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Preliminary investigation of the preparation of repair mortars for the Temple of Diana, Mérida, Spain

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Abstract: In this paper, characterization of the mortars of different constructive elements in Roman Temple of Diana for its conservation is presented. Mortar samples collected from different parts of the Temple were characterized by means of polarized optical microscopy (POM), X-ray diffraction (XRD), thermal analysis (TGA-DSC) and X-ray fluorescence (XRF).

The optical microscopy results revealed that the mortars are composed of lime binder and quartz, feldspar and biotite grains together with granitic and metamorphic rock fragments as aggregates. XRD analysis supports the microscopic observations and adds the information of the presence of actinolite in the aggregates. XRD analysis further indicates the same origin of the granitic and metamorphic rocks from the surroundings and reveals an absence of biotite in the flooring mortar and only a trace of quartz in the masonry mortar of *criptoporticus*. According to results of TGA-DSC and XRF analyses, mortars were used in the channel in front of the Temple, the foundation of granite ashlar, and the inner wall of *criptoporticus* has higher hydraulic character.

For the future conservation of the monument, the information provided here about the composition of original mortars will be useful.

Key words: conservation, Roman mortar, microstructural and thermal analyses.

Investigación preliminar de la preparación de morteros de reconstrucción para el Templo de Diana, Mérida, España

Resumen: En este trabajo se presenta la caracterización de los morteros de diferentes elementos constructivos en el Templo Romano de Diana con vista a su conservación. Las muestras de mortero que fueron recogidas de diferentes partes del Templo se caracterizaron por microscopía óptica polarizada (POM), difracción de rayos X (DRX), análisis térmico (ATG-DSC) y fluorescencia de rayos X (FRX).

Los resultados de la microscopía óptica revelaron que los morteros fueron realizados con un aglomerante de cal, y con agregados de cuarzo, feldespato y biotita junto con fragmentos de rocas graníticos y metamórficos. Los análisis DRX apoyan las observaciones microscópicas y añaden la información de presencias de actinolita en los agregados que indican el mismo origen de las rocas graníticas y metamórficas del entorno geológico de Mérida (España) y muestra la ausencia de biotita en el mortero de suelo y poco cuarzo en el mortero de mampostería de *criptoporticus*. Según los resultados proporcionados con ATG-DSC y FRX los morteros utilizados para la construción del canal frente al Templo, la fundación de sillería de granito y la pared interna de *criptoporticus* tienen alto carácter hidráulico.

Para la conservación futura del monumento es necesario tener presente estas características para el diseño de los morteros de restauración a utilizar.

Palabras clave: conservación, mortero romano, análisis microestructrales y análisis termales.

Introduction

Unfortunately, the practice of heritage conservation is often perceived as the prompt and cursory repairs that coincide with the utilization of restoration work to attract tourism. However, appropriate interventions and compatibility with original building materials are just as indispensable as aesthetic concerns. Therefore, to extend the long term durability of the monuments and historical buildings, detailed characterization of building materials is necessary.

In the conservation intervention decisions of built heritage it is necessary to know the characteristics of building materials to determine the most appropriate restoration techniques (Elert et al. 2002). It is important as much for the joint and filling mortars, coatings, and the foundation as for the building stone. Mortars are the first materials that tend to deteriorate and end up lost, in other words they are the scarifying materials in a building complex. This criterion is necessary to keep in mind in all interventions but especially concerning the monuments of Roman times where mortars were used in construction with a very strict care. Hydraulicity and the proportions of components in the mortars differ according to the function of the construction. These mortars are usually preserved in very good condition until the present day, but it is often necessary to use repair mortars for restoration. For this purpose, it is essential to use lime mortars with the addition of appropriate aggregates that allow the physical, chemical properties and degrees of hydraulicity to be compatible with the original Roman mortars (Rodriques and Henriques 2002; Veiga et al. 2009; Stefanidou 2016).

The most compatible materials are those that have the same composition as existing materials or incorporate compounds that do not generate risks of damaging the original materials (Maravelaki-Kalaitzaki et al. 2005). To this end, there is a tendency to incorporate a variety of organic additive components in repair mortars such as natural fibres or synthetic polymers (Maravelaki-Kalaitzaki 2007; Soufi et al. 2016), inorganic nanoparticles (Rao et al. 2015; Arizzi et al. 2015), or bio-consolidants (Ducasse-Lapeyrusse et al. 2015). Such additives increase the repair mortars' consistency and may improve their qualities by increasing their resistance to deterioration.

Although previous studies have been conducted on the Temple of Diana, no information exists on the composition of the original mortar used to construct this Roman monument. This paper aims to characterize the original mortars with different constructive functions in the Temple of Diana of the Roman city of *Emerita Augusta* with a view to its future restoration.

Temple of Diana

The Temple of Diana is located in the Colonial forum (*forum Coloniae*) (Ayerbe et al. 2009) (Álvarez Martínez and Nogales Basarrate 2003) situated approximately 500 meters northwest of the theatre and amphitheatre of the Roman city of *Emerita Augusta* [figure 1]. The city was founded in 25 BC by the emperor Octavian Augustus as a retreat area for Veterans. When the city was converted into the capital city of the Roman province of *Lusitania*, it reached a great splendour. Currently the city retains many preserved monuments: the theatre, amphitheatre, circus, and several aqueducts, the bridge over the Guadiana River, the House of Mitreo, the Arch of Trajan, neighbourhoods like Morerías, part of the Roman wall, and walkway of the Via de la Plata. Today, the city is called Merida and in1993 was declared a UNESCO World Heritage Site (Barroso and Morgado 1996).

Figure 1.- Map showing the location of Temple of Diana.

The Temple of Diana represents the typology of such temples of the era, having a rectangular plan with a hexastyle *portico*, *peripteros* with 11 columns, which consist of 10 m high Attic bases, fluted shafts and Corinthian capitals that stand upon a 3 meter high podium. The dimensions of the NE-SW oriented temple are 40.75 m in length and 21.90 m in width, its main façade faces south and lies back to *decumanus maximus* of the city [figure 1]. A portico surrounds the temple on three sides (*cryptoporticus*) and encloses a garden (*themenos*). Within the garden area, parallel to the western and eastern fronts, there were two tanks with their channels. The temple was built entirely of granite; the podium has large granite ashlars forming a well-built *opus quadratum*. The *cryptoporticus* was built in granite masonry in regular courses.

With its architectural features, the Temple of Diana fits into the Augustus period or the early years of the Julio- Claudia dynasty (de la Barrera 2000), the lineage having shown their allegiance to the imperial cult (Álvarez Martínez 1991; Mateos Cruz 2006).

Based on archaeological excavations, it is estimated that the monument was abandoned during the 5th century, between period of Constantine and Theodosius (Álvarez Martínez 1991). In the late 15th century the Palace of the Corbos was built inside the temple, using the podium and columns as structural support, maintaining that function until 1972 when it was expropriated by the State. The temple was declared a National Monument on the 13th of December 1972.

Various archaeological excavations have been undertaken in the temple. Among others, the most important were carried out by José María Álvarez Martínez (1972-1975 , 1985-1986 , 1992) and Felix Palma from 2001 to 2011 (Álvarez Martínez 1991; Palma 2003). Based on these excavations, the most significant restoration was undertaken by Jose Menendez-Pidal in 1975, in which architectural elements were replaced with granite, and some parts of the Palace were removed.

Later, several interventions were carried out by Dionisio Hernández Gil between the years 1985 and 1992. A partial reconstruction of the temple was constructed based on the findings of archaeological excavations. There have been recent developments in landscape regulation and urbanization, by the architect José María Sánchez García, which began in 2011.

The objective of this study is to improve the knowledge of the composition and durability properties of the mortars used in the construction of the Temple of Diana, which is necessary for its conservation.

Materials and Methods

Samples of five mortars were taken from different parts of the Temple of Diana according to the function of the mortar in the structure [table 1].

The M1 sample is light brown-beige colour mortar that constitutes 5 mm to 4 cm diameter coarse aggregates (*caementa*) and lime lumps 1-4 cm in size. Poorly sorted aggregates in the porous lime binder are angular to sub-angular, which indicate crushed fragments of the rocks [figure 2]. Sample M2 is a whitish mortar that has coarse 5 mm to 2 cm diameter aggregates (*caementa*). These poorly sorted aggregates are sub-angular to round in shape. The whitish-yellow M3 mortar sample contains 4 mm to 1 cm diameter coarse aggregates (*caementa*), and 1.5 – 4 cm lime lumps. Poorly sorted aggregates in the porous lime binder have angular to sub-angular forms. Sample M4 is whitish in colour and contains smaller, (0.5 to 5 mm diameter) aggregates. Moderately sorted aggregates in the porous lime binder have angular to sub-angular forms, indicating crushed rocks. Sample M5 is whitish-beige mortar with smaller aggregates (0.5 to 5 mm in diameter) and 2 cm lime lumps. Moderately sorted aggregates in the porous lime binder have angular to rounded forms. Although no ceramic fragments were visible to the naked eye, lime lumps were observed in the samples M1, M3, and M5.

Samples were characterized through different analytical techniques including Polarised light optical microscopy (POM), X-ray diffraction (XRD), X-ray Florescence (XRF) and thermogravimetric analyses.

Figure 2.- Image of Temple of Diana.

POM was conducted on an Olympus BX51 microscope fitted with an Olympus DP12 digital camera. Entire mortar samples, including binder and aggregates were analysed using this method.

Mortars were grounded and the powder samples were divided into three groups. The first group of samples were used in XRD analyses. A Bruker D8 Advance X-ray diffractometer fitted with a copper anode tube and PC-ADP diffraction software was utilized. XRD patterns were acquired by operating at 40 kV and 30 mA at 2θ angles of 2–68° with a 0.020-step scan, at a speed of 2° per minute, using a CuKα radiation and a graphite monochromator. EVA X-ray diffraction analysis software was utilized for the interpretation of graphs.

The second group of powder samples were formed into pellets for use in the XRF analyses. A portable (EDTRX) THERMO NITON model XL3T X-Ray Fluorescence device was used. For a better comparison with the literature, the results were converted into traditional XRF desktop equipment results, according to the correlation methodology implemented in the previous research (Ergenç et al. 2016).

Hydraulicity cementation indices were calculated for every sample to gain insight into the degree of hydraulicity [equation 1, equation 2].

Table 1.- Percentage content (%) of major elements and Hydraulicity Index (HI) and Cementation Index (CI).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	HI	CI
M1	33.65	3.54	3.17	25.69	4.63	1.33	3.12
M2	21.88	2.21	1.39	35.65	5.33	0.62	1.50
M3	38.95	4.15	3.28	20.51	<LOD	2.26	5.65
M4	18.39	2.72	4.01	39.44	<LOD	0.64	1.45
M5	37.84	2.78	2.53	21.61	<LOD	2.00	5.13

Equation 1. Hydraulicity Index (HI) (Boynton 1980; Elert et al. 2002).

$$HI = \frac{SiO2 + Al2O3 + Fe2O3}{CaO + MgO}$$

Equation 2. Cementation Index (CI) (Eckel 2005; Lindqvist 2005; Elsen et al. 2012)

$$CI = \frac{2.8SiO2 + 1.1Al2O3 + 0.7Fe2O3}{CaO + 1.4\,MgO}$$

The third group of powdered mortar samples was analysed using Thermal analysis (TG-DSC), performed with a TA Instruments SDT-Q600, DSC Q-200 and General V4.1C DuPont 2000 thermogravimetric analyser, in a nitrogen atmosphere at a heating rate of 10 °C/min.

Results and Discussion

—*Polarized Optical Microscopy*

Microphotographs show the aggregates of quartz, calcite, feldspar, biotite, and rock fragments encased in a cryptocrystalline micritic matrix, that indicates use of a lime binder. Except for sample M3, all other observed mortar binders show dark-brown colour and carbonated appearance under the microscope. Granitic rocks, quartzite, slate and schist comprise the observed rock fragments [figure 3], which coincides with the geology of the surroundings (Robador and Arroyo 2013; Mota López 2015). Regarding the fragments; sample M1 contains polycrystalline quarzitic rock fragments, sample M2 has elongated metamorphic rock fragments with rounded edges and rounded opaque minerals, samples M3 and M4 has bigger edged feldspar grains, granitic rocks , and elongated schist, slate and limestone were observed in sample M5 [figure 3].

According to visual examination of thin sections, binder aggregate ratios are estimated 1:4 in M1, M2 and M5 and 1:3 in M3 and M4. 1 to 3 cm lime lumps are encountered only in the samples M1, M3 and M5. Presence of lumps either indicates dry slaking, lack of water in slaking, or poor workmanship (Silva et al. 2006; Moropoulou et al. 2016).

—*X-Ray Diffraction*

The mineral compositions of mortar samples determined by X-ray difraction is presented in figure 4. Difractograms of the samples show that the main constituents of the mortars are quartz, calcite, and feldspar (albite, anorthite). Biotite is the accessory mineral, which is omnipresent in all samples except M2, and is present in greater intensity in sample M3. Proportions of calcite and quartz vary in every sample: in M1, M3 and M5 quartz is predominent, while M4 has very a little amount of quartz. M1, M4 and M5 have the same peaks of actinolite (Ergenc and Fort, in press).

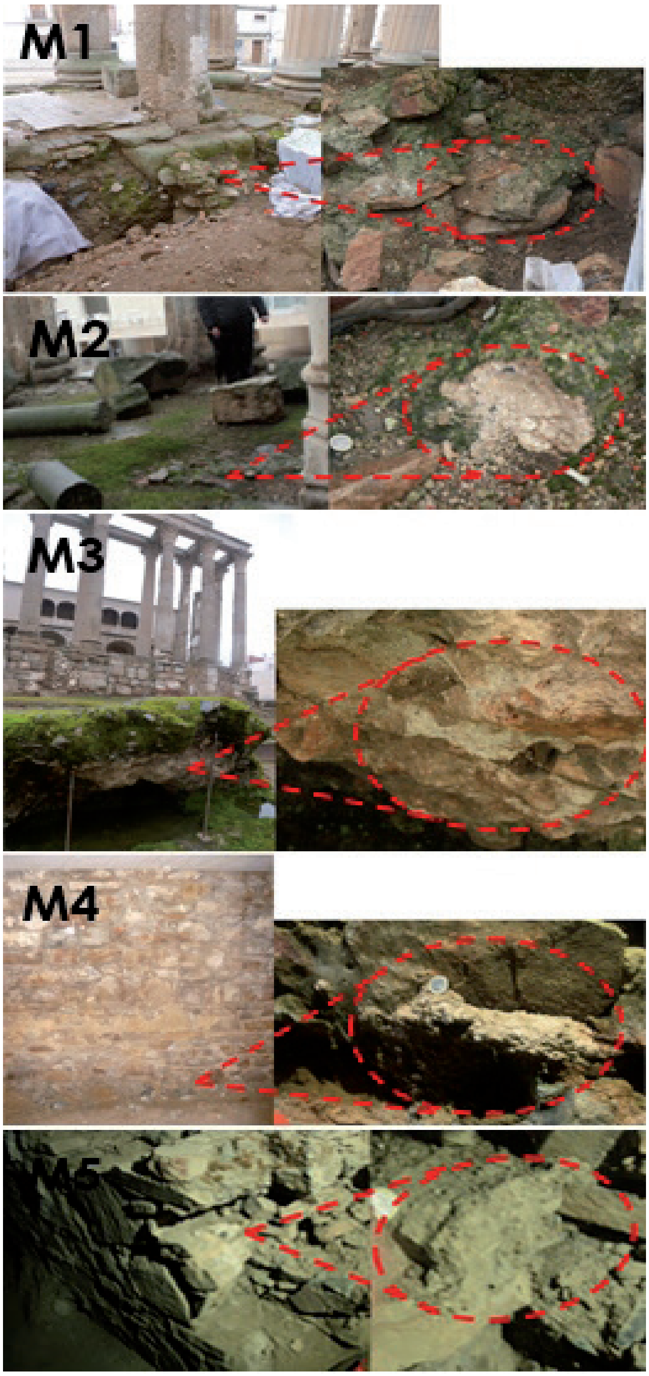

Figure 3.- Photos of collected samples with their location in the temple M1: Foundation of the granite ashlar, M2: Flooring, M3: Vault of fountain or channel in front of the temple, M4: Masonry wall of Criptoporticus, M5: Inner wall in Criptoporticus.

Results of X-ray diffraction analysis supports the petrographic analysis under polarized optical microscopy. In the XRD patterns, calcite, coming from the binder and quartz, feldspar, biotite and actinolite arereleased due to the granitic and metamorphic rocks that were used as aggregate.

—*X-Ray Fluorescence*

According to results of chemical analysis of principal major elements, samples M3 – M5 and M2 – M4 can be grouped

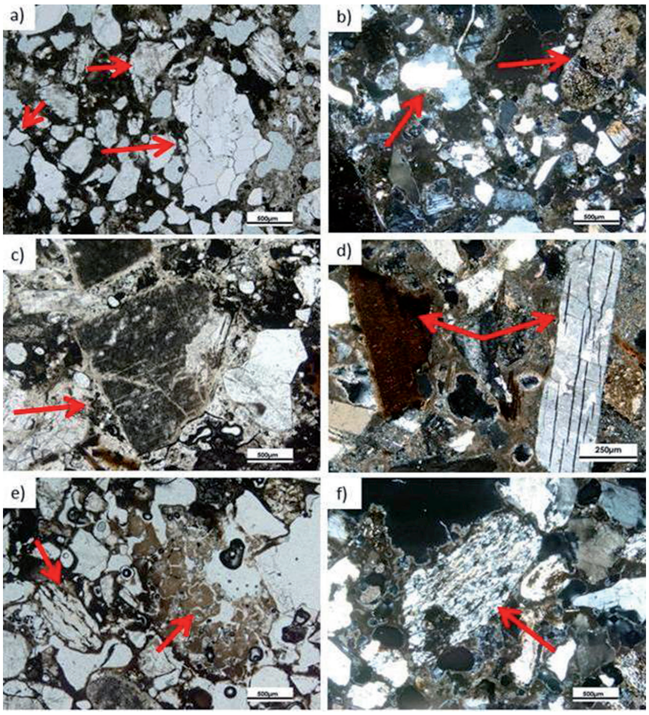

Figure 4.- a) Quartz and feldspar grains together with quartzite fragment embedded in carbonated lime binder in M1 under plane polarized light b) dark brown coloured lime binder with rounded to angular metamorphic rock fragments and quartz and feldspar grains in M2 under cross polarized light c) square edged altered feldspar in M3 under plane polarized light d) Biotite on the left and fragmented plagioclase on the right in M4 under cross polarized light e) Lime lump with recrystallization in its cracks in M5 under plane polarized light f) Schist fragment, quartz feldspar aggregates and large pores in M5 under crossed polarized light.

together. M3 has the highest hydraulic and cementation properties, which is due to the presence of the alkali reactive calcium-aluminum-silicate-hydrates (C-A-S-H). The highest MgO values are detected in the M1 and M2 samples, whereas the highest CaO and Fe_2O_3 and the lowest SiO_2 values are seen in sample M4 [figure 5]. M2 and M4 have the least hydraulic character with low HI and CI values (Elsen et al. 2012).

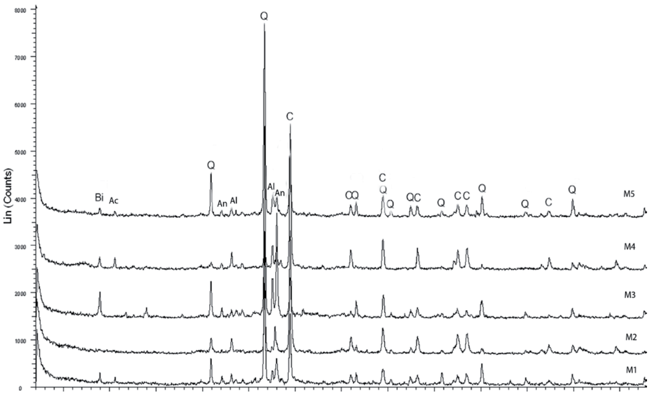

Figure 5.- XRD Patterns of mortar samples of Temple of Diana (Q: quartz, C: calcite, Al: albite, An: anorthite, Bi: biotite, Ac: actinolite).

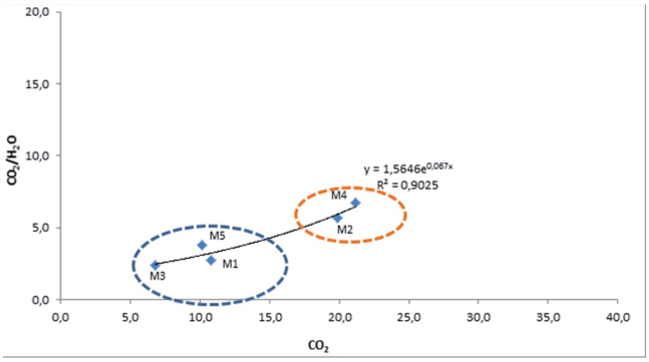

Figure 6.- CO_2/H_2O vs CO_2 plot.

—Thermal Analyses

Weight-loss percentages were calculated from the TGA–DSC curves within selected temperature ranges. In the temperature range below 120°C weight-loss is due to adsorbed water; from 120°C to 200 °C the weight-loss of water from hydrated salts or from gypsum occurs; between 200°C and 600°C the weight loss is due to structurally bound water from the hydraulic compounds and finally, the weight loss due to the release of CO_2 as a consequence of the decomposition of calcium carbonate ($CaCO_3$) takes place at temperatures above 600°C (Moropoulou et al. 2000; 2016).

The hydraulicity of mortars is represented by the inverse relation of decomposition of carbonates (CO_2 - weight loss% above 600°C) to structurally bound water (H_2O - weight loss% between 200°C – 600°C) (Bakolas et al. 1995; Moropoulou et al. 2000; 2016; Biscontin et al. 2002; Böke et al. 2008). Figure 6 shows the exponential increase of inverse hydraulicity with CO_2. This correlation allows classification of mortars according to their hydraulic properties. Since none of the samples show CO_2/H_2O higher than 10 and higher CO_2 values, this demonstrates that all have hydraulic character.

As shown in figure 6, two groups are glittered: the first group includes samples M1, M3 and M5, which are suited to 5 – 15% CO_2 and CO_2/H_2O below 5%, and the second group including samples M2 and M4 fall into the ranges of 15 – 25% CO_2 and 5 –10% CO_2/H_2O. First group can be classified as highly hydraulic (Bonazza et al. 2013) or natural pozzolanic mortars (Moropoulou et al. 2000; 2005; Genestar et al. 2006). The second group can be classified as hydraulic mortars (Bonazza et al. 2013), or artificial pozzolanic mortars (Moropoulou et al. 2000; 2005; Genestar et al. 2006).

According to Moropoulou et al. (2000) weight-loss after 700°C implies the complete decomposition of calcium carbonates in mortar as additive or aggregate or re-carbonation, so weight-loss near 750°C of in sample M3 indicates re-carbonation of lime concerning the lighter colour binder observed under the microscope as no calcitic aggregate is observed. Samples M1, M3 and

M5 reveal exothermic peaks between 200°C and 400°C which indicate C-A- S-H formation (Bruno et al. 2004) and show well marked transformation peaks of quartz at 573°C.

Conclusions

In this study, various analytical techniques were utilized to characterize mortars that were collected from the Roman Temple of Diana in Mérida. Results of macroscopic and microscopic analysis show that they are lime mortars with moderately to poorly sorted aggregates embedded in a cryptocrystalline micritic matrix which indicates an original lime binder composition. Quartz and feldspar grains are the main constituents in aggregates. In addition, the presence of actinolite in the diffractograms indicates the provenance of the aggregates is the granitic and metamorphic rocks from the local surroundings. XRD analysis also shows the absence of biotite in the flooring mortar and rarity of quartz masonry mortar used to construct the *criptoporticus*. According to TGA-DSC and XRF analyses, mortars were used in the channel in front of the Temple, the foundation of granite ashlars and the inner wall of *criptoporticus* has higher hydraulic character. Flooring and masonry mortars consist of predominantly poorer raw materials and so have lesser hydraulic properties. For the implementation of the conservation and restoration of the temple, repair mortars should be prepared in accordance with the characteristics revealed by this study.

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