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MicroCT vs. Hg porosimetry: microporosity in commercial stones

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Decay of rocks, due both to extrinsic and intrinsic factors, can show up in several different forms, such as neoformation of minerals, decohesion of grains and/or crystals, magnification of previous defects, new discontinuities, etc.
Intrinsic factors include the type of material, its properties and microstructure, in particular porosity and microporosity. Extrinsic factors relate to atmosphere and usage of the material itself.
Rock degradation has several heavy consequences for commercial stones, such as increase of permeability, loss of material, loss of mechanical strength; these consequences are of crucial importance for conservation of historical buildings.
Aim of this study is to compare microporosity of some massive commercial stones by means of X ray microtomography, a non destructive technique, and Hg porosimetry.
Nine of the most used Spanish limestones and dolostones have been analysed. The lithotypes have been chosen for their homogeneous mineralogical composition (calcitic or dolomitic) and for their low porosity; some of them have been widely used in Spain for historical buildings.
Different lithotypes have been described in thin section:
Ambarino (A) and Beige Serpiente (BS): brecciated dolostone, composed by microcrystalline dolomitic clasts, in a dolomitic and/or calcitic microcrystalline matrix.
Amarillo Triana (AT): yellow dolomitic marble, with fissures filled up by calcite and Fe oxides.
Blanco Alconera (BA): a white-pink homogeneous limestone, with veins.
Blanco Tranco (BT): a homogeneous white calcitic marble, without any fissures and/or fractures.
Crema Valencia (CV): a pinkish limestone, characterized by abundant stilolythes, filled mainly by quartz (80%) and kaolin (11%).
Gris Macael (GM): a calcitic marble wiht darker and lighter beds, conferring a strong anisotropy.
Rojo Cehegin (RC): a red fossiliferous limestone with white calcitic veins.
Travertino Blanco (TB): a massive white calcitic travertine.
Prismatic samples of about 2x1x1 cm have been cut and scanned by means of a X ray microCT system before and after mercury saturation with Hg porosimeter.
The microCT system used is a BIR Actis 130/150 with nominal resolution of 5 micron; for our samples resolution is of 25 microns. Generator and detector are fixed, while the sample rotates; the scanning plane is horizontal. Samples reduce the X rays energy passing through, as a function of its density and atomic number. X rays are then collected on a detector, which converts them into light radiations; a digital camera collects light radiations in raw data and send them to the computer, where they are processed as black/white images.
The Hg porosimeter used is a Pascal 140/240 Thermo Fisher. Samples were first degassed and then intruded by Hg. Apparent density, bulk density, porosity and open pore size distribution (pore diameter between 3.7 and 58000 nm) of each sample have been computed using the PASCAL (Pressurization with Automatic Speed-up by Continuous Adjustametnt Logic) method and the Washburn equation; this equation assumes: cylindrical pores, a contact angle between mercury and sample of 140°, a surface tension of mercury vacuum of 0,480 N/m and mercury density equal to 13.5 g/cm^3.
MicroCT images and porosity data from Hg porosimeter have been compared by several authors both for rocks (Klobes et alii, 1997) and for artificial materials with medical applications (Lin-Gibson et alii, 2007)
In samples with no density/composition differences microCT images are homogeneous and gives no information on the internal structure of the sample. This is the case of massive samples (such as BA, BT, GM and TB) and of

samples without any significant density differences between clasts and matrix (A and BS) or rock and veins (RC). MicroCT images of the same sample after mercury saturation offer a detailed map of microporosity of the rock, due to the high density contrast between mercury (13.6 g/cm3) and the rock (2.71 g/cm3 for calcite and 2.86 g/cm3 for dolomite). In some cases microporosity coincide with structural features of the rock, such as stylolythes (CV), fissures (AT) and veins (RC).

This method works for samples with low porosity (less than 1%); on the other hand for samples with higher porosity (4%; A and BS) microCT images after Hg saturation present artefacts, due to the spreading of Hg within the sample, which obliterates the true structure of the rock.

References

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