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Carbonation of low-alkalinity mortars: Influence on corrosion of steel and on mortar microstructure

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ABSTRACT

The ability of carbonated mortar to provide corrosion protection of embedded steel was studied on mortars prepared with low-alkalinity binders. Three mortars were investigated, mortar based on ordinary Portland cement, mortar based on high volume of fly ash and silica fume and mortar based on slag and nano-silica. Analysis of impedance spectra after carbonation revealed an increase in electrical resistivity of mortar based on OPC and significant decrease in electrical resistivity of low-alkalinity mortar. After exposure to cycles of wetting and drying steel embedded in carbonated low-alkalinity mortars has lower open circuit potential, lower charge transfer resistance and significantly higher corrosion current density compared to steel embedded in OPC mortar. To explain differences in corrosion protection properties, an extensive microstructural testing of carbonated mortar was performed. Difference in corrosion properties were explained by the changes in mortar microstructure, mainly changes in pore size distribution.

1. Introduction

Nowadays, there is a strong trend of using alternatives to Portland cement clinker [1,2], substituting part of the cement with supplementary cementitious materials (SCMs) [3–5] or adding certain additions into the binder [6–9], all with the aim of enhancing properties of concrete at the same time contributing to the reduction of emissions associated with Portland cement clinker production. The utilisation of certain waste by-products, generated in different industries, in concrete technology has already proven to be efficient in the enhancement of penetrability properties of cementitious materials [10–12]. This enhancement in penetrability properties is usually attributed to the hydraulic and pozzolanic properties of SCMs, because of which additional amount of C-S-H is formed through the reaction of SCMs with portlandite (CH), as well as to their favourable particle size distribution [3,13]. Significant decrease in chloride diffusion coefficient is one of the most frequently reported improvements of concrete incorporating SCMs, compared to Portland cement concrete [11,14]. Since a decrease in chloride diffusion is evident, positive influence of the addition of SCMs on chloride-induced corrosion of steel in non-carbonated concrete is also expected and proven by research [15,16]. Here, however, the fact that concrete is non-carbonated is the prevailing factor, since the pH of pore solution is still sufficiently high to enable stability of passive film formed on the surface of the steel.

For embedded steel surrounding concrete presents both chemical and physical protection from corrosion [17]. Chemical protection consists of a highly alkaline pore solution, ensuring stability of the passive film forming on the surface of the steel. During carbonation of concrete, CO₂ from the atmosphere is dissolving in water and forming carbonic acid that reacts with the different calcium bearing phases inside cementitious matrix, mainly CH and C-S-H [17]. Consequently, initially high pH of the pore solution can be significantly reduced, leading to the weakening of the chemical barrier and consequently leading to loss of stability of the passive film. In general, the carbonation process in civil engineering concrete structures, with the expected service life of 50 to 100 years and based on Portland cement, is considered to progress slowly, mainly due to the sufficient availability of CH for the reaction with the CO₂. Furthermore, it is generally accepted that the physical barrier provided by surrounding concrete becomes even stronger during carbonation. Mainly, properties of Portland cement concrete are considered to enhance during carbonation, through the decrease of concrete permeability and increase in surface hardness, mostly due to the generation of carbonates that fill the pores in concrete microstructure [17,18]. Carbonation was mainly considered as one of the main risks causing premature degradation of structures in nuclear engineering structures, where during several centuries long-term exposure to underground conditions a change in the chemical environment around reinforcement due to the carbonation is inevitable [19].

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With the introduction of numerous alternatives to Portland cement as a binder in concrete and their lower content of CH, many recent studies started to focus on the effect of carbonation on concrete with SCMs, even for civil engineering application [20–26]. Some studies consider that addition of SCMs lowers permeability of concrete, and that the effect of slowing down penetration of CO₂ in concrete is prevailing over faster rate of carbonation of concrete with SCMs [27]. However, the most recent studies clearly show faster carbonation of concrete based on SCMs [22,25,26] and additionally, a considerable influence of carbonation on concrete properties. In the study by Papadakis [11] it was concluded that when SCMs are added as aggregate replacement the carbonation depth decreases, and the effect is inverse when they are added as cement replacement. Research performed by Auroy et al. [22] revealed that during carbonation a decrease in permeability is observed for Portland cement paste, but a significant increase in permeability was obtained for blended cements, attributed to the significant micro-cracking due to the carbonation of C-S-H [23]. A question remains whether this cracking pattern would change once aggregates are introduced in the paste. Additionally, if initially lower permeability of concrete with SCMs is increased due to the carbonation, a question remains what influence will that have on other degradation mechanism, such as corrosion.

Therefore, the aim of this research was to investigate the influence of accelerated carbonation on corrosion of steel embedded in mortar based on Portland cement and two mortars based on high addition of silica rich phases (fly ash, silica fume and nano-silica). The corrosion behaviour was monitored using EIS, linear polarisation (LP) and through monitoring of changes in corrosion potential. The changes in the ability of mortar to provide protection to steel were correlated to the properties of non-carbonated and carbonated mortar, mainly electrical resistivity obtained through impedance spectroscopy (EIS). Lastly, the changes in mortar properties were connected to the changes in concrete microstructure, revealed using thermogravimetric analysis (TGA), X-ray diffraction (XRD) and pore size distribution using mercury intrusion porosimetry (MIP).

2. Experimental program

2.1. Materials

The experimental program was performed using three different mixes (Table 1). The first mix was reference mix prepared with ordinary Portland cement (CEM I 52.5 N Lafarge Le Havre). The second mix labelled “lpH” mix was prepared with blended binder (40% of CEM I 52.5 N Lafarge Le Havre, 30% of fly ash and 30% of silica fume). This mix was studied in previous research [28] as an example of low-alkalinity mix compatible with the environment of the French deep geological repository planned for long-lived nuclear waste [29]. The third mix labelled “nSi” was prepared with slag cement (CEM III/A 42.5 N Holcim Heming) and with the addition of 20 wt% of colloidal suspension of nanosilica (50% in weight). This mix was similar to low-alkalinity mix which was developed for better compatibility with the clayed

Table 1
Mortar mix design.

| Materials | OPC | lpH | nSi |
|-----------------------------------|------|------|-------|
| Cement | 450* | 180 | 450** |
| Fly ash | – | 135 | – |
| Silica fume | – | 135 | – |
| Nanosilica slurry (50% in weight) | – | – | 90 |
| Water | 225 | 225 | 210 |
| Superplasticizer | – | 4.5 | 4.5 |
| Sand | 1350 | 1350 | 1350 |

* CEM I 52.5 N.

** CEM III/A 42.5 N.

environment in the storage of high-level and long-lived radioactive wastes in deep geological repositories [8].

The amount of water and superplasticizer were adjusted to reach similar workability of the mixes. Superplasticizer Masterglenium 27 and calcareous aggregates with maximum grain size 4 mm were used in all mixes. The addition of calcareous aggregates influences all the microstructural analysis, because calcite is found as a product of carbonation, but also in the aggregate. Nevertheless, it was decided to use this aggregate to evaluate whether the same influence of carbonation will be obtained on mortar, compared to the influence previously established on pastes [22,26], especially in the context of paste cracking due to carbonation. Since calcareous aggregate it is the most used aggregate in European countries, mixes prepared present realistic mixes used in the practice.

2.2. Sample preparation and exposure

From each mortar mix, two batches of 2 L of mortar were prepared. First the aggregate and part of the water were mixed together to allow wetting of aggregate. Next, the cement was introduced and the rest of the water was added during mixing, together with the needed amount of superplasticizer. Two mixing speeds were used, first slower (125 rpm) and then faster (250 rpm), to ensure homogeneity of the mix. The mortar was then poured into moulds for analysis of mortar properties and reinforced mortar samples for corrosion study, and vibrated to remove entrapped air bubbles. Next day after mixing, the specimens were demoulded and were then cured for 28 days in curing chamber (90–95% RH and 20 °C).

For microstructural analysis mortar was poured in cylindrical polypropylene mould, diameter 30 mm and 70 mm height. After 28 days of curing, disks for microstructural analysis using XRD were cut from cylinders, using diamond saw cooled with water. The same was done after 6 months of carbonation in carbonation chamber with 3% of CO₂, 55% RH and 25 °C [30]. After XRD, the mortar sample was first cut into smaller pieces and then parts of the pieces were crushed into powder. On pieces of mortar and mortar powder solvent exchange was performed using isopropanol to ensure stoppage of hydration at pre-defined times. Afterwards, the samples were placed into chamber with nitrogen, to avoid carbonation and to dry the sample. Thermogravimetric analysis (TGA) was performed on the mortar powder and mercury intrusion porosimetry (MIP) on pieces cut from the disks.

Reinforced mortar samples were prepared with Ø6 mm rebar placed in the Ø30 mm mould, 70 mm high. Rebars were 50 mm long, with only 30 mm being exposed inside the mortar and the rest of the steel protected first with a layer of epoxy and then with thermo-retractable polymer mixture Araldite 2000. Before protection, the rebar samples were mechanically cleaned and then immersed in 0.5 M H₂SO₄ in order to remove the initial rust. Finally, they were washed with water and weighted on a scale of mg precision. On one side of the rebar a copper wire was welded for electrical connection. The weld was also protected with a layer of epoxy and a layer of polymer mixture Araldite 2000. This connection was necessary to monitor the corrosion potential and obtain impedance spectra of the steel. Nine replica samples were prepared as described and were cured in a humidity chamber for 28 days with controlled humidity (90–95% RH) and temperature (20 ± 2 °C). After 28 days specimens are put into chamber for accelerated carbonation, with 3% of CO₂, 55% RH and 25 °C, from which one group of samples was taken out after 1 month and other group after 6 months. Samples that were taken out from the carbonation chamber after 1 month, were afterwards kept in the climatic chamber (T = 25 °C, RH = 55%) until age of 6 months. After 6 months, all samples were placed in the wetting/drying chamber, programmed to cycles of 30 min of rain and 72 h of drying, to accelerate corrosion propagation.

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