RECH6 - 6th International Meeting on Retouching of Cultural Heritage
Valencia, Spain | 4th-6th November, 2021
Doi: https://doi.org/10.4995/RECH6.2021.13533

RETOUHING MURAL PAINTINGS IN HYPOGEUM: PRELIMINARY STUDY AND FIRST RESULTS

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ABSTRACT

This study was carried out during the ICR conservation project involving two of the mural paintings of the Saint Peter and Paul's hypogeum in Matera. Retouching mural paintings preserved in such a peculiar environment as hypogea is not an ordinary operation. In these contexts, relative humidity approximating to 100% makes hygroscopic materials less durable, favouring biological growth on them. In addition, severe climatic conditions can lead to a fast degradation of retouching materials. Watercolours, extensively employed for retouching mural paintings, are not completely recommendable in such humid environments, so a research was planned to find a compatible and alternative binding media. A study was carried out on laboratory samples to select the most suitable binding media among the following: two synthetic resins, Laropal A81 and Regalrez 1094, and two natural products, Funori and arabic gum. Each binder was blended with two different pigments. For each binder four different dilutions were tested, in order to find out how these factors could have affected the analysed properties. These products have been investigated in relation to their optical properties, wettability, vapour and water permeability, resistance to salt crystallisation and bioreceptivity. Some tests were repeated after an artificial ageing process, based on cyclical alternation of humid-cold and dry-hot exposure conditions. In addition to the laboratory tests, some in situ applications were performed. Amongst four binders, Regalrez 1094 showed the best results. Nevertheless, its bioreceptivity and applicability issues make necessary to continue and develop further research.

Keywords
Hypogeum, reintegration, Laropal A81, Regalrez 1094, Arabic gum, Funori.

1. INTRODUCTION

The Saint Peter and Paul mediaeval church is one of the most ancient hypogeum in Matera (Southern Italy). On

this site, completely carved into the local calcarenite, the XIII century church was erected and named after San Francesco d'Assisi. The church and the hypogeum are connected only by a narrow manhole, placed in one of the chapels of the upper church. A series of stairways allow visitors to reach the underground rooms where some noticeable frescoes, dating back between the XIII and XIV century A.D. [1] are preserved.
These frescoes were made according to the traditional mediaeval technique of rupestrian paintings.
The constitutive materials have been studied through analytical investigations [2]. The paint layer was applied on a single lime-based plaster (average thickness of 1 cm). The artist used a poor palette of colours, mainly based on earth-pigments.
This paper describes the 2021 preliminary study of retouching materials for intervention on two of these fresco paintings, during a thesis work at Istituto Centrale per il Restauro (ICR). Both paintings showed a severe lack of image integrity, due to the extensive loss of paint and plaster layers, to such an extent that a reintegration treatment had to be carefully planned.

Hypogea are characterised by peculiar environmental conditions which strongly affect restoration treatments; they are generally dark places, with high humidity levels and often subjected to water condensation phenomena. These conditions are favourable for the growth of certain types of microorganisms, such as streptomyces [3]. In some cases, karstic phenomena may occur and determine the formation of calcitic encrustations [4]. Other damages can be caused by the alteration of the microclimatic stability, due to air circulation or to visitors' presence. The permanence of visitors increases carbon dioxide concentration and consequently that of carbonic acid [5]. In addition, visitors can be vehicles of spores and microbiological contaminants that can colonise rocky and painted surfaces [6]. Hence, before the intervention in Ss. Peter and Paul hypogeum, microclimatic and biological surveys have been performed. The one-year monitoring survey revealed that the hypogeum has 97-99% relative humidity and a minimal seasonal temperature variation due to a considerable thermal inertia (14-20 °C). Finally, the microbiological survey demonstrated the lack of a vital microorganisms colonisation.
These stable conditions can be easily altered by the introduction of new materials so the selection of the intervention products should be properly done. When choosing the suitable binder for colour reintegration in hypogea some important factors have to be considered:

1) high humidity level requires a binder with both good permeability and water resistance;
2) the humid environment, favourable for microorganism growth, requires products scarcely sensitive to biological attack;
3) health issues for operators: toxic solvents and products should be avoided in environments with low air exchange;
4) short chain alcohols can activate a microorganisms colonisation [7,8,9].

A bibliographic survey demonstrated that there is no tested and traditionally used material for retouching in hypogea and that the most commonly used binders belong to two different categories: natural polysaccharides polymers and synthetic ones. Several documented treatments (from 1976 to 2020) show that watercolours have been widely used, despite their high sensitivity to humidity and biodegradability. As they tend to fade or turn colour [10] in a short time because of severe climatic conditions of hypogea, from the 90s onwards, restorers have experimented with alternative solutions. More recently, the use of pigments dispersed in pure water, in biocide products or in water lime have been documented, as well as the use of synthetic resins, like urea-aldehyde polymer contained in Gamblin Conservation Colors.
Furthermore, the bibliographic survey did not reveal any scientific study about the long-term behaviour of retouching products in the peculiar climatic conditions of hypogea.
Therefore, an experimental study was carried out to test four binders, selected among the others for their properties or for their tested use even in retouching mural paintings: two natural polysaccharide polymers (Arabic gum, Funori) and two synthetic resins (Laropal A81, Regalrez 1094).

2. MATERIALS AND METHODS

2.1 Materials

As generally known, natural organic binders commonly used for reintegration and based on polysaccharides are less resistant to water effects and biodegradability than synthetic resins solutions. The latter, on the other hand, could form an impermeable film and are often dissolved in solvents that should be avoided in a hypogeum environment, both for healthy and conservative issues.
The binders tested during this experimental study were selected taking into account issues related to the

described environmental conditions and the bibliographic data concerning the most used binding media in past interventions.

It has been established not to investigate commercial formulations because some of them can contain additives. In addition, self-made colours obtained from raw binding materials, made it possible to dissolve them in the proper solvent and to choose the suitable concentration for each binder.

Laropal A81 is an urea aldehyde resin, recently used in the field of retouching of mural paintings. Its water insolubility [11] makes this product suitable in contexts with high humidity levels. The temperature of glass transition (Tg= 57°C) is higher than that commonly detected in hypogea [12]. Moreover, the adjusted mixture of powder colours and Laropal A81 gives back a matt surface [13], comparable with the optical features of frescos. A wide range of solvents can be used to dissolve this resin [14], allowing the selection of the less toxic for the restorers and to avoid the alcoholic ones, able to stimulate the formation of microbial colonization.

Regalrez 1094 is an aliphatic resin mainly used as varnish for easel painting. There is not a wide bibliography concerning its use as binding material [15], but its scarce sensitivity to water made it a potential binder to be used in environments with high presence of moisture. Its Tg value (33°C) is lower than that of Laropal A81, but still higher than temperature values commonly detected in hypogea. Its refraction index is higher than that of Laropal A81 [16], producing a transparent veil similar to watercolours. Regalrez 1094 ensures stability and reversibility with the solvent in which it has been dissolved, as proved in previous studies [17,18].

Funori is a polysaccharide obtained from a natural seaweed, mostly used in the consolidation treatment of mural paintings [19,20,21] and as a binding material [22] due to its gluing properties. As it produces matt films [23] and resists to yellowing [24], it is also suitable for the reintegration of matt surfaces. On the basis of its proven good resistance to biodegradability, this product was selected for the experimental study [25]. This polysaccharide was extracted by raw seaweed and purified according to the method developed at Tuscia University [26].

Arabic gum is a natural polysaccharide used in the formulation of watercolours. It is the most widely used product for retouching mural paintings. Hence it was tested to work as experimental reference for the evaluation of the behaviour of the other binders.

Each binder was mixed with two different pigments: Ultramarine blue and Burnt Sienna. These two pigments are included amongst the twelve selected by the ICR for retouching wall paintings [27]. In addition, this choice makes the reading of comparative colorimetric measurements easier: the blue facilitates any yellowing and bleaching detection, while the red earth makes any colour changes more evident.

The binder/solvent ratio, as shown in table 1, was established considering the lowest amount of product necessary to obtain a sufficient cohesive strength of the paint film and suitable optical and handling properties.

Table 1 – Binders/solvent ratio of the tested binders.

Binder	Proportion %	Solvent
Laropal A81	**30**	**White Spirit D40/ Ethyl-lactate (1:1)**
Regalrez 1094	**30**	**White Spirit D40**
Funori	**2**	**Water**
Arabic Gum	**10**	**Water**

Each binder solution was blended with pigments in four different volume ratios: 1:1, 1:2, 1:3, 1:4.

All the binders were applied on a lime mortar layer which had a similar composition to that of the original plasters previously determined by analytical investigations.

The supporting plasters of the analysed wall paintings are made of air hardening lime and calcareous sand and therefore they are very similar to each other. The petrographic study with a polarising light microscope on thin sections revealed the size of the clasts (ranging from 150 to 400 μm) and the binder/aggregate ratio which show an abundant use of lime rather than aggregates. According to this result, the mortar of the experimental samples was realized with lime and calcareous sand, in a 1:2 mixing volume ratio.

2.2 Methods

The scientific research was aimed at testing the behaviour of each binder when put in environmental conditions simulating the ones present in hypogea.
The following properties were investigated:

- optical properties by colorimetric measurements
- hydric behaviour by contact angle measurement and wet sponge test
- salt damage resistance by salt crystallisation test
- bioreceptivity

All measurements and tests were performed in a controlled laboratory environment (20°C and 55% RH). Unless the salt crystallisation test, they were carried out before and after artificial ageing in a climatic chamber (CH250 CLIMATEST ARGOLAB). Alternated 6 hour cycles of cold/humid (T=13°C and R.H.=90%) and hot/dry (T=26°C and R.H.=60%) simulated and intensified the real environmental condition. UV light ageing was not performed, because photochemical degradation was considered negligible in a hypogeum environment.
Each sample underwent 900 hours of T/RH artificial ageing.

2.2.1 Colorimetric measurement

Measurements were carried out by means of a Minolta® CM700d spectrophotometer. A template was prepared to be applied on the sample before the measurement, in order to repeat the test at the same point before and after the ageing. Measurements were also performed on films made of pure binders without pigments, applying them on a transparent slide of glass.
The modifications that occurred on the surface were taken as an indicator of the microscopic alteration.

2.2.2 Contact angle

Wettability is a necessary information when selecting a binder to be used in an environment with relative humidity values close to saturation. The interaction between a drop of water and a material reveals a hydrophilic or hydrophobic surface. The wettability of the painted films was investigated by contact angle measurements, according to NORMAL-33/89. The acquired images were processed with Angle Metre 2.0 software.

2.2.3 Wet sponge test

Wet sponge test was performed according to the UNI 11432 standard to evaluate if the selected binders applied on a sample allow the transfer of liquid water. Some test procedures were modified:
- the dimension of the sponge was reduced to better match the samples (2,7 cm diameter and 0.85 cm in thickness).
- Samples were not conditioned at 60 ± 2 ° C, as recommended by the standard because the integrity of the films could have been compromised at that temperature. Therefore the imposed conditions in the climatic chamber were: 23 °C of temperature, 50% of relative humidity.
According to the results of some preliminary tests, 1 ml of water was introduced in the sponge and the contact pressure was applied for 1 minute. The sponge was changed after approximately 40 measurements.

2.2.4 Vapour permeability test

Vapour permeability test was performed following the UNI EN15803 2010 standard to evaluate if the selected binders allow the transfer of water vapour.
The test cup vessel was filled with a 30% potassium nitrate (KNO_3) solution, leaving a 1.5 cm gap between the sample and the surface of this solution. The potassium nitrate, dissolved in demineralized water, keeps constant the relative humidity value at 93.2%. Before the test, samples were conditioned in a climatic chamber at T 23 ± 1° C and R.H. 50 ± 3%. The water vapour amount flowing through the sample was evaluated by measuring the weight differences of the sample-holder system over time. The stationary condition was reached when the curve in the weight/time diagram assumed a constant slope passing through the last five points of measurement.
Two batches of samples were consecutively analysed: firstly, NOT COATED samples (NC), i.e. the plaster itself, and then COATED samples (C), i.e. the same plaster coated with a paint layer applied by brush.

2.2.5 Salt resistance test

Salt resistance test evaluated the pictorial films resistance to salts disintegration. This test did not follow specific standards, but it was designed by mixing procedures from different standards: capillarity absorption (UNI EN15801), vapour permeability (UNI-

EN 15803) and salt ageing resistance (TC Rilem 127-MS).

Samples were prepared as follows: a 1 cm thick layer of mortar was spread on a 5 × 5 × 4 cm stone block; the different selected binders were applied over the dried mortar.

Each sample of mortar, after being sealed on the four side faces with an impermeable strip, was placed in a container, with the film facing upwards and the underlying stone immersed for 3 cm in a solution of sodium sulphate (3% of conc.). The surface of the saline solution was sealed with oil, in order to force the evaporation of the solution only through the sample surface, where the crystallisation of the sodium sulphate necessarily occurred (Fig. 1).

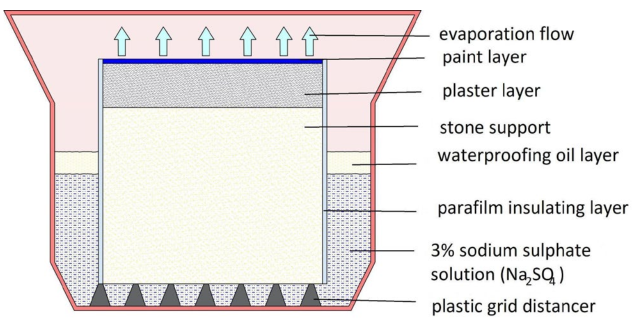


Figure 1 – Scheme of the sample-holder used for the salt resistance test.

The described system was placed in a climatic chamber at controlled humidity and temperature conditions (20 ° C and 50% RH). Throughout 2 weeks, the weight variation of the system was measured at 24 h intervals, in order to obtain an indirect measure of the solution evaporation rate. After each weighing, the surface of the sample was dusted, removing the crystallised salts on the surface and part of the disintegrated film. After this operation, the system was weighed in order to determine the loss of material due to the salt damage.

2.2.6 Bioreceptivity

The bioreceptivity test evaluated if the pictorial films favour the microorganisms growth. This test consists in exposing the samples to the airborne contaminants present in the crypt, placing each sample horizontally on the floor. *S*amples were monitored every week for four months. During this period, any biological growth was detected and photographed.

At the end, all samples were carefully observed under a stereomicroscope equipped with a digital acquisition system.

3. RESULTS AND DISCUSSION

3.1 Colorimetric measurement

Colorimetric measurements performed on pure binders showed that Arabic gum, Laropal A81 and Regalrez 1094 have similar colorimetric coordinates (Tab. 2). Funori is less bright than the other binders and tends to have a yellow tone.

Table 2 – Colorimetric coordinates of pure binders applied on glass slides.

Binder	L*(D65)	a*(D65)	b*(D65)
Laropal A81	88,60	1,59	-3,50
Regalrez 1094	88,92	1,60	-3,49
Funori	84,98	0,76	0,10
Arabic Gum	88,42	1,43	-2,65
glass slide	90,08	0,77	-3,57

Table 2 – Colorimetric coordinates of pure binders applied on glass slides

When Funori is mixed with pigments and applied on mortar samples, its chromatic coordinates are comparable to those of Regalrez 1094 and Arabic gum binders' pigmented films. Laropal A81 films are darker than others, confirming technical literature results [16] (Tab. 3).

Table 3 – L*a*b* coordinates of the four selected binders registered for 1:1 mixing ratios blue films.

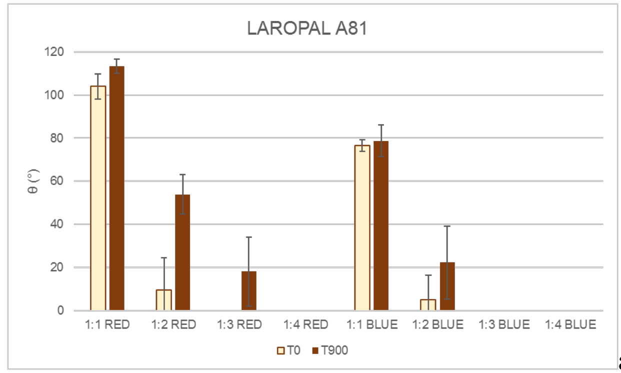
Binder	L*(D65)	a*(D65)	b*(D65)
Laropal A81	34,91	22,16	-63,46
Regalrez 1094	41,15	16,34	-63,01
Funori	44,23	15,10	-63,35
Arabic Gum	41,78	16,92	-63,42

After artificial ageing in the climatic chamber, no visible chromatic changes were detected on any of the binder films, since ΔE values were always below the human eye limit (ΔE<3). The accelerated ageing

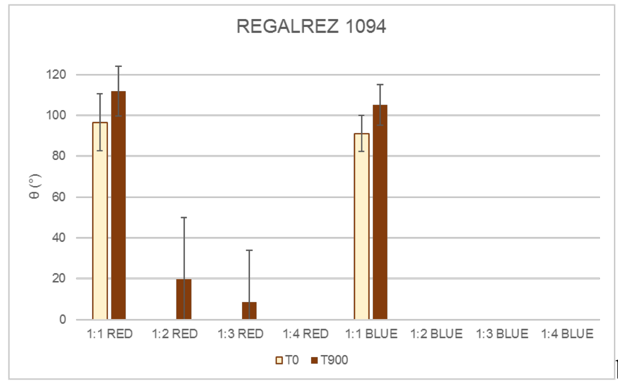
process did not chromatically affect the pigmented binder films applied on mortars.
Anyway, natural binder films have suffered considerable morphologic alterations: Arabic gum films cracked and detached from the underlying glass slide, while Funori films folded.

3.2 Contact angle

This analysis highlighted the hydrophilic behaviour of films obtained from natural binders (Arabic gum and Funori). On these films, no contact angle values were registered as every water drop was immediately absorbed. On the contrary, the synthetic binders showed an opposite trend due to their chemical nature. In fact the acquisitions carried out before ageing gave back an evident hydrophobic behaviour for both Laropal A81 and Regalrez 1094 resins, although limited to the samples with a binder/pigment mixing ratio of 1:1. The water drops on the surface produced contact angle between 75° and 105° for the former and between 86° and 100° for the latter resin. After ageing, there was an increase in the water repellence of both synthetic resins (Fig. 2a). Specifically, it was noted an increase in the Laropal A81 hydrophobicity: in particular, in 1:1 dilution, contact angle increased considerably, up to 120°. Regalrez 1094 also showed a general increase in water repellence after ageing (Fig. 2b). While in the 1:1 samples the drop persisted on its surface with a contact angle between 100° and 120°, the higher dilutions were characterised by less hydrophobic behaviour and every water drop was immediately absorbed.



a)



b)

Figure 2 a,b – Laropal A81 (a) and Regalrez 1094 (b) contact angle (θ) before (T0) and after the artificial ageing (T900)**.**

3.3 Wet sponge test

The wet sponge test demonstrated that all films are permeable to liquid water, especially in higher dilutions (Tab. 4). Regalrez 1094 produced the most permeable film. UR-T ageing caused a general increase of the absorption coefficient (Wa).

Table 4 – G*- Arabic Gum, F*- Funori, R*-Regalrez 1094 L*-Laropal A81 –Absorption coefficient Wa (g/cm^2 x s) before (T0) and after the artificial ageing (T900).

	T0		T900	
Binder	**Burnt Sienna**	**Blue**	**Burnt Sienna**	**Blue**
G*_1:1	**0.83**	**2.03**	**4.01**	**11.48**
G_1:2	**0.98**	**2.47**	**5.67**	**2.47**
G_1:3	**0.95**	**1.81**	**5.86**	**8.01**
G_1:4	**1.96**	**1.69**	**7.86**	**11.83**
F*_1:1	**1.61**	**2.81**	**4.05**	**2.72**
F_1:2	**3.32**	**2.58**	**5.65**	**5.57**
F_1:3	**3.36**	**3.80**	**7.13**	**7.16**
F_1:4	**12.54**	**1.39**	**7.68**	**4.55**
R*_1:1	**1.14**	**0.46**	**12.43**	**12.45**
R_1:2	**1.15**	**4.78**	**22.74**	**12.96**
R_1:3	**1.10**	**6.28**	**16.81**	**26.73**
R_1:4	**1.13**	**10.73**	**19.29**	**22.84**
L*_1:1	**0.69**	**0.82**	**0.83**	**1.78**
L_1:2	**0.51**	**1.89**	**2.65**	**1.97**
L_1:3	**0.60**	**1.25**	**3.35**	**3.90**
L_1:4	**0.58**	**4.73**	**7.86**	**4.54**

3.4 Vapour permeability test

Permeability test detected no significant differences in permeability values between natural and synthetic binders (Fig. 3). Coated samples (C) showed permeability values similar or slightly lower than those of not coated (NC) ones.

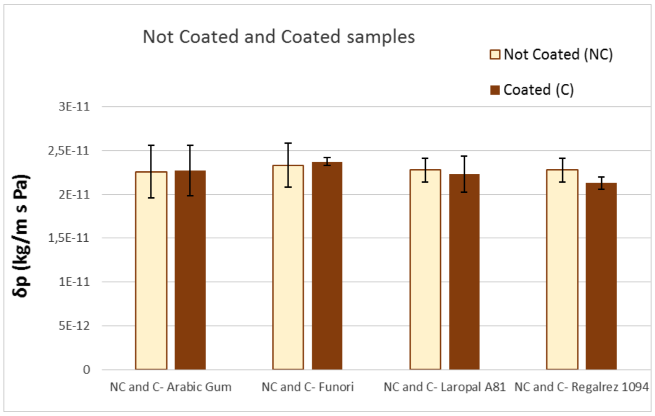
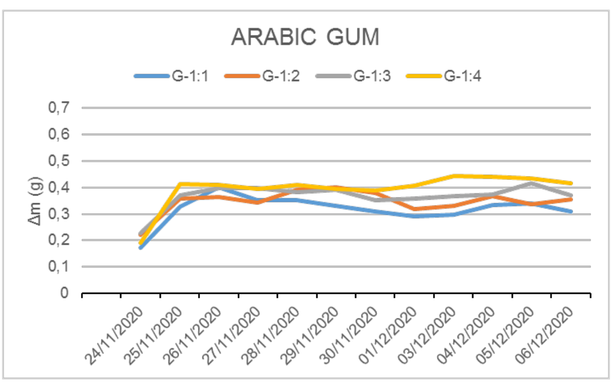


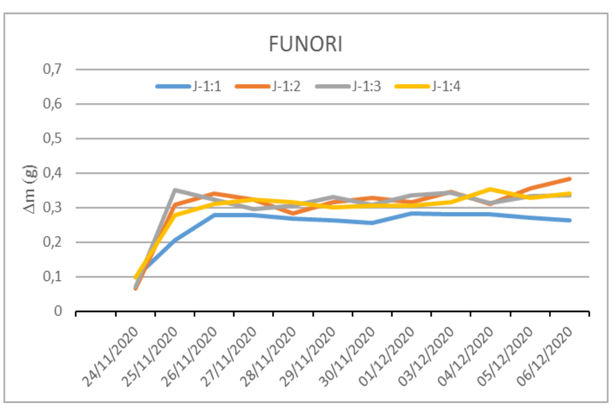
Figure 3 – Permeability (δp) of NC and C samples.

3.5 Salt resistance test

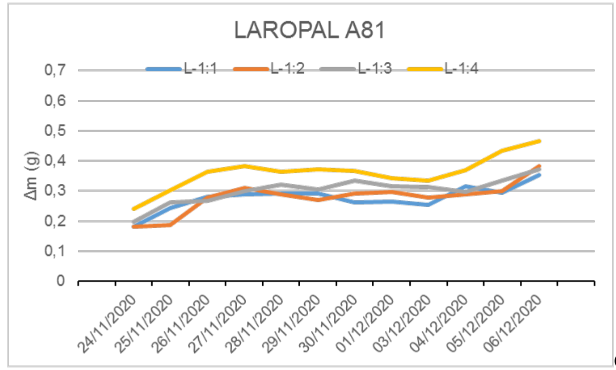
All films in all the tested dilutions allowed the transfer of the salt solution through the samples and, as a consequence of its evaporation, salt crystallisation on the surface. The higher the dilution the higher the amount of material disintegrated by salt: 1:4 lose more weight than 1:1 dilution films. Anyway, synthetic resins showed an overall material loss which was higher compared to natural binders. (Fig. 4 a, b, c, d)



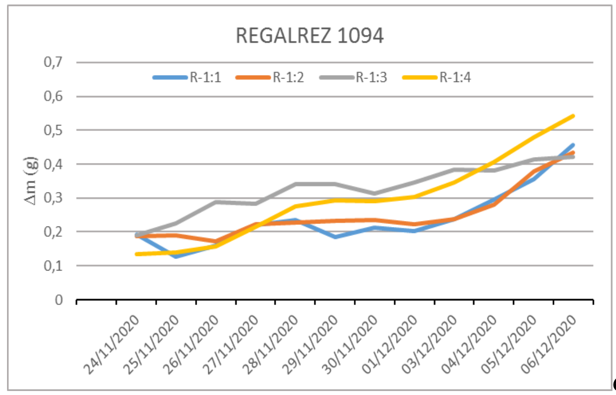
a)



b)



c)



d)

Figure 4 a, b, c, d – Weight loss (Δm) over time of Arabic gum(a), Funori (b), Laropal A81 (c) and Regalrez 1094 (d) samples due to sulphates disintegration.

3.6 Bioreceptivity

The growth of microorganisms was observed and documented on natural binder film surfaces already after 1-month exposure in the hypogeum. After two months, microorganisms also appeared on Laropal A81 and Regalrez 1094 films. Dilutions directly affected the extent of microorganisms presence, as a higher quantity of binder corresponded to a higher microbial growth on the surface (Fig. 5). Among all the selected binders, Regalrez 1094 showed the lowest microorganisms spread on the samples surface.

4. CONCLUSIONS

In conclusion, the above-mentioned experimental results showed that all the tested binders (Arabic gum, Funori, Laropal A81 and Regalrez 1094):

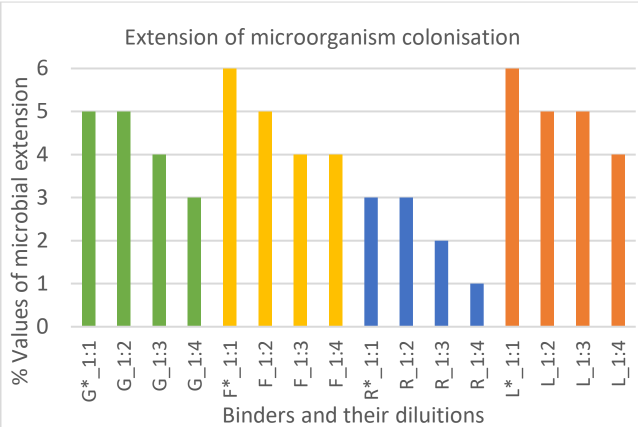


Figure 5 – Extension of microorganism growth on the films surface, expressed as percentage of colonized area: (1 = <10%; 2= 20-30% =2; 3 = 30-40% = 3; 4 = 40-50% = 4; 5= 50-60%; 6 = 50-60%); G*- Arabic Gum, F*- Funori, R*- Regalrez 1094, L*-Laropal A81 – (%).

- have good chromatic stability;

- do not interfere with the transfer of liquid and vapour water;

but:

- are bioreceptive, although at different degrees.

Regalrez 1094 showed the best performance, both when blended with Ultramarine blue and Burnt Sienna. However its bioreceptivity (the lowest compared to the other binders) makes it unsuitable for applications in hypogea. In addition, Regalrez 1094 presented some technical problems when applied on the humid mortar of the crypt: it created a not uniform painted surface and tended to form translucent stains all around the film borders.

Concluding, none of the tested binders is suitable to retouch mural paintings in hypogea. However, further research is needed to improve Regalrez 1094, which showed the best experimental results. Specific biological inhibitors and rheological modifiers that could overcome the handling issues of this product can be designed in future research. For instance, a new formulation should be identified combining Regalrez 1094 with the advantages of a biological growth inhibitor product (i.e. silver nanoparticles or Biotin R1+R2), that does not significantly alter the binder properties.

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