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INTEGRATED STUDIES FOR THE EVALUATION OF CONSERVATION TREATMENTS ON BUILDING MATERIALS FROM ARCHAEOLOGICAL SITES. APPLICATION TO THE CASE OF MERIDA (SPAIN)

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Abstract

The application of conservation treatments, such as consolidation and protection ones, has been demonstrated ineffective in many cases, and even harmful. Evaluation studies should be a mandatory task, ideally before and after the intervention, but both tasks are complex and unusual in the case of archaeological heritage. This study is mainly focused on analyzing changes in petrophysical properties of stone material from archaeological sites of Merida (Spain), evaluating, both on site and in laboratory, effects derived from different conservation treatments applied in past interventions, throughout the integration of different non-destructive techniques (NDT) and portable devices of analysis available at the Institute of Geosciences (CSIC,UCM). These techniques allow, not only assessment of effectiveness and alteration processes, but also monitoring durability of treatments, focused mainly on 1996 intervention in the case of Roman Theater, as well as different punctual interventions from the 90's until date in the House of Mitreo. Studies carried out on archaeological sites of Merida permit us to compare outcomes and also check limitations in the use of those equipments. In this paper we discuss about the use of some techniques, their integration and limits, for the assessment of conservation treatments, showing some examples of Merida's case study.

Keywords: *NDT; Conservation; Treatments; Archaeological sites; Evaluation*

Introduction

Remains of Roman Augusta Emerita, located in Merida (Spain), have gone through many interventions since the first archaeological campaign in 1910. Some of them have demonstrated already to be harmful [1, 2], whereas others, more recent, must be evaluated in order to determine their effectiveness and durability, considering that many of those treatments are currently still applied. For this purpose a range of parameters, such as color, surface hardness and roughness, physic-mechanical or hydric properties, etc., have been measured on the original material (granite, marble and mortars mainly), with and without treatment, in order to analyze changes. The study is being conducted in the laboratory (Petrophysics Laboratory within IGEO) as well as *in situ*, on selected archaeological sites of Merida (Theater and House of Mitreo).

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The comparison of results collected, on one hand by comparing untreated and treated areas of the sites, on the other, pre- and post-treatment laboratory test carried out on samples, allows the distinction of variables that affect the interaction between products and building materials, issues such us effectiveness and durability of treatment and its validation or dismissal. The work in progress is being developed in two main work packages (Fig. 1).

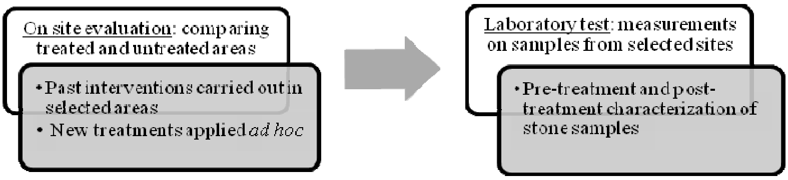

Fig. 1. The working packages.

NDT for assessment of conservation treatments in Merida's sites

1. Measurement of ***Ultrasound Pulse Velocity (UPV)***, in direct and indirect mode (Fig. 2). The equipment used was PUNDIT CNS Electronics, with an accuracy of 0.1 microseconds. The ultrasonic wave will cross the material according to its density and internal strength and cohesion, thus, the denser, the faster. This allows to characterize the stone material, but also check the internal state of conservation and different degrees of deterioration (compared to unaltered areas of the same material), by locating differences like voids, cracks, delamination, hidden elements or any other anomaly. It is also possible to analyze some mechanical properties by the correlation with the modulus of dynamic elasticity [3].

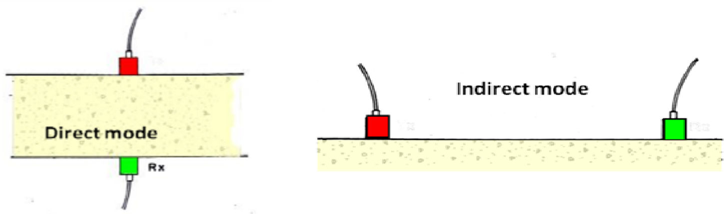

Fig. 2. In the direct mode ultrasounds propagation, transducers are placed in parallel and opposite faces. In the indirect mode the transducers are placed in the same face.

Regarding the assessment of conservation interventions UPV is especially useful, fast and easy handling:

- The degree of deterioration/preservation of materials can be checked before intervention, in order to search for compatibility of materials. The anisotropy of the material determines some deterioration processes as it strongly influences some kind of behavior, such as the capillary absorption one. Thus, it should be considered when placing and orienting new stone material on ancient structures as a preventive conservation method [4]. When studying restoration mortars, UPV permits to analyze how they work on site, their performance in the structure, in terms of compatibility or efficiency. In the case of Merida many types of mortar have been used, for structural consolidation, both in the reconstruction of the Front stage of the Roman Theater (Fig. 3) and in the restoration of mural paintings and mosaics at Mitreo's House. By analyzing the state of junction and the different internal strength between the original material and mortar restoration, and comparing with different types of plaster used (including the original Roman plaster), it is possible to determine the most compatible one, and detect, or prevent, future degradation processes regarding the quality of those ones. In this case, direct mode of UPV, when possible, provides more representative outcomes.

- UPV is especially useful when evaluating the efficacy of consolidative treatments [5, 6]. Measurements, in indirect mode, have been carried out on some wall paintings from Mitreo's House using transducers with a flat contact surface diameter of 12 mm and 1 MHz of energy. In the example showed in figure 4, comparative analysis results in a remarkable difference between treated (with Paraloid B72©) and untreated areas of the same panel. Values will allow us to assess the effectiveness of consolidative treatments applied in terms of higher strength of this area; nevertheless it should be correlated with other parameters, such as, for instance, chromatic measurements, in order to confirm a positive judgment.

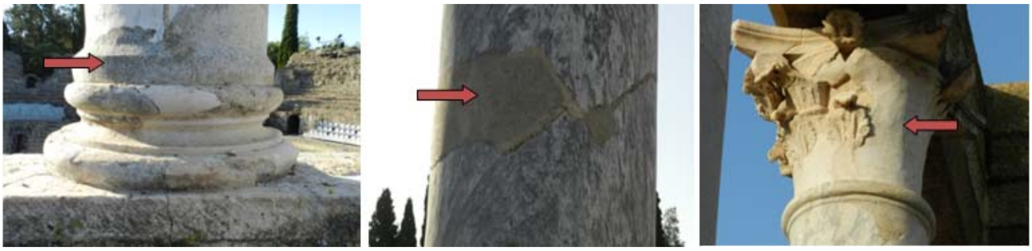

Fig. 3. Restoration mortars used in different periods for the structural consolidation of the Front stage in the Roman Theater of Merida (Photos by Natalia Perez)

Treated area: 2182 m/s　　　　Untreated area: 1611m/s

Fig. 4. UPV measurements on SDH13MA wall from Mitreo's House (Photo by Natalia Perez)

Limitations:

- Values may vary in different contexts; thus, the water content or the presence of salts will influence results according to day time or season, and does not provide data proportional to the amount of moisture retained in the material [7]. So outcomes may not be stable, and therefore not repeatable. In this sense, the use of complementary tools, such us moisture measuring instruments or karsten tube testing, allows to relate obtained values with moisture content and water absorption. It has been proved that there is an indirect relation between water absorption and UPV, that is, the lower UPV, the faster the absorption [7]. In each case it is essential recording also the casuistry of each measurement (date, weather issues, location, etc.).
- There are differences between the results obtained from direct and those obtained from indirect mode, so they are not strictly comparable. In the indirect mode the wave velocity is slightly slower than in the direct mode, due to the longer path in the former than in the latter.
- Deposits in the surface may also influence results.
- There is no data about how the coupling agent placed between the transducer and the material surface generally used in these measurements (plasticine, gel, honey, etc.) influences in outcomes. Regarding measurements on archaeological elements on site the use of these materials are limited, either because of difficulties inherent to each site

(position, location, shape, function, etc., of stone elements), or to the use of fouling material as the above mentioned, which, beyond staining or greasing the surface, it can lead to the loss of material, as peeling effect, on disintegrated surfaces.

2. The ***Roughness-meter*** provides micrometric data, both in image and numeric format, for the measurement of superficial texture. The equipment used for this study is an optical surface roughness-meter (TRACEiT, Innowep GMBH), a non-destructive and portable device, which uses white light (3 white lights at an angle of 120º). It allows 3D roughness topographic maps (at a micrometer scale; 1 micron of deep/height accuracy by 2.5 micromenters in both the X and Y axes) and obtains values for Ra, Rq and Rz roughness parameters according to DIN EN ISO 4287 standard:

- *Ra*: this parameter is defined as the arithmetic average of all the measurements that the equipment has acquired.
- *Rq*: it is defined as the square root of the average deviation of the measured profile, the root of the average of the irregularities and imperfections in the length L of a sample.
- *Rz*: represents the distance between the maximum and the minimum height measured, the extent of irregularities or imperfections that we measured over a length L.

The superficial roughness of stone material largely determines its degree of weathering and the interaction between the surface and the external agents. This is more accentuated when considering archaeological materials exposed to oudoor conditions. Thus, the greater the material surface roughness, the higher the specific surface interacting with degradation agents, so greater weatherability. A rougher surface is much more prone to microbial colonization [8] or to water and soil retention, due to the above mentioned higher specific surface. Some cleaning treatments are especially aggressive in this regard, both mechanical and chemical. Others modify the surface texture of the material, forcing to a readjustment of the relationship between the surface and external environment.

The roughness-meter is a very useful tool to assess changes on these roughness parameters due to the application of conservation treatments [9, 10]. Nevertheless measurements carried out comparing treated and untreated areas of marbles from the Roman Theater cannot be confronted because of the differential state of conservation in treated areas, mainly affected by sugaring (and consolidated for this reason), and untreated ones, with a well-preserved smooth surface. Results are not representative of the effects derived from application of treatments in this case. By contrast we can compare the results of measurements taken on several mural painting fragments from Mitreo's House. In the first case different fragments of mural painting preserved in the National Museum of Roman Art of Merida (MNARM) were measured attending punctual consolidation with PB72© (recently applied) and comparing color by color treated and untreated areas. In the second case three samples of mural painting from this same site were moved to IGEO's laboratory and measured pre- and post-treatment. The results show a slight increase in roughness for the case of fragments treated with PB72© (Fig. 5), and a significant increase in the case of those treated with ethyl silicate (Fig. 6).

Limitations:

- Ra, Rq and Rz values are obtained from the attainment of multiple shots of a three white lights device. This requires that the measuring unit cell rests, as far as possible, on flat surfaces to avoid the entry of outside light. In this sense, there are some physical limitations when it comes to measurements on site or on non flat surfaces in general.
- Comparative analysis requires the measurements been performed on surfaces with similar conditions. It might be necessary to clean the surface before measuring, and in cases where hard crusts or deposits exist outcomes may not be representative.
- To obtain representative values it should be ideal to develop several sets of measurements in order to get an average value from a selected area. The surface measured is small (5×5 mm^2), so, in general, the more measurements the better.

MNARM		IGEO	
Untreated sample (232) Rz=14	Treated sample (238) Rz=20	Pre-treatment(MCM25) Rz=22	Post-treatment(MCM25) Rz=26

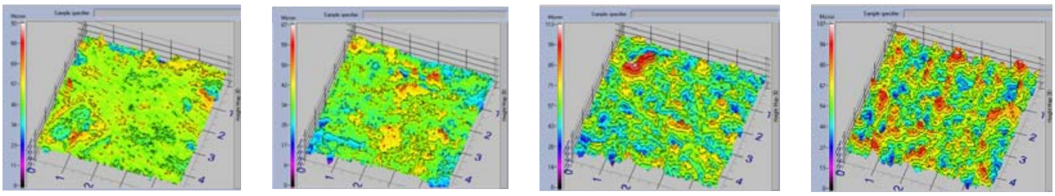

Fig. 5. Rz values and topographic maps from different fragments of wall-paintings from Mitreo's House comparing treated and untreated areas in the MNARM's case, and pre- and post-treatment measurements in the IGEO's case, in both cases with PB72©.

Pre-treatment (MCM23) Rz=20	Post-treatment (MCM23) Rz=40

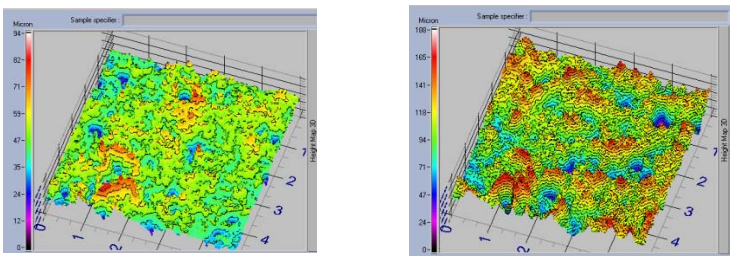

Fig. 6. Rz values and topographic maps from different fragments of wall-paintings from Mitreo's House comparing pre- and post-treatment with Estel 1000© on IGEO case

3. The ***Spectrophotometer*** has been largely developed for superficial measurements of chromatic parameters in the field of cultural heritage. The parameters used in the CIELab system (CIELAB 1976) are L* (luminosity/lightness), a* (chromatic coordinate from red to green), b*(chromatic coordinate from blue to yellow), as well as the standard *UNE-EN 15886:2011 Conservation of cultural property - Test methods - Colour measurement of surfaces* for the methodology and presentation of outcomes. Global change of the colour, defined by the parameter $\Delta E^*(\Delta E^* = \sqrt{(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2})$, which allows to set a numerical comparative value between two samples. It is understood that the change is visible to the human eye when it exceeds the value 5, although it may vary according to author. The equipment available in the IGEO is the Minolta CM-700d/600D, with a COLOR DATA SPECTRAMAGICTM NX CM-S100W Software.

It is well known the fact that some restoration treatments may cause superficial changes such as yellowing, as in the case of acrylic resins, darkening or brightness variations [1]. The measuring of chromatic parameters change caused by the application of conservation treatments has been successfully proved and represents an easy and quick way of evaluation [11-14]. Spectrophotometry also allows monitoring the efficiency and suitability of cleaning methods [11]. It requires a clean point as a reference for chromatic coordinates, equivalent to that which is wanted to be achieved.

Tests carried out in our case study confirm previous studies when focusing on wall painting and mosaics from Mitreo's House. Results of measurements comparing treated (A1-T1) with Acril33© and untreated areas (A1-ST2) on the Cosmologic Mosaic, and those regarding wall painting fragments from the MNARM (232 and 238) are showed in Table 1. The final value (ΔE*) is obtained by calculating the average of all measured points.

Table 1. Changes on chromatic parameters from selected areas of the Cosmologic Mosaic and wall painting fragments from Mitreo's House.

Samples	ΔL*	Δa*	Δb*	ΔE*
A1-T1/A1-ST2	-6.4	-1.7	-2.3	**7.0**
MNARM 232NT-rose/MNARM 238T-rose	-13.6	12.8	3.7	**19.0**
MNARM 232NT-red/MNARM 238T-red	-0.8	2.5	1.6	**3.1**
MNARM 232NT-white/MNARM 238T- white	19.2	-12	-1.4	**22.7**
MNARM 232NT-blue/MNARM 238T- blue	2.3	-7.8	-4.9	**9.5**

Limitations:

- The instrument must be tight against the surface to prevent light from entering and distorting the measurements, so the flatter the surface, the better.
- When measuring very heterogeneous material, such as granites, where the minerals are varied in composition and color, it may requires accurately location of each measured point to repeat the measurement after treatment, as well as precise location of a point with the same characteristics when comparing a treated and untreated to obtain a representative mean value. Heterogeneous materials usually require a higher number of measurements than homogeneous ones to get representative results.
- Roughness greatly influences the perception of chromatic parameters [15], so it is very useful to carry out previous measurements with the roughness-meter in order to relate these two aspects, preferably located on the same point.
- The moisture content may also affect the color values, therefore, when it comes to *in-situ* measurements, it must be taken into account and register the moisture content of the material, along with the environmental conditions at the time of measurement.

4. There are different ***strength testing methods***, but generally destructive techniques, such as *DRMS* (Drilling Resistance Measurement System), or the Pull-out test for calculating adhesive tensile strength. The *sclerometric techniques*, such as the ***Microhardness tester*** Equotip 3, are based on the measurement of the rebound energy derived from the impact of a plunger striked against the material surface. This device also allows using different hardness scales, *Vickers or Knoop scales*, depending on the needs. We chose *HLD scale* (Leeb hardness scale) which is the one given by default for the equipment. It is very useful to obtain mechanical properties of the surface, opportune when evaluating consolidating treatments. Nevertheless, as following mentioned, there are some limitations when measuring on site.

Limitations:

- It may give errors or misleading results when dealing with very rough or heterogeneous surfaces.
- It is considered NDT, nevertheless it could leave marks, little dots, on flat and soft surfaces.
- The influence of the support should be taken into account, since the way in which the object being measured bears on its base may vary the rebound energy value. A clear example is showed by some painted panels from Mitreo's House; in some cases the panel lacks contact with the wall-support, in other cases mortar junction with the wall is perfectly preserved, and therefore there is no air between panel and wall. Consequently, results, even if realized on the same panel, may vary according to how each zone bears on the wall.

In this case tests carried out on laboratory will be essential in order to compare same samples on same conditions pre- and post-treatment. On site it has been not possible to get representative values of the effects arising from conservation treatments, comparing treated and untreated areas, due to the differential state of conservation of those areas. Thus, disaggregation of treated marbles gives us lower values than untreated ones (Table 2).

Table 2. HLD values for hardness measurements on white marbles from the Front Stage of the Roman Theater

Treated areas	Untreated areas
493 HLD (± 60.5)	537 HLD (± 53.7)

Conclusions

The research project in which this study is integrated is still in process, and therefore some results are still partial and preliminary. However, the main objective of this article was to show the different options using various nondestructive and portable techniques showing some examples of its uses, advantages and disadvantages.

No one single method gives definitive results, and should be always compared to other parameters that influence the data gathering process. Hence, it is important to remark the utility of integrating, as far as possible, techniques that allow, at least, make valid hypothesis, reasoned and contrasted, as well as figure it out the influence of external parameters when evaluating conservation treatments.

Consolidating treatments for mural paintings in Mitreo's House have proved effective in terms of strengthening the internal cohesion, but showed superficial changes in color and roughness that may be considered as an alteration leading to degradation processes, and also the change of the original surfaces should be considered. Nevertheless, in the case of the Roman Theater we find no prove of changes regarding on site treated and untreated areas. However, areas where consolidative treatments were applied are now affected by the same disaggregation that motivated the previous intervention. It proves the limited duration of these treatments, and the need for periodical monitoring required in order to maintaining their effectiveness.

The starting premise of this study was to analyze *in situ* the effects, in terms of effectiveness and durability, of past treatments. These on-site measurements are priorities and preferred in order to get outcomes representative from its original environmental context. However, we find certain limitations, as mentioned, and complementary laboratory tests are crucial in order to understand those effects.

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