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Effects of dielectric heating of fresh concrete on its microstructure and strength in the hardened state

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HIGHLIGHTS

- Dielectric heating of fresh concrete with radio-waves is introduced.
- A uniform, rapid and accurate, temperature-controlled heating was achieved.
- High early-age compressive strength was measured.
- Microstructural characterisation showed changes due to heat treatment.

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ABSTRACT

By means of radio-wave technology it is possible to allow the uniform, rapid and accurate, temperature-controlled heating of fresh concrete. This results in the significantly accelerated release of hydration heat. The heat release from this second source can be easily compensated by the temperature-controlled reduction of RF energy input. The consequences are accelerated hydration and higher early-age compressive strength. Early-age concrete, heat-treated by radio waves, have a denser microstructure with a higher gel-pore content and more C-S-H phases than untreated concrete. When cured at the maximum temperature of 80 °C, secondary ettringite can be formed in concrete, whereas this is not the case for the treatment temperatures of 40 °C and 60 °C. At the age of 28 d, heat-treated concretes exhibit a looser microstructure than does untreated concrete. Larger portlandite crystals and secondary ettringite are its characteristic features as a consequence of the much faster reaction rates in the early-age concrete. This results in lower values of the compressive strength of the heat-treated concrete.

1. Introduction

For economic reasons, short station times are necessary to enable the quick reuse of formwork in the production of precast concrete elements. Hence, concrete must reach high early strength values in such applications. To accelerate the hydration process, optimised concrete mixtures – typically with high cement content and/or with a cement of a high strength class – are used [1,2]. Another common method involves increasing the temperature of fresh or early-age concrete by external heating [3,4]. Such heating processes mostly use fossil fuels and require very large heating chambers or tunnels in which the precast concrete elements are placed [5]. The heat transfer to the concrete elements is based on convection and thermal conduction and accordingly runs slowly [6]. In addition to the concrete elements themselves, the air volume in the heating chambers as well as the formwork must be heated as well.

The subject of the present study is an innovative technology for heat treating fresh concrete using radio-wave [RW] technology, which allows for an energy-efficient, temperature-controlled heating procedure. When using radio-wave technology, an electromagnetic field in the radio frequency [RF] in a range of MHz is created. Its interaction with the material acting as dielectric results in the direct dielectric heating of the concrete and enables a uniform increase in temperature throughout the whole volume of the element. By measuring the temperature and controlling the RW

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power, defined characteristic temperature fields can be established inside the concrete member. Exact and continuous temperature control ensures that defined maximum temperatures are quickly reached and not exceeded.

The intention of the research at hand has been to characterise the effects of dielectric RW heating on the microstructure of hardened concrete as well as on its early-age and standard-age strength properties. To investigate the microstructure, various methods were used: the determination of the pore size distribution by using mercury intrusion porosimetry, microscopic investigations with an "environmental" scanning electron microscope (ESEM, including EDX), and the characterisation of hydration products both qualitatively and quantitatively by means of X-ray powder diffraction (XRD) with Rietveld refinement.

2. Experimental investigations and materials

2.1. Radio-wave technology

As in the case of a microwave oven [7], RW technology is based on the working principle of dielectric heating. In both cases, due to the rapid, repeated reorientation in an external electromagnetic field, the interaction between charged species or dipoles within the material acting as lossy dielectric causes volumetric heating. For this reason and in contrast to conventional heating methods, dielectric heating techniques do not need thermal conduction [8–10]. The efficiency of the energy absorption depends on the properties of the material, namely on the relaxation time of the reorientation processes related to the inverse frequency.

The work of Makul et al. [11] comprehensively investigated the behaviour of various blended cement and concrete mixtures during a 24-h first-hydration period applying thermal treatment with microwaves of (2.45 ± 0.05) GHz [7]. The novelty of the study at hand is to use an essentially different frequency in the high-frequency (or radio-frequency) range. While the microwave technologies apply frequencies ranging from 300 MHz to 300 GHz [7], a frequency in the range of 13.56 MHz has been applied here (Fig. 1). This leads to a significant difference not only in the penetration depth but also in the mechanisms of energy absorption. In particular, materials containing no free water phase such as dry brickwork can be efficiently heated by radio-frequency application.

In order to place the compacted concrete in the RW field for dielectric heating, a common-size ($15 \times 15 \times 15\ \text{cm}^3$) plastic mould was equipped with two RF electrodes (Fig. 2a). Between these parallel metal plates, the electromagnetic field was established via a connection with the RF matchbox by a broad copper band (Fig. 2b). This electronic matching network (PFM 10000) was connected to the RF generator (TruPlasma RF 3003, frequency 13.56 MHz, maximum power 3 kW, both from Hüttinger Elektronik Freiburg/Brsg./DE) by a coaxial cable. Fibre optical temperature sensors (Neoptix, Quebec/CA, see [12]) were used to measure the concrete temperature continuously inside the specimen during the experiments. These sensors did not interfere with the applied electromagnetic field. Due to the use of thermal insulation around the form, heat losses due to heat conduction into the environment were minimised. Fig. 2a also shows the two heat sources in fresh concrete. In addition to the RW energy from an external source (Q_{RW}), the hydration heat of the cement Q_{Hydr} develops within the volume of the concrete sample [13,14].

2.2. Materials and experimental procedures

An ordinary concrete was designed without any special features. Table 1 gives its composition; no admixtures were used. The properties of fresh concrete are summarised in Table 2. They were tested according to the European standard DIN EN 206-1 [15].

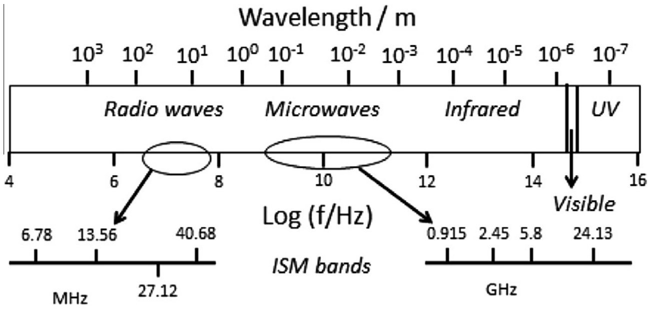

Fig. 1. Frequencies used for dielectric heating and mentioning of ISM bands [8].

The parameters for the heating process were chosen according to various guidelines [15–17]. Three different maximal temperatures (40 °C, 60 °C and 80 °C) were used. The heating rate was 20 K/h, the time of pre-storage at 30 °C was 1 h in the test series at hand; see also Fig. 3. Based on these parameters the following abbreviated description of the different heat treatments was selected: as an example, RW80 corresponded to the maximum temperature of 80 °C, a heating rate of 20 K/h and a pre-storage time of 1 h). Four parallel samples were used for every treatment procedure and testing age (altogether 32 samples).

The temperature program was performed using a special software [18] to control the RW power input on the basis of temperature values obtained from the sensors. After heat-treating the concrete, the cubes were either demoulded and tested for compressive strength at an age of eight hours (8 h strength) or demoulded after 24 h and stored in a water bath for 27 d. The untreated cubes were stripped 24 h after casting and stored in the water bath for 27 d as well.

2.3. Microstructure investigations

2.3.1. Mercury intrusion porosimetry

Mercury intrusion porosimetry (MIP) is an indirect testing method used in characterising the pores by measuring the response of the porous system to Hg pressure [19,20]. The high measuring range (3.5 nm–500 μm [21]) enables characterising the pore volume and the pore size distribution with a low dispersion [22]. In contrast to nitrogen adsorption at low temperature (BET analysis, Belsorp Mini, Rubotherm, Bochum/DE) larger pores are also accessible by MIP. Moreover, reference BET measurements confirmed the efficacy of both methods for small pores in the nm-range [13,23].

Before MIP measurements, parts of the treated concrete cubes were crushed into small pieces (2–4 mm). After extraction from the concrete block, the raw samples were prepared for the microstructure characterisation and particles rich in cement paste (and cement stone, respectively) were selected. The distinct features of the hydration products became significantly more evident when using this sample preparation procedure. After 24 h of vacuum drying at 20 °C, sample masses of approximately 3 g each were used for the investigations. A high-pressure mercury porosimetry device, PoreMaster 33/60 (Quantachrome, Odelzhausen/DE) with a maximal pressure of $p_{max} \approx 230$ MPa was used in the measurements.

2.3.2. Environmental scanning electron microscope (ESEM)

The ESEM ("environmental" scanning electron microscope) is a variation on the theme of scanning electron microscopy. The speciality of this method is that metallization of the sample surface is not required in the ESEM mode in contrast to a conventional SEM. Additionally, dehydration is inhibited in the ESEM because the sample is not exposed to high vacuum but is surrounded by a gaseous atmosphere containing a significant partial pressure of water vapour [24].

For the present investigations, an XL 30 ESEM (Philips, Eindhoven/NL) was used. It was equipped with detectors for secondary as well as back-scattered electrons and an EDX system for element analysis (Quantax 400, Bruker, Berlin/DE).

The investigations with the ESEM were carried out using the concrete fragments with sizes of 2–4 mm prepared as described above.

2.3.3. X-ray diffraction

X-ray diffraction (XRD) is an established method in characterising crystalline substances. It can be used to identify crystal phases both qualitatively and quantitatively. The line widths of the XRD spectrum enable as well the obtaining of information on the size of the crystalline fragments within the material.

In this study, the mineral phases were characterised by X-ray powder diffraction (XRD 3003 TT, Seifert, Coventry). Quantification was performed using 10 wt.% ZnO as an internal standard and calculation according to the Rietveld method [25].

3. Results

3.1. RF power input and temperature development

By using automated temperature-controlled heating the RW technology enables the exact spending of the required RF energy for heating concrete along the targeted temperature curve (Fig. 4). After heating concrete to 30 °C (pre-storage temperature) during the first 30 min, the RF input decreased to a minimum energy input of 5 W required for controlling the matchbox parameters (Fig. 4a). After 90 min, the energy input increased in order to follow the intended heating rate in the concrete specimen until the target temperature was reached. For RW40 and RW60, the maximum RF energy had to be applied just before reaching the maximum temperature. In the RW80 experiment, however, the energy input could be decreased again after approximately 150 min and reached