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Composition and porosity study of original and restoration materials included in a coastal historical construction



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H I G H L I G H T S

- New materials used for the restoration works of a building close to the sea were studied.
- Four different kind of materials were analyzed.
- For the material chemical characterization XRD, Raman microscopy and WD-XRF were used.
- The physical and mechanical test based on MIP and IRT was also carried out.
- Rendering mortar (micropores < 0.1 μm) is more porous than other restoration materials.

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A B S T R A C T

The materials used for the building constructions can suffer during their life numerous deterioration processes induced by different anthropogenic and natural factors. For this reason, optimal restoration works are vitally necessary for a correct Built Heritage preservation process. One of the most critical environment is the marine atmosphere (marine aerosol impact following dry and wet depositions) where the climatic conditions are very aggressive. For this reason, the new building materials that will be used for a restoration must be selected taking into account the possible deterioration sources that are present in the vicinity of the building or construction. Among marine aerosol, other factors such as biodeterioration, water infiltrations, physical stress, even pollutants than can be deposited from the surrounding environment must be taken into account. This work is focused on the analytical and physical study of the different building materials used for the restoration works carried out in 2014 in the Tower of La Galea Fortress (Getxo, Basque Country, Spain), a historical construction placed in front of the sea. The analyzed materials were the weathered sandstone (original sandstone) and the one used to replace it (restoration sandstone), as well as restoration joint and rendering mortars. The results presented in this work offered a global idea of the correct material selection, taking into account the different factors that can affect the integrity of the whole building in the future. In order to test the durability of these materials against the surrounding environment, different analyses were carried out. On the one hand, X-ray Diffraction (XRD), Raman spectroscopy and Wavelength Dispersive X-ray fluorescence (WD-XRF) were used for chemical characterization of the materials. On the other hand, Mercury Intrusion Porosimetry (MIP) was used to understand the porous system and the thermal behavior was studied using Infrared Thermography (IRT). Finally, the thermal fatigue test was carried out to determine if temperature cycles could have impact in the structural integrity and aesthetic appearance (i.e. color changes) of these materials during time.

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1. Introduction

Restoration of historical buildings is an intervention that seeks above all respectful recovery of the cultural heritage, requiring specialists in the field for the development of projects, conducting relevant research and the necessary analysis and supervision of the work, in order to conduct the interventions proposed in the project correctly.

As it is known, historical buildings, through their materials and construction systems, are subject to suffer during time the action of different agents that alter them. These alterations can take place through the affection of its aesthetic appearance (visual and or conceptual) and by the action of physical, chemical, biological and/or human or anthropogenic agents that produce deterioration (physical or chemical degradation) promoting a progressive loss of the useful property. These agents can be presented acting isolated (in the minority of cases), one followed each other, or through the simultaneous or synergistic combination of several of them (in most cases). Therefore, the alteration will depend both on the composition of the construction material and on the nature of the agents acting on it [1,2].

There are different causes and alteration agents that product impact on buildings and constructions. The causes can be classified as extrinsic and/or intrinsic. On the one hand, the extrinsic causes comprise both agents that have long-term action on the property such as wind [3], UV exposure [4], weather conditions [5], water infiltrations [6], air pollutants [7–9], frost action [9–12], temperature or thermal variations [13,14], etc. as well as natural disasters (earthquakes [15], hurricanes [16]), floods [17], fires [18] and biological agents [19,20]. On the other hand, the intrinsic causes to the building are those related to the position of the building and inherent to its structure. These agents can be physical [21,22] and chemical [23,24] or biological [25], which can produce a series of pathologies in the building.

For these reasons, it is crucial the optimal selection of the restoration materials and the previous studies carried out in the original materials that will be replaced or restored inside the building or construction under study. In order to avoid the reiteration of the same decay patterns after restoration, the knowledge of such materials regarding their chemical composition, durability, petrographic composition, texture, physical properties, etc. is necessary to know how they will be preserved in the future, and the suitability of the restoration techniques (material's compatibility and lifespan) to be applied are required. In this way, the combination of the analytical and physical techniques seemed to be an appropriate methodology to undertake such study. Examples of this are the previous researches carried out in La Galea Fortress, all of them using different analytical tools, for the characterization of the different deterioration processes caused mainly by the influence of marine aerosol (wet and dry deposition) [26], infiltration waters and salts migrations [27], atmospheric acid gases impact and accumulation of hazardous elements [28], biological colonizations [29] and non-appropriated selection of the materials used for La Galea Fortress construction [30].

Moreover, in addition to the elemental/molecular characterization of the materials used for restorations, the knowledge of the pore systems used in construction materials is also crucial due to their influence on physical and mechanical behaviors and consequently on their durability [31,22]. In this way, the pore size distribution is an useful parameter that allows to predict the decay of construction materials when they are exposed to water [32], acid atmospheres [33], even to predict the materials thermal fatigue [34,13]. Therefore, related with the aesthetical damages, many works deal with the color changes in the construction materials exposed to different decay environmental factors [35–36,8] or pos-

sible impacts to fires [37], observing the different response of the material. This also helps to know the possible changes in their appearance during time.

In this work, an analytical and physical study of the original materials and the new materials used for the restoration conducted in the Tower of La Galea Fortress (Getxo, Basque country, Spain) is presented. In order to test the durability of these materials in the actual surrounding environment, chemical analysis by means of X-ray Diffraction (XRD), Raman Spectroscopy (RS), Wavelength Dispersive X-ray fluorescence (WD-XRF), and a more physical study of them using Mercury Intrusion Porosimetry (MIP) and Infrared Thermography (IRT) were applied.

2. Materials and methods

2.1. La Galea Fortress

La Galea Fortress is located in the cliff overlooking the Bay of Abra, in Getxo (north of Spain) at about 50 m above sea level. The Fortress is an 18th century military building and took the place of an old watchtower that already existed in the 16th century. It is considered as one of the most important military building in Biscay and Basque Country. The sandstone was the chosen material for the building erection in the 18th century. Due to the poor preservation state several pathologies were observable, and consequently in 2014 a restoration was conducted using new materials. The climatic conditions of the location where La Galea fortress is immersed are the following ones: average temperature of 18.3 °C, average minimum temperature of 7 °C and average maximum temperature of 20.3 °C. Moreover, the annual precipitations are around 1200 mm in which at least, during 128 days rainfall above 1 mm are recorded. The annual average relative humidity is around 72%, but in some periods the humidity reaches 90% or higher values. The annual sunshine hours are 1532 and the average wind speed is 5.6 kts, where the two preferential wind directions are the South-southeast (SSE) and the West-northwest (WNW) [38].

2.2. Samples

To undertake this study, four different samples were analyzed: the original sandstone (originally maintained thanks to its good conservation state), the restoration sandstone (replaced) and the new added restoration joint and rendering mortars. Observing the preservation state before and after the tower restoration (see Fig. 1), the differences were clear. Before restauration, an intense erosion of the original sandstone was evident on the entire height of the tower, with typical associated pathologies such as alveolarization accelerated by the wind and salts presence. In addition, salt efflorescences were found on the sandstone as expected in marine environments.

2.3. Instrumentation

XRD analyses were performed with a powder diffractometer PANalytical Xpert PRO, equipped with a copper tube ($\lambda_{\text{CuK}\alpha\text{media}} = 1.5418 \text{ \AA}$, $\lambda_{\text{CuK}\alpha1} = 1.54060 \text{ \AA}$, $\lambda_{\text{CuK}\alpha2} = 1.54439 \text{ \AA}$), vertical goniometer (Bragg-Brentano geometry), programmable divergence aperture, automatic interchange of samples, secondary monochromator from graphite and PixCel detector. The measurement conditions were 40 kV and 40 mA, with an angular range (2θ) scanned between 5 and 70°. For the data treatment of the diffractograms and the identification of the mineral phases present, the specific software X'pert HighScore (PANalytical) in combination with the

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