Characterization of the porous network structure of concrete

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The porosity and specific surface area of a small intact core of concrete (~ 0.2 g) was characterized non-destructively by micro-computed tomography (μ-CT), N_2/BET and destructively by Mercury Intrusion Porosimetry (MIP).

Biological shielding concrete that has been subjected to neutron radiation is prone to degradation via the alkali silica reaction.[1] This has two implications: loss of mechanical strength (which impacts service life), and a change in the porous structure and reactive surface in terms of the sorption characteristics and transport of radionuclides (either occurring *in situ* via neutron activation or *ex situ* due to contamination of leaked reactor cooling water). The objective of this work is to investigate the characterization of pore structure on intact specimens of concrete (as small as possible) so that reactive transport processes can be directly correlated.

EXPERIMENTAL. Non-radioactive concrete (ca. 48 years old) from the dome section of a reactor pressure vessel forming the base of the reactor was acquired during the dismantling of a decommissioned nuclear power plant, Stade, in Hamburg, Germany. A diamond-coated core driller (dry; 200 rpm) was used to remove a small core (Ø = ~ 5 mm; L = 2 mm; 0.17 g) from a larger drill core (Ø = 73.6 mm; L = 150.7 mm) that remained intact and without any visible cracks after being subjected to a standard test (EN 13412:2006-11) for modulus of elasticity under compression (21.7 GPa). This small core was analyzed non-destructively by μ-CT and Scanning Electron Microscopy (SEM) (sample surface: rough, uncoated), then dried at 100 °C overnight prior to an N_2/BET measurement in triplicate. MIP was subsequently conducted on the small intact core in accordance with ISO 1509-1. μ-CT scans were acquired on the large and small drill cores using a Nikon XT H225 employing a tungsten target. A SEM image was obtained using a JEOL JSM-F100. The Pascal 140 and 440 Series from Thermo Electron Cooperation were used to conduct MIP. Specific surface area determined by N_2/BET was performed using a Thermo Scientific Surfer Analyzer.

RESULTS. The results are summarized in Figures 1 and 2. It is clear that the methods of MIP and μ-CT yield pore-size information on different scales with a small overlap in the region 4 – 75 μm. The specific surface area calculated using the cylinder and plate model from the MIP results is 9.94 m^2/g, whereas N_2/BET yields a value of 6.07 m^2/g (s. d. = 0.54). A higher value for MIP is due both to the ink-bottle effect (large pores with narrow throats)—smaller pores are over-estimated at the expense of larger pores—and to fracking. The hysteresis of the mercury intrusion and extrusion curves suggests needle-like pores.[2] The initial constant volume with decreasing pressure and the fact that 51.5 % of the mercury remains in the sample after extrusion is indicative of ink-bottle pores, as well as non-equilibrium compression and decompression conditions. Larger cores (i.e. ~ 1 g) revealed similar pore features in their differential pore-size distribution curves, but cores < 0.1 g displayed erroneous results. While MIP yields information on the connectivity of the transport pores, the specimen is unfortunately unusable for further reactive transport experiments.

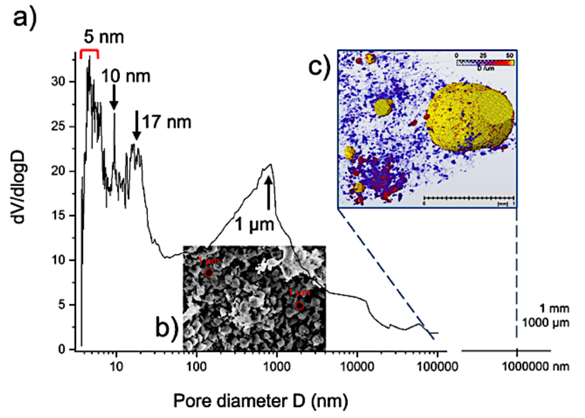

Fig. 1: a) Differential volume pore-size distribution curve of a small intact pore of concrete determined by mercury intrusion porosimetry illustrating on the logarithmic scale gel pore features (5 - 17 nm) and capillary pores of ~ 1 μm, which can clearly be seen in the b) SEM image of the cement matrix and c) a thickness map of a sub-volume of the small concrete core obtained from μ-CT portraying air voids (yellow) in millimeter range, and micropores of ~ 40 μm (red) and ~ 20 μm (blue).

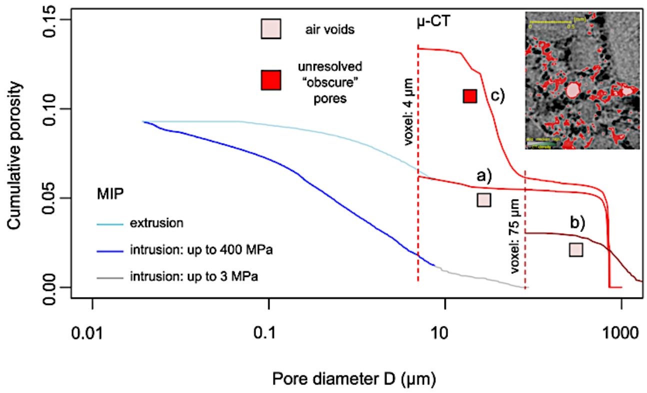

Fig. 2: Accumulated porosity versus pore-size on the logarithmic scale determined by MIP and μ-CT. True air voids and pores are calculated with background greyscale values (inset: pink) for a) and b), respectively for a sub-volume of the real small core (with voxel size 4 μm) and a digitally extracted volume from the big drill core including the small core as sub-volume (with voxel-size 75 μm). Between the cement matrix and immediately adjacent to the aggregate exists a greyscale that is neither air nor grain (inset: red).[3] This greyscale is not well resolved and could suggest obscure pores, possibly in a network of unresolved needles. This volume fraction is illustrated in c) for the real small core.

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