

# LEAF COMPRESSIONS FROM THE LATE MIOCENE SECTIONS OF NW ITALY: RESEARCH ON AN EFFICIENT, EASY AND QUICK CONSOLIDATION TREATMENT

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Abstract: Compressions of fossil leaves on marl blocks soaked with water are tricky palaeobotanic findings to deal with. In fact, this peculiarity makes the findings exposed to serious degradation: the evaporation of water causes shrinkage of the matrix and the leaf leading to fragmentation, delamination and exfoliation of the fossil, until its final disappearance. The aim of this research was to identify a consolidation method which satisfies museum needs and can be utilised for all conditions of hydration of the object. Following a survey of several natural science museums in Northern Italy, Paraloid<sup>™</sup> B72 and PEG4000 along with other resins never previously used on this kind of fossil were tested. At the end of the research, two products gave adequate or good results, preserving the leaf from further degradation whatever the hydration condition of the object: the acrylic resin Paraloid<sup>™</sup> B72 and a polyurethane resin.

Key words: Fossil leaves, consolidation, Paraloid<sup>™</sup> B72, polyvinyl acetate, aliphatic polyurethane

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#### Introduction

The marl deposits formed during the final part of the Miocene (late Messinian, 6.0 to 5.3 Ma), and abundantly cropping out in NW Italy, in particular around the town of Alba (Dela Pierre et al. 2011), are very rich in fossil plant remains. The stratigraphy and the characteristics of these deposits have been recently described by Lozar et al. (2010) and Dela Pierre et al. (2011). Fossil plant remains have been collected from several localities and studied by Cavallo et al. (1986), Martinetto et al. (2000, 2007), Bertini and Martinetto (2008).

In particular two localities (Text-fig. 1) yield abundant leaf compression remains: the Govone and Pollenzo sections (Lozar et al. 2010, Dela Pierre et al. 2011), where the late Miocene strata have been repeatedly eroded by the Tanaro River. This natural excavation brings to the surface hundreds of fossils, destined to rapid decay.

In fact, the main conservative issue regarding freshly collected fossil leaves is related to the water content of the matrix which provides plasticity to the leaf and the embedding sediment. When dehydration occurs, the muddy support and the leaf contract differentially, due to their different properties. Thus, the leaf becomes brittle and breaks, especially along the midrib and the veins. Furthermore, in the Messinian marls of NW Italy an additional problem is the abundant growth of gypsum crystals on the leaf surface.

The local museums of Alba and Asti have often attempted recovery of material at risk, but conservation was hampered by the rapid degradation of the fossils. Although examples of specimens successfully prepared for museum exhibition do exist, satisfactory preparation of fossil leaf compressions is recorded as being extremely difficult. Two cases are reported here as examples of possible preparation methods. The first method suggested considers a leaf which underwent a successful treatment: it is the pinnate palm foliage from Pollenzo recently reported by Teodoridis et al. (2015), now exhibited in the Alba Archaeological and Natural Sciences Museum "F. Eusebio". The acrylic resin Paraloid<sup>™</sup> B72 (5% w/w diluted in acetone) was applied once the object was partially dehydrated in order to allow the product to penetrate into the substrate without forming a whitish film over the surface (E. Bonelli, personal communication). On the contrary, a leaf assigned to Engelhardia orsbergensis (WESSEL et WEBER) JÄHNICHEN, MAI et WALTHER, figured



Text-fig. 1. (A) Map showing the location of the main macroflora-bearing sites in northern and central Italy. 1–5, sites of the Gessoso-Solfifera Formation near Alba: Castagnito, Guarene, Monticello, Piobesi and Scaparoni. 6, Corneliano d'Alba. 7, Nizza Monferrato. 8, Banengo. 9, Monte Castello. 10, Carbonara Scrivia and Torre Sterpi. 11, "Stradella" and Portalbera. 12, Tossignano and Monte Tondo. 13, Polenta. 14, Monte Castellaro. 15, Senigallia. 16, Ancona. 17, Gabbro. 18, Palena. The dimension of the leaf symbols is proportional to the taxonomic diversity in each plant-bearing locality. (B) Locations ( $\star$ ) of the two Messinian plant-bearing localities that provided the material studied in this paper. 1: Pollenzo; 2: Govone. Shading indicates the distribution of Messinian sediments in the vicinity of Alba (NW Italy).

by Bertini et al. (2014), which was in good condition when photographed in the field, but not treated in any way completely broke up after a few weeks. In 2014, a rich late Miocene assemblage of leaf compressions was exposed by the Tanaro River in the Govone section, ca. 23 km NE from the Pollenzo section: a few specimens in danger of destruction (Pl. 1) were recovered by the application of a commercial cyanoacrylate glue applied by brush to the top of the leaf with the aim of preparing them for the exhibition at the Asti Museum. Issues connected with such consolidation treatment are the rigidity and fragility of the polymeric material which scarcely follows the micromovements of the object. Although Messinian plant macrofossils, with an analogous preservation state to those presented here have been recovered not far from the studied area (Sachse 2001), no precise information regarding preparation was published.

From a more scientific point of view, the importance of these fossils is related to the palaeofloral, palaeoclimatic and palaeoenvironmental signals that can be obtained through their study. For this reason, consolidation treatment also needs to be reversible after some years in order to permit new analyses. The preservation of fossil leaf cuticle is of primary importance not only for taxonomic studies, but also as a record of carbon dioxide palaeoconcentration.

Usually palaeobotanists wrap samples carefully in order to keep them in a microenvironment as similar as possible to the sedimentary deposit; later they put them in water as a temporary arrangement, with a low concentration of acid and/or biocide (Collinson 1987). Later, specimens are stored in an aqueous solution of glycerol or polyethylene glycol (PEG) 200/600. Another storage system involves the application of PEG4000 in water (1:1 ratio) directly on the surface, followed by placement of the sample in a polyethylene bag under vacuum as shown in Pl. 2, Fig. 1 (Cilia 2005). However leaves could be damaged: alcoholic solutions can degrade organic material, while PEG can lead to even more serious problems as reported by Collinson (1987). Nevertheless, PEG is reversible, allowing scientists to analyse findings by carefully removing the polymer in limited areas, and eventually taking a sample. However, the conservation methodologies described do not satisfy museum needs because the leaves cannot be exposed in these conditions. Several synthetic polymers have been tested for consolidation treatment: some were soluble in water, such as polyvinyl alcohol (PVAl), whereas others, e.g. polyvinyl acetate (PVAc), acrylic and polyester resins, were soluble in organic solvents or available as aqueous emulsions. The objective was to achieve fixation of the leaf on the matrix by the application of a product which could be viewed as the definitive solution regardless of the state of hydration of the fossil (wet or partially/totally dried).

PVAc aqueous dispersions (e.g. commercially available under the trade name of Vinavil®) have been used to repair fractures or as a support for whole pieces (Collinson 1995); they have also been applied to the leaf surface to consolidate this fragile layer on the matrix (Mastrorilli 1965, Collinson 1987). Solvent-soluble varnishes, fixatives or acrylic sprays can sometimes preserve the fossil from further degradation when already exfoliated, but it is important to guarantee the correct moisture condition in order to avoid the coating clouding phenomenon (Collinson 1987, Lepage and Basinger 1993). Unfortunately, the consolidant tends to form a shiny layer, which is disturbing from an aesthetic point of view and also hindering observation under an optical microscope. In general application to partially or completely dried samples is pointless (Collinson 1987). Lepage and Basinger (1993) sprayed samples with a nitrocellulose lacquer diluted in acetone or in high-quality lacquer thinner, such as Lucite® series by Dupont. This treatment produces good results, however, some nitrocellulose formulations

were found to contain abnormal amounts of phenolic resins causing darkening of the coating. Although nitrocellulose is known to be quite unstable (Selwitz 1988), a lifetime of up to a century can be hypothesized in museum environments (Shashoua et al. 1992) and even though it is not frequently employed, conservators' discussions regarding its value are still ongoing (Koob 1982).

Silicon oils have also been tried out. Since these are not compatible with water, wet samples must be previously immersed in a series of alcohol baths, then treated with toluene and finally with silicon oil. In addition, samples need to be washed before being studied (Collinson 1995), so this alternative turns out to be both complicated and expensive and does not satisfy either museums or scientists.

Some attempts have been made on previously desiccated samples. Acrylic resins and water based dispersions were tested, and also a poly(p-xylylene) polymer whose low reversibility limited application (Collinson 1995).

There is very little literature about the preservation of this kind of findings and the long term efficacy of the treatments, thus a short survey was carried out among a number of Italian museums with similar fossil collections.

Potassium silicate was tested by Leidi, but the product created a crumbling vitreous layer on the surface affecting the adhesion and the entirety of the leaf (Leidi 2004). The author also suggested the use of an aqueous solution with 4% of PVA1 and about 1% dichlorophen as the biocide. The Bergamo Museum of Natural Sciences (Italy) applied this formulation to some findings: after four years, the leaves with thinner cuticle were already degraded (B. Leidi, F. Confortini, personal communication).

The procedure used at the Civic Museum of Natural History in Milan involved the application of a polyester resin used as an adhesive to restore cracks and crevices (G. Teruzzi, personal communication). The same resin, crosslinked with styrene, was occasionally used as a consolidant. Other objects were treated with PVAc producing a film on the surface which did not stop degradation of the leaf as it did not penetrate into the porosity of the marl. At the Palaeontological Museum of Montevarchi a few fossils with exfoliated and detached areas were re-moistened and attached again to the matrix by the application of an aqueous PVAc adhesive, diluted in water (5%) (M. Rustioni, personal communication). Apparently, the extended degradation was also due to a previous consolidation treatment, the details of which are unknown. Other examples of objects treated with PVAc were observed at the Civic museums of Natural Science "G. Doria" in Genova, at the Experimental geological museum in Giaveno and at the Civic museum of Natural Sciences "Craveri" in Bra, with opposite results: the consolidation was successful with fossils fixed to the matrix after final dehydration. In fact, no colour changes or defoliation could be noticed 3 years after the treatment of a large *Platanus* leaf sample continuously exposed to moderate solar light. Despite these results, the application of the polymer when not dispersed sufficiently in water created surface films that shrank resulting in physical stress on the fossil being preserved (Cimino 2015).

From what has been revealed by the survey, the conservative approach seemed to be more related to empirical restoration practices rather than to scientific systematic studies. The work introduced here was based on the information so far obtained and aimed at researching a conservative methodology suitable for museum-exhibition purposes of this type of fossil. Therefore, research on a suitable preparation technique for such material was carried out, aimed at finding a compromise between an acceptable result for museum exhibition and a reversible preparation which allows cuticular studies. Attention was focused on seminatural and synthetic products whose formulations could consolidate leaves before water evaporation in order to also protect the fossils against mechanical stresses. The compatibility with the aqueous environment was therefore a selection criterion.

## Materials and methods

The different treatments were carried out on compressions of fossil leaves from the Govone and Pollenzo stratigraphic sections, collected in layers of muddy sediments formed during the Messinian age. Fossilization took place under two conditions: a permanently anoxic environment and secondly an environment with increasing pressure due to sediment load. The Govone specimens were collected from a single stratum of marls sampled by one of the authors, and therefore named "Damarco bed"; the Pollenzo specimens were collected from several marly layers with similar characteristics within the section described by Dela Pierre et al. (2011).

The experiments were mainly aimed at defining more accurate preparation methods. Two consolidation approaches were followed:

- using a polymer which forms an elastic film on the surface to make the leaf and, at least, the upper part of the marl more elastic in order to improve resistance to mechanical strains resulting from the evaporation of the water;
- attaching the leaf to the outer most marl layer using a resin inclusion which has the double effect of securing it on the substrate and protecting it from mechanical stresses.

Due to the importance of these palaeobotanical objects, during this experimentation only incomplete or partially degraded leaves were treated.

Six chemical classes of consolidants were tested:

- Polyethylene glycols: PEG4000 (distributed by Gentaur Europe, Milan, Italy), chosen because occasionally it has been employed as a quick intervention on this type of fossil. It creates a polymeric film which may be solubilised in warm water.
- II. Cellulose ether compounds: Klucel G (supplied by Bresciani srl, Milan, Italy) is a hydroxyl-methyl-propyl cellulose. It is well known as a consolidant when applied to cellulose-based materials (paper and wood) (Flieder et al. 1988, Jackson and Watson 2010); it forms a thermoplastic and elastic film. Two different polymer solutions were tested: in water and in ethyl alcohol 99,9%, both with 2% (w/w) polymer.

- III. Acrylic resins (Chiantore et al. 1996): among the possible products available, three were chosen for this study, all sold by Rohm and Haas company:
- Paraloid<sup>TM</sup> B66: methyl methacrylate/ butyl methacrylate (MMA/BMA) copolymer, ca. 1:1 ratio;
- Paraloid<sup>TM</sup> B72: methyl acrylate/ ethyl methacrylate (MA/EMA) copolymer, ca. 3:7 ratio;
- Paraloid<sup>™</sup> B82: methyl methacrylate/ ethyl acrylate (MMA/EA) copolymer, ca. 1:1ratio.
   Acrylic resins have already been tested with good results on animal fossils (Paganoni 2003, Lopez-Polin et al. 2008, Beiner and Rabinovich 2013) and in other conservation fields (Charola et al. 1985, Amoroso and Camaiti 1997). In most cases they were solubilised in

acetone, a solvent with a high evaporation rate. Here,

xylene was preferred because of its lower volatility. IV. Silicate-based consolidant (Felix and Furlan 1994): the esters of orthosilicic acid are one of the two main groups of consolidants in this class. In this instance, the tetraethyl orthosilicate (TEOS) Estel 1000 supplied by CPC (Rome, Italy) was tested.

TEOS is often used in conservation treatments, especially on stones (Horie 2010). It comes up in a monomeric form that, in the presence of water, reacts by hydrolysis followed by a condensation step. At the end of the process, precipitation of amorphous silica occurs. TEOS was tested because of its well-known properties:

- hydrophilicity of the solution and of the precipitate;
- limited dimensions of the starting product so that its penetration of the marl is facilitated;
- the consolidant is able to create bonds with free OH groups in the matrix of silicate-type samples (Goins et al. 1996, Silva et al. 1997).

The application of this product may be affected by the high humidity of the support which promotes the hydrolysis phase, but limits condensation.

- V. Polyurethane resins: three aqueous dispersions (supplied by Euraflex, Turin, Italy) were chosen (30% w/w of polymer):
- Flex PU 2130: aliphatic, cationic, pH 5, used in leather manufacturing;
- Flex PU 2230: aliphatic, anionic, pH 7;
- Flex PU 4230: aliphatic, anionic, pH 9, used in varnishes for wood treatment.

Polyurethane resins are not yet widespread in the field of conservation, but several tests have been reported aimed at modifying their chemical and physical properties in order to obtain suitable products (D'Orazio et al. 2001).

VI. Thermosetting resins: an epoxy resin, bisphenol A (RenLam M-1, sold by Huntsman, USA), cured with a polyfunctional amine was employed. Epoxy resins are well known for their excellent adhesive strength and great mechanical properties (Horie 2010). Despite a low photochemical stability leading to yellowing, this resin was tested because of its structural rigidity.

Application of aqueous products should be favoured because of better interaction and easier diffusion into the matrix of the wet object, but with the awareness that, when water dispersions are used, the size of micelles could be greater than some of the pores in the marl. Furthermore, the high concentration of salts (and therefore the possible high ionic strength) may cause collapse of the product. This also seems to be confirmed by the results obtained by Leidi (Leidi 2004) with Primal AC-33, an aqueous acrylic dispersion: the surface film was not able to prevent fossil degradation due to limited penetration or sudden collapse of the polymer on the surface.

Three different methods of application were tested: "brush-dropping" (i.e. drops of solution applied to the surface by brush, allowing them to fall or gently depositing them on the surface, but never exerting pressure on the leaf through the brushstrokes), immersion in the consolidant solution and thirdly through capillarity by the use of lint. As the application was not always carried out on wet samples, the last method proved to be too risky: when dry, the leaf is very fragile and can be displaced by even the minimum rubbing exerted by the lint. Furthermore, a high evaporation rate of the solvent from the textile causes adhesion of the fossil to the lint.

The brush-dropping method showed several advantages: the consolidant may be applied quickly and it is possible to control the thickness of the film also taking into account the aesthetic appearance.

Only TEOS could also be applied by immersion in order to allow penetration of the solution into the porous structure of the marl so as to have additionally a deposition of silica in the inner part of the support.

35 samples were treated in three different ways: one group was treated immediately after arrival to the laboratory, another group of samples was left to dry for a few days until the beginning of the exfoliation phenomenon, and the last group underwent a double-step treatment, i.e. a first application of PEG4000 aqueous solution and, after one month under vacuum condition, a second treatment with a different product.

After the treatment, samples were observed under optical microscopy to evaluate optical properties, whereas the mechanical behaviour was empirically evaluated by pressing the surface of the sample with a dry brush in order to simulate a moderate abrasive stress. Since the pattern and surface of the samples were irregular, it was not possible to carry out colorimetric and gloss analyses. For this reason, evaluation of the final appearance was performed visually: an overly shiny film prevents clear observation of the object, thus it is better to have a film with as limited a gloss as possible. Another aesthetic aspect is the presence of bubbles formed in thermosetting resins as a consequence of the exothermic curing reaction.

The stability of the two best performing consolidants was evaluated with accelerated ageing experiments involving irradiation at a constant temperature of 50°C for 1000 hours in a Suntest CPS+ (Heraeus, Germany) simulating solar radiation with a xenon lamp and a Suprax filter to cut out wavelengths below 295 nm. Film forming capability and yellowing were evaluated on sample films applied to microscopy glass slides, whereas four rock specimens, two for each product, with and without a fossil leaf, were aged in order to verify the effect of the polymeric layer on the object's appearance, also relating it to the thickness. These marl samples were partially covered during the ageing process with an aluminium sheet to differentiate thermal effects from photo degradation.

All the treated objects were kept in laboratory condition  $(20^{\circ}C, 45\% \text{ Rh})$  in a box and examined again after eight years.

#### **Results and discussion**

The tested consolidants were chosen according to their performance in other branches of the field of conservation and considering the particular nature of the specimens, particularly insidious because of the heterogeneity related to the contemporary presence of hydrated organic material, hydrated inorganic aluminosilicates, water and, most likely, soluble salts. Evaluation of the consolidant performance was based on the appearance of the fossil specimen after the treatment and the permanence of the consolidant on the marl surface. A resin was considered excellent when it created a non-tacky, continuous, homogeneous and transparent surface layer; on the contrary it was considered very poor if the film was not homogeneous along the surface or if the product did not produce a film at all. Similarly, mechanical properties were considered very good if the brush action did not cause the removal of leaf fragments and inadequate if the fossil material detached.

#### **Products performance**

Fossils treated with PEG4000 appeared to be consolidated and initially preserved from the fragmentation phenomenon both in air and when wrapped under a vacuum, but it was noticed that the efficacy remained for only a limited time. After only two years the objects treated and kept in a bag under vacuum showed fractures (Pl. 2, Fig. 1). The presence of PEG, on the other hand, was found not to have any detrimental effect on the efficacy of other resins (epoxy, acrylics, polyurethanes) when subsequently applied for consolidation.

Klucel G appeared to be totally ineffective, with no differences related to the solvent employed. Delamination and fragmentation of the leaf occurred within a few days following application, together with the beginning of dehydration.

Among the acrylic resins tested, Paraloid<sup>TM</sup> B82 did not produce satisfactory results, whatever the hydration state of the fossil. On the other hand, Paraloid<sup>TM</sup> B66 and Paraloid<sup>TM</sup> B72 appeared to be good products when applied to the samples. They formed a film on the surface which acted more as an adhesive than a consolidant: the leaf continued to lose water without detaching from the support. Application of these products on partially dried specimens however was not optimal because the film always lost its mechanical properties over time. Probably in this condition the film-contact with leaf and marl surfaces was not homogeneous, similarly the contact between the marl and the partially detached leaf was also not homogeneous.

Brush application of TEOS produced the worst results: the hydrolysis reaction was probably too fast and the product did not penetrate deeply enough into the marl before the condensation phase began; the amorphous silica precipitated on the surface without consolidating the leaf. In fact, on areas where the fossil was already detached, drops of product accumulated creating a kind of efflorescence, increasing the damage. There was no difference when application was by immersion and, despite the affinity between marl and consolidant, the results were completely unsatisfactory also with respect to the mechanical resistance.

Polyurethane resins Flex PU 2230 and 4230 did not give good results: the former did not prevent the fossil leaves from detachment and fragmentation, while the latter consolidated and preserved the finding from exfoliation only where a very thin layer was created; the polymeric film was not homogeneous and probably for this reason it was not able to maintain the sample and its fragments on the substrate. Furthermore, this irregular pattern is an unwanted effect from an aesthetic point of view. The third polyurethane resin, Flex PU 2130, gave more promising results in all cases of application. In fact, the polyurethane formed a transparent, continuous and elastic layer, with low gloss, capable of penetrating into fractures and acting as an adhesive between the small fragments and the substrate (Pl. 2, Fig. 3), avoiding their complete detachment even when applied to dry samples. The elastic properties of the film play an important role in the success of the intervention: the Flex PU 2130 resin is much more flexible than the other polyurethanes tested, as also confirmed by the ultimate prolongation value of the material, 800% of the initial value, as reported in the technical data sheet. A continuous contact between the film and the finding is therefore ensured during the drying process.

The epoxy resin gave good initial results, creating a film with good adhesive and mechanical properties (Pl. 2, Fig. 2). The thickness of the film however proved to be a critical issue: with the increase of the layer, the film appearance can change from mat to shiny or even whitish. This behaviour is probably related to the formation of micro bubbles or to a clouding phenomenon due to the humidity, imperceptible when the film is very thin.

Since epoxy resin and Flex PU 2130 appeared to be the best consolidant systems for the particular application investigated here, accelerated ageing was carried out in order to evaluate their chemical behaviour after severe light exposure. In this stage of the trial it was important to identify possible further damage to the objects or visible changes. These two products are not reversible; in particular the epoxy resin yellows considerably with ageing and this affects the visual perception of the fossil, reducing the contrast between the leaf and the substrate. Therefore the change in their stability with ageing is crucial for predicting the suitability of possible application.

After the first ageing step (500 hours), the appearance was observed of a diffused craquelure phenomenon with greater scattering of the light on the film surfaces of both polymers when applied to flat substrates. A general yellowing was observed, especially on the areas exposed to light radiation, and the chromatic variation appeared particularly serious in the case of epoxy resin (Pl. 2, Fig. 4).

All the samples were similarly examined after eight years of natural ageing. Paraloid<sup>™</sup> B66, which had given good results on fossils treated when still wet, did not maintain the

properties necessary to hold the leaf on the substrate and the organic part was slowly crumbling. Samples treated with Paraloid<sup>™</sup> B72, alone or after previous application of PEG4000, were somehow fragile and less resistant to mechanical abrasion stresses. Nevertheless, fossils treated in this way showed no changes in their visible appearance and there were no fragments which were falling apart.

Flex PU 2130 maintained the mechanical properties both after accelerated and natural ageing: the film remained elastic and the resin layer, although broken at some points, could mirror the leaf movements without detaching. The film, where tightly connected to the marl, acted as an anchor, preventing the complete loss of the fragment. Epoxy resin was the only product which did not change during this time period, providing an excellent mechanical support to the finding and without showing colour changes. It must be considered, of course, that eight years represents a very short time period in comparison to the lifetime of an object in a museum collection.

In general, the compatibility between the water in the fossil substrate and the solvent in the consolidant formulation was recognised as being a critical issue. Furthermore, the formation of a superficial continuous film seems to be a necessary condition to preserve the entirety of the fossil. Thus, the consolidant does not need to penetrate deeply into the support, but it must seep into the leaf fractures so as to fix it on the surface due to its adhesive properties. Moreover, drying of the applied product needs to occur in an environment with low air circulation: evaporation of the water should occur slowly in order to avoid the sudden contraction of the leaf-marl system which is the main cause of exfoliation (Collinson 1987, Leidi 2004).

Although products which are completely soluble in water or water-compatible solvents were at the beginning of this work assumed to be the most suitable, the experimentation demonstrated that the opposite option was better. Penetration of the solvent/resin system and the interaction with the wet substrate is an issue which needs further experimentation since the results obtained seem more related to the film properties than to penetration into the marl. It could be that some of the products that appeared totally unsatisfactory might work better if applied using a different methodology or in different solvents and/or different concentrations. In any case, polymeric films with notable elastic behaviour were demonstrated to be more suitable for the objects investigated here due to their ability to stretch and adapt to the dimensional changes in the system.

The accelerated ageing allowed the authors to differentiate between the two best performing products in terms of "aesthetic performances" and chemical stability. The chemical stability of the polyurethane Flex PU 2130 was surprisingly good, whereas the light-fastness of the epoxy resin ranks the latter as inadequate. It is important to note that polymers tested in this research are not specifically developed for the purpose of consolidating fossil findings. It is possible to predict that there will be resins on the market with comparable mechanical and adhesive properties but more chemically stable and, therefore, worth considering further.

## Conclusions

The main aim of this study was to identify a successful preparation technique for fossils that, although interesting and suitable for museum exhibition, are rarely collected for this purpose because of the unquestionable difficulties posed by their preservation state. From the survey carried out on the consolidation practice in some Italian natural science museums, it appears that PVAc can be used for an effective "emergency" preparation with rapid treatment in the field or in poorly equipped laboratories in order to control dehydration which occurs to naturally exposed fossil leaf compressions. Nevertheless, its use is discouraged because, when not dispersed in sufficient water, it may create surface films capable of causing unacceptable stresses on the fossil.

The consolidation tests on fossil leaf compressions with waterlogged marl as the substrate were carried out by applying commercial polymeric products not developed for this particular purpose. According to conservation needs, consolidants for fossils treatment should be generally reversible.

The acrylic resin Paraloid<sup>™</sup> B72 was revealed to be a good product. Despite the poor resistance to mechanical stresses, that are assumed not to happen in museum showcases, its use can be suggested for future preparation of wet fossils of the type described here, in particular because of its reversibility. The application of a thin film may be carried out before water evaporation occurs by brush-dropping on the whole upper surface of the leaf-bearing sediment sample and can be eventually preceded by application of a concentrated solution of PEG4000.

The results of the investigation also showed that a waterbased aliphatic soft polyurethane resin was suitable for consolidating fossils on their marl substrates, guaranteeing an acceptable appearance of the objects, even though they exhibited a shiny surface and moderate yellowing after accelerated ageing.

Following these preliminary investigations, it seems worthwhile to continue experiments to assess different resin formulations, considering a larger group of commercial products. Experience, consideration and evidence from the work described here could represent basic guidelines for a conservation protocol for the preparation of this peculiar type of fossil. In particular, the organization of a new collection of fossil leaves from the Govone section may be a suitable starting point for testing these conclusions regarding preparation procedure.

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# **Explanation of the plates**

# PLATE 1

Fossils from the Damarco bed of Govone, Palaeontological Museum of Astigiano and Monferrato

- 1. Fossil leaf of *Acer tricuspidatum*. 1a, photographed shortly after extraction; notice the occasional black material indicating original organic remains. 1b, after needle preparation and consolidation with a thin film of cyanocrylate.
- 2. Two fossil leaves (left: *Dicotylophyllum* sp.; right: *Pterocarya paradisiaca*). 2a, photographed shortly after extraction; notice the abundant black material indicating original organic remains in the left specimen, contrasting with its scarcity in the right specimen. 2b, the same specimens after consolidation with a thin film of cyanocrylate.
- 3. Leaf compression of *Laurophyllum* sp. before (3a) and after (3b) treatment with cyanocrylate. Film application made the cracks in the fossil leaf lamina less apparent.
- 4. Leaf compression of *Engelhardia orsbergensis* before (4a) and after (4b) treatment with cyanocrylate.
- 5. Fossil leaf of *Dicotylophyllum* sp. with a well-preserved, thick cuticle; conversely, the mesophyll was largely lost during fossilization. Specimen shown before (5a) and after (5b) treatment with cyanocrylate.

# PLATE 2

Fragments of fossil leaves

- 1. Leaf compression wrapped in a polyethylene bag under vacuum when still in a humid state (year 2004, photographed 2008). Notice that the vacuum condition did not stop leaf lamina fragmentation. The specimen is not really suitable for museum exhibition, but can provide material for cuticular study. To the left, the cataloguing number PU105246.
- 2. Leaf compression from the Pollenzo section before (2a) and after (2b) treatment with epoxy resin.
- 3. Leaf compression from the Pollenzo section before (3a) and after (3b) consolidation with polyurethane Flex PU 2130.
- 4. Muddy sediment from the Pollenzo section treated with epoxy resin after accelerated ageing. Part of the object (upper half) was covered during the ageing process in order to verify if the behavior was due only to thermal exposure. The yellowing phenomenon is more evident in those areas where the film applied is thicker (left half).

PLATE 1



# PLATE 2

