



Influence of cellulose ether on hydration and carbonation kinetics of mortars



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ABSTRACT

The hydration process of Portland cement and retarding effect of cellulose ether (CE) on hydration and carbonation were studied. The degree of CE-substitution is a major parameter which plays an important role in terms of retardation of both hydration and carbonation. For the hydration process, this CE-effect was highlighted through the results of an experimental campaign based on thermogravimetric analysis (TGA) performed on mortar samples conserved in an ambient air in which the atmospheric CO₂ was absorbed by whitewash solution. This type of conservation is chosen in order to make precise the measurement of dehydration rate by TGA tests. While for the carbonation mechanism, the CE-effect was identified by the measurement of carbonation depth with phenolphthalein spraying.

This paper aims to determinate a coefficient of retardation of hydration according to the CE-rate used in the manufacturing of mortars. This coefficient may be taken into account in the calculation of the reaction rate of anhydrous constituents of cement in order to determine a precise hydration degree of mortars. Consequently, this delay in cement hydration delays the carbonation processes because of the lack of hydrates to react with CO₂.

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1. Introduction

Cement is the most important construction material used nowadays as a binder in different building materials formulations. Its importance is not only due to its binding function in all cementitious materials including polymer modified mortars (PMM), but also to its relatively low costs. In comparison with ordinary cement-based mortars, PMM are more specialized and more sophisticated.

One of the constituent of PMM could be cellulose ether (CE), which is used as an addition in many construction applications. It is introduced into industrial mortars formulations to adjust their workability by controlling the water balance and to provide rheological properties for enhancing applications easiness. Moreover, CE is the most important regulator for aqueous systems because of its high water retention capacity [1]. It increases the viscosity of the liquid as a function of molecular weight and addition level.

However, it induces a retardation of cement hydration [2]. Quantifying this delay is necessary for the control of hardening and its optimisation.

The control of the hydration process [3] and then the carbonation attack, to which coating mortars are particularly exposed [4–6], are one of the many factors which govern the quality of CE-modified-mortars and their physical properties [7]. The study of hydration kinetics of cements has been the objective of many researches. Indeed, Papadakis et al. [8] and Tennis and Jennings [9] consider that each of the four anhydrous constituents of cement hydrates independently of the others. This result was confirmed by Copeland et al. [10] and Granju and Grandet [11] who show that the evolution of hydration degrees is different from one cement constituent to another. Papadakis et al. [8] propose the relationship (1) to calculate the reaction rate of anhydrous constituents of cement.

$$\bar{R}_i = \frac{k_i}{[\bar{i}]_0^{n_i-1}} [\bar{i}]^{n_i} \quad (1)$$

where \bar{R}_i (mol m⁻³ s⁻¹) is the reaction rate of the anhydrous compound i , $[\bar{i}]_0$ and $[\bar{i}]$ (mol m⁻³) are the initial and instantaneous molar concentrations of i , respectively, k_i (s⁻¹) and n_i (–) are the fitting

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parameters (see Table 1). Only few works deal with the quantitative investigation of the CE effects on hydration kinetics and carbonation process in CE modified mortars. Therefore, the first objective of this study is to highlight experimentally the influence of CE on hydration kinetics of mortars used as a thin overlay for repairing damaged structures or in building front coating. For this purpose, thermogravimetric analysis (TGA) method was chosen because it allows quantifying precisely the evolution of bound-water in the material with time. This experimental method was applied on mortar samples at various durations and with different CE-rates (from 0% to 2%). It allows determining the delay coefficient of hydration which must be taken into account in the calculation of hydration degree of cement anhydrous constituents. Consequently, in case of CE containing mortars, Eq. (1) becomes:

$$\bar{R}_i = \alpha \left[\frac{k_i}{[\bar{i}]_0^{n_i-1} [\bar{i}]^{n_i}} \right] \quad (2)$$

where α is an average delay coefficient of hydration of all anhydrous compounds (-). From this study, a relationship was deduced from experimental results. It aims linking the delay coefficient of hydration to the CE-cement ratio. The second objective of this work is to determine the role played by CE on the materials resistance against carbonation, as this phenomenon is depending on hydration degree of the cement [4]. In this framework, an experimental campaign based on accelerated carbonation tests was carried out by considering two different rates of CE in the mortar (0.27% and 0.5%). The phenolphthalein indicator was used to determine the depth of carbonation front while the Scanning Electron Microscopy (SEM) and Mercury intrusion porosimetry (MIP) were performed on mortar specimens to explore their microstructure and to determine their pore size distribution and porosity variation.

2. Experimental program

2.1. Materials

The polymer-modified mortar manufactured had a composition which meets the usual compositions of thin layered PMM [12,13]. It was designed in order to be representative of typical industrial compositions of factory made mortars, especially those used for coatings in buildings. The cement used (300 kg/m³) was a Portland cement CEM I 52.5 R according to the European norms EN197-1. Its chemical composition is summarized in Table 2. Fine siliceous sand, where the chemical composition is given in Table 3, was used (650 kg/m³): 0.15–0.35 mm, with a specific area of 152 cm²/g and a real density of 2.65. Siliceous filler was added to previous components with a proportion of 50 kg/m³ (with SiO₂ > 99%, 6.5 μm mean diameter and 2.7 density). The organic admixture is cellulose ether (methyl hydroxyl propyl cellulose, denoted MHPC), commonly used in factory made mortars to improve their water retention and consistency [12]. Different proportions were added as shown in Table 4, which gives the mass proportions of each solid constituent. The water–cement ratio (w/c) was equal to 1.

After mixing, samples were moulded in prismatic moulds with the dimensions of 160 × 40 × 10 mm. This geometry is used in order to obtain specimens of 1 cm thick, simulating a layer coating. After their turning out, the samples of mortar were covered

with coats of adhesive aluminium on all sides except one (16 × 4 cm²). This side was let in contact with the ambience in the test chamber. For the carbonation tests, the CO₂ diffusion within the specimen is unidirectional through the sample thickness.

For the hydration measurements, some samples were conserved after manufacturing in an ambient air without CO₂. This ambience consists of a chamber with a volume of 0.082 m³ maintained at 20 °C temperature and 65% relative humidity, in which the atmospheric CO₂ is absorbed by a saturated whitewash solution. Otherwise, for the carbonation measurements, other samples were directly submitted to an accelerated carbonation test.

2.2. Tests and protocols

2.2.1. Accelerated carbonation test

The laboratory accelerated test consists in submitting samples to a conditioned chamber with a volume of 0.3 m³ and which provides a controlled CO₂ concentration. The same temperature and relative humidity as the reference chamber without CO₂ were used. The laboratory accelerated carbonation test uses a concentration of CO₂ of 5% given the high porosity of the manufactured mortar due to the high water/cement ratio used in the mix-design.

2.2.2. Thermogravimetric analysis (TGA)

TGA is a type of testing that is performed on samples to determine weight loss in relation to temperature rise. It is the more suitable technique for studying the hydration rate of cementitious materials. This hydration degree can be determined using the following equation [13–14]:

$$\alpha_{\text{hyd}}(t) = \frac{W_n(t)}{M_c \cdot W_n(\infty)} \quad (3)$$

where: $W_n(\infty)$ is the mass of nonevaporable water (g/g of cement) at $t \rightarrow \infty$ corresponding to full hydration of 1 g of cement. It is estimated from the mineralogical composition of cement (Bogue's formula for Portland cement). $W_n(t)$ is the mass of nonevaporable water (g) at time t . It is defined here as the mass loss recorded between 145 and 800 °C [15], minus the mass loss due to CO₂ releases produced by the calcite decomposition between 600 and 800 °C. M_c is the mass of initial anhydrous cement contained in the sample (g).

As it is shown in Eq. (3), the determination of the hydration degree by the thermogravimetric analysis requires the calculation of the nonevaporable water mass (g/g of cement) when $t \rightarrow \infty$ corresponding to full hydration of 1 g of cement. This estimation is a function of the quantity of water consumed by the anhydrous constituents during their hydration given that these different constituents of the clinker do not consume the same quantity of water [16]. Table 5 gives these quantities of chemically bound water for the different anhydrous constituents of the cement used in this study according to [15].

According to Bogue's formula and the cement composition, the water necessary for a complete hydration can be estimated assuming that the four anhydrous are the main constituents of the cement (Table 6).

An experimental study was realized to calculate the delay coefficient of hydration according to the cellulose ether rates used in the manufacturing of mortars (0%; 0.1%; 0.27%; 0.5%, 1% and 2%). TGA were carried out on samples for various times (10; 20; 30 h, 2; 3; 4; 5.5; 7; and 10 days) in order to determine experimentally the hydration degrees for the previous rates of cellulose ether. For each test date, the samples tested were taken from the cross-section of specimens. After being cut, they were crushed to be tested in a powder form and placed in the TGA device. Then, they were heated at a uniform rate of 10 °C/min from 20 °C to 1050 °C in a non-reactive environment (nitrogen gas).

Table 1
Fitting parameters of hydration law according to Papadakis et al. [8].

Constituent i	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
n_i	2.65	3.10	2.41	3.81
$k_i \times 10^5$ (s ⁻¹)	1.17	0.16	2.46	1.00

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