Study on cement paste pore connectivity using Nuclear Magnetic Resonance (NMR)

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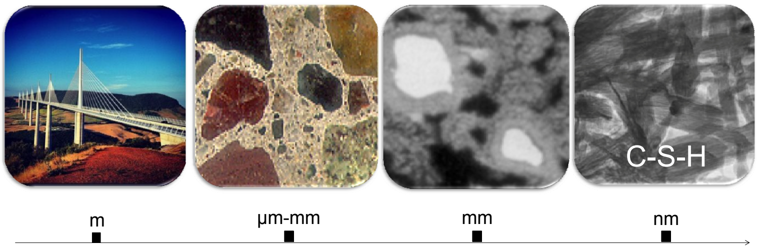
Introduction

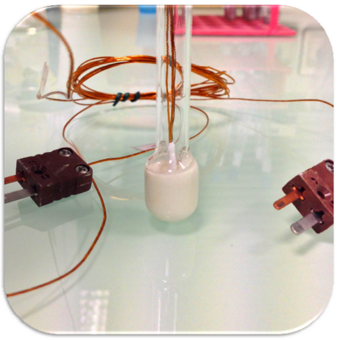
Pores play a key role in concrete degradation, as they form 3D pathways for the water. Despite being used for hundreds of years, its porous space at the nano scale is not known well. By combining advanced NMR techniques, we will be able to explore information of the pore connectivity of the C-S-H – the main binding phase of the concrete.

Scale at which we work

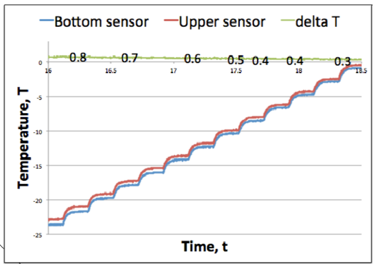
After cement hydrates, about 90% of porosity is at nanometre scale. Water transport goes through the gel pores of the C-S-H, which connect bigger pores to each other.



Cryoporometry



Temperature gradient within 1 C achieved during cryoporometry experiment. This gives us assess to the better pore size resolution

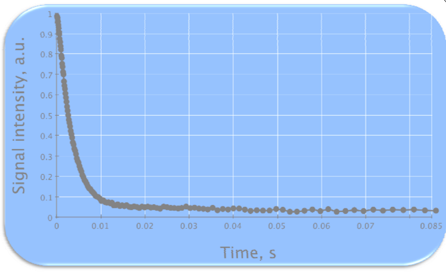


^{1}H NMR is very sensitive to the proton motion

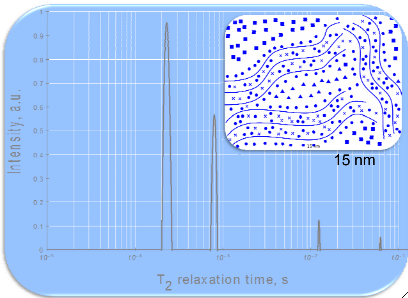
With cryoporometry we measure the quantity of liquid that has melted, as a function of temperature. This technique is based on the Gibbs-Thomson effect, which maps melting depression point to pore size:
$\Delta T_m(x) = k_{GT}/x$, where k_{GT} is the Gibbs Thomson Coefficient.

Relaxometry: relaxation rates (T_2) are strongly dependant on the liquid interaction with the surface. In other words, spins relaxation will occur much faster in the smaller pores. CPMG (Carr–Purcell—Meiboom–Gill) NMR experiment is used, with ILT (inverse Laplace transform) needed in order to obtain T_2 distributions. One can map T_2 time to pore size using $1/T_2=\lambda * S/V$, where S/V is surface to volume ratio.

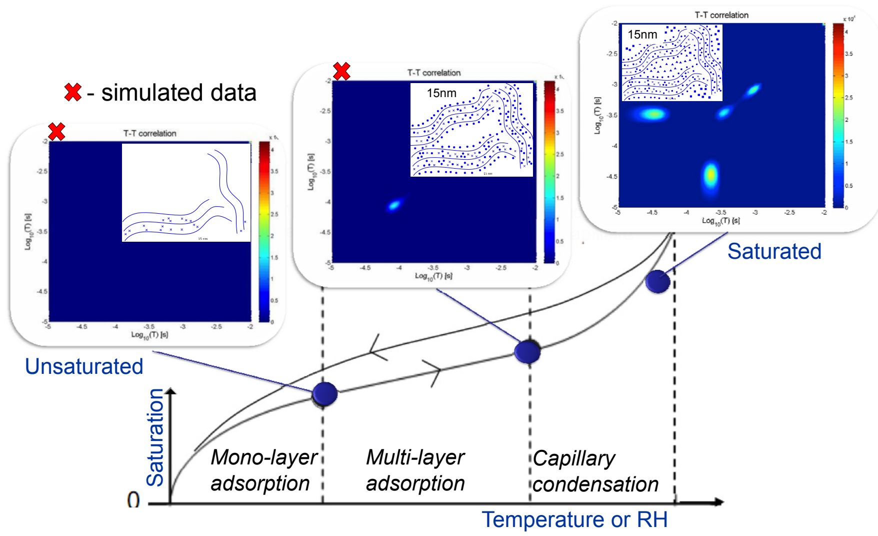
Relaxometry



NMR signal obtained with CPMG pulse sequence. Corresponding T_2 relaxation times for the 4 days old white cement with water/cement ratio = 0.4 are presented below. Peaks from right to left: C-S-H inter-layer, gel pores, inter-hydrate space and capillary pores.



Combined 2D relaxometry (T2-T2) with cryoporometry



The idea is to couple CPMG into 2D NMR experiment and with the cryoporometry. The core of the proposed experiment is to isolate smaller subsets of the porespace of C-S-H by freezing out bigger pores (zooming in). **It will give new experimental data on pore connectivity and diffusivity.**

Future work

- Validate cryoporometry and relaxometry on the reference samples (with known PSD) and well studied liquid, as cyclohexane.
- Compare to the differential scanning calorimetry (TRANSCEND project 10).
- Couple 2 techniques into T_2-T_2 cryoporometry experiment.
- Perform tests on the reference sample with bimodal pores size distribution.
- Experiments on the synthesized C-S-H and C_3S.
- Apply technique to the pozzolan C-S-H. Test on the range of pozzolan C-S-H with difference Ca/Si ratio.
- Apply on the real cements.

Read it on your smartphone

QR code

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