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# Experimentation of a PVA-Borax hydrogel for the removal of Paraloid B72® from artifacts of archaeological interest from the National Archaeological Museum in Naples, Italy

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ABSTRACT
This paper shows the results of an experimentation aimed at the removal of a polymeric film of aged Paraloid B72® originally placed to protect some wooden artifacts belonging to the Egyptian collection of the National Archaeological Museum of Naples (MANN). The study was conducted on two shabtis dated to the 19th Dynasty and a sculpture of Ptah-Sokar-Osiris dating to the late period. Thanks to the collaboration between the National Archaeological Museum of Naples and the Laboratory of Restoration of Wooden Artifacts of the University of Naples Suor Orsola Benincasa, it was possible to conduct an in-depth study of the artifacts, from both an archaeological-historical point of view and from a conservation point of view. This led to the development of the above-mentioned experimentation and to the planning of a proper restoration intervention. The study and experimentation were supported by several diagnostic techniques. In a first stage, non-invasive investigations were carried out to study the execution technique and the state of conservation of the artifacts. This allowed the recognition of the wood species that constituted the three sculptures through optical microscopy, whereas IR reflectography, UV induced fluorescence and X-ray radiation (XRF) were conducted to identify the type and the chemical nature of the pigments. Based on the above analysis, different restoration strategies, based on the removal of Paraloid B72® through a methodology that would act in full respect of the wooden support of the artifacts, were considered. Pros and cons of the different methodologies applied in the recent past for the removal of aged Paraloid from artifact surfaces were analysed. A removal technique based on the use PVA-Borax hydrogel loaded with a solvent mixture was selected. Experiments were conducted to validate the effectiveness of the selected method.

**Section:** RESEARCH PAPER

**Keywords:** Paraloid B72®; Hydrogel PVA-Borax; wood restoration; Egyptian artefacts; Shabties; Ptah-Sokar-Osiris

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## 1. INTRODUCTION

The Restoration procedures aims to preserve the artwork in all its parts, opting for minimal intervention and minimal complexity. Degradation of artifacts, especially when made of organic materials such as wood, is a physiological phenomenon [1]. For this reason, the materials used during restoration processes should be compatible with the materials of the artifact to which they are applied. Moreover, these materials should be stable from aging, as their degradation can accelerate the artifacts' degradation. In any case, these restoration procedures should be reversible [2], in particular when the previous restoration needs to be removed to carry out a second different intervention. The concept of reversibility, however, becomes relative when talking about consolidants. In this area one of the main procedures is the application of consolidating resins, by solution or dispersion in suitable solvents. The consolidating agent penetrates the wood cavities, adhering to the walls and making them more resistant by


Figure 1. Shabti IG648 (a) Shabti IG655 (b), Ptah-Sokar-Osiris IG909 (c,d).

creating an auxiliary microstructure to sustain the cavity walls [3], [4].

Since the second half of the 1960s, the use of polymer resins as consolidating materials in restoration has reached the highest level of popularity [5], [6]. This was particularly true for the resin Paraloid B72®. The main drawback of this and other similar products is related to the aging of the same resin placed on and inside the artifacts. The degradation process of polymer resins consists of chemical transformations due to the action of factors such as light, temperature, interactions with the substrate, humidity, and atmospheric pollution. They cause embrittlement of the protective film, decrease in solubility, yellowing of the resin, increase in polarity and decrease in adhesive strength [1], [7], [8]. For the above reasons, polymer films can cause irreversible damage to artworks, drastically altering the physicochemical properties of the surface to which they have been applied. The film become also increasingly resistant to common solvents, thus limiting the reversibility.

This work aims to develop a methodology for the removal of aged Paraloid B72® that would be well suited to the needs of the three wooden artifacts belonging to the Egyptian collection of the National Archaeological Museum of Naples (MANN), in full respect of their execution technique. The artifacts are two wooden shabtis inventoried under codes IG648 (Figure 1a) and IG655 (Figure 1b) dated to the 19th Dynasty, and a small wooden sculpture of Ptah-Sokar-Osiris in the form of sarcophagus inventoried under code IG909 (Figure 1c, Figure 1d) dated to the late period. Preliminary investigations were carried out on the artefacts to better understand their execution technique and to analyse their state of conservation. With full respect for the materials in question, it was decided not to carry out any invasive investigation, as the artefacts were already in a compromised state of preservation and were largely lacking in detail. The in-depth study of the state of conservation of the works highlighted the problems related to the aging of Paraloid B72, which determines the loss of its native characteristics such as transparency, flexibility and, therefore, its "reversibility" [9]. Accordingly, the need to remove the aged layers of Paraloid, before they could itself turn into a cause of degradation of the artifacts to which it was applied, became apparent. To determine the best removal strategy, several methods of resin removal were studied. At the end of the evaluation, the choice fell on a new cleaning methodology: a system of Hydrogel in PVA cross-linked with borax and loaded with 20 percent solvent, which due to its high elasticity allows its safe removal through exfoliation action that leaves no residue on the surfaces to which they are applied [10]. An experiment was therefore carried out to test the effectiveness of this cleaning system.

## 2. MATERIALS AND METHODS

### 2.1. Analyses for the study of artefacts

*Wood identification*: A taxonomic analysis was carried out to recognize the wood species used to make the artifacts. The investigation was carried out macroscopically for the two shabties (IG648 and IG655), and partially microscopically by the Dino-Lite® portable microscope, which uses the coaxial illumination technique. The observations were carried out particularly on the radial section at the head and feet of the artifacts and on the tangential section along the body of the shabties. In this way, the surface details were investigated using two different lights sources, namely, visible light and UV-induced fluorescence; some photographs were taken and compared with some images of known woods. In the case of the Ptah-Sokar Osiris sculpture, the taxonomic analysis was possible by observing the microscopic characteristics of the wood using a Zeiss Axiolab light microscope equipped with the Nikon Digital Sight DS-L1 digital photographic system [11]. This allows for the morphological analysis of the specimens using different magnifications (10x and 20x). Observation of the microscopic structure was made possible, by the discovery of a small fragment belonging to the sculpture inside the storing box.

*UV-indued fluorescence:* UV radiation-induced fluorescence was used to document the presence of protective layers on the artifacts. UV fluorescence analysis (UVIFL) was carried out with 2 lamps from Wood Photo Ectronics- 400 W UV CURVING EQUIPMENT: single features- 400 W UVA, 230 V / 50 Hz, Abs. 2.5. Fluorescence recording was performed with a Canon Eos 1100D camera completely obscuring any light source other than UV, with the lamps placed at 45° to the centre of the surface of the artifacts.

*X-ray Fluorescence (XRF*): An XRF campaign was carried out to investigate the chemical nature of the traces of red pigment found on the back of shabti IG655 and to look for possible traces of pigment on the other two artifacts [12]. XRF analysis was carried out using a portable XRF-Q Assing spectrometer, with tungsten tube, silicon PiN diode detector with beryllium window and Peltier effect cooling system, with 189 eV resolution at 5.9 KeV and operating conditions 30 kV, 0.5 mA. The survey was conducted at the locations on the artifacts where traces of pigments were visible.

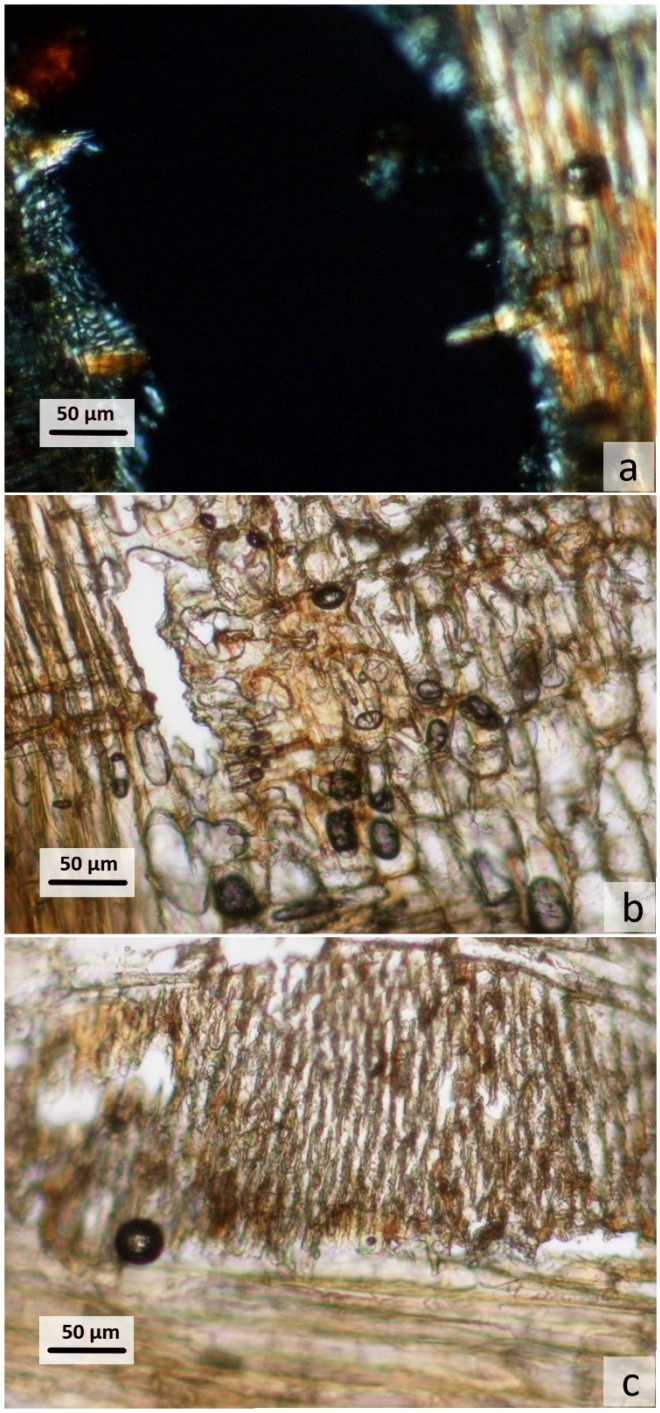


Figure 2. Image of the wooden fragment taken from artefact IG909 under the Zeiss Axiolab light microscope, equipped with the Nikon Digital Sight DS-L1 digital system. Simple perforation plate and large solitary pot, the large pot diameter size (about 200 micrometers) is diagnostic to distinguish Ficus Sycomorus from Ficus Carica (a). In the middle and lower part of the image b, crystals within the bands of the axial parenchyma are visible (b). ray shape parenchymatous, heterocellular (c).

*Fourier Transform Infrared Spectroscopy (FT-IR)*: To check the presence of the consolidant, an analysis by FT-IR was carried out using a Nicolet iS10 Thermo Fisher Scientific FT-IR spectrofotometer, with a dried and sealed optical system and optical bench, equipped with KBr windows with protective CaF2 coating. Michelson-type dynamic alignment interferometer with KBr/germanium beam splitter; interferometer scanning speed: acquisition of up to 40 independent spectra per second with a spectral resolution of 16 $cm^{-1}$; spectral resolution: better than 0.4 $cm^{-1}$; wavelength precision: 0.01 $cm^{-1}$; spectral range: 7,800-350 $cm^{-1}$, suitable for the detection of a wide class of organic compounds.

### 2.2. Hydrogel experimentation

For the removal of the Paraloid B72® layer by means of a hydrogel based on Poly-Vinyl Alcohol (PVA) and borax, loaded with two solvent mixtures was used. The first formulation was a 1:1 mixture of methyl-ethyl-ketone (MEK) and 1-pentanol (1-PeOH) already described in the literature [13]. The second formulation involved the use of alternative solvents, with acetone replacing MEK and ethanol replacing pentanol.

*Products:* Paraloid B72, CTS S.r.l. 100 % acrylic resin in the form of transparent granules. Poly-Vinyl Alcohol, PVA. CTS S.r.l., Borax, Sodium tetraborate, decahydrated.
Bi-distilled water, Carlo Erba Reagents S.r.l. Conductivity ≤ 0.1 µS/cm; Residue on evaporation ≤ 1 ppm; Residue on calcination ≤ 1 ppm.

*Preparation of the specimens:* The application phase of the methodology was preceded by a preliminary optimisation divided into two phases. Firstly, the effectiveness of the methodology and the removal times were studied on slides specially prepared with Paraloid B72® and aged. Slides measuring 5 cm × 7 cm were coated with a double layer of Paraloid B72® solutions in different concentrations, namely, 5 %, 10 % and 15 % w/w, dissolved in different solvents: Acetone, Ethyl Lactate, Nitro Diluent and two mixtures made of Acetone and Ethyl Lactate in 1:2 proportion and Acetone and Ethyl Lactate in 2:1 proportion respectively.

Wooden specimens were prepared to simulate the characteristics of the wood of the artifacts, to carry out removal tests on specimens similar to the real case. To this end, cubes of approximately 2 cm side of *Ficus Carica* L1753 were cut. The wood was selected because it had anatomical characteristics similar to that of the artifacts. Wood consolidation was carried out with Paraloid B72® dissolved in various solvents, at concentrations of 5 % and then 10 % w/w. The specimens were then divided into three groups and subjected to different ageing cycles. The first group (Group 0) was stored in the dark, protected and away from any source of light, heat, and humidity. The other two groups (Group 1 and Group 2) were subjected to two artificial ageing cycles, using the *Angelantoni SU250* aging chamber, set to the values $RH\% = 50\ \%$; $T = 40\ °C$; $UV = 400\ nm < \lambda < 250\ nm$. Ageing was carried out for a time $t = 408$ hours (17 days). At the end of this period, Group 1 was removed from the chamber and stored in the dark, protected and

Figure 3. IR observation of the black pigment.

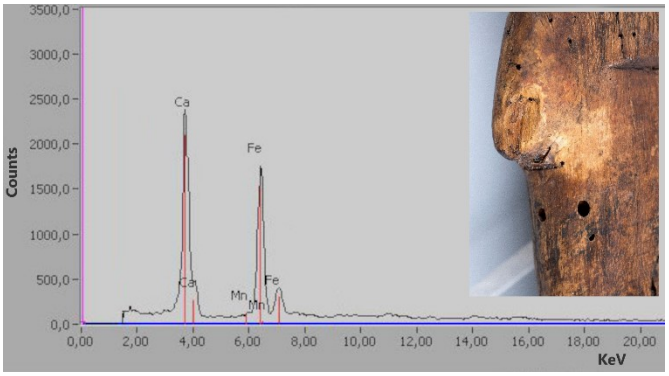


Figure 4. Traces of red pigment on the back of Shabti IG655 (left) and its XRF spectrum (right). Trace amounts of calcium (Ca) and iron (Fe) traceable to stucco residues and red pigment.

away from any source of light, heat, and humidity, while Group 2 underwent a second ageing cycle, in the same chamber using the same parameters, but for a time t=264 hours (11 days) for a total of 672 hours (28 days).

*Preparation of PVA-Borax Hydrogels:* The two hydrogel formulations, containing the two different solvent mixture, were prepared in the same way. The first step involved the preparation of an 8 % w/w polyvinyl alcohol solution dissolved in water under moderate heating to promote solubilisation. After cooling the solution, 20 % of the previously prepared solvent mixtures were added to the PVA solution, for which the ketone and alcohol were mixed in equal parts. Finally, a 6 % solution of water-soluble borax was mixed in a 1:4 ratio, leading to the formation of the gel phase.

*Application of the hydrogel:* For both formulations, slide tests were carried out to evaluate the time of action and the effectiveness of the method. A small amount of hydrogel was placed on the slides, which, as described earlier, had been treated with Paraloid B72® at different concentrations and then subjected to artificial aging. The hydrogel was allowed to act for a predetermined period, starting from a time of 15 seconds up to 2 minutes. Once the hydrogel had been removed, residual swelled Paraloid was removed using a small cotton swab. The removal effect was verified by observation under grazing light and UV. In a second step, the two hydrogel formulations were tested with a setting time of 120 seconds on the wooden samples treated with Paraloid B72® and artificially aged, to test their effectiveness on the substrate simulating the real case. As before, once the gel was lifted, the aged Paraloid residue was removed by the cotton swab. The removal effect was verified by observation of the sample under ultraviolet light.

## 3. RESULTS AND DISCUSSION

### 3.1. Analyses for the study of artefacts

Thanks to taxonomic analysis, an affinity with the wood of *Ficus Sycomorus* was hypothesised for the shabties according to the information contained in the archival-documentary material. For the sculpture of Ptah-Sokar-Osiris, analysis of the fragment confirmed the use of *Ficus sycomorus*, known as Sycamore. As far as the microscopic characteristics of the wood are concerned, it can be observed that the growth rings are diffused and porous; the vessels are very large and mostly solitary; when the axial parenchyma is observed in tangential section, it is characterised by large bands containing crystals; the parenchyma rays are very large and heterocellular. (Figure 2).

UV observation did not provide indication answers as to the type of substances present to protect the artifacts. The IR survey returned useful indication as to the type of black pigment, which was present in the form of residues on top of the artefacts (Figure 3); being very dark, it was inferred to be carbon-based. There is a good chance that the specific colour used for the decoration of the artefacts was smoke black, which was widely used in ancient Egypt for the decoration of large and small artefacts.

XRF measurements were carried out on the artefacts, on the area of the back of the shabti IG 655, where the presence of a red pigment had already been noted. The presence of a ferrous residue attributable to the use of an iron-based pigment was investigated, which from the optical comparison was most likely traced back to the use of a red earth. Other areas of the same artefact were also investigated with the same technique, namely, on the back of the elbow and the side of the foot, where residues of an ancient stucco were present. (Figure 4).

The detection of elements, such as calcium and sulphur, typical of the composition widely attested in literature [12], allowed this intervention to be recognised as belonging to the original execution technique of the artefact. The hypothesis was confirmed through the observation of these areas with the Dino-lite portable microscope, thanks to which the presence of the pigment layer above the plastering was highlighted.

The same investigation conducted on artefact IG 909 was useful for the historical-chronological framing of the piece. The front surface of the lid was characterised by a colour lighter than the inside and the back of the artefact.

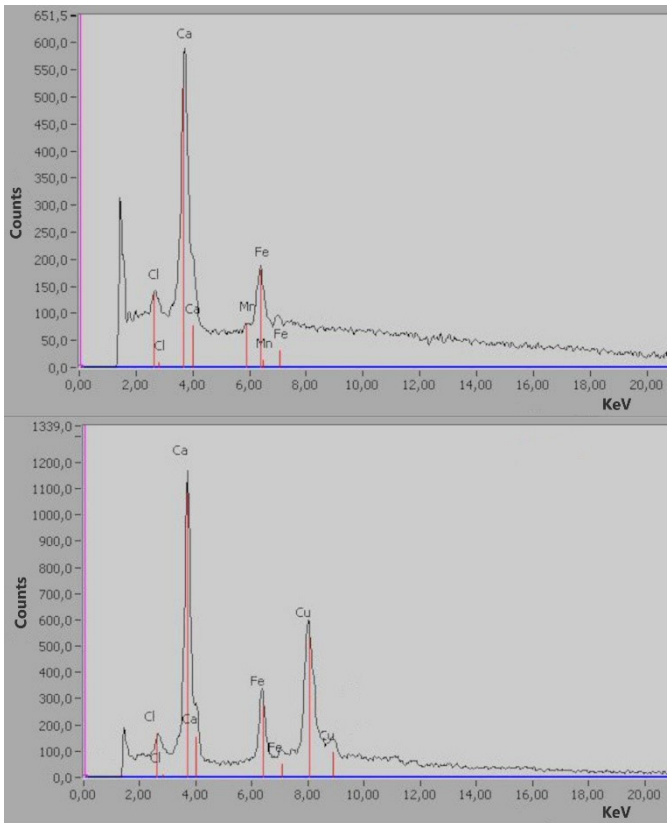


Figure 5. XRF Spectrum IG909: Trace amount of Calcium (Ca) and Iron (Fe) traceable to stucco residues and red pigment (top); XRF Spectrum IG909: Trace amount of Calcium (Ca) and Copper (Cu) traceable to stucco and blue or green pigment (bottom).

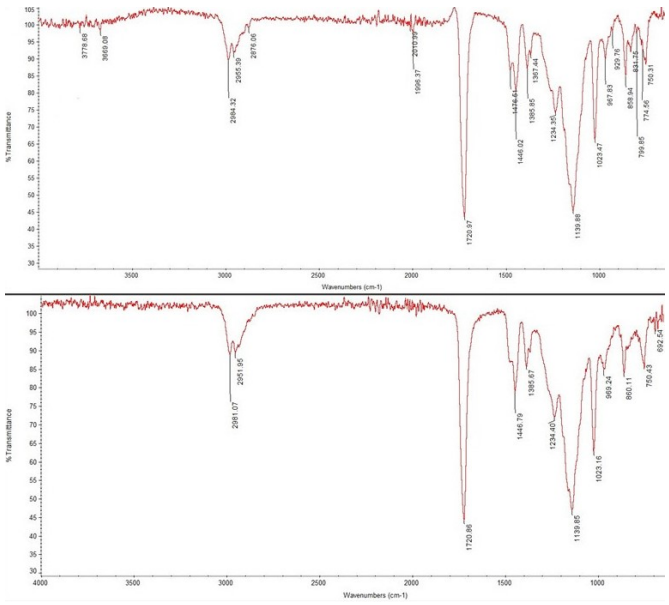

Figure 6. FT-IR spectrum of Paraloid B72® solubilised in 10 % Acetone prepared in the laboratory (top); FT-IR spectrum of a resin sample taken from the surface of the IG909 artefact in which bands attributable to Paraloid B72® are evident (bottom).

The initial hypothesis of the presence of plaster on the entire surface of the lid was confirmed by the widespread presence of traces of plaster. On several areas of the lid, traces of other elements were detected, such as iron, which was generally diffused over the entire 'body' of the artefact (Figure 5, top); at the level of the 'xSx n bik' necklace, the presence of copper was detected, deriving from an original green or blue pigment (Figure 5, bottom).

The presence of these elements suggested the original presence of pigments, in this case red iron-based pigments used to colour the body of the entire artefact and green or blue copper-based pigments used for the decorations painted on the artefact. Thanks to above analysis, it was possible to orient the historical-chronological research, allowing the Ptah-Sokar-Osiris sculpture to be placed in a specific typology. According to Raven's classification, the sculpture would fall into Type IV, which is where the scholar encloses all those artefacts characterised by a red-painted body, decorated with a blue or green necklace and a gilded face.

FT-IR investigation, allowed to detect the presence of Paraloid B72®. Small samples of resin were taken from the surface of the artefacts and analysed by FT-IR spectroscopy. The resulting spectrum was compared with the FT-IR spectra of samples of specially prepared consolidating resins in the laboratory (Figure 6). The presence of the characteristic bands of Paraloid, around about 2980, 1720 and between 1445 and 750 $cm^{-1}$, allowed the resin used as a consolidant for Egyptian sculptures to be recognized.

### 3.2. Testing for hydrogel

From the study of the execution technique and the state of conservation of the works, it became evident that the presence of degraded Paraloid B72® could itself become one of the causes of degradation of the materials to which it had been applied, thus posing a threat to the conservation of the works under examination. The next step was therefore to verify the effectiveness of the cleaning system that had been selected. At first, the action times of the hydrogel were evaluated through


Figure 7. Comparison of wood specimens consolidated with Paraloid and subjected to artificial aging before (left) and after (right) cleaning with Hydrogel PVA-Borax loaded with Acetone and Ethanol in a 1:1 ratio.

tests, whereby it was verified that: within the first 60 seconds of contact, the Hydrogel was only able to partially solubilise the aged Paraloid layer, regardless of the thickness of the film; complete removal of the resin was not observed in either case [14]. After the first minute of contact, the 5 %-diluted Paraloid was completely swelled while the 10 %-diluted Paraloid needed more insistence during the swab action to remove the Paraloid residue. After the second minute, the polymer film, both at 5 % and 10 %, was completely swollen, and through the gently action of the swab, any residual aged Paraloid B72® was completely removed from the glass surface. In the view of the results obtained, it was decided to use the same formulation of Hydrogel with an application time of 120 seconds on the wooden samples treated with Paraloid B72® and subjected to artificial ageing, to test their effectiveness on the substrate simulating the real case and possibly investigate differences between the two different formulations (Figure 7).

A similarity was found between the two gels as revealed by observation in visible an ultraviolet light of the post cleaning samples with the system tested. The choice of the formulation of Hydrogel to be used for the cleaning of the artefacts was oriented towards the formulation loaded at 20 % with the 1:1 Acetone and Ethanol mixture because it was simpler in terms of complexity, availability and much cheaper. The efficacy of the system loaded with acetone and ethanol was therefore tested on a naturally aged Paraloid layer applied during an old restoration work on a polychrome wooden crucifix from the early 17$^{th}$ century.

The application of the tested cleaning method led to the swelling of the Paraloid layer and its complete removal after only 120 seconds of application, without affecting any layer of the underlying polychromy as confirmed through observation under the handheld microscope Dino-lite® (Figure 8). Finally, the effectiveness of the system was also verified by FT-IR investigation on the small wood fragment attributable to the Ptah-Sokar-Osiris sculpture and already observed under the microscope for taxonomic analysis. The same fragment was subjected to the FT-IR investigation before and after cleaning by applying a small amount of hydrogel. Comparison of the two spectra (Figure 9) shows, for the specimen analysed after the cleaning procedure (Figure 9, bottom), a decrease in the characteristic bands attributable to Paraloid (around about 2980, 1720 and between 1445 and 750 $cm^{-1}$), which are instead very evident for the same specimen analysed before cleaning (Figure 9, top).


Figure 8. Detail of a portion of the leg of a polychrome crucifix covered with a layer of Paraloid B72® 30 years ago, and then cleaned with Hydrogel PVA-Borax (Acetone and Ethanol 1:1).

## 4. RESTORATION WORK

Thanks to the positive results obtained from the experimentation carried out, it was possible to apply the methodology to the cleaning of the three Egyptian artefacts housed at the MANN. As already pointed out, before the cleaning operation investigations and research were carried out to gain an in-depth knowledge of the works. The study of the execution technique and the state of conservation highlighted the possibility of limiting the degradation caused by Paraloid B72® to the works, and although the experimentation carried out did not allow for in-depth action, even though it was limited to the surface of the artefacts, the benefits in terms of conservation, knowledge and valorisation of the works were more than evident.

The solvent solution was chosen with the intention of interfering as little as possible with the underlying support. To this end, the PVA-Borax hydrogel, loaded at 20 % with a solution of acetone and ethanol diluted in a 1:1 ratio, seemed a valid cleaning system to use on supports of this type, maintaining the action at the interface, with the minimum contribution of humidity and solvent. Prior to the cleaning operation, incoherent residues were removed from the surface of the artefacts; with the help of a small soft-bristled brush, the dust was removed first and foremost, and with it the rosume, the remains of pupae

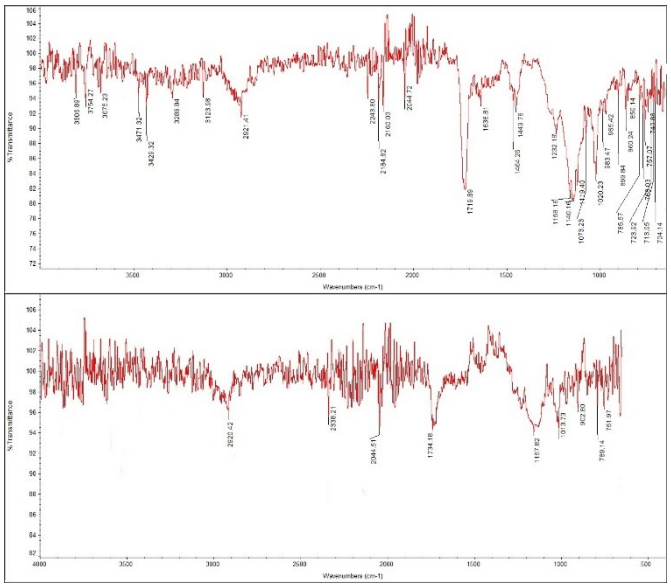

Figure 9. FT-IR spectrum of the wood sample taken from artefact IG909 before cleaning (top). FT-IR spectrum of the wood sample taken from the artifact IG909 after cleaning by application of a small amount of PVA-B hydrogel loaded with the mixture of acetone and ethanol (bottom). Both spectra show background noise due to wood interference.

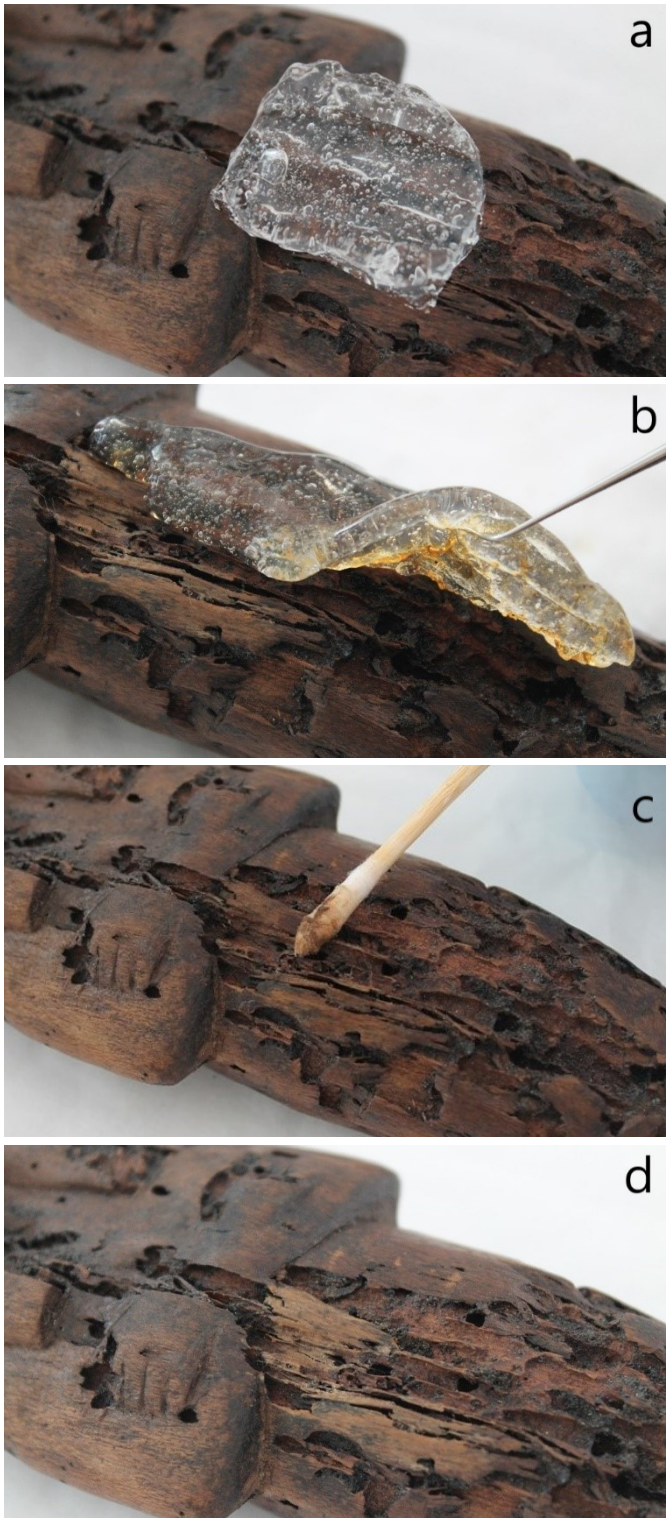


Figure 10. Cleaning treatment of IG648 shabti using PVA-Borax hydrogel loaded with 20 % Acetone and Ethanol 1:1. Application of the gel for 120 seconds (A); removal of the gel (B); removal of residual Paraloid B72® swabs by dry swabbing (C); area of the surface after cleaning (D).

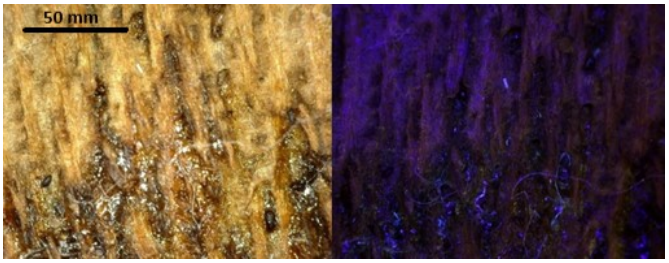


Figure 11. Observation through Dino-lite of treated area. Visible light (left) and in UV (right).

present in the tunnels dug by the insects and not completely incorporated in the substances applied to the works in the past.

The next step involved the application of the tested cleaning system. A small quantity of PVA-Borax hydrogel (20 % acetone+ethanol) was taken with a small spatula and applied to a portion of the surface of the artefact. The hydrogel was held in place for approximately 120 seconds, and then lifted from the artefact. After the gel was removed, it had a less transparent colour, tending to yellow. A small, dry cotton swab was gently rubbed over the treated area to remove the residues of swollen consolidant. The cleaning of the artefacts involved a diversification of the intervention not only for the different artefacts, but also for the different areas of the artefacts themselves.

For the shabty IG648, cleaning was first carried out as described above, with application times of 120 seconds per application. Once the application of the hydrogel over the entire surface was finished, the artefact appeared uneven, showing some resin residues, particularly at the level of the gaps. Therefore, only at the level of the gaps was the cleaning action repeated, placing the hydrogel at the residues, and leaving it to act for a further 60 seconds (Figure 10).

Regarding artefact IG655, due to the presence of the traces of hieroglyphics found on the artefact, it was decided to opt for a gentler cleaning to avoid the risk of losing the last traces of black and red pigment still present on the shabti. The cleaning therefore involved a single application of the hydrogel for a period of 120 seconds, at the end of which the swollen Paraloid was removed from the surface by gently wiping it with a dry cotton swab. At the end of the cleaning operation, the shabty IG655 no longer showed the same glossiness caused by the heavy presence of the consolidating layer.

As for the sculpture of Ptah-Sokar-Osiris in the form of a miniature sarcophagus IG909, differentiated cleaning was carried out here too. On the front section of the sarcophagus lid, very gentle cleaning was carried out, with short contact times, insisting only on those small areas that showed a more pronounced shine; hence, in the recesses and areas where Paraloid had deposited in greater quantities. It was decided to thin out the layer of Paraloid B72® placed on the surface of the artefact so as not to lose the last traces of preparation and paint film that could most likely have been absorbed by the consolidating agent. A small amount of PVA-Borax hydrogel loaded with acetone and ethanol was applied to the lid, left in place for only 120 seconds and then removed. Once the layer of consolidant had been re-swollen, it was removed by the light action of a dry cotton swab. Only on the underside of the artefact was the hydrogel repositioned for a further 120 seconds due to the presence of the thicker layer of Paraloid. All the other surfaces of the artefact, i.e., the back of the lid and both faces of the bed were treated with a double application of the hydrogel due to the presence of the substantial Paraloid layer. All steps of the consolidation removal were verified using the dino-lite handheld microscope, in visible light and UV (Figure 11).

## 5. CONCLUSIONS

The hydrogel PVA-Borax cleaning system used for the cleaning of the artefacts under examination performed particularly well, adhering perfectly to the surface to which it was applied, guaranteeing the complete swelling of the polymer film without leaving residues that are difficult to remove. The feature that guided the choice of this cleaning system, in addition to its mechanical properties, was its ability to retain organic solvents up to a maximum concentration of 30 %. The effectiveness of the system, verified through experimentation, led to the use of this method for the removal of aged Paraloid from the wooden surface of the two shabties and the sculpture of Ptah-Sokar-Osiris in the form of a sarcophagus.

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