The removal of Paraloid® coatings with aqueous based formulations. Practical case in frescoes from els Munts Roman *villa* (Catalonia)

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Abstract

This is the report of a practical case regarding the removal of Paraloid® B72 coatings from Roman frescoes. During the archaeological excavation, the paintings had been consolidated with the application of cotton gauze with an acrylic resin. The article presents the results regarding the effectiveness of the removal of the acrylic resin and the analytical assessment of the possible presence of residues on the work of art. The removal of the acrylic resin was carried out with an aqueous gelled system emulsified with only 10% solvent. The selection of the solvent phase presents fewer toxic alternatives to traditional solvents.

Keywords

Wall paintings, Emulsions, Gels, Cleaning, Green conservation, Paraloid®, Rhodiasolv® Iris

Introduction

The archaeological site of Els Munts is one of the most important Roman sites in Catalonia and a UNESCO World Heritage Site. The *villa* displays signs of occupation from the first to the seventh century and is located in a privileged area of the Catalan coast, next to the sea.

It is a sumptuous *villa*, profusely decorated with mosaics and wall paintings for the elites of the nearby capital of Tarraco. It consists of several sectors: the residential area, the baths area, a garden area or *hortus*, a *mithraeum*, an *ambulatio* connecting them and, from later on, an industrial area with water tanks.

The imperial residential area consisted of a large peristyle, a triclinium and a two-store building of which only part of the semi-subterranean floor remains. It was made up of a corridor and some adjoining rooms. This area is home to the most important set of Roman wall paintings preserved today in situ in Catalonia (Figure 1). These rich paintings were created in the second century, following a visit from the Roman Emperor, Hadrian.

Figure 1 – A general view of the crypto-portico and the annexed rooms where the wall paintings are located. Author: Silvia Marín.

Background

The excavation of the residential area of the *villa* began in the '90s of the last century when this group of wall paintings that decorate a wide and long corridor and five of the adjoining rooms of the crypto-portico were discovered (Tarrats, Macias, Ramon & Remolà, 1998).

During the excavation, and in order to protect the paintings according to the common practice, they were consolidated with various types of mortars and were protected with the placement of fabrics adhered with some type of resin. Once the excavation was finished, these fabrics were removed, but it remained an important thickness of resin covering the paintings.

Taking into account the importance of the paintings discovered, a provisional roof was placed. In spite of these protective measures, after a short period of time important signs of degradation associated with the presence of soluble salts began to be seen. These degradations were literally destroying the wall paintings (Figures 2 and 3).

Figure 2 – Detail of the degradation of the wall paintings. The resin coating on the surface is detaching small fragments of the painting layer. Author: Aleix Barberà.

Figure 3 – Detail of the degradation of the paintings produced by the disruptive effect of soluble salts. Author: Aleix Barberà.

Various restoration interventions were organized, which included cleaning, consolidation and desalination, but to this day they have not been able to guarantee the conservation of the pictorial ensemble or, at least, stop its degradation, which is still clearly active. The principal degradation factors are associated with the presence of soluble salts and their disruptive and disintegration effect on the paintings.

At the end of 2018, and in view of the strong and constant degradation, the Center for the Restoration of Cultural Assets of Catalonia decided to stop the interventions and carry out a preliminary in-depth study in order to exactly understand the origin of the degradations that affect the paintings.

This study, which is still in course, has included 3D digitization with photogrammetry of the whole complex; obtention of orthophotographs, drawing of alteration maps; extraction and analysis of samples; performance of cleaning tests; desalination tests; study of salt distribution patterns; investigation of the previous interventions and an architectural project for the planning of a new permanent roof (Barberà, Marín, Llobet, Rovira & Gracia, 2019). In this article, we specifically present the results of the cleaning studies.

This topic is relevant in this case because the presence of a thick layer of a coating on the wall paintings obstructs the porosity of the matrix and affects the correct circulation of water vapor. Then, it can be assumed that this film is a factor that aggravates the damages produced by salts. On the other hand, under the coating, there is a layer of dirt and calcareous concretions covering the paintings. So, in order to achieve a complete cleaning of the wall paintings, it is needed a double approach that includes both the removal of the resin as the elimination of the concretion layer.

Paraloid® removal tests: solvent selection and methodology

The resin was identified as Paraloid® B72 with FTIR analysis (Figure 4). Therefore, following the common tendency for resin removal, the first tests were based on the use of free solvents (Masschelein-Kleiner, 2004). Paraloid® still showed a high capacity to re-dissolve using common solvents into which it is soluble. Tests were carried out using xylene, acetone, benzyl alcohol and different mixtures, obtaining very similar results at the naked eye and raking light (Figure 5). Principal differences were seen in the time needed for the removal but not to the final result. For instance, benzyl alcohol was very slow compared to acetone or xylene but it could obtain the same removal degree.

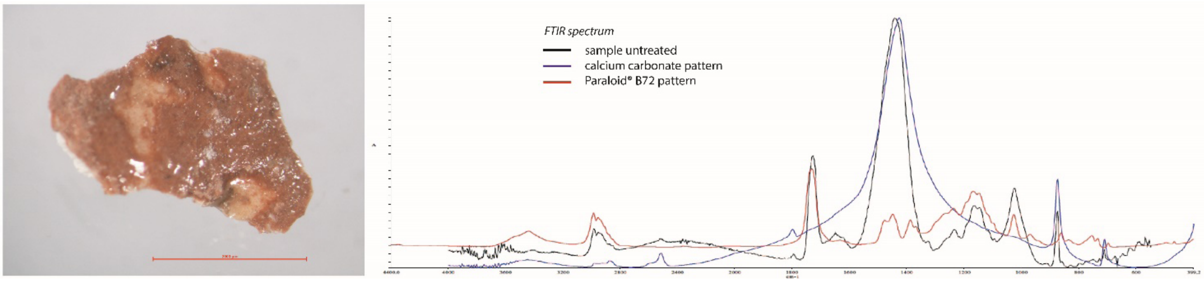

Figure 4 – With the optical microscopy an important presence of a transparent coating on the surface of one sample can be seen. With FTIR analysis with Perkin Elmer Spotlight 300 equipment the coating has been identified as Paraloid® B72. Author: Centre for the Restoration of Cultural Assets of Catalonia (CRBMC).

Figure 5 – Photograph with raking light of some solubility tests of the coating with free solvents. From left to right acetone; acetone and ethanol 1:1; isooctane and acetone 3:7; benzyl alcohol; acetone and benzyl alcohol 1:1 and xylene. All the mixtures are able to solubilize the acrylic resin. Author: Aleix Barberà.

It was also decided to test a considered green solvent according to provider, Rhodiasolv® IRIS. Results were apparently the same as traditional solvents at macroscopic observation, and the solubilization took place really fast and matching the efficiency of acetone or xylene.

To consider this solvent as "green" it must combine good health, safety and environmental characteristics (Capello, Fischer & Hungerbühler, 2007, p. 927).

Rhodiasolv® IRIS is a dibasic ester solvent, based on the short-branched alkyl chain dicarboxylic acids. Specifically, it is a dimethyl 2-methyl glutarate (INCI name: Dimethyl Methylglutarate, CAS number: 14035-94-0), presented by Solvay as a green alternative to many chlorinated and a high volatile organic compound containing solvents. In fact, according to the Hansen solvency parameters, Rhodiasolv® IRIS ($\delta H = 5.0$; $\delta P = 8{,}7$) is very close to the solubility of acetone, N-Methyl-2-pyrrolidone, ethyl acetate and Methyl ethyl ketone, among others (Rhodia, 2009a, p. 5).

According to the tests carried out by Rhodia Novecare, Rhodiasolv® IRIS is a powerful solvent able to dissolve a wide range of resins and polymeric substrates like acrylic, epoxy, polyester, polyurethane, vinyl and alkyd resins (Rhodia, 2009b). On the other hand, it is a powerful solvent to eliminate industrial grease (Rhodia, 2009a, p. 8, 9, 19), and has also been tested with very good results as a paint stripper and in graffiti removal and laundry stains (Rhodia, 2009a, p. 11-16).

To consider this solvent as a green alternative we can take into account that it has non-VOC and nontoxic emissions according to the European directive [1999/13/EC] with Vapor pressure of 6.1 pascals at 20°C and an Evaporation Rate (BuAc=1 at 25°C) of 0.006. It also meets the United States consumer products LVP-VOC exemption criteria established by California Air Resources Board for consumer and institutional products and the federal VOC exemption criteria for consumer products by the EPA 40 CFR 59.203(f)1 (Rhodia, 2011). It's also a non-flammable solvent with Flash Point at 98°C.

According to toxicological data, Rhodiasolv® IRIS has been declared non-carcinogenic and non-irritant or sensitizer. It has no mutagenicity (OECD 471), no skin irritation (OECD 404), no skin sensitization (OECD 429) and no eye irritation (OECD 405). As drawbacks it presents acute oral toxicity, LD50 > 2000 mg/kg (OECD 425) and acute dermal toxicity, LD50 > 2000 mg/kg (OECD 402). And according to ecotoxicological data, this solvent is readily biodegradable, in more than 60% according to OECD 310 (Rhodia, 2010). These values lead us to consider this solvent a safer choice for the environment and the conservators.

Taking into account these characteristics, Rhodiasolv® IRIS is a much safer option than traditional solvents used for the solubilization of Paraloid, such as xylene, and in comparison, with other solvents such as acetone or benzyl alcohol it has a lower evaporation rate and it is nonflammable. Therefore, this solvent could have some advantages for conservation practice because the solvency power for acrylic resins is really high compared to benzyl alcohol and it's also immiscible in water, which is a key factor to emulsion formation.

At the macroscopic level, the removal of Paraloid® using free solvents was supposed to be an effective method. However, the observation of samples with a digital microscope revealed that, despite removing most of the resin from the surface, there were still little residues build up in pores and cracks (Figure 6). The high risk of not only finding traces of resin on the surface but also resin penetrating into the wall painting was corroborated with FTIR analysis (Figure 7).

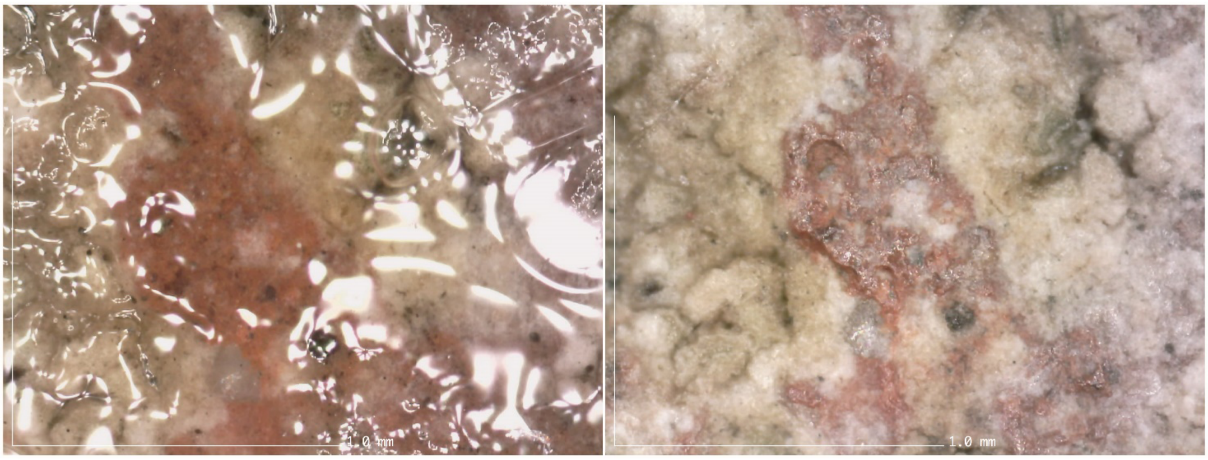

Figure 6 – Images with a digital microscope at 215x before (left) and after (right) removal tests with acetone. Some shiny residues can be seen in pores. Author: Aleix Barberà Giné.

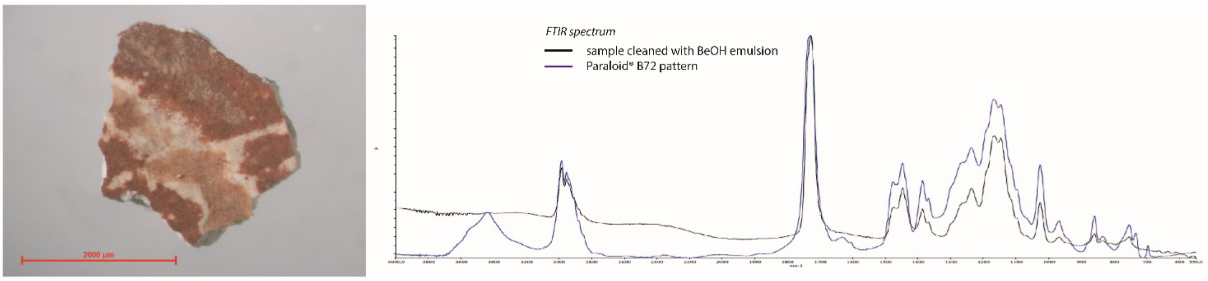

Figure 7 – A sample of an area cleaned with free acetone was analyzed with FTIR with Perkin Elmer Spotlight 300 equipment and great amounts of Paraloid® B72 were identified. Author: CRBMC.

A possible alternative to the use of free solvents is to apply them in a gel form. With this methodology, we can diminish the solvent evaporation ratio, the total amount of solvent required for the process of the penetration into the porous substrate. Unfortunately, traditional solvent surfactant gels (Stulik et al., 2004) don't work on ionic surfaces, and considering the enormous presence of soluble salts in the paintings had to be discarded. In addition, for the rinse of the gels free solvents are also required, so they don't really avoid the risk of resin re-penetrating the wall paintings, although they represent a greener option (Wolbers, 2017).

Apart from using more respectful solvents for health and the environment, it is preferable to first reduce the volume of solvents and then use greener solvents (Jessop, 2011). In order to avoid the use of free solvents and to try to minimize the resin residues on the paintings, we considered the use of emulsions. Traditional emulsions or microemulsions (Carretti, Dei & Baglioni, 2003; Baglioni, Chelazzi & Giorgi, 2015) with an aqueous phase and an oil phase emulsified with a surfactant and/or a co-surfactant were discarded (Brajer et al., 2014). We wanted to prevent the use of any surfactants which could remain as a residue on the porous matrix of the paintings. Instead, xanthan gum gelled polymeric emulsions were tested according to previous successful experiences for the removal of Paraloid® on wall paintings (Barberà & Marín, 2017).

The use of a free surfactant gelled emulsion with xanthan gum offers many advantages for resin removal on a porous substrate.

First of all, in an oil in water emulsion the solvent is not in a free form but encapsulated into the aqueous phase (Moreno, 2013; Wolbers, 2012). When the solvent solubilizes the resin, assisted with a slight mechanical action, the polymer is detached from the surface of the paintings and it's incorporated into the emulsion minimizing the risk of its re-penetration into the porous matrix. None of the resin which is solubilized may come apart from the emulsion and therefore it's a safer solution for the work of art in terms of leaving undesired residues.

Furthermore, with free-surfactant emulsions with xanthan gum there's no risk of leaving residues from our cleaning products, because the rinse of the complex can be carried out simply with an aqueous solution, so, with this methodology, there is no need of using free solvents.

On the other hand, in an emulsion, we can use a very low amount of solvent and at the same time, it shows a minimum evaporation ratio into the environment becoming a less toxic and a more efficient methodology. The same result can be obtained with less product.

Not only the solvent phase can be useful in an emulsion, but we can also look at the aqueous phase to adjust pH with a buffer to be in a safer range for the carbonated surface of the wall paintings, control conductivity or even add other chemicals such us chelators or enzymes to help us reach our goal. With this methodology, we are including another factor to the equation that can clean the wall paintings superficial dirt or concretions at the same time. Therefore, we can solve in a one-step process both the removal of the acrylic resin and the concretions layer.

And finally, in a gel emulsion form, we can more precisely control the application of our cleaning product (Figure 8). Gels help us to control time applications; to avoid diffusion into the porous matrix or to facilitate application with a soft brush.

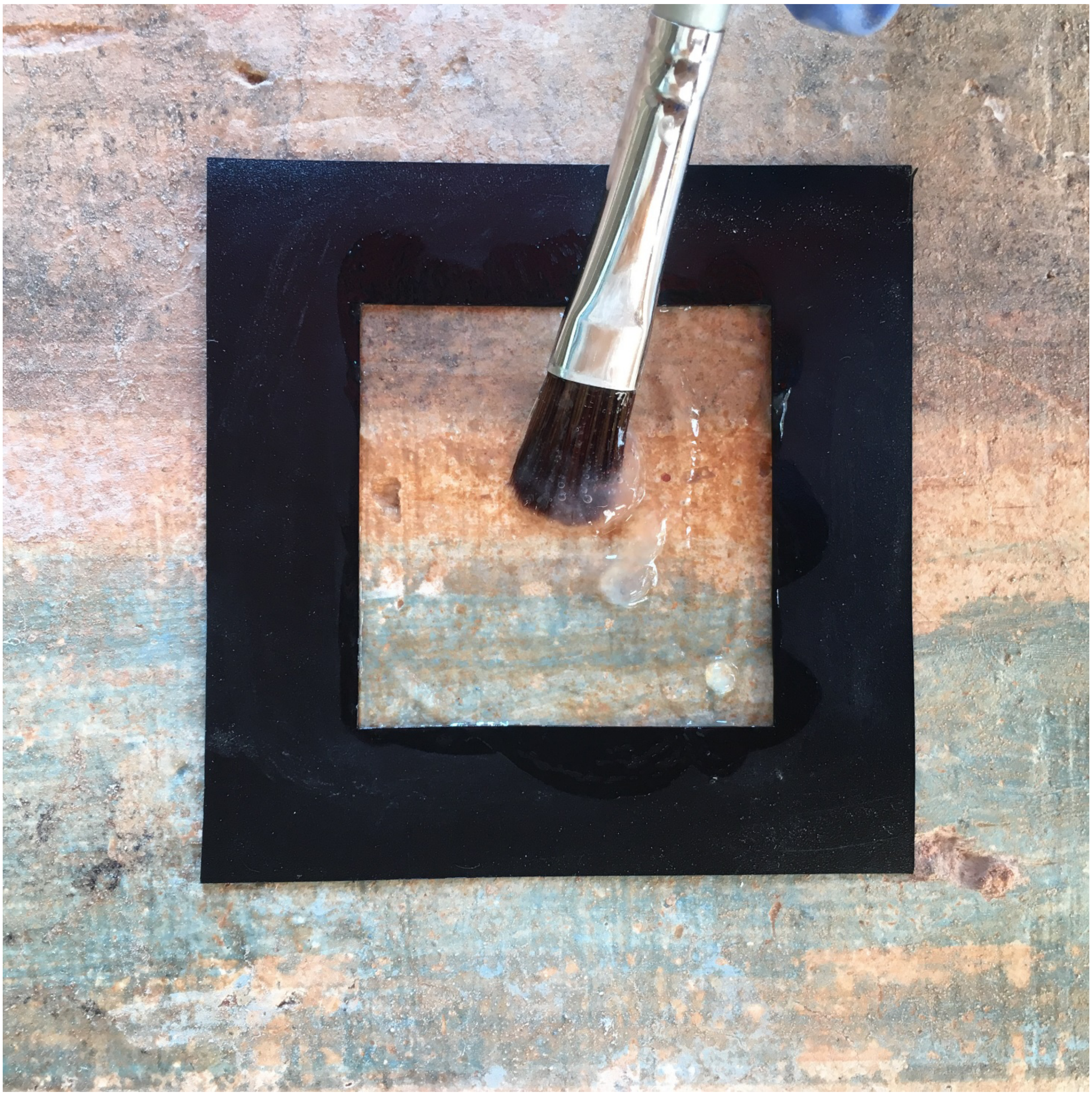

Figure 8 – Application of a polymeric xanthan gum emulsion by brush. Author: Aleix Barberà.

All these considerations make the free surfactant emulsion with a tailored aqueous phase the best choice for resin removal according to the green chemistry principles (Anastas & Warner, 1998).

Nontoxic formulation for Paraloid® removal

Taking into account that in an oil in water emulsion we are working with aqueous systems, we evaluated the pH and conductivity values of the surface based on absorption tests with agarose gel at 5% in deionized water (Wolbers, 2018). Results were obtained with 20-minutes applications and measured with Horiba® LAQUAtwin PH11 pH meter and Horiba®

LAQUAtwin EC11 conductivity meter, getting a pH ranging from 7.5 to 8, and a conductivity ranging from 150 to 12.000 μS/cm^2. The high conductivity variations were explained for the huge amount of soluble salts in the paintings, one of the most important degradations factors of the ensemble (Figure 9).

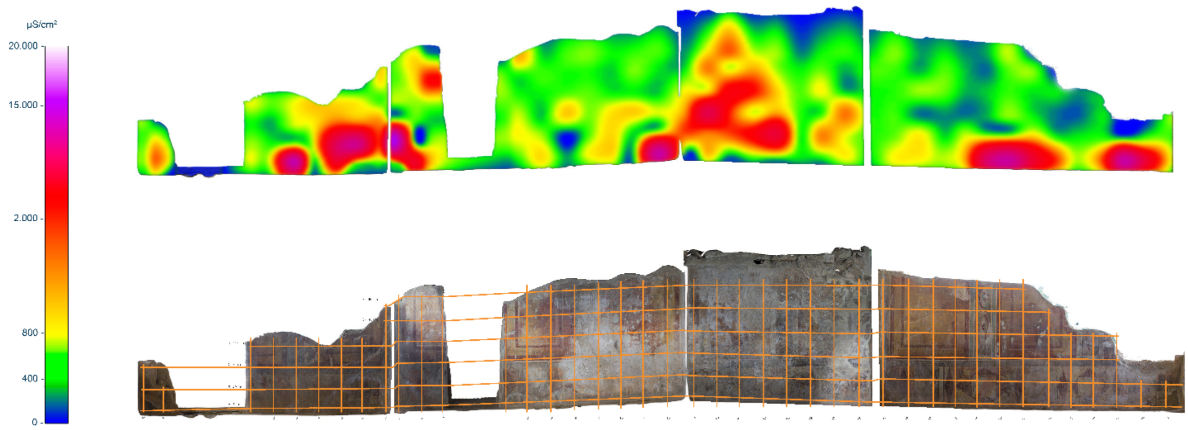

Figure 9 – Soluble salt content in the wall paintings expressed with statistical color maps on ortho-photographs in one of the rooms. Values obtained with agarose gels extraction in order to study salt distribution patterns on the paintings. Author: Jaime Salguero.

Considering these results, we designed an aqueous phase based on a sodium borate buffer solution at pH 8.5 with the addition of DTPA in conductivity of around 3.000 μS/cm^2. This solution, matching the sensitivity of a fresco surface, maybe also able to slightly remove carbonate concretions without affecting the pictorial surface. The solution was then gelled with 2 wt% Vanzan® NF xanthan gum.

For the solvent phase it is required an immiscible solvent in water able to solubilize Paraloid®. In this sense, acetone and mixtures of acetone and ethanol can't form emulsions in water and were discarded. Different solvents and mixtures have proven able to remove the resin in an emulsion form with less than a 10 wt% solvent phase, such as xylene, benzyl alcohol, or a 1:1 mixture of acetone and benzyl alcohol (Figure 10). The solvent phase is added into the aqueous gelled phase and stirred with an agitator. The application of the product can be carried out with a soft brush and needs a slight mechanical action and a short working time. The rinse is carried out first with dry cotton and then with a sodium borate buffer solution at pH 8.5.

Figure 10 – An area cleaned with a xanthan gum emulsion with a 10 wt% solvent phase consisting of a 1:1 mixture of acetone and benzyl alcohol. Author: Aleix Barberà.

We consider that in these working conditions the general toxicity of the cleaning product has clearly diminished in relation with the use of free solvents or solvent gels. The total amount of solvent used is around 10 wt% of the cleaning product but it could be also diminished. And we have to take into account that the solvent presence is constrained in an emulsion form and the rinse is always carried out with aqueous methods. The removal capacity of the resin is evident at macroscopic and microscopic levels (Figure 11). And in terms of Paraloid® residues, the results are improved with the emulsion cleaning than with free solvents according to FTIR analysis (Figure 12).

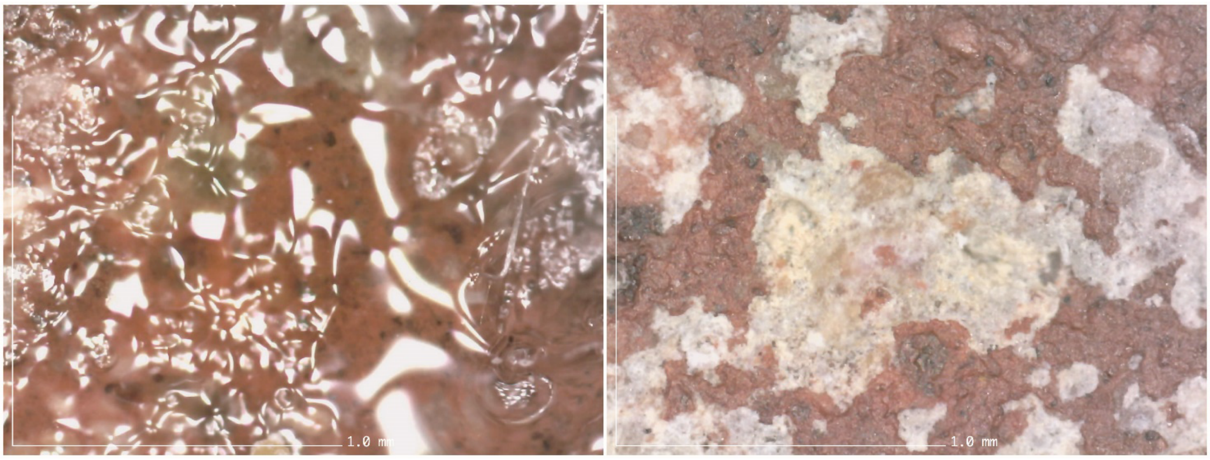

Figure 11 – Images with a digital microscope at 215x before (left) and after (right) removal tests with the xanthan gum emulsion with a 10 wt% Rhodiasolv® Iris. There are no resin residues visible. Author: Aleix Barberà Giné.

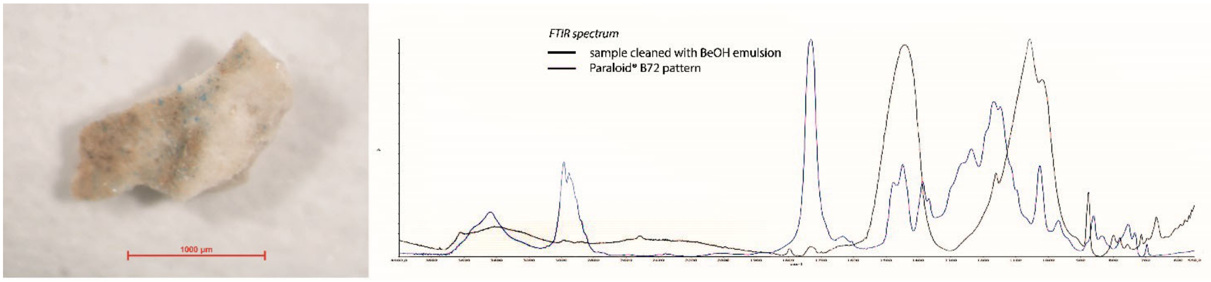

Figure 12 – A sample of an area cleaned with a xanthan gum emulsion with a 10 wt% solvent phase consisting of a 1:1 acetone and benzyl alcohol mixture was analyzed with FTIR with Perkin Elmer Spotlight 300 equipment. Although it could still be found Paraloid® B72, the values were lower than the cleaning with free solvents. Author: CRBMC.

This cleaning system for Paraloid removal could consider any type of solvent immiscible in water. First tests were carried out with solvents with low toxicity, such as benzyl alcohol and acetone mixtures, but also Rhodiasolv® Iris was tested. This solvent can form an emulsion with the former aqueous phase gelled with xanthan gum. Although it has some solubility in water in a ratio of 2.5% (Rhodia, 2009b) it doesn't really affect at the emulsion formation and the soluble fraction in water is also incorporated in the gel. This final cleaning product has very low toxicity according to actual legislation and is perfectly able to remove an acrylic resin coating (Figure 13).

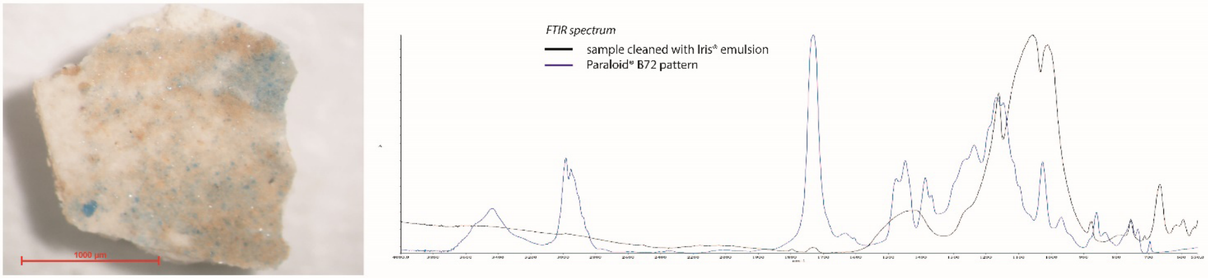

Figure 13 – A sample of an area cleaned with a xanthan gum emulsion with a 10 wt% solvent phase Rhodiasolv® IRIS was analyzed with FTIR with Perkin Elmer Spotlight 300 equipment. Although it could still be found Paraloid® B72, the values were lower than the cleaning with free solvents. Author: CRBMC.

Conclusions

Although all the systems tested allowed, with some variations, to quite efficiently remove the Paraloid-B72 coating from the wall paintings, we have concluded that the use of a xanthan gum emulsion with just a 10 wt% of solvent offered the best results in terms of efficiency, respect of the work of art and safety for the environment and professionals.

The degree of resin removal with the free surfactant emulsion was really effective, matching or even improving the traditional systems according to digital microscope observation and FTIR analysis. Moreover, the fact of working with no free solvents prevented the possibility of re-dissolved resin penetrating into the porous system. In addition, the use of really small amounts of solvent, confined in an emulsion, reduced the toxicity of the process, both for the environment and for conservators.

Furthermore, this working methodology allows the specialists to properly select the solvent according to safety, efficiency and environmentally criteria. Emulsions with mixtures of benzyl alcohol and acetone or new green solvents available like Rhodiasolv® IRIS have proven perfectly able to remove Paraloid® from the wall paintings surface.

In addition, when we are using an emulsion, we are combining the solvent capacity to remove the resin with the chemistry of water which we can use to achieve other goals, such as the surface cleaning of the paintings or the removal of other layers. The efficiency of the process is then completely improved with this approach.

Finally, the definition of the cleaning system based on the preliminary analysis of the surface, the use of a buffered solution with pH and conductivity control in ranges to protect the fresco painting is a cleaning methodology that guarantees the integrity of the work of art.

In conclusion, the use of free-surfactant emulsions is a more efficient system for the removal of resin layers on wall paintings, because it can drastically minimize the use of solvents and it avoids the repenetration of the resin inside the pore matrix. In addition, it allows us to remove the resin and the layers of concretion or superficial dirt in a one-step process (Figure 14).

Figure 14 – An area cleaned with a xanthan gum emulsion with a 10 wt% solvent phase of Rhodiasolv® Iris. Author: Aleix Barberà.

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