Assessing the Performance of Hydrophobing Agents on Concrete using Non-Destructive Single-Sided Nuclear Magnetic Resonance

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Abstract
Single-sided nuclear magnetic resonance is a non-destructive analytical technique by which the ingress behaviour of hydrophobing agent as well as the properties and performance of the resulting hydrophobic layers can be assessed quantitatively. The present results indicate that the layer thickness is the most important parameter for the performance of hydrophobic layers provided that the layer is free from defects. The results further demonstrate that single-sided nuclear magnetic resonance is an excellent non-destructive tool for the quality assurance when applying such protective layers.

Keywords: Hydrophobing agents, silanes, nuclear magnetic resonance, NMR MOUSE®

1. Introduction
Hydrophobing agents are the compounds of choice in civil engineering whenever an invisible water-protection layer is required. However, a proper application of a continuous layer of sufficient thickness can be tricky as it depends on a number of factors. Today, the properties of hydrophobic layers are either qualitatively estimated by the roll-off behaviour, which is only a superficial effect, or by taking drill cores. In this context, single-sided nuclear magnetic resonance allows to follow the ingress of hydrophobing agents and to determine the thickness of hydrophobic layers in a non-destructive way.

2. Materials and Methods
The experimental programme, sample preparation, and test methods follow that described in [1]. Samples included here were prepared from Portland cement and allowed to carbonate. Amplitude-depth profiles were recorded by single-sided nuclear magnetic resonance using a NMR MOUSE® PM 25. The hydrophobing agent was n-octyltriethoxysilane provided by DOW Corning.

2. Results and Discussion
Proton nuclear magnetic resonance can in principle detect all substances containing hydrogen atoms. In practice, the detectability is associated with the relaxation time of the hydrogen nuclei, which is linked to the molecular mobility. In practice, hydrogen rich liquids such as water can be easily detected, amorphous polymer work at reduced signal to noise ratios and crystalline solids are invisible with the device used [2].

2.1 Ingress of the hydrophobing agent
The hydrophobing agent is a liquid, which is applied neat to the surface of the specimens in a quantity of 127 g pure active compound per m$^2$. The ingress of the liquid can, therefore, be followed using the NMR MOUSE® (Fig. 1). Initial ingress appears to proceed rapidly and the front of the penetrating layer reaches a depth of 4 mm after 18 min as indicated by the intersection of the profile with the background noise of the dry sample (dashed line). However, redistribution of the hydrophobing agent evidenced by a shift of the intersection to greater depths and a decrease in amplitude appears to be considerably slower and the transport process comes to a halt after approx. 22 h. Any further decrease in amplitude is solely due to the condensation reaction of the silane, which in the case of $n$-octyltriethoxysilane can continue for several weeks.

Figure 1. Ingress of $n$-octyltriethoxysilane into a concrete specimen followed over time. The background noise of the dry sample is shown as dashed line.

2.2. Hydrophobic layer thickness

The thickness of the hydrophobic layer is assessed by submersion saturation in water. The amplitude value indicating the transition from non-wettable (hydrophobic) to wettable (hydrophilic) pore system was found to be 100, determined independently by spraying a fractured surface with water. It should be noted, however, that this amplitude is only valid for the instrument settings used in this study. For $n$-octyltriethoxysilane applied under the conditions mentioned above, the thickness of the hydrophobic layer was found to be 6.5 mm (Fig. 2A), which matches the intersection of ingress and dry profile at the end of the transport process (cf. Fig. 1).

2.3 Water transport through the hydrophobic layer

The water transport through the hydrophobic layer is assessed in suction experiments, by following the increase in moisture behind the hydrophobic layer [3]. After almost one year in the footbath, the amplitude-depth curve does not significantly deviate from that of the dry specimen indicating this hydrophobic layer to be practically impermeable to water.
3. Conclusions

Single-sided nuclear magnetic resonance is a powerful tool for the non-destructive testing of hydrophobic layers. The ability to follow the ingress of applied compounds offers the possibility for on-site quality assessment during or shortly after application, while the NMR technology holds the potential to detect faults in aged layers facilitating rehabilitation.

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References and Notes

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