CHARACTERIZATION OF MOISTURE TRANSFER IN UHPFRC-CONCRETE COMPOSITE SYSTEMS AT EARLY AGE

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Abstract

Better understanding of the hydration process and hydration kinetics in advanced cementitious materials such as UHPFRC is required. Furthermore UHPFRC used for rehabilitation not only would be subjected to the climatological conditions (temperature and humidity) of the site but also the microclimatic state of the substrate (moisture and thermal conditions).

This paper presents information related to (1) the development of the free water consumption in plain UHPFRC from the time of casting up to 48 hours, (2) The water transfer processes between the UHPFRC and a dry concrete substrate from the time of placing up to 48 hours. Both objectives were achieved by utilizing the nondestructive Nuclear Magnetic Resonance (NMR) setup at Laboratory Navier.

For the first objective, Proton NMR (1^{H}-NMR) relaxometry was performed on sealed plain UHPFRC samples using two different sequences. By Inversion Recovery (IR) sequence and treatment of data using inverse Laplace transformation, water mobility in UHPFRC was investigated. Free Induction Decay (FID) pulse-acquisition sequence was used to investigate the rate and development of the free water consumption from the time of casting which represents the hydration kinetics. With regard to the second objective, Magnetic Resonance Imaging (MRI- 1D) with Single Point Imaging (SPI) was used to visualize profiles of water transfer exchanges between the UHPFRC and a dry concrete substrate.

The outcome is to understand the mentioned occurring phenomena which then would assist in modeling the hydration development and the hygral exchanges in UHPFRC-Concrete composites.

1. INTRODUCTION

Hydration plays a crucial role in every aspect of cementitious materials development, from the formation of the microstructure to the rate and magnitude of the development of mechanical properties and later on durability issues. Although there are still some unsettled issues with the cement hydration mechanism, well-established understanding of the related phenomena has been accumulated over the recent years [1, 2, 3]. However when it comes to the use and the effect of supplementary cementitious materials (SCM) on the hydration process, only general understanding has been attained [1]. This understanding, lessens when it comes to advanced cementitious materials such as Ultra High Performance Fibre Reinforced Concretes (UHPFRC) where significant amount of SCM, i.e., silica fume (SF) is incorporated in the recipe with high cement content, very low water to binder ratio and significant amount of superplasticizer [4]. From the aforementioned, it is probable that the hydration kinetics, the rate and magnitude of the phase development in UHPFRC different from that of concrete [5]. The evolution of free water content and water consumption could be evaluated using NMR [6, 7]. Since the rate and magnitude of the free water consumption during the hydration is directly related to the hydration kinetics, NMR techniques can be used as advanced methods to characterize the hydration kinetics and microstructural developments in UHPFRC.

Implementation of on-site application of UHPFRC for rehabilitation involves the existing concrete substrate. That is why the fresh UHPFRC not only is subjected to the climatological conditions (temperature and humidity) of the site but also to the microclimatic state of the substrate (moisture and thermal conditions) [8]. Although care is taken to ensure proper moist curing of the UHPFRC top surface in early stages after placing, the assumption is that the impair happens from the bottom, i.e. through interactions with the substrate moisture conditions which as a result might have detrimental influence on the development of the earlier mentioned properties of the UHPFRC. MRI-1D with SPI was used to visualize profiles of water transfer processes between the UHPFRC and a dry concrete substrate. Note that although the rule of thumb in practice is for the substrate to be moisture saturated but have a dry surface prior to UHPFRC application, using a totally dry substrate considers the worst case scenario that could be encountered in practice.

The objective of this paper is to understand the occurring phenomena in view of modeling the hygral exchange in UHPFRC-Concrete composite members at early age, which could lead to microstructure gradients within the UHPFRC layer. The perspective is to come up with construction practice solutions that would resolve any negative microclimatic effect of the substrate on the development of UHPFRC properties so that this advanced cementitious material reaches its full potentials in terms of material properties.

2. MATERIALS

Typical strain hardening UHPFRC used for cast-on site rehabilitation applications have usually 4 to 9 % steel fibers such as CEMTEC multiscale®, depending on the components used in the mix design. However, one of the limitations of NMR is that the sample under investigation must not have any paramagnetic components. That is why experiments had to be carried out to replace the steel fibers with aramid fibers to come up with a comparable recipe but with non-metallic fibers. The composition of adapted UHPFRC with Aramid fibers is

given in Table 1. Note that the water to cement ratio is 0.180, silica fume over cement is 26% and total amount of superplasticizer is 2.5% of the weight of cement. Chemical analysis and physical properties of cement and silica fume in percent are given in Table 2.

Table 1: Composition of UHPFRC with Aramid fibers

Raw materials	Quantities (kg/m^3)	More information
OPC	1112	CEM I 52.5 N PM ES from Lafarge (TEIL)
Sand	643	Fontainebleau type MN30 Quartz; Dmax < 0.5 mm
Silica fume	289	Zirconium from SEPR
Superplasticizer	28	Zementol TKK (SL) ; Polycarboxylate-25% Solids content
Total water	200	
Aramid fiber	42	Technora; 20 mm 800 dtex fibres
Air	40	4% air

Table 2: Chemical and Physical properties of Cement and Silica fume used

Chemical Constituents	Cement(%)	Silica Fume (%)
SiO_2	22.75	93.5
ZrO_2	-	2.4
Al_2O_3	2.7	3.5
Fe_2O_3	1.9	0.15
Na_2O	0.15	0.10
CaO	67.1	0.02
K_2O	0.2	0.06
MgO	0.75	-
SO_3	2.1	-
Physical Properties		
Density (g/cm^3)	3.15	2.2
Specific Surface (m^2/g)	0.34 (Blaine)	12 (BET)

A 5 liter Type 32 Perrier paddle mixer having a 0.35 KW power was used for mixing the UHPFRC. Initially Cement and silicafume were dry mixed for 2 minutes with a speed of 61.5 rpm (revolution per minute). Then sand was added and the dry components were mixed for another 2 minutes. The water and superplasticizer were added and entire mixture was mixed for approximately 7 minutes after the water addition; note that the speed of mixing was 61.5 rpm from the start of the batch up to 6 minutes (change of the consistency of the mix) and 123 rpm thereafter, until 11 minutes. Finally the Aramid fibers were added and mixed for one minute with the speed of 61.5 rpm. Total mixing time was 12 minutes.

3. EXPERIMENTAL METHODS

3.1. NMR equipment

Relaxation and Imaging experiments were performed by using a vertical imaging spectrometer DBX 24/80 Bruker, operating at Larmor frequency equal to 0.5 T (20 MHz proton) with a gradient strength of 50 mT.m^{-1}. Specimens were placed in a radio frequency birdcage coil with an inner diameter of 200 mm. It should be noted that since there are no

pretreatments and these techniques are nondestructive and non-invasive, the measurements were started immediately after casting of the samples. All tests were carried out at constant room temperature of 20 ± 0.1 ^{o}C. More detailed information regarding the NMR equipment could be found in [6, 7].

Two different methods were used: 1) NMR relaxometry (T_1 relaxation measurements) applied to plain UHPFRC samples and 2) MRI method with SPI profiles applied to both composite UHPFRC/concrete samples and plain UHPFRC samples.

3.2. NMR relaxation technique

Two different sequences were implemented with the NMR relaxation technique. For both sequences, same sample was used. The samples were cast in cylindrical shape containers with dimensions 63 mm in diameter and 75 mm average height. After casting, the UHPFRC was sealed by using the container's cap to avoid any drying. Note that one additional sample with the same geometry was cast to monitor the temperature evolution due to the heat of hydration. Preliminary analyses showed that there was no significant difference between the results of the UHPFRC matrix and matrix with fibers. That is why in this paper the results of the NMR relaxometry presented are only for the matrix of the modified UHPFRC.

3.2.1. Inversion Recovery (IR) sequence

The 1st sequence, Inversion Recovery with data treatment by Laplace inversion was used to monitor the longitudinal relaxation time T_1 and its distribution. The treatment was obtained by a self-developed program [6] based on well-known CONTIN program, by assuming 256 different predefined T_i (a list of predefined relaxation time) values logarithmically distributed between 75 μs and 40 ms.

The T_1 distribution before setting is related to the mobility of free water between cement grains and silica fume particles for the long T_1 values (6 ms). The T_1 distribution during and after setting is an indication of the development of pore size distribution and thus the microstructure, as schematically shown in Figure 1a [6, 7].

Furthermore by obtaining the evolution curve of the logarithmic mean longitudinal relaxation time (for simplicity is called the main T_1 value) from each distribution, the different stages of hydration and the setting progress could be identified and correlated to the temperature evolution due to the exothermic nature of the hydration process [6, 7].

3.2.2. Free Induction Decay (FID) sequence

The 2nd sequence was the pulse-acquisition which is also called Free Induction Decay, showing the evolution of free water content during the hydration process which signifies the hydration kinetics. By integrating the FID, the free water content inside all the pores of various sizes was accumulated for each measurement. Note that the same logarithmical distribution of T_i values between 75 μs and 40 ms were used in the case of FID measurements as was in the case of the monitoring of T_1 distribution. [6].

3.3. Magnetic Resonance Imaging (MRI) technique

On the old cylindrical concrete substrate having dimensions of 110 mm diameter and 50 mm height (thickness) a fresh UHPFRC having dimensions of 110 mm diameter and 30 mm height was cast as shown in figure 3. SPI sequence was used to visualize profiles of water exchange between the UHPFRC and the dry concrete substrate during the setting and

hardening of the fresh UHPFRC. Detailed information regarding the MRI technique could be found in [9]. Note that only the MRI SPI results of the matrix of the modified UHPFRC are presented here and figure 3 is just a schematic representation of the UHPFRC matrix composite system.

4. RESULTS AND DISCUSSION

4.1 UHPFRC hydration - NMR relaxometry

Figure 1 shows the development of T_1 distributions in the first 48 hours after casting. From the time of casting and before the initiation of setting, the long T_1 values in each distribution are characteristic of the free movement of water located between cement and silica fume particles. On the other hand as shown in figure 1a, short T_1 values are correlated to the presence of water within agglomerates of silica fume and cement grains or also might be related to the early hydrates forming around cement grains (early CSH) where water has less freedom to move.

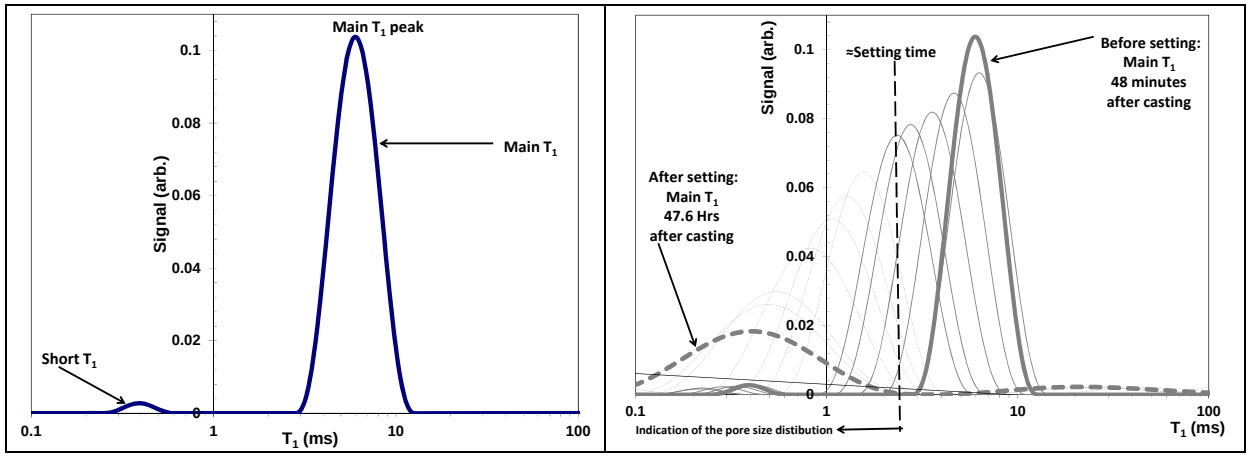

Figure 1: a). Schematic of a T_1 distribution b). Evolution of T_1 distributions in the first 48 hours after casting

However as the material sets, the T_1 distributions shift to smaller values and the signal amplitude of the main peak decreases. Aforementioned is an indication of the development of the pore size distribution, i.e. the microstructural development during and after setting [6, 7]. In order to better quantify these findings, Mercury Intrusion Porosimetry (MIP) technique will be used later on to correlate the evolution of the T_1 distributions to pore size distribution.

Figure 2 shows the development of free water content by FID up to 30 hours, the evolution of main T_1 value and temperature changes due to heat of hydration up to 48 hours. It can be seen schematically that, start of the rapid reduction of the water content, corresponds to the reduction of the main T_1 and the rise of the temperature due to the hydration. Note that by 30 hours, approximately 50% of the free water was consumed.

Figure 2 also demonstrates that by monitoring the main T_1 value, the well-known stages of hydration can be schematically distinguished but in a noninvasive manner (1: initial reaction, 2: dormant, 3: acceleration stage where the initial and final setting time takes place, 4: deceleration period, and 5: slow continued reaction), as accurate as using conventional

methods such as monitoring the temperature evolution due to hydration reactions. Furthermore Faure et al. (2008) have shown that by using the main T_1 vlaue, more detailed information could be revealed about the different stages of hydration [6]. It could be seen that in step 4 and 5, the evolution of the main T_1 peak has reached a stabilized descent and the development is less pronounced than the other steps (steps 1 to 3).

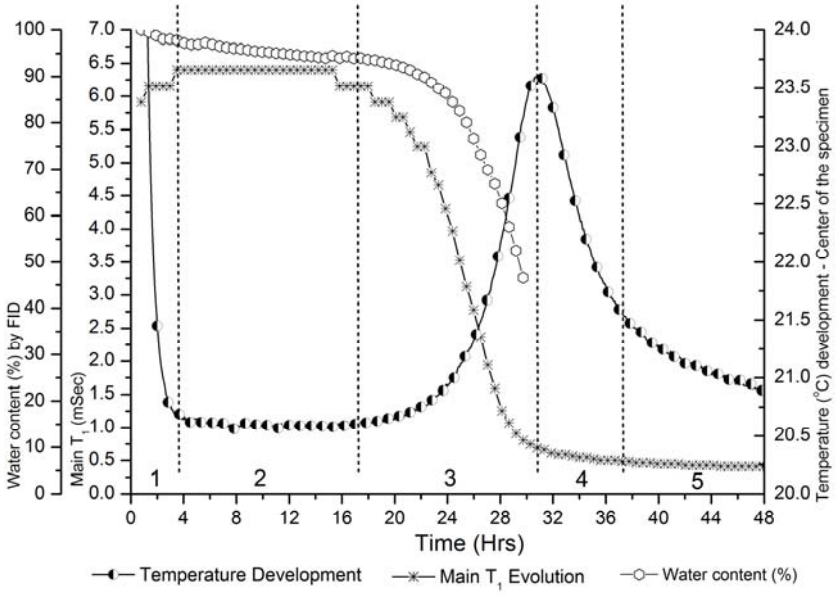

Figure 2: Evolution of the main T_1 peak, temperature changes due to the heat of hydration and consumption of free water content

4.2 Moisture profiles mapping at early age - Magnetic Resonance Imaging (MRI)

Figure 3 shows a side view of the UHPFRC composite system resting on top of the plain UHPFRC, with some space between the two provided by the cap that seals the unbounded sample. Figure 4 shows the results obtained from the MRI SPI measurement for the matrix of UHPFRC. On the left-hand side of figure 4, water exchange between the matrix and the substrate takes place while on the right-hand side of figure 4, the profile of the unbounded UHPFRC matrix could be observed. Note that at the boundaries there are some signal distortion due to the edge effect in both unbounded matrix and the composite system; however this distortion diminishes as the measurement scans take place away from the boundaries.

In the case of the unbounded sealed sample, the uniform decrease in the signal, which represents water consumption, is only due to the hydration reaction taking place. Whereas the signal decrease in the bonded UHPFRC matrix of the composite system is due to two reasons: 1) Exchange of water between the fresh UHPFRC matrix and the dry substrate and 2) Water consumption due to hydration reaction phenomena. After the fresh UHPFRC is cast on the dry substrate, the transfer of water to the dry substrates starts and water from the UHPFRC slowly continues to penetrate in the dry substrate. It is interesting to note that the rate and amount of water being transferred for regular concretes, shown in [9] seems much faster than the results presented here for a UHPFRC matrix.

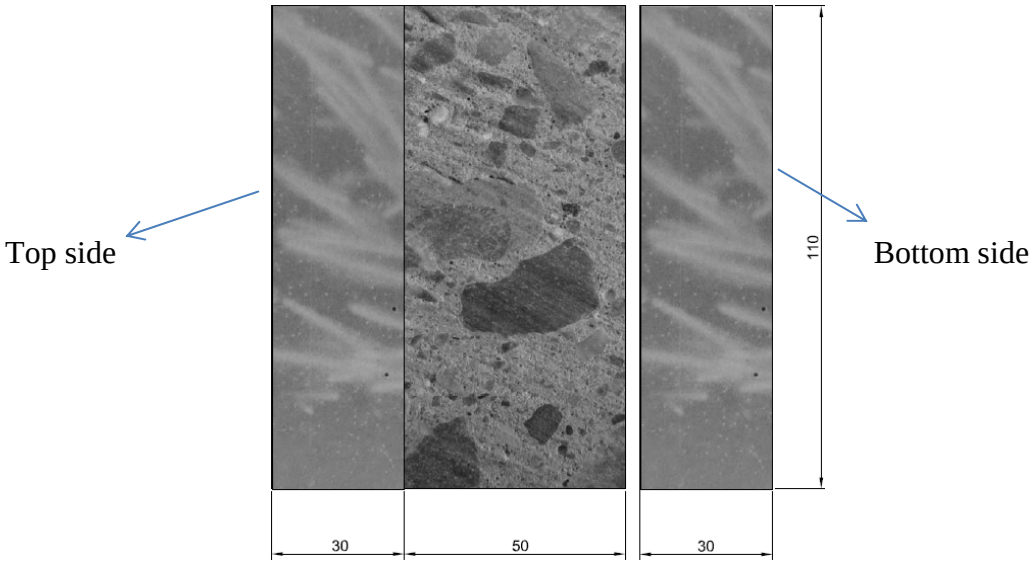

Figure 3: Side view schematic and geometric dimension of the UHPFRC composite system and the unbounded UHPFRC used in the MRI measurement

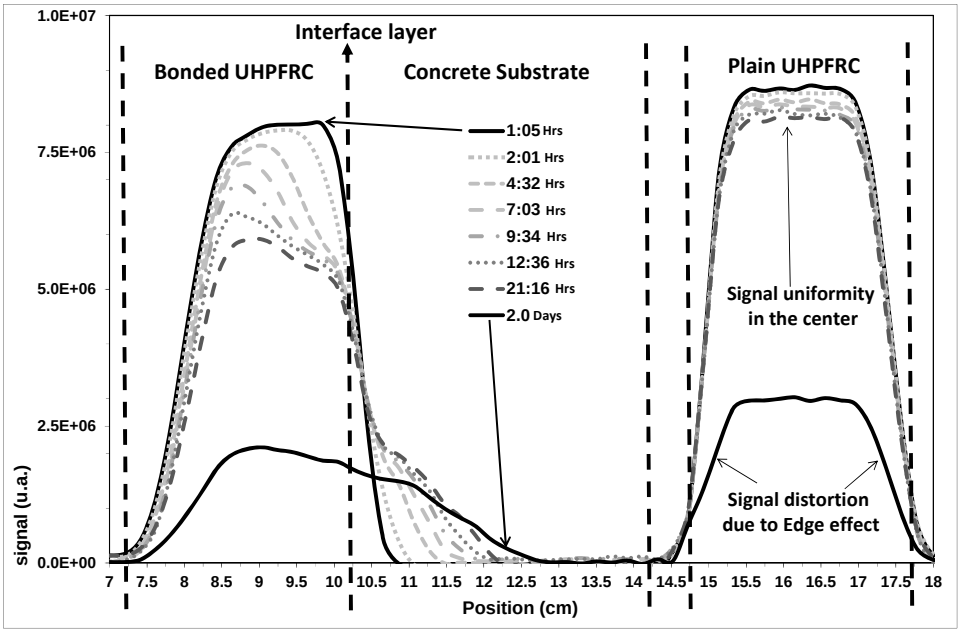

Figure 4: MRI SPI Profile of the UHPFRC matrix

As the setting starts, due to self-desiccation of UHPFRC, strong internal capillary pressure dominates and slows down the transfer of water. Some of the water that was transferred to the substrate tends to find its way back to the interface and to UHPFRC as can be seen between the moisture profiles at 21.16 hrs (just after setting) and 2 days, for the substrate between positions 10.2 cm (interface) and 11.5 cm.

5. CONCLUSIONS

- Following the main T_1 peak, not only the well-known stages of hydration could be identified as accurately as with conventional methods, but also based on [6], more detailed information could be revealed about the different stages of hydration.
- Results obtained by NMR relaxometry analysis showed the rapidness of the hydration kinetics and micro structural development in UHPFRC.
- Results from the MRI technique showed that compared to ordinary or high performance concrete the rate and magnitude of water transfer to the dry substrate are much slower and lower respectively. Furthermore, during the hydration, due to the internal capillary forces, the rate of water transfer from the UHPFRC to the substrate slows down and some of the water migrates back to the interface during and after setting.

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