



In-situ analytical study of bricks exposed to marine environment using hand-held X-ray fluorescence spectrometry and related laboratory techniques

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ABSTRACT

In this work, the degradation processes that take place in bricks exposed to marine environments have been studied. Taking into account the importance of this building material where the silicates present in the final product act as stabilizer in the porous material itself, it is necessary to understand the decay processes that occur in these aggressive environments. As is known, the marine aerosol carries different types of salts, such as chlorides, sulfates, nitrates, etc., present in surrounding environment exerting a negative influence on the materials producing cracking and disintegration processes of the material and consequently loss of brick wall stability. Nowadays the development of portable devices is taking much more importance helping researchers to resolve problems in the field in a fast and easy way. In order to extract fast and satisfactory results about the conservation state of different bricks from Punta Begoña Galleries (Getxo, Basque Country, Spain), an in-situ analytical methodology was developed based on the use of hand-held Energy Dispersive X-ray fluorescence spectrometry (HH-ED-XRF) assisted with other laboratory techniques (μ -ED-XRF and X-Ray Diffraction) in order to corroborate and complement the information obtained in-situ. This construction undergoes the influence of marine aerosol, industrial port, power generation plants, and a fuel refinery among others. The pathologies visually observable in these bricks are disintegration, breakup and detachment of the bricks. The presence of deterioration compounds in the bricks has been studied according to the orientations of the bricks inside the construction.

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1. Introduction

Since ancient times, bricks are some of the most used materials in constructions. In 5000 BCE, the brick appeared as building material, and around 3500 BCE, the fired brick was finally created. Thanks to the industrialization in the 19th century, the use of brick as building material increased and acquired great importance, being one of the most used material for the building constructions [1].

Bricks used for constructions are made from clay, which is kneaded with 20–25% water. The resulting paste is molded and the bricks obtained are dried in the air [2]. Although several processes of bricks production exist, various mixtures of raw clays (mainly composed of illite-chlorite, quartz, calcite and/or dolomite, sodium and potassium feldspars, and iron oxides/hydroxides) are fired at about 900–1025 °C in order to obtain the final brick [3]. Their characteristic reddish-orange color, is indicative of the amount and type of iron oxides and

oxyhydroxides present in the clay used for its manufacture [4]. However, other chemical elements such as Sb, Pb, Ca, Fe and traces of Cu have a key role in the reddish orange color of the bricks [5]. In this sense, the composition and microstructure of the bricks are some of the most important characteristics to preserve their durability [6,7]. Moreover, the clay must have a low amount of impurities and especially must have a low content of calcium carbonate to be plastic enough, and to avoid the presence of unreacted lime after firing. The presence of calcium carbonate in the bricks can cause cracks when the clay is mixed with water. For this reason, usually the clay must contain calcium carbonate in a proportion of <8–10%. During firing, a series of mineralogical transformations take place depending essentially on the initial composition, kiln temperature, heating grade, firing time and prevalence of oxidizing or reducing reactions [8–10] redefining the mineralogical composition and microstructure. In this way, after firing at 900–1025 °C, kaolin is dissociated into silica and alumina and also into calcium and aluminum silicate. One of the critical factors in the conservation state of bricks is the content of calcium silicate that provides stability to the structure and resistance to temperature changes. Consequently, the bricks fire or

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cooking temperature is established as one of the most important steps in the bricks construction [11,12].

Bricks, as all building materials, can experiment different chemical reactions that could promote changes in their appearance and conservation state. Bricks can suffer the effect of humidity [13,14], biocolonizations [15], the attack of atmospheric acidic gases [16], infiltrations by aqueous solutions [17–19], the impact of marine aerosols [20,21], dry and wet depositions [22–24], freeze-thaw cycles [25], etc. These sources of deterioration, can promote several chemical and physical problems as well as efflorescences formation [17], loss of material [26], etc.

In the literature, there are many works that analyze bricks in order to extract conclusions about their conservation state [27,28], the provenance [29], original composition [30], the brick composition influence on decay compounds [31], etc. Moreover, different techniques such as X-Ray diffraction (XRD) [32], Raman spectroscopy [1,19,33], infrared spectroscopy [34,35], Inductively Coupled Plasma Mass Spectrometry (ICP-MS) [36], Laser ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) combined with Instrumental Neutron Activation Analysis (INAA) and X-ray fluorescence (XRF) [37], XRF [38] and Laser-induced breakdown spectroscopy (LIBS) [39], have been used to carry out these types of analyses. In all these case studies, the analytical instruments used were laboratory benchtop devices and there are no works that use hand-held/portable instruments to analyze bricks and to evaluate their pathologies. Sometimes the extraction of samples is a critical step, mainly due to sampling restrictions. For this reason, the application of portable devices is taking much more importance, helping researchers to carry out different in-situ analyses obtaining satisfactory results without the necessity of taking samples [18,40–42]. Example of these types of devices are the hand-held Energy Dispersive X-Ray fluorescence spectrometers (HH-ED-XRF), for which its use is being increased in the last years, because they are user friendly and offer results in few seconds or minutes [43–45]. As a consequence of this, many worldwide scientific groups are improving and developing new analytical methodologies based on these kinds of devices [46,47].

In this work, a simple, fast and non-destructive methodology based on the use of a hand-held Energy Dispersive X-ray fluorescence spectrometer (HH-ED-XRF) was applied to perform a field analysis of bricks from Punta Begoña Galleries (Getxo, Basque Country, Spain) which show different deterioration degrees depending mostly on their location in front of the industrial port of Bilbao and diverse factories emitting atmospheric pollutants. In order to confirm and complement conclusions extracted in-situ, additional analyses were conducted in the laboratory using micro-ED-XRF spectrometry and X-Ray diffraction (XRD) techniques.

2. Experimental

2.1. Punta Begoña Galleries

Punta Begoña Galleries are located in Getxo, close to Ereaga beach, in Biscay (Basque Country, Spain). These galleries were built in 1918 by an industrial tycoon, Horacio Echevarrieta [48]. It is one of the most significant construction in Biscay and Basque Country of the 20th century. Nowadays, it is abandoned and it is poorly preserved (see Fig. 1). The construction is composed by two galleries: the upper gallery or “Northwest Gallery” (NG) and the lower gallery or “Southwest Gallery” (SG). All the construction is placed in front of the sea; there is a beach in front of the NG and a leisure port in front of the SG, together with the industrial port of Bilbao at 1–8 km and several factories (refinery, power station plant of liquid hydrocarbons, and metallurgical companies), emitting acid gases and metallic particles to the atmosphere, at not >10 km from the building. The whole building is made by different materials such as concrete, ceramic, mortar, cement, limestone and brick.

2.2. In-situ analyses

The in-situ analyses were carried out in both galleries by means of HH-ED-XRF. For this purpose, seven areas from the Northwest Gallery (coded samples NG1–NG7) and additional seven from the Southwest Gallery (coded samples SG1–SG7) were selected (see Fig. 1). As can be observed in Fig. 1, bricks show a better or worse conservation state depending on the orientation.

2.3. Instrumentation

The in-situ elemental analysis of the bricks was carried out using an XMET5100 (Oxford Instruments, UK) hand-held energy dispersive X-Ray fluorescence spectrometer (HH-ED-XRF). The instrument is equipped with an Rh tube working at a maximum voltage of 45 kV. The spot size of the emitted X-Ray beam is 9 mm. The analyzer includes a Peltier-cooled silicon drift detector (SDD) of high resolution that is able to provide an energetic resolution of 150 eV (calculated for the Mn K_{α} line). The analyzer contains a PDA to control the spectrometer and also to save the spectra and semi-quantitative information. To determine the presence of the heaviest elements ($Z > Ti$) the voltage and current of the X-Ray tube was set at 40 kV and 15 μA respectively and the spectra were acquired during 100 s (real time) in order to improve the limit of detection for the identification of trace elements. Additionally, to remove the Bremsstrahlung and possible Bragg diffraction peaks, a 500 μm Al filter was used. In order to improve the detection of the lighter elements ($Z < Ti$), additional measurements were performed without the Al filter and at lower voltage (13 kV) and higher current (40 μA) during 70 s. In order to determine possible contributions from detector materials and possible contaminations of the XRF analyzer window, 20 repetitive spectra of an instrumental blank (a PTFE block) were acquired before each measurements batch. For the repetitive measurements, the same spectral conditions (voltage, current, filter and test time) as those used for the analysis of the bricks were considered. Although the software is based on the use of Fundamental Parameters quantification methods, in this work, the net counts of K_{α} lines of each detected element in the spectrum were considered following a normalization process. See elsewhere for further details [43]. To extract representative results, ten measurements (9 mm area each one) were performed randomly distributed on seven areas from the NG (NG1–NG7) and on additional seven from the SG (SG1–SG7). In order to evaluate the elemental composition of the bricks without extracting any sample, semi-quantitative information given by the HH-ED-XRF was avoided. Instead of that, a specific spectral data treatment based on the normalization of the net counts of each detected element was conducted [43]. For all the detected elements, net counts associated with K_{α} line were considered, except for Pb for which L_{β} line was used. Considering that Fe is a major element which showed an almost constant concentration in all the measured areas, its Fe K_{α} line was used to normalize the net counts of light elements ($Z \leq 20$) in the spectra. For the heaviest elements ($Z \geq 26$), the net counts of their K_{α} lines were normalized against the net counts of the Compton line (incoherent scattering). This normalization process was done in order to correct possible matrix effects and variations in the positioning of the measuring sampling interface. In order to avoid spectral interferences between the As $K_{\alpha 1}$ and Pb $L_{\alpha 1}$ lines, $K_{\beta 1}$ (11.7 keV) and Pb $L_{\beta 1}$ (12.6 keV) lines were considered for As and Pb respectively.

In order to extract more conclusions, the elemental composition was also studied in the laboratory by using the M4 TORNADO Energy Dispersive X-ray Fluorescence spectrometer (Bruker Nano GmbH, Berlin, Germany). This instrument is equipped with a micro-focus side window Rh X-ray tube powered by a low-power HV generator and cooled by air. The spectral acquisitions in this work were performed at the maximum voltage (50 kV) and current (600 μA) that the X-ray source allows. This equipment can work using polycapillary lenses, which allow performing both single point measurements down to 25 μm of lateral/spatial

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