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Research Article

Innovations in the Use of Paraloid B-72® As a Surface Adhesive Method for Conservation of Bones and Fossils

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Abstract

Consolidation and adhesion processes are key in the structural conservation of bones and fossils. It is therefore necessary to choose the most suitable products to ensure proper preservation of these cultural assets, evaluating their adhesion power, chemical compatibility, reversibility, stability and minimal interference with future analyses. For this purpose, we are putting forward a new way of using Paraloid B-72®, presenting a new application format as an adhesive in sheets, which can be prepared with different concentrations, formats and drying times. Its adhesive efficacy, penetration capacity and reversibility have been tested, showing good results for its application in the conservation and restoration treatments of these cultural assets.

Keywords: Adhesion; bone; fossil; sheets Paraloid B72®

Introduction

Adhesion and consolidation treatments are common in bone and fossil conservation and restoration. They allow these usually friable and fragile materials to be handled, studied and observed. However, any adhesive or consolidant must meet the conditions of stability, reversibility, compatibility and effectiveness [1]. The first interventions carried out on fossils were performed with triterpenoid resins such as dammar, and other natural products: shellac, waxes and organic glues of animal origin [2-4]. These substances cause chromatic changes on the surface and yellowing, and they have shown a low compatibility with the fossil substrate in the long term, mainly because of their lack of plasticity [5]. These problems have led to their replacement by synthetic adhesives such as epoxy resins, cyanoacrylates, polyurethanes, fluorinated resins, and especially acrylic and vinyl polymers [6-11].

Paraloid B-72® is possibly the most widely used organic adhesive and consolidant in this field. It is an acrylic polymer based on ethyl methacrylate, which is sold in pellets, and it is applied diluted in a liquid medium, usually acetone, though it is also soluble in esters and aromatic hydrocarbons. When the organic diluent evaporates, the product hardens inside the fossils and they are thus strengthened, acting as a filler for small cracks and an adhesive. Its stability has been shown in many fossils treated since the second half of the 20th century and in accelerated ageing tests, showing good results [1,12-18]. Paraloid B-72® can be used both as a method of consolidation and adhesion. At a low concentration with respect to the volume of solvent, it consolidates the internal structure of the piece. If its concentration is increased with respect to the solvent, it acts as an adhesive method, although in this case it will have a higher viscosity, a lower penetration capacity and a greater fixing power [19].

Compared with other consolidants and adhesives, Paraloid B-72® shows advantages such as its ability to form thin films [20] as well the possibility of being resolubilized if desired. The latter is a very important property in restoration treatments, since others are more insoluble in the long term, such as water-based acrylic and vinyl adhesives, or those that dry by chemical reaction, like cyanoacrylates or epoxy resins. The main drawback in using this and other synthetic polymers is their lack of chemical compatibility with bone matter, which can cause irregular dilation gradients and generate external and internal stresses. Furthermore, acrylic resins undergo photodeterioration upon continuous exposure to light sources, causing changes in hue and loss of elasticity and adhesive strength [5,21,22].

As for studying paleontological heritage, adhesion and consolidation treatments with Paraloid B-72® and other products may make it difficult to perform and interpret their analyses such as with optical and electron microscopy, molecular biology or stable isotope studies [23,24]. Likewise, acrylic resins involve a carbon contribution (Takahashi and Nelson 2002) that can alter the results of C14-based techniques [5,9,25,26]. In this sense, a previous study has been carried out [27] whose objective was to determine the penetration capacity and the risk of contamination in consolidation treatments with Paraloid B-72®, Nanorestore® and Estel 1000®. The products studied showed a good capacity for penetration, which was greater in Nanorestore®, though they never exceeded 4 mm in depth. This suggests that these treatments are effective in medium-to-large-sized fossils or bones with no risk of contamination. However, in the case of microfauna bones whose cortical bone thickness is 2 mm or less, these procedures may be too invasive.

This study aims to propose a simple, effective protocol to improve the structural conservation of bones and fossils with minimum invasiveness. It makes use of the multiple functional advantages of Paraloid B-72® by using a new method of application via surface adhesion sheets, similar to adhesive tape. These sheets are placed over the fissures. They are semi-transparent in appearance and can reinforce the piece via a reversible treatment with minimal physicochemical impact. This new application format of Paraloid B-72 has not been previously described. Related procedures have been proposed, based on the product in a solid state subsequently resolubilized with acetone [28,29], although the aims and the method used are not the same as those described in this study, which uses a flexible, superficial adhesive that ensures good preservation with minimal intervention in the original materials.

Materials and Methods

Samples

Four types of samples were used to evaluate Paraloid B-72® sheets (Table 1). Bone fragments of deer metapods (type 1 samples, Figure 1) were chosen because they are modern bones in a good state of conservation. The study of the sheets' behaviour with this type of very stable and dense sample ensures their efficacy in fossil pieces, which are more delicate and lighter.

Figure 1: Study samples: Type 1: Deer metapod fragment; Type 2: Plaster cubic specimen; Type 3: Plaster bone trabecula.

Table 1: Samples used in the study, treatments and studies applied.

Sample Type	Description	Treatment	Study
1	Deer metapods	Paraloid B-72® sheets 5%-10%-20%	-Adhesión -Drying Time -Penetration Capacity -Reversibility
2	Cubic plaster simples		
3	Irregular plaster samples		
4	Dinosaur footprint	Paraloid B-72® sheets-20%	-Adhesion (extreme)

To complete the study, two types of plaster samples were added (type 2 and 3, Figure 1). The plaster was obtained by mixing high purity ceramic gypsum ($CaSO_4 \cdot 1/2H_2O$) (Bedmar Plasters, Barcelona, Spain) and water in a ratio of 1.20:1 (kg/L). Cubic samples measuring 5×5 cm were prepared with this material (type 2 samples). They resemble the cortical layer of bone due to their texture, which is very consistent and even, so that it allows for evaluation of the product's penetration capacity. Likewise, in order to study the behaviour of the sheets in porous and curved shapes, which are more similar to natural ones, irregular plaster samples of 5×5 cm were prepared, simulating the trabeculae of the spongy area of a bone (type 3 samples). In this case, cubic plaster specimens were drilled randomly using an electric drill with a 0.8-1.6 mm drill bit and a 1.6 drill bit. In addition, 120-grit wet sandpaper was used to increase the porosity of the entire sample, both externally and internally. All types of samples and the adhesion, resolubilization, and penetration tests were done in triplicate to check the reproducibility of the results. Finally, to evaluate the adhesive strength of the Paraloid B-72® sheets, tests called "extreme adhesion tests" were performed, where two plaster dinosaur footprints weighing 3,764 and 4,008 g with microcracks and a central fracture were used as samples (type 4). For this test, an adhesion system was performed by cross-laying sheets of various concentrations and drying times (Figure 2).

Figure 2: Plaster dinosaur footprints (type 4 samples).

Paraloid B-72® sheets

The sheets were prepared using a 15×18 cm industrial plastic mould, though the method can be adapted to obtain formats of any size, weight, thickness and shape (Figure 3). The preparation started with mixtures of Paraloid B-72® and acetone (CTS, Madrid, Spain) in the proportions usually used in paleontological excavations: 5%, 10% and 20% w/v [27]. Acetone was stained with safranin (Sigma-Aldrich, Madrid) in a proportion of 0.5% by volume to help measure the proposed treatments' penetration capacity [27].

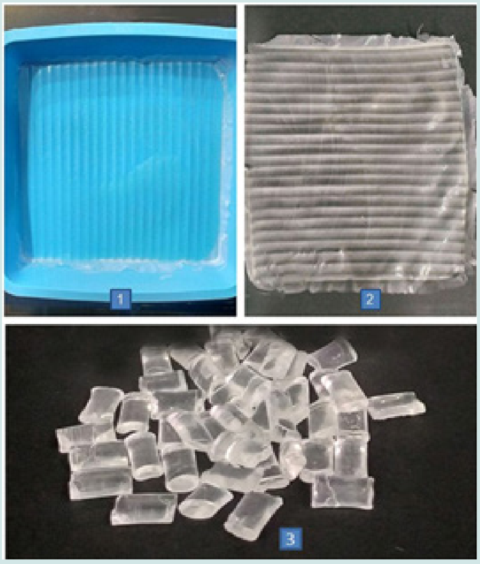

Figure 3: Obtention of Paraloid B72® sheets (preparation in mould 1-2 and final product 3).

After obtaining the solutions, they were kept for 24 hours at 20°C while stirring for the product to completely dissolve and eliminate air bubbles. Subsequently, the mixtures were poured into the mould and allowed to solidify for 24 hours, in the case of 10% and 20% sheets, and for 48 hours for those prepared at 5%, to ensure proper consistency. Then they were removed from the mould and the central pieces were cut with a length of 3 cm, a thickness of between 0.45 and 0.55 mm, and a weight of between 0.1 and 0.2 g. and then immediately used for the study (Figure 3). Regarding the use of the sheets for on-site use, they can retain their properties for at least 48 hours if they are kept in any closed container or wrapped in polyethylene film to slow down the polymerization process.

The properties of the sheets were evaluated by carrying out various assays in which their capacity of penetration, polymerization time, treatment reversibility and adhesive strength were tested. Each test was carried out three times to ensure the studies could be reproduced. The samples of types 1, 2 and 3 were cut crosswise with a toothed saw (Figure 4), and the resulting pieces underwent surface adhesion treatment with Paraloid B-72® sheets in the three concentrations described, measuring the drying times in each case. In the case of the dinosaur footprints, no cuts were made, since both footprints were fragmented into several parts, presenting microcracks and cracks with greater separation of between 1 and 2 cm.

Figure 4: Cutting of type 2 (1) and 3 (2) samples.

The placement of Paraloid B-72 sheets to achieve effective adhesion of a fissure or crack depends on the width and shape of such structural alterations. In more uniform cracks with a width of less than 0.5 cm and a length of no more than 4 cm, as is the case of type 1, 2 and 3 samples, the sheets were placed longitudinally (Figure 5). If the crack has an uneven shape exceeding 4 cm in length and 0.5 cm in width, as in the case of dinosaur footprints (type 4 samples), it is advisable to overlay and crisscross the sheets (Figure 6). The first layer was placed perpendicularly to the orientation of the crack, and the second layer was applied longitudinally, following the same direction as the crack and adapting to its shape. It is convenient to place the sheets at the points of the crack that are closest to each other. In this way, there is greater efficiency ensured in the reinforcement for the matter being restored, especially in samples weighing more than 0.5 kg.

Figure 5: Application of Paraloid® B72 sheets in type 1 samples.

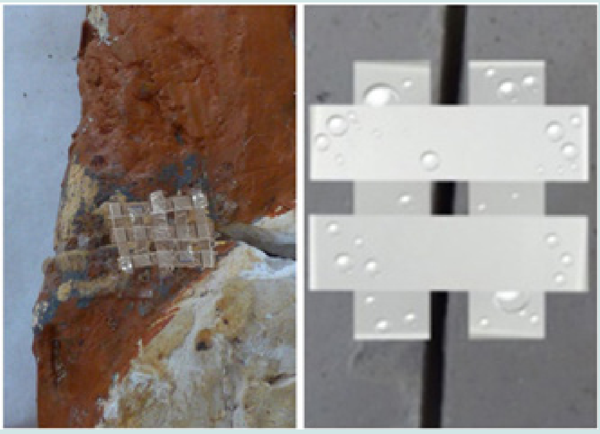

Figure 6: Design of adhesive structures.

Following the adhesion study, a resolubilization test was carried out, which involved removing the sheets to study the treatment's reversibility. The test was carried out at different times after the start of the adhesion process: 1 hour, 24 hours and 30 days. To resolubilize the sheets, acetone was used as a solvent, applied with a brush to the sheets with the highest concentration of Paraloid B-72® (10% and 20%) and by means of a 5 ml syringe to the ones with the lowest concentration (5%), thus achieving a less abrasive interaction for the surface and a more efficient application of the product. After completing the resolubilization process, the sheets were removed (Figure 7). After separating the pieces, the penetration capacity of Paraloid B-72® with the different concentrations of the sheets studied (5%, 10% and 20%) was measured with a digital calliper and a Leica GZ6 binocular loupe with a built-in Leica DFC 320 camera. The magnifications used to perform the tests were 6×, 12×, 25× and 50× [30,31].

Figure 7: Resolubilization of Paraloid B72 sheets in type 1 samples.

Results and Discussion

Adhesion tests

The Paraloid B-72® sheets showed great flexibility in all of the concentrations studied, especially at 5%, gradually increasing their rigidity with the polymerization and drying process, which varies according to the concentration tested. In the case of sheets prepared at 5%, in type 1 samples good adhesion was achieved in a short time (5-7min) since they adapted perfectly to the curved bone surface. In type 2 specimens, 5 minutes were enough to achieve adhesion, given the evenness and great porosity of their surface, whereas in type 3 samples, which are much more uneven, the adhesion of the separated parts requires more time to obtain good fixation, between 7 and 10 minutes [32-35].

In the sheets prepared at 10%, the adhesion time was shorter in the three types of samples, with a maximum of 5 minutes, while in the 20% sheets it was less than one minute in the type 2 samples and less than two minutes in the irregularly shaped ones (types 1 and 3). In all cases, the stability achieved was adequate for fossil handling and storage. As regards interventions in archaeological sites, a waiting time of 20 minutes is advisable in order to attain sufficient consistency for the fossils treated with any of the concentrations tested. In general, after one hour the transparency and elastic properties of the Paraloid B-72® sheets were maintained, allowing for easy removal if necessary. After 24 hours, the sheets almost completely lose their elastic properties and part of their transparency, showing a whiter tone and more intense adhesion (Figure 8) [36-39].

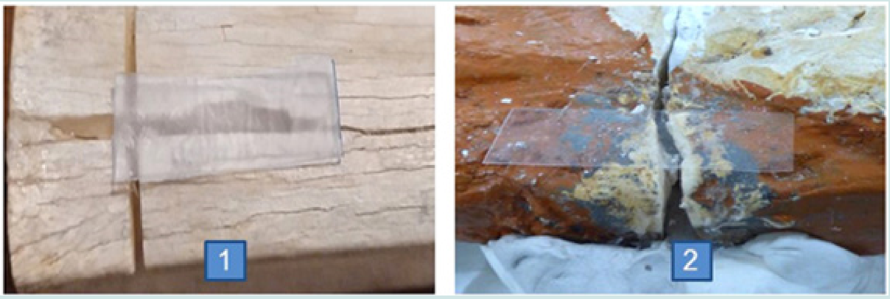

Figure 8: Paraloid B-72® sheets (10%) applied to type 1 (1) and 4 samples (2) after 24 hours.

Fifteen days after application and up to 30 days, a slight increase in opacity and a gradual loss of elasticity were observed, giving the fossils a strong adhesion and great stability, very suitable for handling and museum display, which is the objective of the study without the need to apply specific tests, such as micro-drilling, peeling tape or Vickers hardness, with which we have carried out several preliminary studies that have shown little reproducible results in these types of materials. This application format allows for great freedom in handling the product, in moldable and cuttable sheets with the desired concentration of adhesive, shape, thickness and dimension. In the extreme adhesion test, the qualities of the Paraloid B-72® sheets were used as much as possible to aid the fixation of the pieces of the dinosaur footprint with irregular morphology (Table 1). As a basis for closure of the larger cracks, sheets at 20% were used, arranged in a crisscross pattern in the form of a net (Figure 6) although other systems are possible. These sheets were placed at key points of the fissure; that is, clear fit areas where a better grip of the fragments was ensured. Once all the parts are disposed correctly, smaller and more concentrated sheets can be used, including those prepared at 5% (Figure 9). It is advisable not to apply many overlapping sheets, since this can cause poor fixing of the sheets to the piece because they are kept wet for longer [40-42].

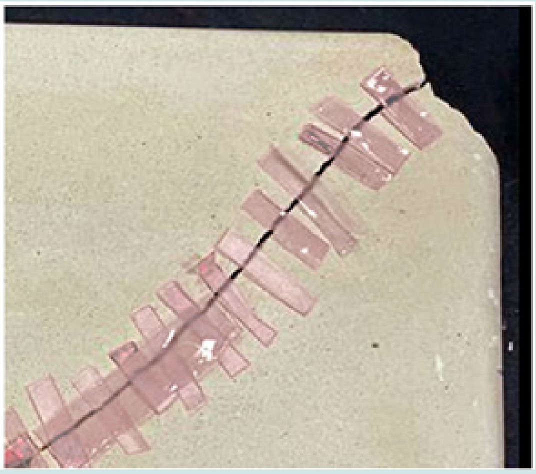

Figure 9: Application of Paraloid B-72® sheets in a fissure (type 4 samples).

Reversibility tests

The reversibility tests were carried out on the different types of samples in times of 1 hour, 24 hours and 30 days after the application of the sheets. After one hour, the reversibility with acetone was achieved in all cases with great simplicity (Figure 10). This is helped by the sheets being easier to locate compared to other adhesion techniques, as well as using a minimal thickness of adhesive sheets, while they have not completely dried in such a short period of time. The sheets prepared at 5% were removed by applying acetone with a syringe, allowing the solvent to act for less than thirty seconds in all types of samples. The sheets prepared at 10% can be completely removed in a maximum time of 3 min after applying the solvent, while in the case of those prepared at 20% the time ranges from 3 to 5 min. On the other hand, the use of the brush in these cases involves a greater risk of detachment of fragments from the outermost layer of the piece, due to the traction force exerted when removing the adhesive. The resolubilization of the sheets after 24 hours was more difficult, since the solvent's action must be maintained for around 30 minutes. In any case, the proposed application system allowed for a relatively simple reversibility process compared to traditional treatments in a liquid or viscous state using a brush or syringe, in which it is often impossible to locate and completely resolubilize the adhesive.

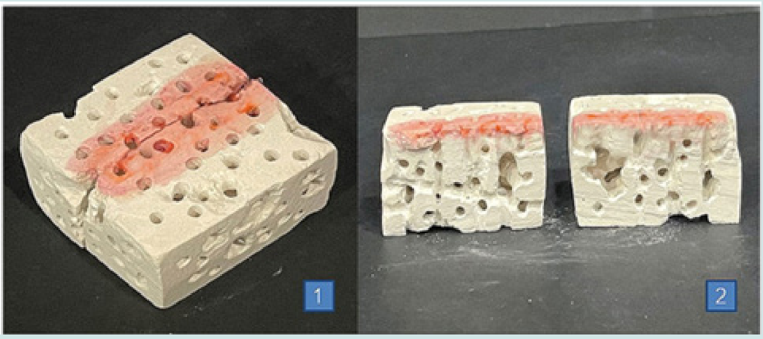

Figure 10: Resolubilization of Paraloid B72 sheets in type 3 samples.

Penetration capacity

The penetration of the product occurred in all types of samples at a very superficial level in the three concentrations tested, always reaching less than 1 mm of maximum depth (Figure 11). This is a significant decrease with respect to traditional application systems [27] and therefore a significant reduction in the addition of foreign matter to these cultural assets.

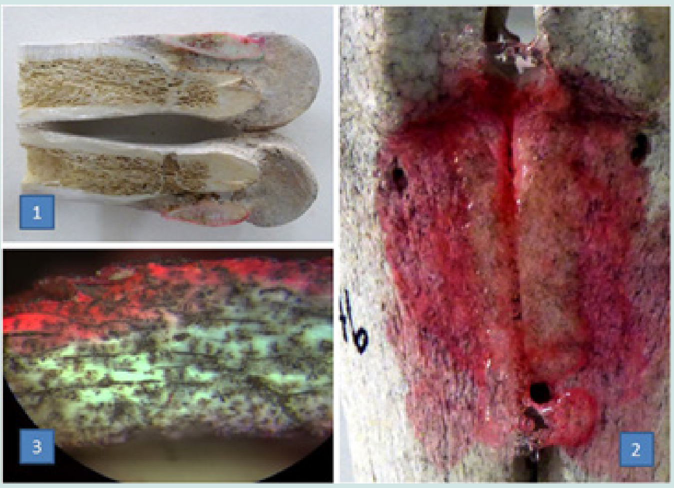

Figure 11: Type 1 samples (deer metapods), detail of staining after removing a Paraloid B-72® sheet (1,2) and study of penetration capacity by marking with 50x safranin (3).

Conclusion

A new curative conservation method has been proposed for the most widely used adhesion methods on bones and fossils, with the intention of preserving the pieces' original physical and chemical properties, as well as their biological information. Paraloid B-72® sheets have been shown to be a very stable, reversible and effective adhesion method, which limits the application of the product to the most superficial layer, thus keeping contamination of the sample under control. The possibility of using different concentrations of Paraloid, as well as sizes and thicknesses of sheets, enables the system to adapt to different types of fossils. Moreover, it is simple to handle and easily applicable in interventions in archaeological sites, allowing to work quickly with a product well known as adhesive material and safe for cultural assets and restorer's health [28-42].

Conflicts of Interest

The authors declare no conflicts of interest. The funders played no part in designing the study or in collecting, analysing or interpreting the data, in writing the manuscript, or in the decision to publish the results.

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