Vertical Photo Microscope (VPM)*

Stereomicroscopic examination was carried out to investigate the fine artistic details of the paint layers, the granulometry

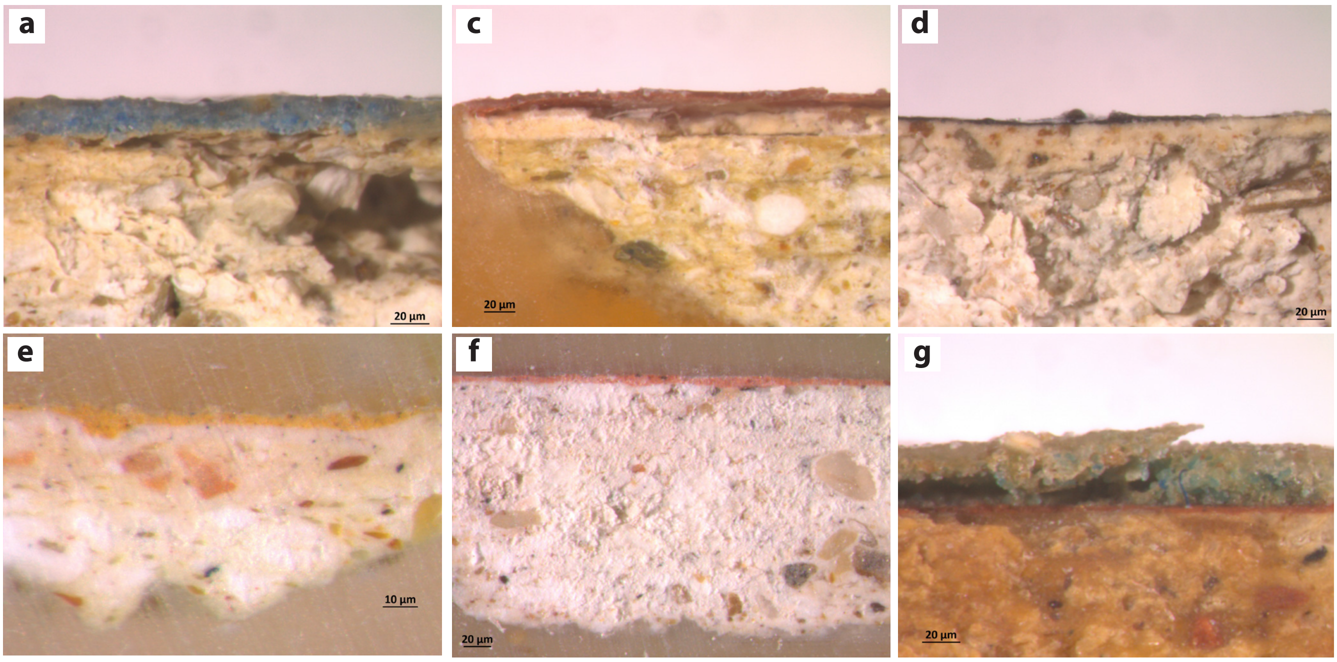

Figure 1.- Cross-section examination of paint layer samples showing: (a) blue sample; (b) red sample; (c) black sample; (d) yellow sample; (e) light pink sample; (f) green sample. (Source: Authors own work).

of the pigments, and to assess deterioration phenomena. The observations revealed that the paint layer was smoothly applied, suggesting that the pigments were finely ground to achieve this finish. Various signs of deterioration were detected, including surface dirt and contaminants, as well as darkening and colour alterations in certain pigment particles.

For example, the green pigment sample [Figure 2a] showed darkening, appearing almost brown, while the blue pigment [Figure 2b] displayed a shift to a pale blue, milky hue, or even green. Some red pigment particles [Figure 2c] had changed to yellow or orange. Efflorescence of salts was also observed, likely due to the migration of sulfate salts from the ground into the paint layer under the combined influence of humidity and heat. This process may lead to sulfation, where calcite crystals are replaced by gypsum (Ali *et al.* 2021). Biological activity could also be sources of sulfates. The black pigment sample [Figure 2d] showed evidence of possible biological colonization, warranting further investigation.

Microbial activity can also contribute to pigment discoloration through the production of metabolic pigments and by utilizing organic compounds within the paint as nutrients (Moussa *et al.* 2021). Evidence of insect infestation, likely from termites, was observed as holes and tunnel-like structures affecting the blue and black paint layers as well as the ground layer [Figures 4b, 4f] (Abd El-Tawab & Abu El-Hassan 2021). Powdering of the yellow pigment layer was also noted [Figure 2e], and traces of a substance, possibly a consolidant from previous restoration, were identified in the red and black samples [Figures 2g, 2h].

The observed surface characteristics—such as chromatic muting, micro-craquelure due to shrinkage, and certain darkening—are consistent with the natural ageing of tempera mural paintings and with original execution techniques. These features should not be systematically interpreted as deterioration requiring corrective intervention. In accordance with conservation ethics, it is essential to distinguish between patina, natural material ageing, and actual structural damage. The objective of conservation is neither to recover the original appearance nor to intensify colour saturation, but to stabilize the material condition while preserving historical authenticity and visual integrity.

- *Scanning Electron Microscope (SEM)*

Scanning electron microscopy revealed poor cohesion between pigment particles in the green, blue, and yellow samples [Figures 3a–3c], with numerous voids and pores. Sulfate salt crystals were observed, likely originating from the preparatory layers as a result of temperature and humidity fluctuations. Dust particles and surface contaminants were also present, distributed irregularly across the surface. In contrast, the red pigment particles (Fig. 3d) appeared cohesive and uniformly distributed, forming a more compact and homogeneous layer compared with the green, blue, and yellow samples. This indicates that the red pigment was better mixed and likely contained a higher binder content. Nevertheless, fine cracks and voids were present in the red layer, suggesting environmental stress or mechanical damage as contributing factors.

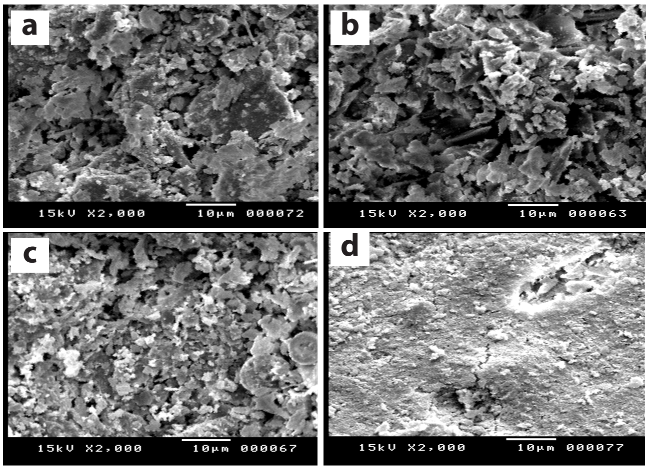

Figure 3.- Scanning electron microscope (SEM) micrographs of paint layer samples showing: (a) green sample; (b) blue sample; (c) yellow sample; (d) red sample.

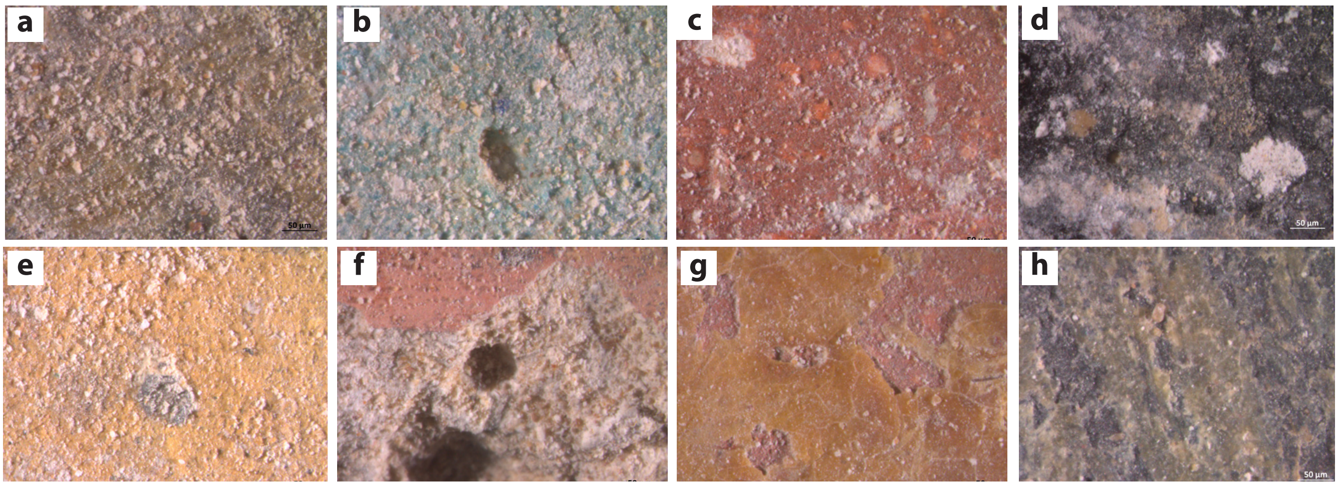

Figure 2.- Stereomicroscope examination of paint layer samples showing: (a) green area; (b) blue area; (c) red area; (d) black area; (e) yellow area; (f) ground layer; (g) another red area; (h) another black area. (Source: Authors own work)

— *The Case Study Mural Painting Composition and Technique*

- *Painting Layer*

- Pigment Materials:

Pigments used in ancient Egypt offer a fascinating area of study, as the Egyptians employed both natural and synthetic materials. Analysing these archaeological pigments provides insights into ancient technological advancements, cultural exchange, and informs conservation and restoration practices (Marey 2009). X-ray diffraction analysis of the pigment samples revealed that the artist used Egyptian green ($CaSiO_3$) as a green pigment [Figure 4a] and Egyptian blue ($CaCuSi_4O_{10}$) as a blue pigment [Figure 4b]. Red ochre (hematite, Fe_2O_3) served as the red pigment [Figure 4c], graphite (C) as the black pigment [Figure 4d], and yellow ochre (goethite, FeO(OH)) as the yellow pigment [Figure 4e].

- Paint Medium:

ATR-FTIR analysis of the mural paint layer indicates it is likely animal glue tempera, evidenced by strong protein-associated bands (Amide I, II, III). Key bands were observed at Amide I: 1622 cm^{-1}, Amide II: 1507 cm^{-1}, and Amide III: 1320 cm^{-1}, characteristic of collagen-based materials (Derrick, Stulik,

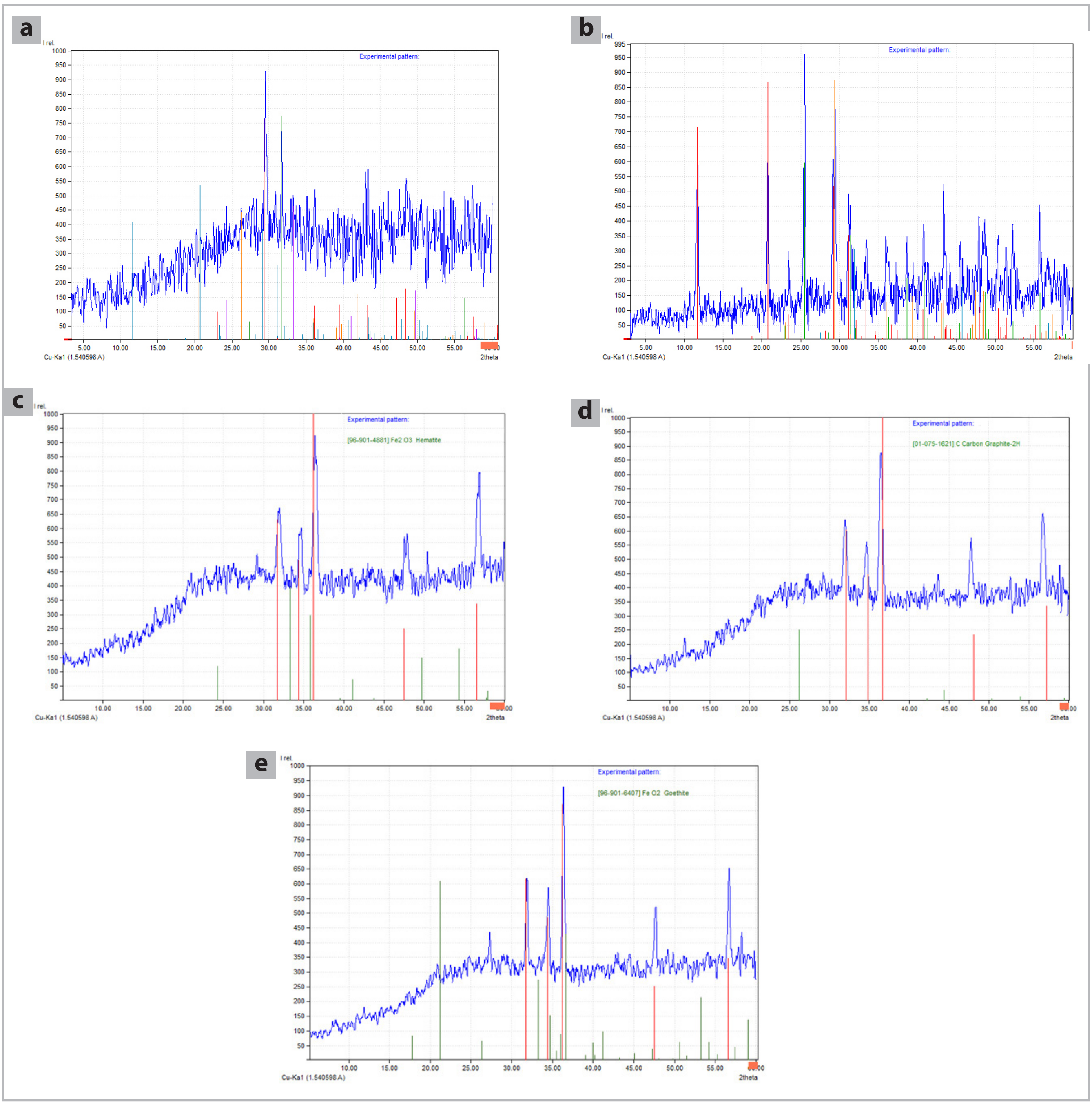

Figure 4.- XRD spectra of paint layer samples showing: (a) green sample; (b) blue sample; (c) red sample; (d) black sample; (e) yellow sample.

and Landry 1999). The spectrum [Figure 5a] shows a broad O–H/N–H stretching band at 3400.56 cm^{-1}, with distinct Amide I (C=O stretching), Amide II (N–H bending/C–N stretching), and Amide III (C–N/N–H) bands, confirming a polypeptide backbone typical of historical tempera binders (Derrick, Stulik, and Landry 1999; Mazzeo *et al.* 2008; Stamboliyska 2021). Peaks in the fingerprint region (1107.05, 1031.07, 874.28 cm^{-1}) may indicate carbohydrate residues, 798.80 cm^{-1} suggests C–H bending in aromatic residues, and 912, 889, 867 cm^{-1} indicate Si–O vibrations from substrate or pigments (Cortea *et al.* 2024). The ATR-FTIR spectrum of a yellow layer on red and black pigments [Figure 5b] reveals a lipidic or plant-derived resinous medium, likely a drying oil or resin-oil mixture from past restoration (Colombini and Modugno, 2009). Ester C=O stretching at 1735.09 cm^{-1}, aliphatic C–H stretches at 2854–2924 cm^{-1}, broad O–H at 3400.30 cm^{-1}, and C–O–C bands at 1244.91, 1156.47, and 1097.63 cm^{-1} confirm the presence of aged lipidic or resinous material (Mazzeo *et al.* 2008).

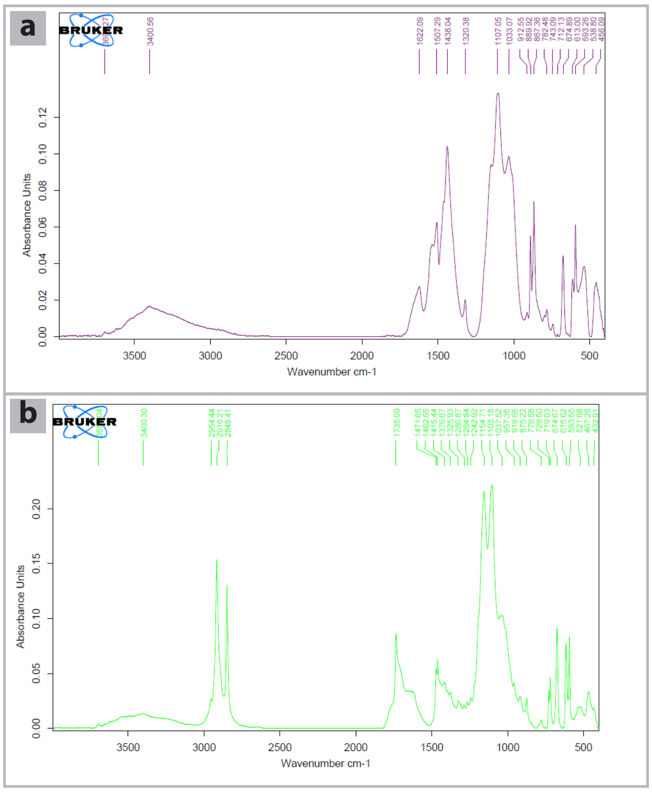

Figure 5.- ATR-FTIR spectra showing: (a) binding medium of the paint layer; (b) consolidant material, possibly applied during a previous restoration intervention or used as a varnish layer.

• *Ground Layer*

Artists typically did not apply pigments directly onto the stone substrate but first applied preparatory plaster layers topped with a whitewash layer (Moussa *et al.* 2021). X-ray diffraction analysis of the ground layers revealed that the whitewash consisted of calcium sulfate dihydrate ($CaSO_4{\cdot}2H_2O$, gypsum) (Fig. 6a). The presence of anhydrite ($CaSO_4$) suggests gypsum transformation under elevated temperatures (Korany 2016). The fine plaster layer was composed of calcium carbonate ($CaCO_3$, powdered limestone) and gypsum (Abdelaal 2018) [Figure 6b], whereas the coarse plaster layer contained calcium carbonate, gypsum, and quartz (SiO_2) [Figure 6c]. Both coarse and fine plaster samples also showed traces of halite (NaCl).

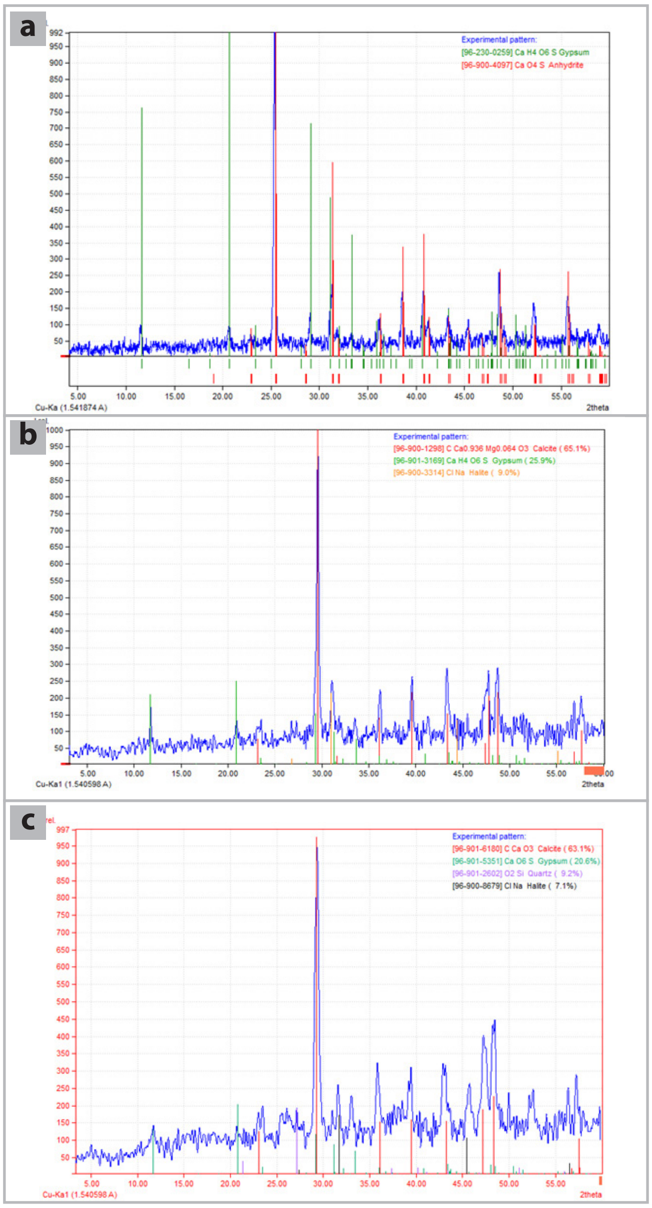

Figure 6.- XRD spectra of preparatory ground (priming layer) samples showing: (a) whitewash sample; (b) fine plaster sample; (c) coarse plaster sample.

— *Preservation State of the Case Study Mural Painting*

The mural shows deterioration due to climatic, biological, geological, and anthropogenic factors. Past vandalism [Figure 7a] likely weakened the structure, and undocumented restoration efforts may have further affected stability and visual integrity. Geologically, the tomb ceiling shows faulting [Figure 7b] and is partly built in the expansive Esna Shale Formation, which undergoes swelling and shrinkage, causing irreversible deterioration (Wüst & McLane 2000). Arid conditions further fracture the rock, while limestone and shale weathering fills fractures with secondary minerals (Moussa 2007).

Biological threats include wild bee nests and droppings[Figure 7c], introducing staining and biochemical damage. Salt migration, driven by heat, low humidity, and biological activity, contributed with the paint deterioration (Emara & Korany 2016). Thermal stress has caused cracking of plaster and paint layers, while ceiling instability and original application flaws added mechanical stress [Figures 7d–7f]. Fine plaster and whitewash show flaking [Figure 7g], peeling [Figure 7h], and blanching [Figure 7i], worsened by seasonal temperature–humidity shifts. Salt efflorescence crystallizes and rehydrates, expanding and causing cracking, powdering, and flaking (Marey 2009).

XRD analysis confirmed halite recrystallization, while gypsum dehydrates to anhydrite, weakening plaster and causing fissures (Korany & Elhazmy 2019). Pigment fading—especially blue and yellow—is evident [Figures 7j–7k]; Moussa *et al.* 2021). Water, debris, and dust from rain or flash floods also damage surfaces [Figures 7l–7m]. Artistic techniques, such as applying red over undried white, produced colour shifts [Figure 7n].

In summary, TT81's heterogeneous mural layers, combined with high temperature, humidity fluctuations, and geological instability, undergo differential expansion and contraction, producing micro-cracks, delamination, and progressive deterioration (Doehne & Price 2010).

The observed deterioration phenomena—such as pigment powdering, microcracking, salt efflorescence, and chromatic alterations—are common in mural paintings located in subterranean contexts. These conditions do not

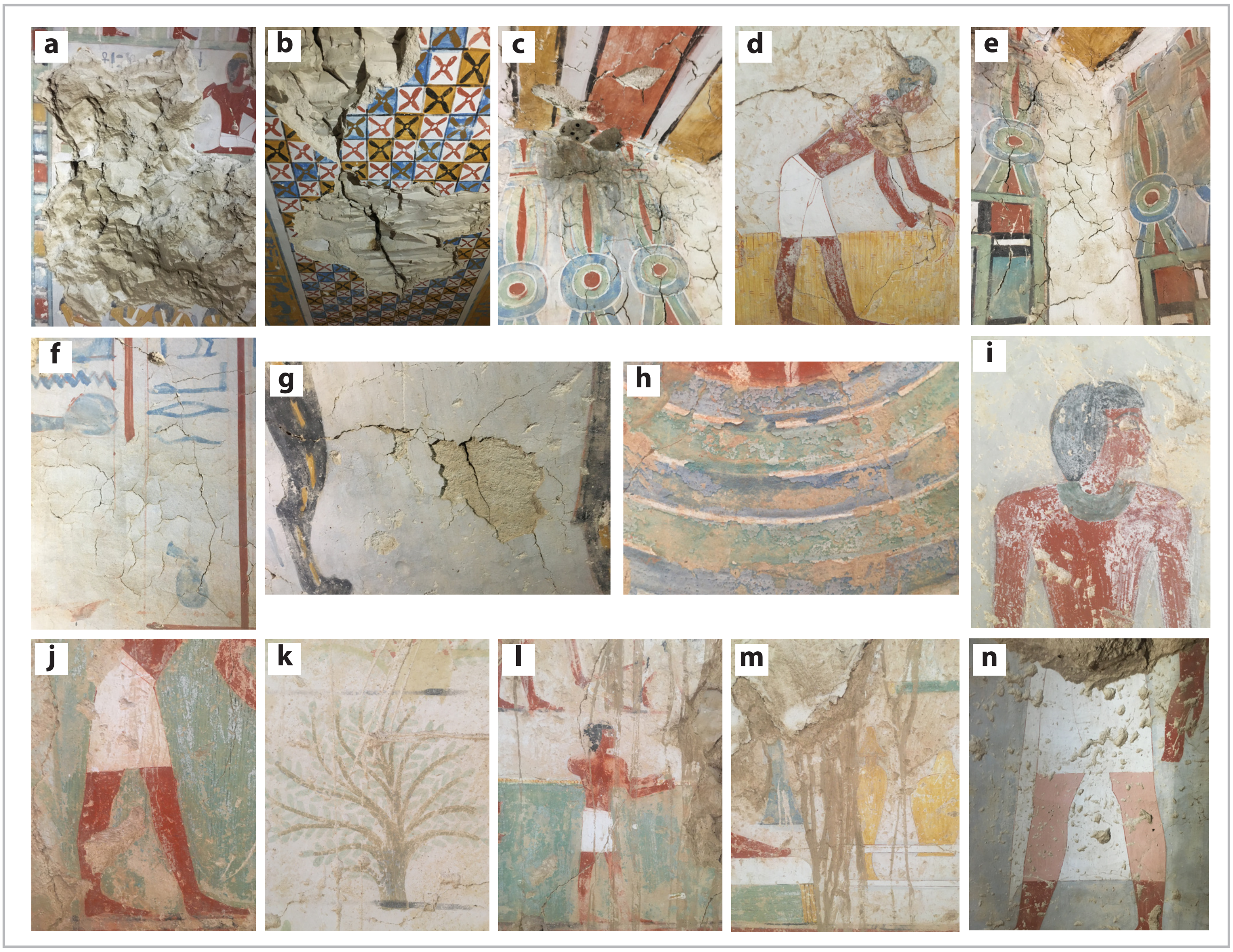

Figure 7.- Illustrates various forms of deterioration in the TT81 tomb: (a) intentional human-induced damage or vandalism and destruction from earlier periods; (b) a geological issue, specifically separation or faulting in the tomb ceiling; (c) biological deterioration, namely the presence of wild bee nests or droppings; (d) cracking in the paint layers caused by geological issues; (e) cracking in the paint layers due to improper techniques employed by the artist during application; (f) cracking in the paint layers resulting from fluctuations in high temperature and relative humidity; (g) separation or flaking of the preparatory ground and paint layers; (h) flaking of the paint layer; (i) blanching of the paint layer or efflorescence of salts on the surface; (j) colour change in the paint layer; (k) fading of the paint layer; (l) impact of water and debris from rain or sudden flash floods on the deterioration of the tomb's mural paintings; (m) further impact of water and debris from rain or flash floods on mural deterioration; (n) improper technique employed by the artist in applying red paint over the white layer before it had dried. (Source: Authors own work)

necessarily justify general consolidation treatments. In many cases, localized desalination, surface cleaning, and removal of extraneous materials represent more appropriate conservation measures. Accordingly, any potential use of MMA/HPMA should be strictly limited to localized areas exhibiting active flaking or powdering, and it must never be employed as a preventive treatment or for extensive surface coverage.

— *Evaluation of MMA/HPMA Efficiency in the Conservation of Experimental Mural Painting Samples*

• *Characterization of the Morphology and Microstructure of the Prepared MMA/HPMA*

TEM micrographs of the prepared MMA/HPMA copolymer showed well-dispersed, nearly spherical nanoparticles with relatively homogeneous sizes ranging from 66 nm to 71 nm, confirming their nanoscale dimensions. The particle boundaries were clearly defined with minimal agglomeration [Figure 8a]. Figure 8b reveals a uniform distribution and dense, coherent arrangement of particles, indicating excellent film-forming properties. These morphological characteristics confirm that the emulsion polymerization process successfully produced stable nanosized MMA/HPMA copolymer particles with uniform shape and distribution. The nanoscale size is advantageous for improving penetration and adhesion in consolidation treatments. Furthermore, the absence of large clusters or phase separation indicates successful copolymerization of MMA and HPMA into a homogeneous hybrid material, beneficial for crack treatment, pore filling, reduction of material powderiness, and creating a water-repellent layer.

While the nanoscale dimensions of the MMA/HPMA particles may favour penetration into microcracks and intergranular voids under laboratory conditions, the formation of continuous or dense polymeric films must be regarded with caution. In mural painting conservation, particularly in hypogean environments, the development of occlusive layers may interfere with vapour permeability and exacerbate moisture-related deterioration. Therefore, film-forming behaviour observed at higher concentrations should be considered a potential risk factor, reinforcing the necessity of restricting application to minimal, localized zones and avoiding surface coating effects.

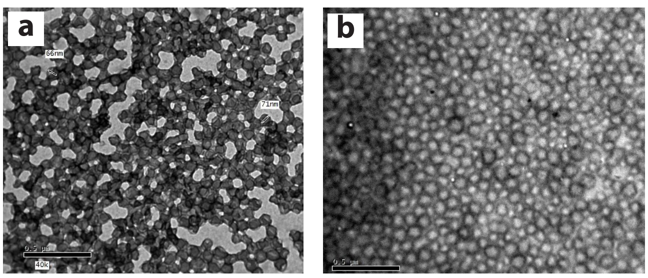

Figure 8.- TEM micrographs of the prepared MMA/HPMA showing: (a) MMA/HPMA particles morphology after the synthesis process; (b) homogeneous dispersion between MMA and HPMA.

• *Scanning Electron Microscope (SEM) Investigation of Experimental Samples Before and After Treatment*

SEM micrographs of the treated aged samples, compared with the untreated control [Figure 9a], showed that the 1% concentration formed a thin, irregular surface layer with incomplete coverage, though it penetrated pores and filled cracks [Figure 9b]. The 2% concentration produced a more uniform surface layer and moderately filled pores and cracks, maintaining the surface properties [Figure 9c]. The 3% concentration formed a noticeably thicker and more continuous layer [Figure 9d].

SEM observations indicate that increasing MMA/HPMA concentration leads to progressive filling of pores and microcracks; however, higher concentrations also promote the formation of a more continuous surface layer. While this behavior may improve short-term cohesion, it also highlights a critical conservation concern. The formation of continuous or semi-occlusive polymer films may interfere with vapor permeability, which is essential for the long-term stability of mural paintings, particularly in underground environments where moisture exchange is unavoidable. Therefore, concentrations leading to excessive surface film formation should be considered unsuitable for in situ application.

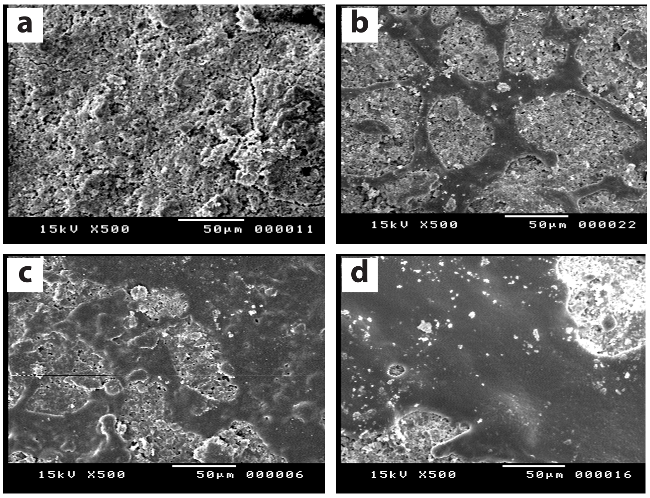

Figure 9.- Scanning electron microscope (SEM) micrographs of the experimental samples showing: (a) aged untreated sample; (b) aged treated sample 1%; (c) aged treated sample 2%; (d) aged treated sample 3%.

• *Stereomicroscope Investigation of Experimental Samples Before and After Treatment*

Microscopic examination of the treated aged samples, compared with the untreated control [Figure 10a], revealed no visible change in appearance. The 1% MMA/HPMA treatment did not fill microcracks, nor did it form a uniform protective layer, resulting in limited surface protection [Figure 10b]. In contrast, the 2% treatment effectively filled cracks, achieving an optimal balance between pore penetration, crack filling, and surface layer formation [Figure 10c]. The 3% treatment not only filled cracks but also formed a thin surface film in some areas, creating a thicker, more homogeneous layer with noticeable material accumulation within cracks [Figure 10d].

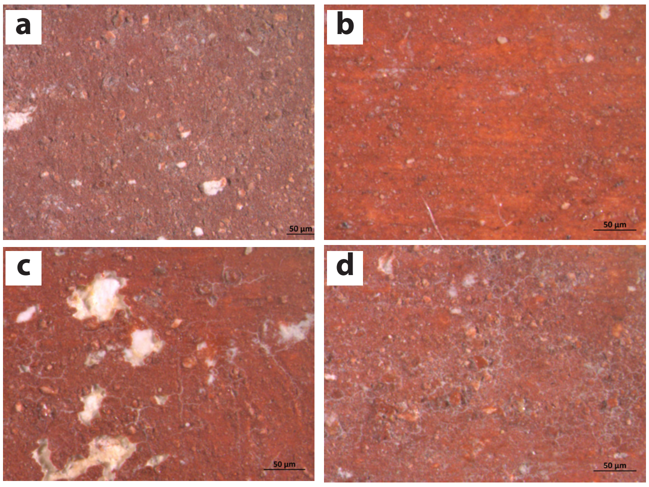

Figure 10.- Stereomicroscope examination of the experimental samples showing: (a) aged untreated sample; (b) aged treated sample 1%; (c) aged treated sample 2%; (d) aged treated sample 3%. (Source: Authors own work)

• *Colorimetric Measurements of Experimental Samples Before and After Treatment*

The colorimetric analysis of artificially aged experimental samples before and after treatment is presented in Table 1. The untreated sample showed baseline values of L* = 50.97, a* = 14.01, and b* = 8.43, reflecting darkening from artificial ageing.

Following treatment, colour changes varied with MMA/HPMA concentration. The 1% sample showed moderate change (ΔE = 8.00), primarily due to increased redness (Δa* = +4.47), yellowness (Δb* = +6.20), and slight darkening (ΔL* = –2.38). The 2% sample exhibited the highest shift (ΔE = 20.29) with decreased lightness (ΔL* = –4.99) and strong increases in chromatic components (Δa* = +11.86, Δb* = +15.69). The 3% sample showed milder impact (ΔE = 12.86).

All treated samples exceeded the perceptibility threshold (2 < ΔE < 3) (Limbo & Piergiovanni 2006). The pronounced ΔE for the 2% treatment reflects enhanced red pigment saturation rather than a detrimental effect, consistent with reports that consolidants can intensify red hues without altering pigment structure (Baiza *et al.* 2021; Becherini *et al.* 2018).

The ΔE difference between 1% and 3% treatments is only 4 units, within visually acceptable limits, confirming the consolidant does not significantly alter appearance. ΔE ≥ 5 is generally acceptable in conservation practice (Baiza *et al.* 2021).

The chromatic changes observed after treatment, particularly the increase in color saturation, should not be interpreted as a conservation objective. Variations such as color fading, micro-craquelure due to shrinkage, and surface darkening are often inherent characteristics of aged tempera mural paintings and may form part of the historical patina. These features should not be systematically classified as deterioration. The aim of conservation is not to recover the original appearance or enhance chromatic intensity, but to stabilize the material while respecting the natural ageing processes of its constituent materials.

Sample	Colour values			Colour difference			Total colour difference
	*L**	*a**	*b**	*ΔL**	*Δa**	*Δb**	*ΔE*
aged untreated sample	50.97	14.01	8.43	---	---	---	---
aged treated sample 1%	50.03	19.26	15.07	- 2.38	4.47	6.20	8.00
aged treated sample 2%	45.48	27.19	25.68	- 4.99	11.86	15.69	20.29
aged treated sample 3%	47.82	21.40	18.47	- 1.87	7.21	10.48	12.86

Table 1.- Table 1. Shows the colour change values of aged untreated and treated experimental samples.

• *Contact Angle Measurements of Experimental Samples Before and After Treatment*

Water contact angle (CA) measurements for the experimental samples before and after treatment [Figure 11] showed that the untreated sample (0°) absorbed water readily. Treatment improved water resistance: 1% MMA/HPMA yielded a CA of 31.7°, 2% reached 53.3°, and 3% achieved 58°, indicating progressively enhanced repellency.

CA is a key indicator of surface wettability, with regimes defined as superhydrophilic (CA < 5°), hydrophilic (5° < CA < 90°), hydrophobic (90° < CA < 150°), and superhydrophobic (CA > 150°) (Manoudis et al., 2017). Lower angles reflect higher water spreadability; higher angles indicate greater water repellency.

Although all treated surfaces remain hydrophilic (CA < 90°), the increasing CA with higher MMA/HPMA concentrations demonstrates reduced water absorption. Symmetry between left and right CA values confirms uniform application of the consolidant and homogeneity of surface properties.

Although contact angle measurements demonstrate a reduction in surface wettability with increasing MMA/HPMA concentration, all treated samples remain within the hydrophilic range. This finding confirms that the copolymer does not render the surface impermeable; however, even partial reduction of surface permeability may pose risks in the presence of internal moisture pathways. In cases of sudden water ingress, such as flash floods, the application of synthetic copolymers may exacerbate deterioration by hindering evaporation and promoting salt mobilization. Consequently, MMA/HPMA cannot be considered a protective solution against flooding or moisture-related damage.

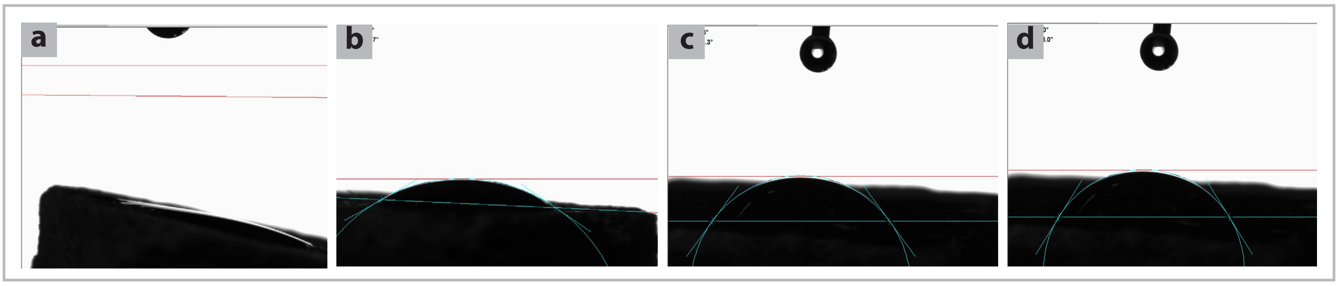

Figure 11.- Static contact angle measurement (wettability) of the experimental samples showing: (a) aged untreated sample; (b) aged treated sample 1%; (c) aged treated sample 2%; (d) aged treated sample 3%.

Conclusion

This study provides a comprehensive assessment of the materials, techniques, and deterioration of the mural paintings in tomb TT81, offering insights into their historical execution and current conservation challenges. Analytical results confirm the use of animal glue tempera with traditional pigments, including Egyptian blue, Egyptian green, red and yellow ochre, and carbon black. Documented deterioration—colour changes, cracking, flaking, vandalism, and residues from previous restorations—highlights the need for targeted conservation strategies. Experimental evaluation of Polymethyl Methacrylate/Hydroxypropyl Methacrylate (MMA/HPMA) copolymer showed that 2% and 3% concentrations provide effective consolidation, balancing structural reinforcement with minimal visual alteration while significantly improving water resistance. These benefits are particularly relevant for mitigating risks from flash floods, moisture fluctuations, and extreme temperatures combined with microclimatic variations. The findings support integrating MMA/HPMA copolymer at optimized concentrations into conservation protocols for mural paintings at environmentally vulnerable heritage sites, contributing to their long-term preservation.

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Author/s

Mahmoud Sayed Korany
mahmoudsayed.sca@gmail.com
Conservation Department, Faculty of Archaeology, Luxor University, Luxor, Egypt.
https://orcid.org/0000-0001-6384-0910

Mahmoud Sayed Korany is a member of the academic staff in the Conservation Department, Faculty of Archaeology, Luxor University. He holds an M.A. and a Ph.D. in Archaeological Conservation from Cairo University. He has supervised M.Sc. and Ph.D. theses in archaeological conservation and has published extensively in international peer-reviewed journals. He also serves as a reviewer for several international journals and as an editorial board member of a number of academic periodicals. He is the author of several books and has taught courses on the conservation and restoration of archaeological artifacts through part-time appointments at various faculties and institutes. In addition, he has participated in national and international research projects on cultural heritage preservation and management. He has also taken part in several restoration projects of historic buildings and in the conservation of museum artifacts, as well as in numerous archaeological excavation missions within and outside Egypt. Moreover, he is a member of the expert committees responsible for inspecting architecturally significant historic buildings and for developing conservation and management plans for archaeological sites in Egypt.

Wael Sabry Mohamed
wsabry1976@yahoo.com
Polymers and Pigments Department, National Research Centre, Dokki, Giza, Egypt.
https://orcid.org/0000-0001-5246-7620

Wael Sabry Mohamed is a Professor in the Department of Polymers and Pigments at the National Research Centre. He holds a Ph.D. from the University of Stuttgart, Germany, specializing in the application of nanotechnology in polymers. He has served as principal investigator for numerous internal scientific projects and participated in several external and international projects. He has supervised many M.Sc. and Ph.D. theses and has authored over 100 publications in high-impact international journals. He has also presented at numerous international conferences, both in Egypt and abroad.

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