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X-ray tomography and chemical–physical study of a calcarenite extracted from a Roman quarry in Cartagena (Spain)

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ABSTRACT

This paper is focused on a calcarenitic stone quarried from ancient time in Cartagena, Spain. The chemical and mineralogical characterization of the stone is performed by X-ray spectrometry and thermogravimetric analysis. Microstructural features of interest, such as open/closed porosity, variation in partial porosity and equivalent diameter of the pores were studied using X-ray tomography in combination with 3D analysis software (Morpho+). The segmentation of the air phase confirmed that the stone contains interconnected pores classified as open pores in Morpho+. Scanning electron microscopy and 3D software for visualization showed that the stone microstructure is mostly composed of micrite, sparite, quartz and skeletal grains. Ultrasonic pulses, uniaxial compressive tests and capillary water absorption measurements were performed in samples extracted from a sane block. Consolidation by means of sonicated $Ca(OH)_2$ water–isopropanol suspensions improved the stone's physical behavior and particularly, its capillary water uptake.

1. Introduction

The Tabaire stone is a calcarenite quarried in Canteras a locality of Cartagena, Spain. The inspection of the Roman quarry (Figure 1a) shows deterioration patterns that are normally observed in calcarenitic rocks, such as alveolization (Figure 1b), sanding and peeling (Figure 1c) (ICOMOS-ISCS, 2008). Moreover, salt crystallization is frequently observed in deteriorated blocks of monuments built with this type of stone (Cardell et al., 2008). The quarries were exploited by Carthaginians and Romans as can be seen in the Punic wall (225–220 BC) or the Roman theatre of the city. Despite the fact that the stone was widely used in historical buildings, little is known about its fundamental properties. As many other sandstones, the stone is prone to weathering (Camuffo, 1995; Siedel et al., 2010; La Russa et al., 2011; Anania et al., 2012) so that, protective methods are needed to preserve the blocks' surface in historical buildings and constructions (Cultrone et al., 2007; Ventolà et al., 2012).

Conventional microstructure analysis of rock samples is usually carried out by studying polished 2D thin sections with optical microscopy or scanning electron microscopy (Cnudde et al., 2011a). However, when looking at two dimensional images, it is necessary to keep in mind that the shape of minerals and pores and their spatial relationships can be much more complex in three dimensions. Although it is possible to interpret data derived from 2D images by powerful quantitative tools for the characterization of 3D structures, many quantitative and qualitative aspects of structures remain inaccessible from 2D images (Russ, 2002). In order to overcome this problem, 3D characterization tools like micro-CT in combination with powerful 3D analysis software, like Morpho+, can be used. The advantages of this technique reside in the fact that X-ray CT is an ideal tool to characterize the internal structure of a rock in 3D in a nondestructive way at resolutions up to submicron scale (Cnudde and Boone, 2013). Although petrographic investigation of geomaterials by means of optical microscopy remains very important for a detailed microscopical study, it remains largely restricted to 2D observations as acquisition of direct 3D is very labor intensive by means of, for example, serial sectioning (Cnudde et al., 2011b). Micro-CT has the advantage of nondestructively providing stacks of more than 1000 2D cross-sections of the sample under investigation at high resolution in a much faster and more accurate way than serial sectioning. When one wants to study the pore-size distribution in detail, besides micro-CT other important techniques exist, including the Brunauer–Emmett–Teller (BET) technique, image analysis of thin sections, mercury intrusion porosimetry (MIP) and NMR microscopy (Cnudde et al., 2009a). All these analysis techniques, including micro-CT, have their advantages and disadvantages. By combining the correct techniques often is the best way to clearly characterize a material.

The use of X-ray microtomography (micro-CT) in characterizing building stone has been reported by Jacobs et al. (1995) and Jacobs and Cnudde (2009). In micro-CT, the scanner acquires multiple 2D X-ray absorption images (projections) that are recorded by a detector while the sample rotates (Landis and Keane, 2010). Once the X-ray absorption images are acquired, they are reconstructed using a

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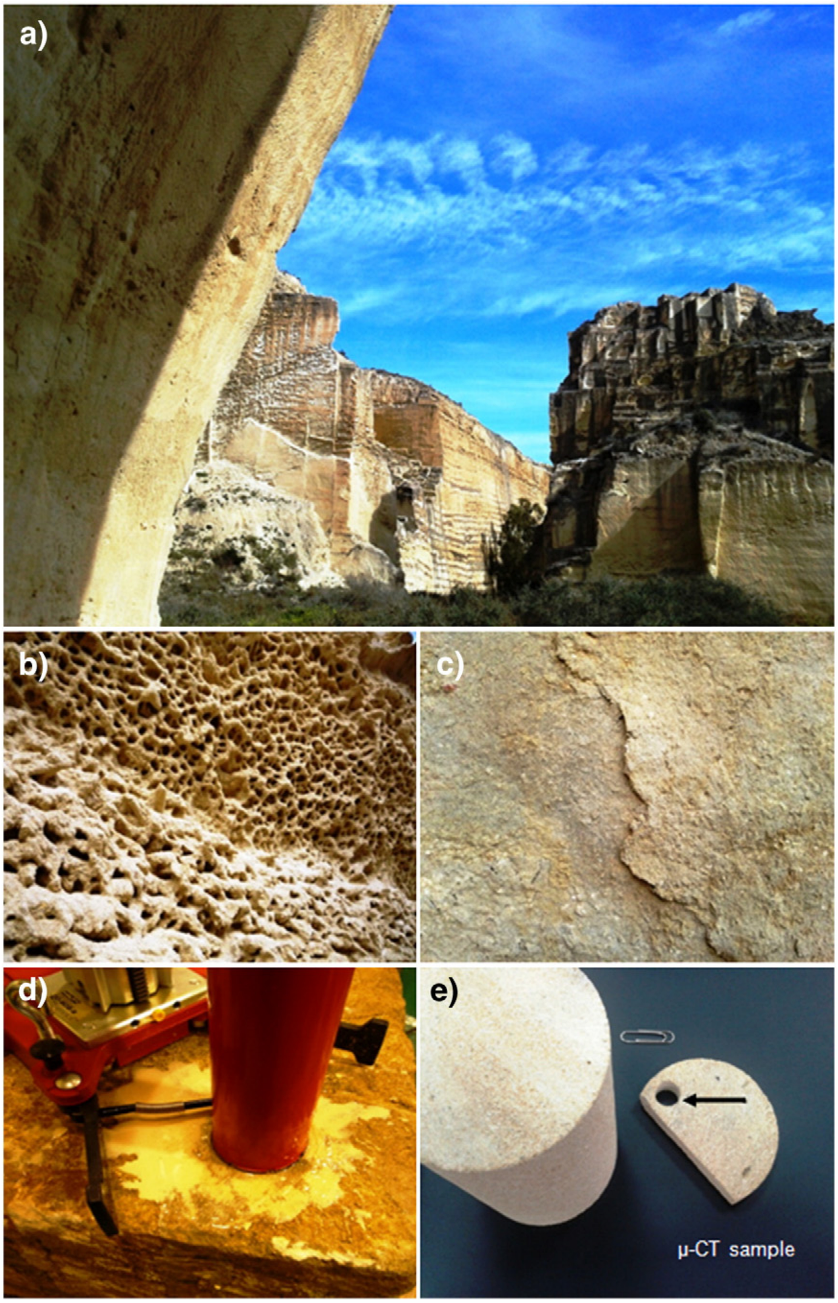

Fig. 1. a. General view of the quarries; b. *Alveolization*; c. *Sanding* and *peeling* deterioration; d. Drilling machine extracting cylindrical cores to carry out physical tests; e. Subsample used in the micro-CT measurements.

mathematical procedure from hundreds of the projections taken at different angles. The phases are differentiated from each other by their brightness, which is a function of X-ray absorption. Therefore, in micro-CT images the dark spots correspond to lower density phases such as voids or fissures, whereas the lighter areas correspond to solid components (Christe et al., 2011). The resolution of the images is expressed in voxels and the acquired 2D slices can be treated with software for 3D analysis. In this way, micro-CT in combination with analysis software has been used to evaluate open/closed porosity and pore size of geological materials (Cnudde et al., 2009a, 2011b; Cnudde and Boone, 2013). Micro-CT has been also used for visualization of water movement inside sandstone and to study the distribution of water repellents and consolidants in natural stone (Cnudde et al., 2009b; Ruiz de Argandoña et al., 2009). Creation of non-destructive three-dimensional imaging is one of the most important advantages of micro-CT since relevant properties inside the studied material can be visualized with special software to create 3D solids (Cnudde and Boone, 2013).

A number of consolidants, such as lime water, barium hydroxide, polymers, silicone-based treatments, epoxies and acrylics, are used for stone conservation (Doehne and Price, 2010). The selection of a consolidant is not an easy task since many requirements must be fulfilled. A consolidant must be effective and compatible with the substrate as well as easy to use, safe and durable (Hansen et al., 2003). Besides, the color of the original substrate should be preserved as much as possible. Lime water treatments are widely used in the conservation of limestone and related materials. In lime water consolidation, a saturated solution of calcium hydroxide, $Ca(OH)_2$, is applied to the stone to form in situ $CaCO_3$. The new material formed within the pores is similar to the stone and therefore, compatible with the original substrate. On the