Micro-CT and Mercury Intrusion Porosimetry Characterization of the Fabric of Roofing Slate

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ABSTRACT

The petrological fabric of roofing slate determines the main properties of the slate as a construction material, such as durability and waterproofing. Roofing slates are rocks derived from the low-grade metamorphism of lutites, with a characteristic lepidoblastic fabric. However, little attention by petrologists has been paid to the role of this fabric in the behaviour of roofin slates. This work characterizes the fabric and pore system of two roofing slate varieties from Spain, using two different techniques, each with its advantages and disadvantages: X-ray microtomography, useful to find heterogeneities and mineral inclusions on the slate bulk,and mercury intrusion porosimetry, which defines the pore system. The differentiation of mineral inclusions is very useful for predicting the weathering of a slate, while the definition of the pore system may help to understand how the slate will behave during its service life.

1. INTRODUCTION

Roofing slates are a very popular construction material in both historic and modern buildings all over Europe (Prieto et al., 2011). Nowadays, most of the production is located in Spain (García-Guinea et al., 1997), with about 80% of the world´s production. The characteristics that make a slate suitable for roofing are a fine textural homogeneity, which also gives waterproofing, and a well-defined, slaty cleavage, which gives a characteristic lepidoblastic texture to the slates, making it possible to manufacture plane and light tiles. These two features determine the pore system of the slates, which is closely related with the durability of the tile (Cárdenes et al., 2010). However, the pore system of slates has not been studied in detail, maybe because of the widely extended supposition that there is no open porosity. This pore system can suffer important changes during weathering, by the action of iron sulphides oxidation (Fischer and Koch, 2005) or by the effect of successive freeze-thaw cycles (Cárdenes et al., 2012a). These changes may compromise the integrity of the slate tiles, or even the waterproofing of the cover. Thus, the evolution of the pore system during the slate´s life service greatly affects its performance. An indirect way to measure this evolution is through the changes in the water absorption, which can be used to give an estimation of the life service of the slate (Walsh, 2002).
In this work, the pore system of two slate varieties from Spain is studied by means of optical microscopy, X-ray diffraction (XRD), X-ray computed microtomography (micro-CT) and mercury intrusion porosimetry (MIP), finding the main characteristics for this special pore system.

2. EXPERIMENTAL

Slates were sampled directly at the quarries of North Spain, in the Domain of the Truchas Syncline, the largest outcrop of roofing slate in the World. Samples were taken from fresh blocks extracted from the quarry. The first sample, LMO, is from an Upper Ordovician slate known for its low content in weatherable minerals such as iron sulphides and carbonates, while the second slate, MRA, is from

Middle Ordovician, and in this case is known to have significant quantities of these weatherable minerals.
The mineralogical and petrological analysis were developed at the Geology department of the Oviedo University, while the MIP was performed *at GEA Asesoria Geologica*, a company specialized in restoration of historical heritage. Micro-CT scanning was performed at the Centre for Tomography of the Ghent University (www.ugct.ugent.be).
For the petrological characterization, thin sections of 30 µm thickness were elaborated and examined with a Carl-Zeiss® Universal Polarized Light microscope. Thin section images were taken with a Leica DC 300F camera. The mineralogical semi-quantification was done by X-ray diffraction (XRD). The XRD analyses were carried out using a Philips diffractometer PW 1830, Cu cathode with a wavelength of $K\alpha = 1.54051$. The angular scan was recorded from 2º to 15º 2θ, with a digital register Philips PW 1710. The diffractograms obtained were studied with the software Xpowder for the qualitative and quantitative analysis.
MIP was performed with an AUTOPORE III 9400 porosimeter, obtaining the specific surface, pore diameter, pore access radius, percentage of connected porosity, and real density of the samples. For the micro-CT analysis, small core drills of 8 mm diameter were taken from the slates and scanned at the UGCT, an X-ray tube FeinFocus was used, at 120 kV. A voxel size of 6.50 µm could be obtained.

3. RESULTS AND DISCUSSION

The petrological (Figure 1) and mineralogical analysis (Table 1) show the typical features for an Ordovician Spanish roofing slate (Cárdenes et al., 2012b). The petrological texture goes from porfiro-lepidoblastic to lepidoblastic, while the proportions of the main minerals can be considered normal for these type of slates. Optical examination showed a very small grain size and pore size, under the resolution of the optical microscope. Other techniques, such as SEM, may give more accurate information about other facts of the fabric.

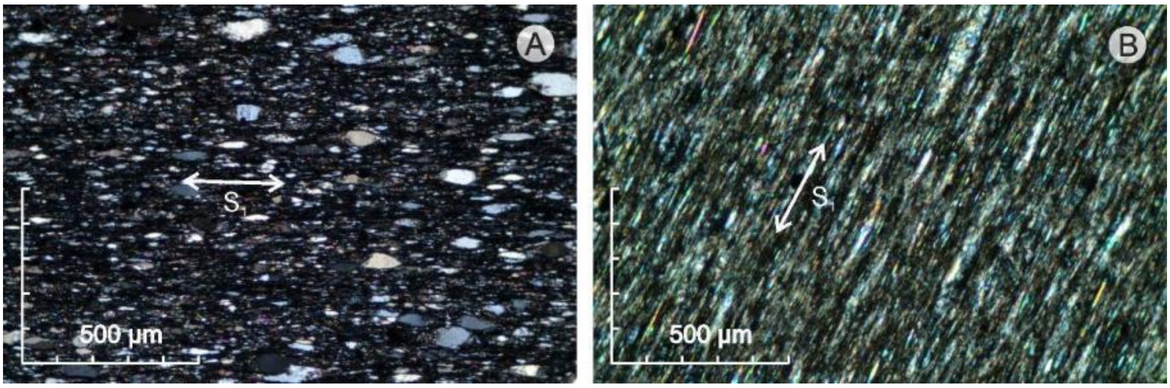

Figure 1: *Petrological microscope images (crossed polarizers and 63x magnification) of the slate samples MRA and LMO. A) MRA: The texture is porfiro-lepidoblastic, where the quartz grains stands out the chlorite and muscovite matrix; B) LMO: This texture is lepidoblastic, all the mineral components have similar size.*

Table 1: *XRD mineralogical determination of the slate samples.*

Slate	**Mineralogy (%)**					
	Quartz	Chlorite	Muscovite	Feldspar	Iron sulphides	Carbonates
MRA	27.8	20.8	42.1	7.0	1.1	1.2
LMO	24.9	24.6	43.3	7.2	<1	<1

The MIP analysis show low porosity values for both slates. The cumulative intrusion curves (Figure 2) present different shapes, for MRA the curve adjust to a Gaussian distribution, with a pore radius access biased to the higher values, but for LMO the curve shape highlights that there are pore sizes below the limit of detection of the porosimeter. Thus, the real % porosity should be a little higher than 1.24 % (Table 2). Thus, LMO has more microporosity than MRA, and hence a slightly higher specific surface.

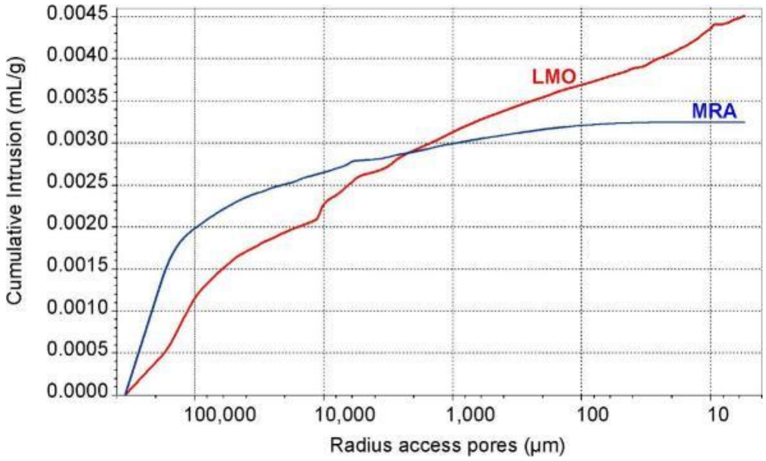

Figure 2: *Cumulative Intrusion curves for MRA (blue) and LMO (red).*

Table 2: *MIP pore system parameters.*

Slate	**Specific surface (m^2/g)**	**Average access radius (μm)**	**Median access radius (μm)**	**Porosity (%)**
MRA	0.04	195.7	114.5	0.89
LMO	0.69	82.5	11.6	1.24

Finally, micro-CT has proven to be more effective for visualizing joint veins or mineral inclusions on the slate bulk than for determining the pore system itself, due to the very low size of the existing pores (Figure 3).

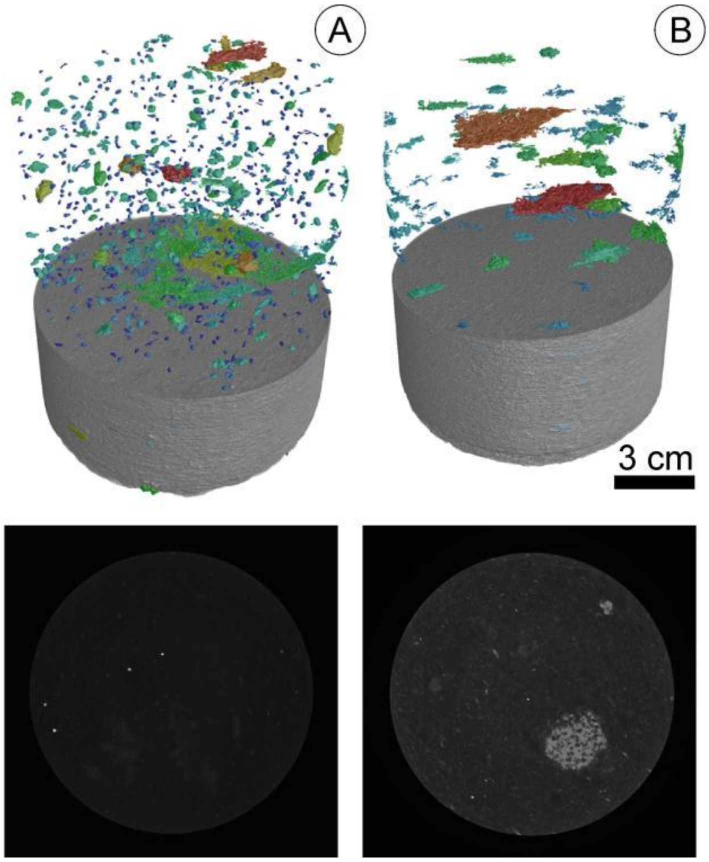

Figure 3: *Rendering of scans of samples LMO (A) and MRA (B). While LMO is characterized by a lot of small, dense inclusions (pyrite and pyrrhotite), These smaller inclusions are less common in MRA, which is characterized by larger inclusions (carbonates). Colours indicate similar grain size of the inclusions. Below both renders there are a representative cross-section for each sample.*

4. CONCLUSIONS

Roofing slates have a very small grain size and pore radius access which makes it difficult to study their pore system. MIP analysis has shown low porosity values, while micro-CT analysis has allowed characterizing mineral inclusions and other heterogeneities in the slate matrix. Micro-CT can determine the size, distribution and abundance of these mineral inclusions, giving important information about the potential response of the slate to mineral weathering, which is one of the main problems for the slate sector.

5. REFERENCES

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