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Cleaning plaster surfaces with agar-agar gels: evaluation of the technique

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Abstract: Cleaning plaster surfaces represent a challenge for conservators. It should only be performed following fully tested methods that guarantee the conservation of such fragile material. The goal of this work is to establish a suitable cleaning method for this type of artwork from the tested concentrations and time of applications, using agar gels on plaster supports.

Morphological, porosity and weight variations have been studied. Confocal and stereomicroscopy have been used as analytical techniques, as well as the measurement of water vapor permeability and weight have been taken on the samples.

Key words: agar, gypsum, plaster, gels, cleaning.

La limpieza de superficies de yeso-escayola con geles de agar-agar: evaluación de la técnica

Resumen: La limpieza segura y eficiente de las superficies de yeso constituye un reto y una responsabilidad para el conservador-restaurador, y debe llevarse a cabo siguiendo métodos testados que garanticen su correcta conservación. La intención de este trabajo es determinar, a partir de las concentraciones y tiempos de aplicación ensayados, cuáles serían los parámetros óptimos para la ejecución de una limpieza eficaz e inocua empleando geles de agar-agar sobre soportes de yeso.

Se han comprobado las posibles variaciones morfológicas de la superficie, las variaciones de la porosidad y del peso, así como la presencia de residuos, para lo cual se ha empleado la microscopía confocal, microscopía binocular, la medida de la permeabilidad al vapor de agua y la medida del peso de las muestras.

Palabras clave: agar, yeso, escayola, geles, limpieza.

A limpeza de superfícies de gesso com géis de agar-agar; avaliação da técnica

Resumo: A limpeza segura e eficiente das superficies de gesso constitui um desafio e uma responsabilidade para o conservador-restaurador e deve ser levada a cabo seguindo métodos testados que garantam a sua correcta conservação. O propósito deste trabalho é determinar quais serão os parámetros óptimos para a execução de uma limpeza eficaz e inócua sobre suportes de gesso, a partir das concentrações e tempos de aplicação de géis de agar-agar.

Verificaram-se as possiveis variações morfológicas da superficie, as variações da porosidade e do peso, assim como a presença de resíduos, tendo sido empregue a microscopia confocal, a microscopia binocular, a medida da permeabilidade do vapor de agua e a medida do peso das amostras.

Palavras-chave: agar-agar; gesso, géis, limpeza.

Introduction

Cleaning plaster surfaces is challenging. It should only be performed following fully tested methods that guarantee the conservation of such fragile material. Establishing a suitable cleaning method for this type of artworks is indeed very challenging to say the least.

Existing cleaning methods have been problematic when used on plaster. Considering the physicochemical properties of this type of surfaces, and a cleaning procedure pursuing selectivity and respect to the integrity of the artwork, aqueous solutions are usually not a viable option because plaster is partially soluble in water, so any moisture accompanied by the slightest removal action can wear away the surface causing irreversible alteration of its original qualities. Besides water solubility, plaster surfaces are extremely fragile. They can be easily eroded by the action of a cotton swab and do not usually react well with the direct contact of poultices, so cleaning options are very limited.

The research conducted by Dr. Cremonesi and his team have served as a starting point for this study. They have worked on the use of agar in conservation and their experiences are published in two "Cuadernos" by Cesmar7 (Centro per lo Studio dei materiali per il restauro) (Cremonesi *et al.* 2007, 2008). In this article agar is presented, as an alternative cleaning method. Agar is the dry extract obtained from several kinds of rodophicea algae, with a great gelling power that when used in its gel form is capable of supplying water in an extremely controlled way, allowing aqueous cleaning of porous surfaces, such as plaster, in an efficient and innovative way, while conserving the intrinsic characteristics of the substrate. Their first tests were run using rigid agar gels but soon they started to use agar in a viscous liquid phase so they could be applied to tri-dimensional objects. The results of cleaning on plaster objects were very promising, providing appropriate levels of cleanliness, while treated surfaces seemed to show no signs erosion when observed with optical microscopy, or any other sign of alteration.

However, it was considered necessary to conduct a thorough study of how these surfaces could be affected by contact with these aqueous gels, considering the frailness and water solubility of the material.

In this research we have systematically studied plaster surfaces, in terms of porosity and morphology variations, and presence of residues, that have been treated with agar gels at different concentrations and application times by observation with confocal microscopy (topographies and profiles, with micron resolution), and performing water vapor permeability test, before and after each treatment. The study was designed so that variations in weight of the treated samples. Observations were made under stereoscopic microscope (x40, x25, x16) under white and ultraviolet light, to be able to evaluate this cleaning technique with a scientific approach.

Objectives

The intention of this paper is to determine, based on the concentrations and application times tested, what would be the optimal parameters of a safe and efficient cleaning procedure using agar-agar gels on gypsum, specifically in terms of:

- Morphological changes on the surface.
- Porosity changes.
- Presence of residues.

To establish damage threshold parameters concerning:

- Concentration.
- Application time.

Methodology

The tests should be designed to assess and interpret the results obtained away from subjectivity. Parameters that may vary like the type of dirt, particular surface characteristics and operator skills should be avoided. This means that the samples for this research were made following the same procedure, using the same plaster material and plaster/water ratio, and were left unstained, so they would all have similar surface characteristics. All the tests were performed on unsoiled plaster taking under account cleaning efficiency was not to be assessed in this study.

Two types of samples have been used: samples specifically made for this study and some real fragments, to better illustrate the results.

For the sample specifically made for this study, a total of 21 samples were selected. These samples were plaster discs of 6 cm in diameter and a thickness between 10 and 13 mm. They were made using two similar water-solid mixes that once hardened needed to be dry before testing started. They were left to air dry for two weeks and then placed into a drying chamber with silica gel for two months to eliminate the maximum amount of water possible. A drying stove was not used to speed up the drying process due to the low temperature of calcination of calcium sulphate (around 100 ° C) to avoid the risk of molecular dehydration.

Alamo 50 gypsum[1] was used for the making. This gypsum is considered "plaster quality". This material was selected because it is the one currently used by the Casting Workshop of the Royal Academy of Fine Arts of San Fernando to make their casts.

To better illustrate how the cleaning procedure works, some tests have been conducted on real plaster cast objects. They are fragments donated by Professor Pedro Terrón from the School of Fine Arts modeling workshop at Universidad Complutense of Madrid. They all showed abundant surface pollution and clay deposits.

For the cleaning tests, we worked with two concentrations of agar gels: 2% and 4% (w/v) in distilled water. The gels were made using *Agar-agar* (UPS) PRS-CODEX 141792.1209 from the commercial firm Panreac, prepared with 4 g of powder in 196 ml distilled water (2 g in 98 ml x 2 for the 2% gel) and analogously using 8 grams of dry powder in 192 milliliters of distilled water (4 g in 96 ml x 2, for the 4% gel). The water was then heated up to 85-90°C. At that temperature agar will dissolve in water, so the powder is poured in the liquid and allowed to dissolve for about 5 minutes, stirring with a glass rod. This mixture is left to cool and reheated a second time at 90 ° C to ensure the dissolution has been complete. It is then poured into a beaker and allowed to cool until a hard gel is formed.

Agar gels can be used in a broad temperature range depending on the degree of gelling required, so this will allow for gels to be applied as a viscous liquid using a brush. The higher the temperature, the more fluid the mixture will be, and as temperature decreases there is an increase in the viscosity until the mixture becomes rigid around 35 ºC. To make a successful application by brush the ideal temperature is about 40-45 °C (measured with a thermometer.) To the touch the mixture is perceived as lukewarm. At this temperature it could be considered safe to use on most materials that make up the artwork. After waiting for the application time established according to the tests, the gels are then removed.

On the test samples 2% and 4% agar gels were applied with three different application times: 1T = 2 minutes, 2T = 20 minutes, and 3T = 24 hours(totally dry). Each treatment was applied on groups

of three different samples. For the real fragments a 2% concentration was always used, with application times of 2, 5, 10, 20, 30 minutes and 24 hours (totally dry). Also a treatment based on two successive applications of 2 minutes was tested for the 2 minutes application.

The disc samples are identified by the concentration of agar gel used and application time in each case. For example "sample 2% T1" would indicate that 2% agar gel has been applied, and it was left on the surface for 2 minutes. Table I shows the different treatment conditions tested on the plaster disc samples.

Table I. Different treatment conditions tested on the plaster disc samples.

Sample Code	**Treatment description (applied on three samples each)**
2% 1T	Agar concentration (w/v) 2% applied during 2 minutes.
2% 2T	Agar concentration 2% applied during 20 minutes.
2% 3T	Agar concentration 2% applied during 24 hrs or totally dry.
4% 1T	Agar concentration 4% applied during 2 minutes
4% 2T	Agar concentration 4% applied during 20 minutes
4% 3T	Agar concentration 4% applied during 24 hrs or totally dry

The treatments effects have been characterized using different observation techniques and test methods. Before any treatment was applied, the material under study has been characterized. The observation techniques used were optical confocal microscopy, the stereomicroscope under white light and ultraviolet light. The water vapor permeability and weight of samples was also measured (both tests reflect permeability and weight variations undergone by the samples after each treatment).

The Confocal Scanning Microscopy (CSM) provides roughness measurements (Ra), height variations allows for the observation of microcracking of the surface of the samples. This microscopy system performs three-dimensional reconstructions of the surface topography with a vertical resolution of hundreds of nanometers and a lateral resolution varies depending on the lens used (x10_0, 935 μm; x20_0, 623 μm; x50_0, 350 μm). Allowing to fully document the surface roughness through images, profiling and numerical values. Ra described as the arithmetic mean of the absolute departures of the roughness profile from the mean line was used in this study, since this value is a universally accepted surface roughness value and appropriate for evaluating the effect of factors on the surface quality (Yavuz *et al.* 2011). The equipment used was Confocal Imaging Profiles Sensofar PL N 2300, with PL N Version 2.1 software based on Windows 2000/XP. The lenses used were x10 for data evaluation and x20 for detail image in figure I.

And initial measurement was taken on one sample from each treatment (a total of six measurements because six different treatments conditions were being tested). After each treatment a second roughness measurement was taken on the surface of the same sample, selecting a representative surface area (4.4 mm^2) in every case. Also in treatments where large variations occur additional measurements were taken to better record them.

The water vapor permeability test was performed using as reference standard from the Italian legislation, NORMAL 21-85. The test was performed on groups of three samples, before and after treatment. The conditions during the test were of an average temperature of 20ºC and chamber gradient (wet-dry) Relative Humidity (RH) of 59% (RH 90%-RH 31%). We applied a thickness correction factor in all samples, and we express permeability values in g/(m x 24h).

Permeability is the ability of a material to transmit through its mass liquids, gases or mixtures of both, under the action of a pressure gradient (Gisbert 2002: 179). This property is related to the porosity and the connectivity of voids (Esbert 1993). Permeability is a characteristic of each material and varies in the untreated material in relation to the treated one. It gives an idea of the extent to which the pores have been clogged due to the presence of residues of different materials or treatments (Gomez Terreros 2000: 33-51), due to the modification of the material surface, variations in roughness, and may also cause changes in the permeability thereof. This data is important to define the ability of the material to "breathe", how easily the moisture retained inside flows to the outside.

The test specimens were weighed before and after the application of the different treatments, with an accuracy of 10^{-4} grams, to assess the loss or gain in weight of the samples after each type of intervention. A scale Mettler Toledo 48206/TACT was used for such measurements.

We have also observed that the samples with long-wave (365 nm; 6W) and shortwave (254 nm, 6W) ultraviolet light, both directly and through stereomicroscope with 16, 25 and 40 magnifiers, with the intention, given the different response given by the support material (gypsum) and agar under this illumination, to determine the presence of residues after the treatments. The quantification of residues has been calculated on the UV visible images of the complete surface of the samples managed with a digital imaging treatment software that allows taking surface area measurements to estimate the presence of residues. These remains appear as very thin films in the peripheral areas. They can be removed using a scalpel.

Discussion of results

The initial appearance of the sample surfaces prior to any treatment is shown in Figure I. The surface is matte, despite being quite polished, and you can see lines (polishing grit) and pores (setting bubbles). For these surfaces the average roughness (Ra) ranging between 1- 1.5 µm.

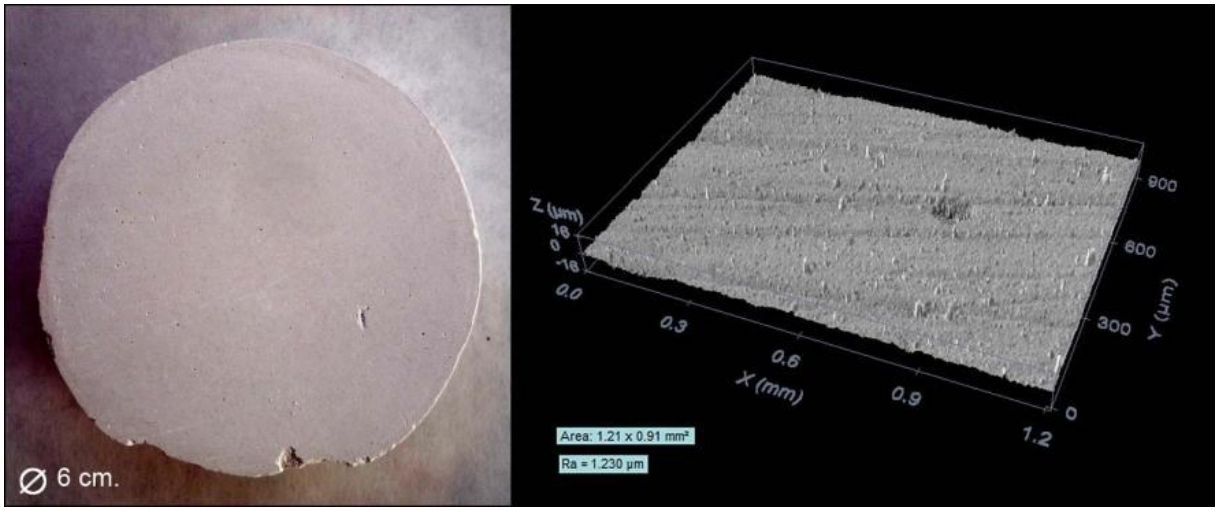

Figure I: Left: Plaster surface. Right: confocal detail topography (x 20) of the sample on the left, in which a pore, polishing scratches and dust particles can be seen on the surface.

For the water vapor permeability the initial average value was calculated for the plaster samples: 1.8 g / (x 24hrs) with a coefficient of variation of approximately 8%.

Figure II: Left: Surface appearance of a sample 2% 1T. Right: Surface appearance of a sample 2% 2T.

Figure II shows the surface of the samples treated with agar gel at 2% concentration left for 2 minutes on the surface (left) and 20 minutes (right). Such treatments have caused no changes in the surface roughness of the samples, detectable by the measurement method used. Variations in permeability values of the samples tested after both treatments are less than 2%, not finding a clear trend of variation. Due to the negligible variation (and the lack of trend) we can consider that the changes in permeability caused by these treatments are not significant. Samples treated with 2% agar for 2 minutes, present no residues or loss of material on the surface. In the case of the 20 minutes application, the material loss is less than 0.01 mg/cm^2 (may be considered negligible) and residues present on the specimen waste samples cover between 0-4% of the material surface.

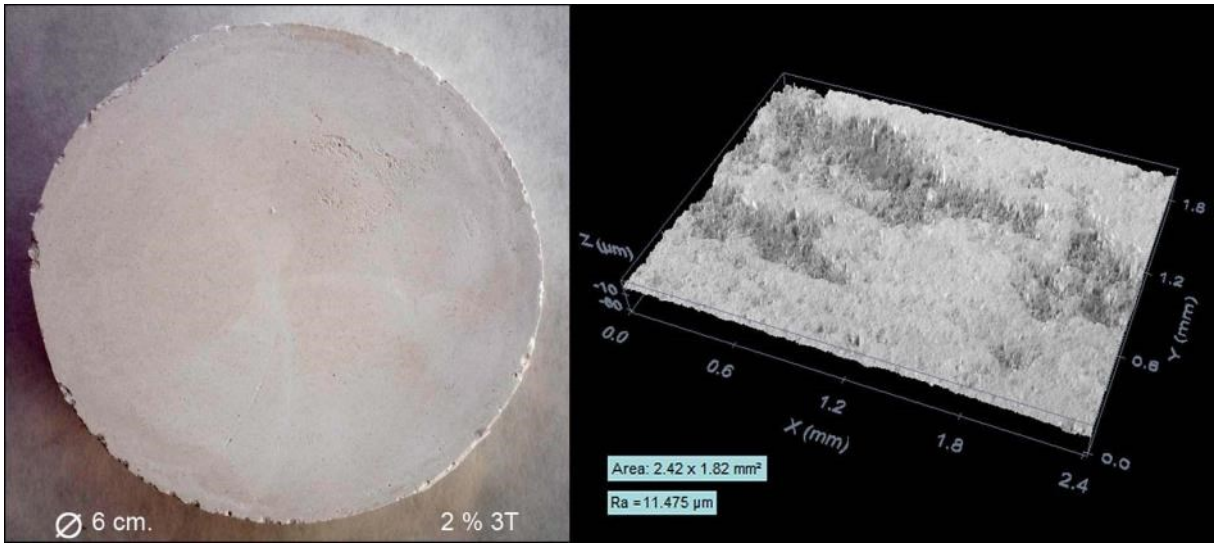

Figure III: Left: surface appearance of sample 2% 3T. Right: confocal topography (x 10; 4.4 mm^2) of the upper central area of the sample to the left where material has been lost.

In Figure III we can see the surface of a sample treated with 2% agar gel concentration left to dry completely. Overall, the roughness of the treated samples under these conditions is very similar to the initial values, but we can observe local areas where there has been an increase in roughness by removal of material. In these areas the roughness values can reach 11.5 µm [Figure III, right image]. Concerning permeability values after treatment, the variations are less than 1% and do not show a clear trend. The weight losses are less than 0.1 mg/cm^2 (average value could be considered small). The presence of residues following the application is estimated between 4-6% of the sample surface, of all treatments tested this is the one leaving more residues behind. As in previous cases the residues appear as thin films on the edges, and are easily removable.

So far we have not discussed the relevance of residue of such a product like agar remaining on the surface. The evolution of these residues is closely linked to atmospheric moisture. In high humidity conditions the evolution of these films could be extremely harmful as they will surely develop heavy biological colonization. In Figure IV the appearance of such biological colonization of the agar gel films in treatment 2% 1T is shown, thirty days after being removed from the surface a plaster sample, kept on filter paper inside a plastic bag.

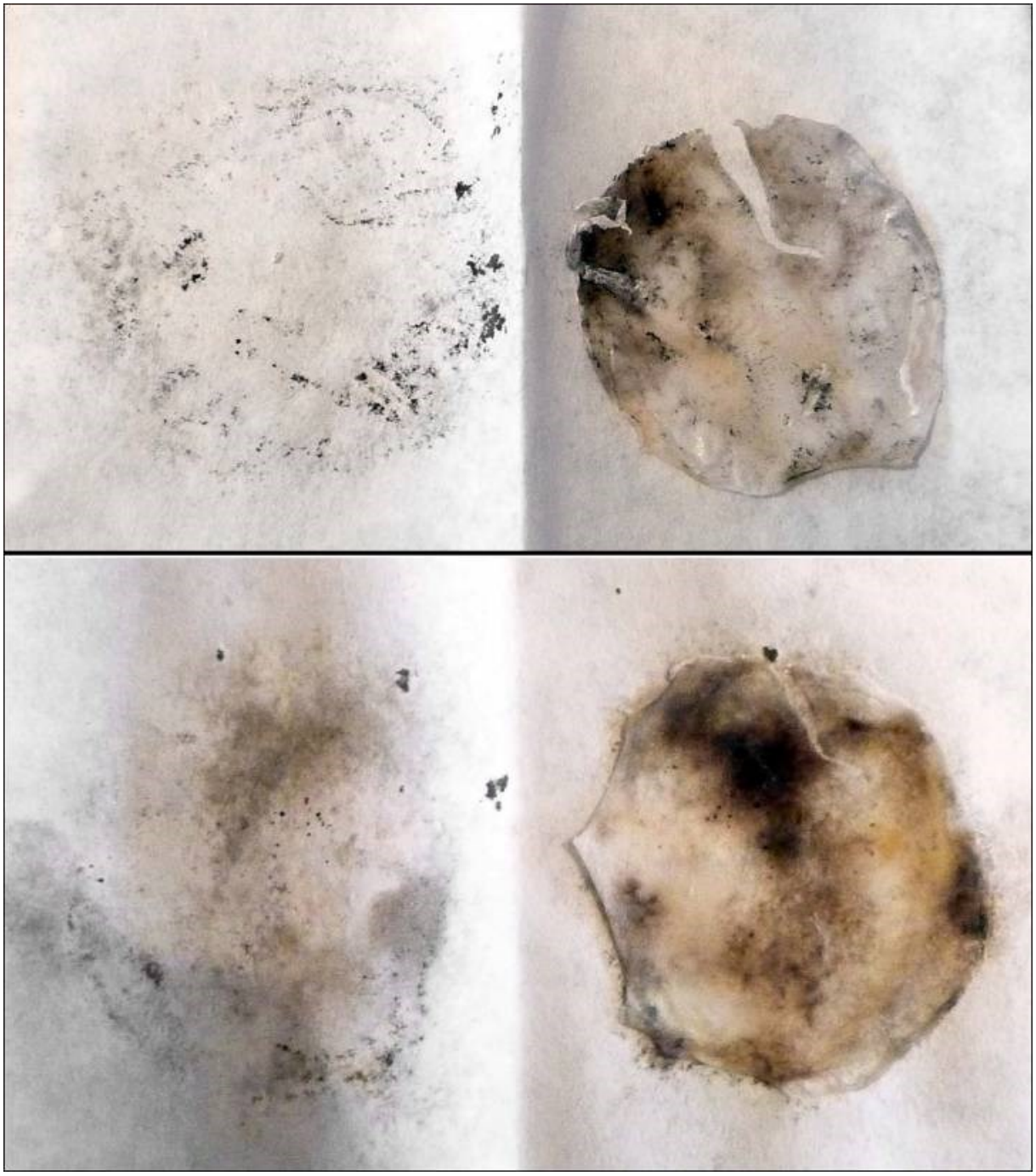

Figure IV: Left top and bottom: filter paper with traces of mold. Right top and bottom: agar film 2% 1T treatment after thirty days of application and removal.

On Figure V we can see a sample treated with 2% 3T under long-wave ultraviolet light. This is the sample on which confocal images were taken and roughness measured. The agar film response can be clearly seen in pale yellow colour, compared to the pinkish tone of plaster.

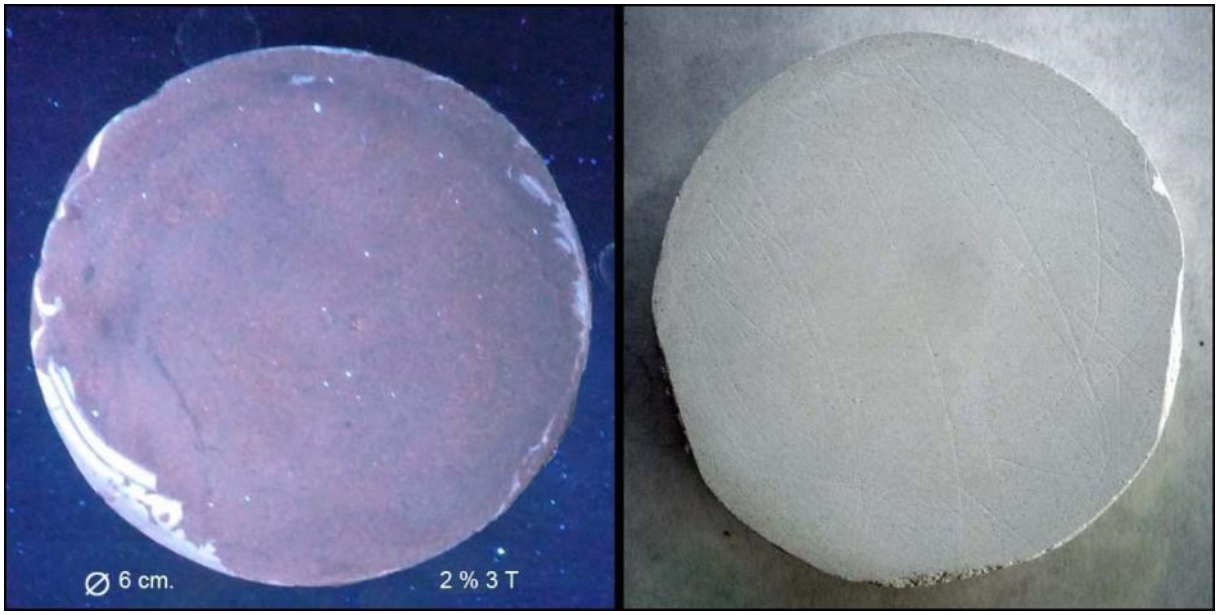

Figure V: Left: image of the surface of a sample treated with 2%3T, under long wave ultraviolet light. Right: the surface appearance of a sample treated with 4%1T.

In treatments with 4% 1T roughness changes have not been detected and variations in the permeability of the samples are around 1%, with no clear trend. The loss in weight is less than 0.1 mg/cm^2, and the remnants of the gel are between 0 and 2% of the sample surface, with a similar location to the above cases.

Treatment with 4% 2T [Fig. VI] has caused an increase in surface roughness of the sample. In general roughness values are around 3 microns with peak values reaching 7 μm (center-top of the sample).

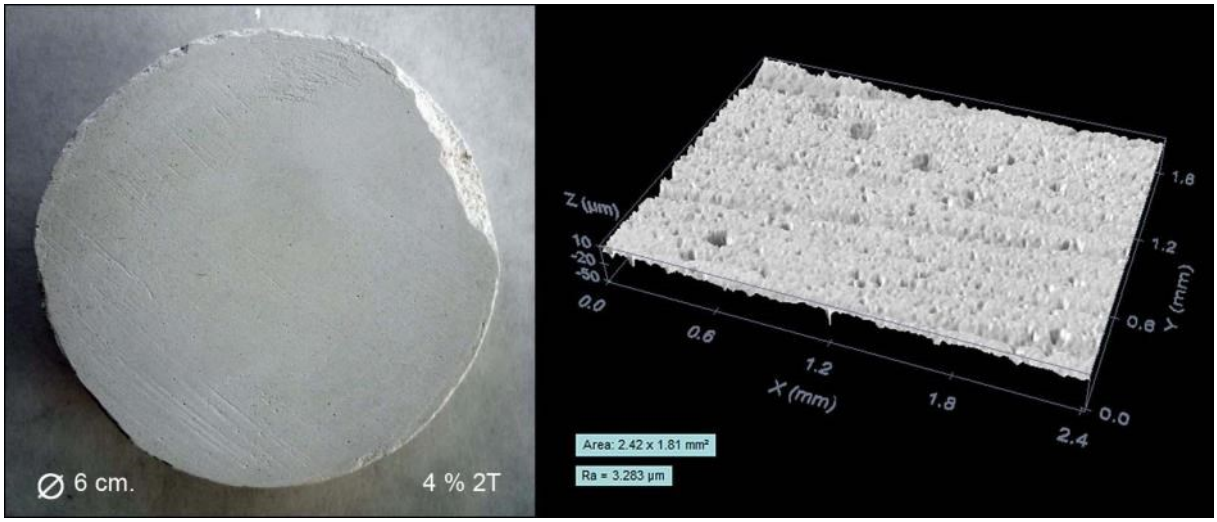

Figure VI: Left: surface appearance of sample 4% 2T after treatment. Right: confocal topography (x 10; 4.4 mm^2) of the same sample surface.

The overall increase in roughness is due to the loss of small amounts of material that occurs preferably on preexisting grooves and bubbles, making them bigger. Rougher areas correspond to where there has been further loss of material. Even so, the loss of material per unit area remains small (less than 0.1 mg/cm^2). Regarding permeability, with this treatment variations close to 8% have occurred, having a clear negative trend. This decrease in the permeability begins to be significant. The surface residues location is similar to the previous cases and its extension is 2-4% of the sample surface.

Figure VII: Left: surface appearance of sample 4%3T after treatment. Right: confocal topography (x10; 4.4 mm^2) of the same sample.

In figure VII we can see the effects of treatment 4% 3T on the sample surface. As seen in the photograph, this treatment has proved to be frankly aggressive, the samples reaching roughness average values at around 45µm, with areas in which this value may increase to about 150 µm. In some points a vertical drop of around 500 microns has been observed.

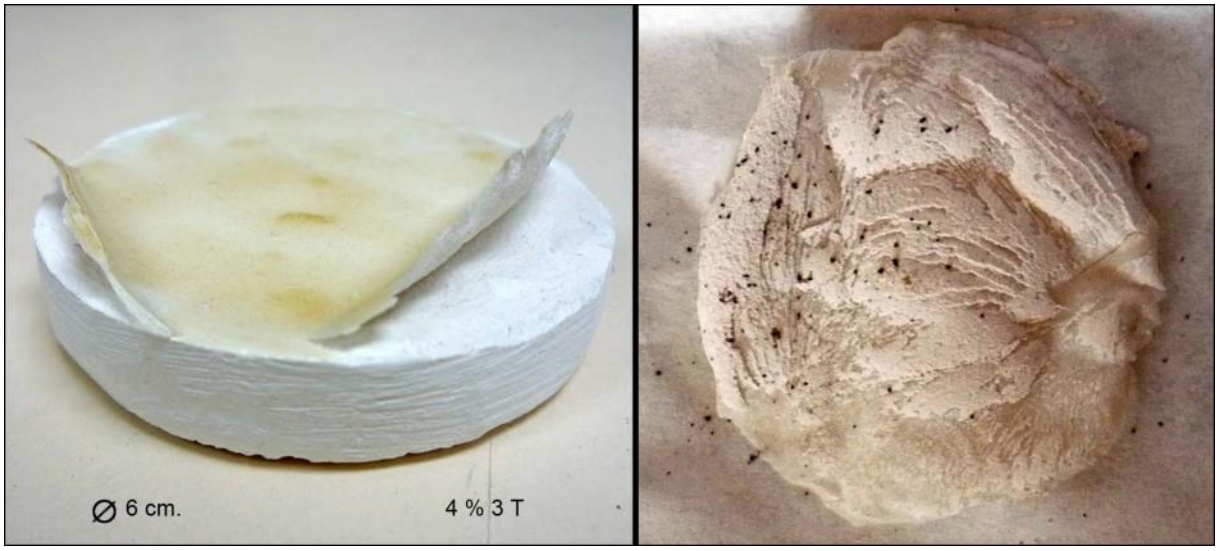

Figure VIII: Left: appearance of the sample treated with agar film 4%3T, showing the retraction of the agar layer. Right: agar film once removed from the surface of the sample with quite a lot of plaster and some fungi (black dots).

Again a decrease in water vapor permeability occurs. In this case it is around 12%. The material losses are significant (4.74 mg/cm^2), the shrinkage and *strappo* capacity of the treatment under these conditions should be emphasized. Remaining residues cover approximately 4% of the sample surface with a similar distribution to the above. Notice that the black spots on Figure VIII right, have appeared within thirty days after treatment, when the film was removed and stored on filter paper in a plastic bag. There is quite a difference in the intensity of the biological colonization on this sample compared to the samples previously seen, which could be due to variations in humidity.

To better illustrate how the cleaning procedure works, some tests have been conducted on real plaster cast fragments [Figure 9]. They all showed abundant surface dirt and clay deposits. For these tests we used a concentration gel of 2%, with application times of 2, 5, 10, 20, 30 minutes and 24 hours (totally dry).

On these samples soil does not appear as separate layer. Under the optical microscope we can see it as pores filling (decreasing surface roughness) and in some areas as patches (areas up to 2 mm^2) between 5 and 30μm thick. Occasionally we find accumulations up to 80μm in height with an area of about 1mm^2.

On these samples localized measurements and confocal topographies have been taken, as well as *visu* observations on the surface of the samples before and after treatment.

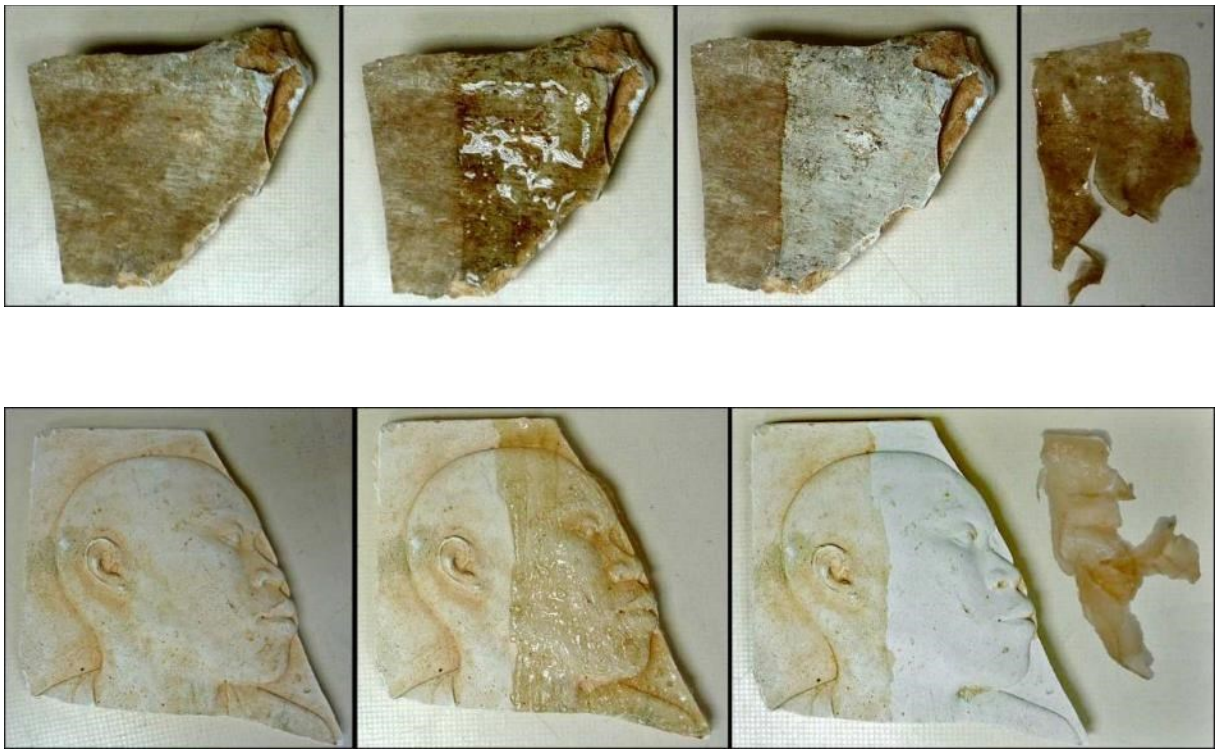

Figure IX: Upper: Application of 2% agar gel for 5 minutes. Observe the migration of dirt on the borderline between the treated and untreated areas. Lower: Application of 2% agar gel for 20 minutes.

In all treatments applied, regardless of the duration of treatment, we have found there has been a loss of support material. In applications less than 30 minutes, we could describe these losses as acceptable. In the case of treatment with two successive applications of 2 minutes, note that the second application has removed a greater amount of support material, possibly due to softening of the surface.

From these experiences it is advisable to apply the product with substantial thickness to make its removal easier in order to reduce the amount residues when working with this type of gels. Indicate that the residues are concentrated in the recesses of the samples.

Conclusions

Concerning variations in the surface morphology of the samples, an average value of initial roughness Ra=1.325 μm was calculated (coefficient of variation: 18.7%), measured over an area of 4.4 mm^2, on a total of 6 samples and prior to any treatment applied [Table II]. After the various tests conducted, we have found that 4% treatments of longer duration, 2T (20 minutes) and 3T (totally dry), the material changes throughout the treated surface. For the 20 minutes treatment the roughness increases to 3.283 μm, with the appearance of a pitted surface. The 3T, totally dry, treatment causes more severe effects, generating an abrupt surface texture, with mean values of Ra=45.5 μm.

Table II. On the first two left columns initial roughness measurements are shown. On the two right columns treatments showing significant variations are recorded as general and localized values (in parenthesis).

Initial Roughness		Treatment	Final roughness
Average (6 samples)	1,33μm	4%3T	45,45μm (148,68μm)
Standard Deviation	0,25μm	4%2T	3,28μm (7,03μm)
Coeficient of Variation	18,70%	2%3T	1,12μm (11,48μm)

The modifications discussed above affect the entire sample surface. In both cases, 4% 2T and 4% 3T, localized variations in roughness occur (greater than the overall value), as well as in the 2% 3T (totally dry) treatment, with Ra=11.5 μm, although their general values stay in the initial range. For the 4% 2T (20 minutes) application time, Ra=7 μm values are reached. When using 4% and 3T, totally dry, treatments Ra goes up to 148.7 μm [Table II]. When evaluating the rest of the treatments the roughness values remain within the range of values established initially. In all cases the areas where roughness changes have occurred are those of plaster material loss.

Experimental results show that treatments 4% 3T and 4% 2T reduce the permeability of the material (12% in the first one, and approximately 8% in the second). In the case of treatments 4% 2T and 3T the trend in all samples is to reduce their permeability (all the samples tested gave lower

values of permeability after treatment) [Table III]. As a hypothesis, this decrease is considered to be possibly caused by the increased in surface roughness which results in larger surface area with a consequent increase of phenomena of surface adsorption of water vapor molecules, slowing down their ascent to the atmosphere.

In all other cases, although the average value indicates a positive direction, there is not a clear trend for these treatments, since in all of them some of the samples show a decrease in permeability. Moreover, in these cases, it must be said the variations between the permeability values obtained before and after each treatment are less than 2% (i.e. very small. 5% is the maximum percentage variation that is allowed between two consecutive weightings when calculating the value of water vapor permeability of a material). Therefore, due to the small variation in the results, we can say that the changes in permeability caused by these treatments are not significant.

Regarding the loss of material [Table IV] only the applications of longer duration (totally dry) and with higher product concentration (4%) cause a significant loss in the sample weight after treatment. It should be emphasized that none of the material losses are homogeneous across the surface of the sample. As the reference average value, it was calculated that the loss of material per unit area which amounts to 4.74 mg/cm^2. In treatments with 2% total dry, a small localized loss of material has occurred (0,08 mgr/cm^2).

Table III. Variations of the water vapor permeability for each treatment.

Permeability	Befote	After	Variation
Treatment	(gr/m x 24h)	(gr/m x 24h)	(%)
4% 3T	2,058	1,810	-12,07
4% 2T	1,919	1,767	-7,91
4% 1T	1,818	1,838	1,13
2% 3T	1,668	1,684	0,96
2% 2T	1,801	1,832	1,73
2% 1T	1,683	1,713	1,79

Table IV. Loss of material produced by each treatment and agar remains after each treatment (% of the surface covered).

Treatment	Weight loss (mg/cm^2)	Residues (% surface)
4% 3T	4,74	4
4% 2T	<0,1	2-4
4% 1T	<0,1	0-2
2% 3T	<0,1	4-6
2% 2T	<0,01	0-4
2% 1T	No	No

Weight loss measurements cannot be altered by the possible gain of weight due to the presence of agar gel residues. This possibility has been dismissed because of the low density of the product, its scarce presence in all cases, and the film thickness (only about 10μm).

The risk linked to the presence of traces of agar on the samples, has been stated through the agar films removed that were wrapped still moist with filter paper and stored in airtight plastic bags away from light. After 30 days, when they were unwrapped for study, mold had developed.

Observations with ultraviolet light and by visual analysis under x40 magnification show that there are no residues in the pores of the material and the existing ones are in the form of films on the perimeter zones [table IV], especially at the lateral edges of the samples. More residues remain with longer times of application and the thinness of the gel layer applied.

Concerning the tests on real objects, it is interesting to apply the gels in very thick layers, which will greatly facilitate their removal and help prevent leaving any residues on the surface even with short times of application.

As a summary, based on the results of the study, could be considered safe and effective treatments on plaster objects:

- The 2% agar gel concentrations with times of application between 2-30 minutes
- 4% concentrations with application times of less than 5 minutes.

In these treatments it has been proven:

- There is no removal of original material,
- The surface roughness does not change
- Vapor permeability is almost constant.

Not recommended treatments regarding damage threshold:

- Both concentrations leaving the agar gel to dry completely
- 4% application concentration over 15 min.

These treatments:

- Promote material removal
- Increase of surface roughness
- Decreasing its permeability to water vapor (for the 4% concentration)

And also we would like to state that:

- The agar-gel cleaning technique leaves no residues included in the pores.
- Application of thick films to minimize product residues is recommended.
- The presence of traces of agar is an ideal substrate for biological colonization.
- Too hot applications promote border lines.
- Like in any other cleaning technique it is better to make two consecutive short applications, rather than one longer one (ie. two 10 minutes applications rather that one of 20 minutes). It is recommended to leave enough time between applications to allow the support to dry completely.

Notes

[1] Cremonesi, P., *et al. Use of Agarose and Agar for preparing "Rigid Gels"* Cuaderno n. 4. CESMAR 7. Il Prato. Saonara, PD, 2007. Cremonesi, P., *et al. Use of Rigid Agar Gels for Cleaning plaster objects.* Cuaderno n. 6. CESMAR 7. Il Prato. Saonara, PD, 2008.

[2] ALAMO. Ctra. De Fuencemillán s/n. 19292 Espinosa de Henares. Guadalajara.

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