Isopropanol exchange in cement pastes: an NMR relaxation analysis study

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Abstract

To enable reproducible experimental analysis of cement pastes it is necessary to halt hydration. One way to do this is by removing water via solvent exchange. However, the consequent changes to the morphology of the cement are not fully understood[1].

It is accepted that the method of water - isopropanol exchange causes least damage[2]. However, the majority of experiments to show this have been conducted on samples in which both the water and the isopropanol have been removed, with only a few studies reporting data on saturated samples[3,4]. Nuclear Magnetic Resonance (NMR) relaxation and imaging have been used to investigate the microstructure of the cement paste[5] and re-wetting of isopropanol dried samples[6,7] respectively. However, to the best of our knowledge, there is no study of confined isopropanol.

In this work, we examine the change to the microstructure via direct NMR of the confined isopropanol. To separate signals of the water from the isopropanol different combinations of H_2O, D_2O, CH_3CHOH and CH_3CDOD have been used.

The results show that isopropanol acts as a drying agent by removing and replacing water molecules in the interhydrate and capillary pore network. It is also shown, that isopropanol removes water from the C-S-H interlayer and gel pores although it does not fully replace the lost water.

Contact between water remaining in the interlayers and isopropanol residing in the pores after the exchange process, is evidenced by observation of proton - deuteron chemical exchange between the liquids. The solvent exchange also reveals the presence of large capillary pores that are observed in microscopy but which are not normally seen in ^{1}H NMR since water is unable to fill them due to the reduced Kelvin-Laplace radius at the reduced RH of hydrated cement.

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