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Corrosion behaviour of bare and galvanized steel in geopolymer and Ordinary Portland Cement based mortars with the same strength class exposed to chlorides

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ABSTRACT

The corrosion behaviour of bare and galvanized steel reinforcements in geopolymeric and cementitious mortars, at three strength classes, has been investigated throughout the curing period and exposure to wet-dry cycles in 3.5% NaCl solution. During the curing, the high alkalinity of geopolymers prolongs the active state of both bare and galvanized steel. During the chloride exposure, fly ash geopolymers give the highest protection to reinforcements. The higher alkalinity of geopolymers compared to cement mortars seems to decrease the minimum free chloride threshold necessary to induce corrosion for galvanized steel, but it increases that for bare steel.

1. Introduction

The increasing awareness that Ordinary Portland Cement (OPC) production causes elevated CO₂ emissions [1] has induced the development of more environmentally-friendly construction materials, including those known as geopolymers [2]. Geopolymers are cement-like materials obtained by the polymerization of a solid aluminosilicate with an alkaline solution, generally based on hydroxides and/or silicates [3,4]. These materials are a subclass of alkali-activated materials (AAMs). In order to obtain a geopolymer, aluminosilicates should have a SiO₂ + Al₂O₃ content > 80 wt.% and a low CaO content, such as class F fly ash and metakaolin [5].

Geopolymeric materials show better durability than OPC-based ones, especially when attacked by sulphates or acids, as well as in the case of alkali-silica reactions [3,6].

In coastal zones, chlorides promote the corrosion of embedded reinforcements and this phenomenon is considered one of the major cause of premature failure of reinforced concrete structures [7]. Methods proposed to mitigate reinforced concrete deterioration include the use of hydrophobic treatments, due to their ability to make concrete less susceptible to water saturation [8], corrosion inhibitors [9,10], stainless steel rebars [11] which are highly resistant to corrosion but very expensive, and galvanized reinforcements. In particular, galvanization of steel reinforcements is a cheaper prevention method against

corrosion if compared to other anti-corrosion methods [12,13].

Zinc is an amphoteric metal, which undergoes severe attacks both in acid and alkaline media. In particular, in alkaline environments the zinc dissolves as zincate ion Zn(OH)₄²⁻. This dissolution continues until the solution becomes oversaturated by these ions, which turn into the precipitate Zn(OH)₂ (or ZnO), yielding a continuous passivation layer [14,15]. In the presence of Ca(OH)₂, as in the concrete pore solution, the protective layer is not only formed by zinc oxide and hydroxide, but mainly by a compact, protective, and highly chloride-resistant layer of calcium hydroxyzincate (CaHZn) [16–21]. The passivation of galvanized steel largely depends on the alkalinity of the environment and the concentration of Ca²⁺ ions [16,17]. The passivation layer may be less protective due to the presence of chlorides or a high amount of soluble alkalis, whereas the formation process is favoured by oxygen availability [22].

It is well-known that steel reinforcements passivate in the alkaline environment of concrete pore solution, but in geopolymers, where NaOH or KOH concentrated solutions are used to activate the aluminosilicate powders, the alkalinity is much higher than in traditional OPC matrices. Alkalis are highly mobile in the pore system of geopolymers and this effect may significantly limit the durability of embedded reinforcements [23]. Moreover, in geopolymers, the content of calcium, which contributes to the galvanized steel passivation, is much less than in OPC matrices.

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Unlike OPC-based concretes, in geopolymers chlorides able to induce reinforcement corrosion do not bind to cement aluminate hydrates forming calcium chloroaluminates, Friedel's salt ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaCl}_2\cdot x\text{H}_2\text{O}$). However, the higher porosity and the higher surface area exhibited by the sodium aluminosilicate hydrate gel (N-A-S-H), in low-Ca alkali-activated materials, thus in geopolymers provide a greater ability on the surface adsorption of chlorides when compared to the calcium aluminosilicate hydrate gel (C-A-S-H), typical instead of high-Ca alkali-activated materials [24,25]. In alkali-activated fly ashes, the presence of halite (NaCl) in the X-ray diffraction pattern of dried specimens of chloride-penetrated regions could possibly suggest the N-A-S-H gel hosting a chloride-rich pore solution [25,26]. Therefore, the permeation of chlorides through geopolymers depends primarily on the resistance of the binder to their transport and their binding ability on producing specific reaction products [24,25].

The information provided by the literature on steel corrosion in geopolymers is still limited and generally focused on simulated pore solution of alkali-activated concretes [25] or fly ash or slag based mixtures [24,26–32], whereas there are no studies on metakaolin-based geopolymers. Moreover, there are only few studies on the performances of galvanized reinforcements embedded in such matrices [33]. Furthermore, in the literature, geopolymeric mortars/concretes generally are not compared to traditional cement-based ones at the same compressive strength class.

Thus, the main purposes of this study are:

- to investigate the passivation capacity of bare and galvanized steel reinforcements in metakaolin (MK) and fly ash (FA) based geopolymers during the curing time;
- to investigate the corrosion behaviour of bare and galvanized steel reinforcements in MK- and FA-based geopolymers in the presence of chlorides;
- to compare the passivation capacity and the chlorides induced corrosion behaviour of bare and galvanized steel reinforcements in FA- and MK-based geopolymers with that of cement based mortars, at the same strength class.

To achieve these purposes, the corrosion behaviour of such type of reinforcements, embedded in R1 ($R_c \geq 10$ MPa), R2 ($R_c \geq 15$ MPa) and R3 ($R_c \geq 25$ MPa) mortars, was investigated. In a previous work [33], the same mortars were compared in terms of fresh and hardened state properties and durability, whereas only the corrosion behaviour of reinforced R3 mortars during the curing period (1 month) was reported. In this paper, the corrosion behaviour was monitored by electrochemical measurements during both the initial curing period and the following 12 weekly wet-dry cycles in a 3.5% NaCl solution. The pH and the content of penetrated chlorides of mortars were determined. At the end of the exposure to the aggressive environment, in order to validate the electrochemical measurements, visual and metallographic cross-section observations of reinforcements were performed.

2. Experimental

2.1. Materials

Class F fly ash (FA) supplied by General Admixture S.p.A., MetaStar[®] 501 metakaolin (MK) supplied by Imerys Minerals Ltd., and calcium aluminate cement (CAC) from Kerneos Inc. were used as geopolymer precursors. Sodium silicate solution supplied by Ingessil S.r.l. ($\text{SiO}_2 = 29.86$ wt.%, $\text{Na}_2\text{O} = 14.64$ wt.%) mixed with NaOH or KOH solutions were used as alkaline activator. NaOH solution was prepared by dissolving NaOH flakes (97–100% purity, Carlo Erba Reagenti S.r.l.) in demineralised water; KOH solution was prepared by dissolving KOH pellets (85% minimum assay, Carlo Erba Reagenti S.r.l.) in demineralised water.

For OPC mortars, Portland cement CEM II/A-LL 42.5R (OPC), with limestone filler content of 15%, and hydraulic lime (HL) UNI 10892 LIC

Table 1
Chemical compositions (wt.%) of FA, MK, CAC, OPC and HL.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	TiO ₂	SO ₃
FA	44.0	29.1	6.0	5.5	1.5	1.1	0.4	0.9	1.1
MK	55.0	40.2	0.6	< 0.1	0.4	2.4	< 0.1	–	–
CAC	4.5	39.5	15.3	37.0	< 1.5	–	–	0.9	–
OPC	29.7	3.7	1.8	59.3	1.1	0.8	0.3	0.1	3.2
HL	8.4	0.1	0.6	65.7	2.6	0.3	< 0.1	< 0.1	< 0.1

3.0 (only in R1 and R2 strength classes) were used as binders.

Commercial calcareous sand (ESINCALCE S.r.l.) with maximum grain size 8.0 mm was used for each type of mortar.

The chemical composition of powdered materials [33] is reported in Table 1.

2.2. Mortar mixes

Mortars cured at room conditions and belonging to two non-structural classes (R1 ≥ 10 MPa and R2 ≥ 15 MPa) and one structural class (R3 ≥ 25 MPa) were considered. Even if R3 mortars are mainly used in reinforced systems, also R1 and R2 mortars can be reinforced in non-structural applications. R1 mortar is mainly used as grouting for precast reinforced masonry blocks; R2 mortar is used for non-structural repairs and for smoothing concrete surfaces also in the presence of steel reinforcements. Moreover, premixed R1 and R2 mortars usually contain HL as cement partial replacement.

In order to reach the structural strength class (R3), OPC-based mortars were manufactured with water/binder (w/b) ratio equal to 0.65 by weight. OPC non-structural strength classes (R1 and R2) were prepared by replacing OPC with HL (80 and 50 wt.%, respectively) at w/b ratios of 0.50 and 0.65, respectively. All OPC-based mortars were prepared with a sand/binder ratio equal to 3 by weight.

As comparison, three types of geopolymeric mortars (labelled as FANa, FAK and MKK) were prepared with a sand/binder ratio equal to 3 by weight and different water/binder (w/b) ratios. FANa mortars were manufactured with FA and NaOH; FAK mortars were manufactured by replacing 8.0 wt.% of FA with CAC and KOH; MKK mortars were manufactured with MK and KOH. To modulate the mechanical strength of geopolymers, different concentrations of the activated solution were prepared by lowering the w/b ratio and by dissolving different amounts of NaOH or KOH in demineralised water. Details of mortar mixtures are reported in Table 2 [33].

Mortars were maintained at RH = 95 \pm 5% and T = 20 \pm 1 °C for the first 2 days and then at RH = 50 \pm 5% and T = 20 \pm 1 °C.

2.3. Methods

2.3.1. Microstructural analyses of mortars

The pore structure of a binder material plays an important role in the movement of ions from the external environment towards the steel-mortar interface. In order to investigate the pore structure of mortars, three small fragments of each manufactured mortar were tested by mercury intrusion porosimetry (MIP) (Thermo Fisher 240 Pa) after 28 days of curing and the average results are reported.

2.3.2. Electrochemical measurements

The corrosion performance of reinforcements was evaluated by free corrosion potential (E_{corr}) measurements, using a Saturated Calomel Electrode (SCE, +0.241 V vs SHE) as reference. The corrosion resistance of reinforcements was evaluated by polarization resistance (R_p) measurements, using an Amel workstation. The R_p was measured with the galvanodynamic polarization method (scan rate = 0.5 $\mu\text{A}/\text{s}$; $\Delta V = \pm 5$ mV) by calculating the average of the slopes of both anodic and cathodic branches.

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