Cement and Concrete Research 42 (2012) 265–271

Contents lists available at SciVerse ScienceDirect

Cement and Concrete Research

journal homepage: http://ees.elsevier.com/CEMCON/default.asp

Fire spalling of concrete, as studied by NMR

G.H.A. van der Heijden [a], L. Pel [a,*], O.C.G. Adan [a,b]

[a] Department of Applied Physics, Transport in Permeable media, Eindhoven University of Technology, P.O. Box 513, 5600 MB Eindhoven, The Netherlands
[b] TNO, Delft, The Netherlands

ARTICLE INFO

Article history:
Received 17 June 2011
Accepted 28 September 2011

Keywords:
Thermal analysis (B)
Degradation (C)
Permeability (C)
Transport properties (C)
Concrete (E)

ABSTRACT

The moisture transport in concrete subjected to fire is one of the most important processes with respect to fire spalling. The research on fire spalling of concrete is currently lacking experimental information of the moisture transport processes.

We present combined moisture content and temperature profiles of one-sided heated concrete samples measured with our dedicated NMR setup. The concrete samples were equilibrated at different moisture contents ranging from 97 to 50% RH. The moisture content can be measured quantitatively and non-destructively while heating up the sample one-sided to 500 °C.

We present the first experimental proof for the build up of a moisture peak in concrete, and the formation of a saturated layer. The temperatures measured at the boiling front indicate a vapour pressure in the order of 1.8 MPa. A simple vapour transport model was successfully used to describe the speed of the boiling front.

1. Introduction

The moisture content and transport in concrete, together with the vapour permeability, is presumed to be one of the most influential factors for fire spalling. It is generally assumed that below a certain moisture content of approximately 5 vol.% no spalling will occur [1,2]. Numerous heat and mass transfer models have been used to predict the moisture transport and its consequences on the strength and permeability of the concrete [3–11]. However, these models are only of use if they can be validated. For model validation, quantitative measurements of the evolution of moisture, temperature, and possibly pressure distributions in time are needed.

A few (indirect) observations of the moisture content in concrete have been reported in literature. Water has been observed bleeding from the cool side of a concrete wall, indicating that the surface layer is saturated [12]. Jansson et al. report a method in which a concrete cube is heated from one side. By splitting the heated cube in half, a layer with a relative higher saturation is observed by a discolouration of the concrete [13]. In this way, the sample is destroyed by the act of measurement itself. It will be extremely labourious to obtain a time evolution of the moisture content. Each time increment would also be from a different concrete sample. Experimentally determined moisture contents with a spatial resolution of about 20 cm were presented in a paper by Ichikawa et al. [14]. However, the obtained resolution is far too low to validate the model results.

With a dedicated NMR fire spalling setup it is possible to measure both the moisture and temperature distributions non destructively. This setup has been extensively tested in previous experiments on benchmark materials such as fired-clay brick, calcium silicate brick, and gypsum [16,17]. For the measurements on concrete it is necessary to have a validated measurement technique since the moisture content of concrete in equilibrium with, e.g., 50% RH, is typically in the range of 10^{-3} m^3 m^{-3}. Although the low moisture content is challenging the capabilities of the NMR setup, it was possible to measure the moisture transport in concrete.

In this paper we will present for the first time the combined moisture and temperature profiles in concrete. By combining these two parameters it is possible to estimate the vapour pressure inside the concrete samples. The paper is organised as follows. In Section 2 the materials and methods are presented. The results from the one-sided heating experiments are presented in Section 3. A discussion of the obtained results and final conclusions will be given in Section 4.

2. Material and methods

2.1. NMR setup

The fire spalling experiments were performed using a home-built NMR setup, which was especially designed for non-isothermal moisture measurements on building materials. A schematic diagram of this setup is shown in Fig. 1. The setup can be placed entirely in the bore of a 1.5 T whole-body medical scanner (Gyroscan, Philips), which is used only for its main magnetic field. Two coils, in an anti-Helmholtz configuration, with a diameter of 35 cm, provide a constant magnetic field gradient

* Corresponding author. Tel.: +31 402473406; fax: +31 402432598.
E-mail address: l.pel@tue.nl (L. Pel).

0008-8846/$ – see front matter © 2011 Elsevier Ltd. All rights reserved.
doi:10.1016/j.cemconres.2011.09.014

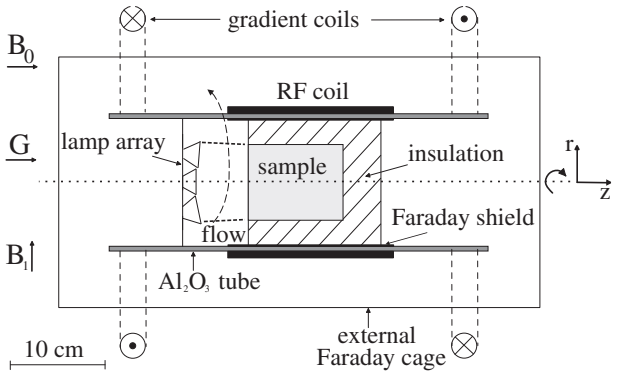

Fig. 1. Schematic diagram of the NMR setup. The setup has a cylindrical symmetry. A whole-body 1.5 T MRI scanner provides the main magnetic field B_0. Two coils in an anti-Helmholtz configuration provide a constant magnetic field gradient G. A bird-cage RF coil with a diameter of 140 mm is used for both sending the RF pulses B_1 and receiving the NMR signal. The bird-cage coil is constructed on an aluminium oxide (Al_2O_3) tube. An array of four 100 W halogen lamps is used to heat the sample, which is thermally insulated and positioned in the bird-cage coil.

G in the direction of B_0. In the experiments the gradient is set to 100 mT m^{-1}, providing a spatial resolution in the order of 3–5 mm.

A home built birdcage coil is used for sending the RF pulses (B_1) and receiving the NMR signal from the sample. The coil is 140 mm long and has a diameter of 140 mm. It is constructed using copper strips which are wrapped around an Al_2O_3 tube. A birdcage type coil is used because it generates a homogeneous B_1 field perpendicular to the long axis of the cylindrical sample. Therefore, the coil can be placed parallel to the main magnetic field, providing optimal use of the available space inside the bore. The coil is designed with an internal Faraday shield, in order to prevent changes in the dielectric constant of the drying sample from de-tuning the RF coil [18]. In this way, quantitative moisture content profiles can be obtained.

The Al_2O_3 tube can withstand temperatures far above the requirements of the experiments (melting temperature 2072 °C). Furthermore, it does not give any background signal, which is important when measuring a moisture content as low as 10^{-3} m^3 m^{-3} in concrete at high temperatures. In order to simulate the conditions as they occur in a fire, the concrete has to be heated up quickly. With an array of four 100 W halogen lamps, capable of generating a heat flux of 12 kW m^{-2}, we are able to mimic a 'fire' inside the NMR setup. The reflectors of the lamps are gold plated to ensure maximum reflection of infrared radiation towards the sample surface. Gold is used because it has a very high infra-red reflectivity of 0.98 and it does not oxidise [19]. To minimise heating of the RF coil, the lamp array is actively cooled. The heat flux from the lamp was calibrated calorimetrically and varies linearly with the applied electrical power to the lamp [16]. The maximum temperature which can be reached at the heated surface is about 400 to 500 °C. Although we do not reach the maximum temperatures occurring in a real life fire (~1000 °C), we are in the region of temperatures where the most interesting moisture related processes occur, such as boiling at temperatures higher than 100 °C, moisture clogging, and dehydration of the concrete. The temperatures are measured with type-K thermocouples, which were fitted in pre-drilled holes approximately 1 mm wide and 5 mm deep.

2.2. Concrete samples

The cylindrical concrete samples were drilled from larger cast blocks of $40\times10\times10$ cm^3 and have a diameter of 80 mm, and a length of 100 mm. The concrete has a strength class of C40, with a water cement ratio of 0.5. The details of the mix are given in Table 1. As can be seen, the largest aggregate size has been reduced to 8 mm to ensure a more representative sample volume. The concrete blocks were stored under water for 3 months before the samples were drilled. After drilling, four samples were first dried, after which they were equilibrated at four different relative humidities of 50%, 75%, 86%, and 97% RH until a constant mass was reached. The samples were 1 year old at the time of the experiment.

Table 1
Concrete mix design.

Constituent	Amount [kg m^{-3}]
CEM I 32.5 R	350
Water	175
Sand (0.125–0.250)	127
Sand (0.250–0.500)	217
Sand (0.500–1)	217
Sand (1–2)	253
Sand (2–4)	380
Grave (4–8 mm)	614
Total	2330

The concrete was characterised with mercury intrusion porosimetry (MIP), moisture sorption, and NMR. The MIP curve and the sorption isotherm of the concrete are shown in Fig. 2. From the MIP curve we can conclude that the dominant pore size is between 10 and 100 nm. The sorption isotherm curve shows an increase in moisture content already at a very low RH, which is in agreement with a dominant pore size in the nanometer range. Before the experiment, the samples were pressed in a PTFE holder to seal all sides except for the heated surface (transversal plane of the cylinder). In this way the moisture transport is limited to one dimension. The flow of heat is limited to one dimension by insulating the sample using mineral wool (4 cm). Numerical simulations indicate that the radial heat flow is limited to 10% of the longitudinal heat flow.

At the start of the experiment one moisture and temperature profile is measured to record the initial state of the sample. The first moisture profile is used to normalise subsequent profiles. In this way the moisture profiles are also corrected for the longitudinal inhomogeneity of the RF field. After the first profile is measured the heating is started. A constant heat flux of 12 kW m^{-2} is applied. To prevent hot vapour from condensing on cold surfaces inside the coil, air of 1–2% RH was blown over the sample surface. The air flow also ensures a constant boundary condition for the sample surface. The moisture and temperature profiles are measured every 7–10 min, depending on the number of averages needed.

The signal profiles measured with NMR have to be corrected for the temperature at which they were measured. Both the magnitude (magnetisation) and the time dependence (relaxation) of the NMR signal are temperature dependent. The correction procedure to obtain a quantitative moisture content was introduced and demonstrated in [15].

2.3. NMR calibration

In order to obtain a quantitative moisture content, the NMR signal must be calibrated against the moisture content of the concrete. For this purpose, the NMR signal was measured with the same settings as during the experiments. A sample was initially vacuum saturated, after which it was dried slowly to keep the moisture homogeneously distributed throughout the sample. Mass and signal were both recorded throughout the drying process. The NMR signal is shown as a function of the normalised sample mass in Fig. 3. For mass ratios between 1 and 0.94 a linear relation between the NMR signal and the moisture content is obtained. This indicates that the NMR signal can be directly related to the amount of free moisture present in the concrete.

After the sample was dried at ambient temperatures a further decrease in moisture content (adsorbed and chemically bound moisture)