



# Corrosion of steel bars induced by accelerated carbonation in low and high calcium fly ash geopolymer concretes



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## HIGHLIGHTS

- Ca content influences the durability of class F fly ash geopolymer concretes.
- Low-Ca class F fly ash geopolymer concrete is suitable for structural applications.
- High risk of corrosion in high-calcium class F fly ash geopolymer concretes.

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## ABSTRACT

Reinforced geopolymer concretes prepared using three fly ashes of varying Ca content were subjected to accelerated carbonation for a period of 450 days. Changes induced in the concretes were determined after exposure to a 5% CO<sub>2</sub> atmosphere, and electrochemical measurements were performed over the time of testing to evaluate the effects of carbonation on the corrosion process of the steel rebars. This study indicates that geopolymer concretes prepared with fly ashes having reduced calcium content are more suitable for the production of durable concrete for structural applications under exposure to carbonating environments, as the properties of these materials seem to mitigate the risk of carbonation-induced reinforcement corrosion in concrete.

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## 1. Introduction

The growing demand for concretes with higher performance, lower cost and reduced environmental impact when compared to those produced with conventional Portland cements has promoted the development of clinker-free alternative cementitious materials including alkali-activated cements, also referred to as 'geopolymers', whose use can contribute to reduction of the carbon footprint of construction projects [1,2]. Geopolymer binders are produced via the chemical reaction of a reactive aluminosilicate source, mainly fly ash derived from the coal combustion process, with an alkaline activator, to produce a hardened monolith that can develop high mechanical strength [3,4]. This reaction can result in the formation of zeolite type phases along with a highly disordered aluminosilicate geopolymer gel [5–7].

Low and high calcium based fly ash stockpiles, classified according to the ASTM standard C 618 [8] as class F when the content of

SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> is greater than 70 wt.% of the total composition of the ash, and class C if the content of SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> is between 50 wt.% and 70 wt.%, can be used for production of geopolymer concretes, although more attention has been given to the activation of low calcium fly ashes. Some studies claim [9] that low calcium fly ashes are more suitable for producing geopolymer materials as these ashes tend to have a higher content of glass and therefore are more reactive; however, different characteristics of the fly ash such as the location of the glass diffraction reflection, degree of vitrification, particle size distribution, the nature and percentage of impurities, and the loss on ignition (LOI) can affect the mechanical properties of the resulting geopolymers [5]. The microstructure and strength development of fly ash geopolymers has been extensively studied over the past decades [10–12], but most of these studies are limited to the assessment of pastes and mortars, even though the ultimate material that is placed in service is usually concrete. There is a general agreement that fly ash geopolymer concretes can develop high mechanical strengths at early age [13–17], which is strongly influenced by the nature of the fly ash [18], the nature and concentration of the activator [19], and

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the curing conditions applied to the material, which can involve high temperature (between 40 °C and 80 °C) [10,15].

The durability of concrete is a key concern in civil infrastructure, as it is strongly dependent on the interactions of the material with the environment, and determines performance during the service life. There is little existing knowledge about the long-term performance of fly ash geopolymer concretes, and this is one of the main limitations facing the adoption of this technology on a large industrial scale [20]. Studies conducted on pastes and mortars reveal that fly ash geopolymers can show remarkable resistance to acid attack [21,22], can perform better than Portland cement when exposed to sulfate solutions [23], and can be highly stable when exposed to high temperatures [24].

Carbonation is defined as the chemical reaction taking place between the reaction products of a cementitious matrix and the CO<sub>2</sub> in the atmosphere, promoting the formation of carbonates [25]. It has been identified that alkali-activated materials are prone to damage when assessed under accelerated carbonation conditions [26]. Criado et al. [27] identified formation of nahcolite (sodium bicarbonate) in low-calcium fly ash geopolymer samples cured under atmospheric conditions, which was associated with the carbonation of the alkalis in the pore solution. Bernal et al. [28] also observed formation of different sodium carbonate products upon accelerated carbonation of low calcium fly ash geopolymer samples, depending on the CO<sub>2</sub> concentration of exposure used. In these fly ash geopolymers, nanostructural changes were not detected after CO<sub>2</sub> exposure; however, fully carbonated samples lost their loading capacity and could be easily destroyed by hand. Carbonation data for high calcium fly ash geopolymers have not been reported in the open literature. This elucidates that the carbonation phenomenon in fly ash geopolymers is a critical aspect of durability that needs to be studied, as it seems to be one of the most detrimental durability aspects of these materials.

Corrosion of steel in reinforced concrete structures is one of the major durability issues faced by civil infrastructure components, and it strongly controls their life-span. Undamaged concrete provides the pH conditions necessary to promote the passivation of the steel rebars, which protects them against the effects of ingress of aggressive agents such as chlorides. In Portland cement-based concretes the pH is mainly controlled by the hydration product portlandite (Ca(OH)<sub>2</sub>); however, in the case of low calcium fly ash geopolymer concretes this phase does not form, and therefore the pH of the system is controlled solely by the pore solution. Upon carbonation of ordinary Portland cements, the consumption of portlandite during carbonation leads to a reduction in the pH and the consequent corrosion of the steel rebars. In the specific case of fly ash geopolymers, it is still unknown what are the effects of carbonation of the matrix related to the corrosion of the metallic component.

The volume of research assessing corrosion resistance of steel reinforcement embedded in fly ash geopolymer matrices is quite limited, although previous studies do suggest that fly ash based geopolymers mortars are able to passivate the steel reinforcement. The stability of the passive layer depends on the concentration of the activator solution [29,30], as this controls the electrochemistry of the pore solution, and it has been observed that reinforced fly ash geopolymer specimens perform similarly to Portland cement systems. Recent studies focused on the assessment of corrosion of carbon steel [31] and stainless steel rebars [32,33] embedded in polluted (carbonated/chloride enriched) fly ash geopolymer mortars, and in simulated pore solution, identified that the stability of the passive layer forming under the pH conditions within these materials is also strongly dependent on the type of activator used, as this factor controls the pore network of the cement and consequently influences the permeability of the material. In fly ash geopolymer concretes, high resistance to corrosion

induced by chlorides compared with Portland cement specimens has been observed [34,35], associated with the low chloride permeability of these materials.

This study focuses on the assessment of the effect of accelerated carbonation on concrete matrices produced by alkali-activation of three different class F fly ashes, and its effect on the half-cell potential and linear polarization resistance of the steel rebars embedded in these concretes over 450 days of CO<sub>2</sub> exposure. Changes in the pH of the concretes are assessed using phenolphthalein and alizarin yellow R indicators, and the pore structure and carbonation products in the interface between rebars and concrete matrix are evaluated using mercury intrusion porosimetry and X-ray diffraction respectively. Corrosion rates of the metallic component embedded in carbonated fly ash geopolymer concrete specimens are thus calculated.

## 2. Experimental procedure

### 2.1. Materials

Three fly ashes, all Class F according to ASTM C 618 [8] were used as main precursors for the production of the geopolymer concretes, obtained from Dolet Hills power generation station (PGS) located in Mansfield, LA (abbreviated DH), Avon Lake PGS, OH (abbreviated OH), and Monticello PGS, Mount Pleasant, TX (abbreviated MN) [36]. The chemical compositions of the fly ashes determined by X-ray fluorescence (XRF) spectroscopy are shown in Table 1. Particle size distributions (PSD) of the fly ashes were determined using a PSD analyzer with a measurement range of 0.02–2800 μm. The results of the PSD analysis are summarized in Table 2.

The activator solution consisted of a blend of a commercial sodium silicate with 14 M sodium hydroxide. The sodium silicate used in this work was 45% dissolved solids by weight, and a ratio SiO<sub>2</sub>/Na<sub>2</sub>O of 2:1. The activator solution was produced as a 1:1 blend by mass of the sodium silicate and sodium hydroxide solutions. For the preparation of the concretes, fine aggregate (commercial sand) and P-gravel were used. The sand had a bulk density of 1680 kg/m<sup>3</sup> and a specific gravity of 2.63. The P-gravel has a maximum particle size of 9.5 mm and a bulk density of 1960 kg/m<sup>3</sup>.

### 2.2. Concrete sample preparation

Geopolymer concrete specimens were produced by mixing the activator solution with the designated fly ash until a homogeneous paste was obtained, followed by the inclusion of fine and coarse aggregates. The geopolymer specimens were formulated with a binder content of 489 kg/m<sup>3</sup> of fresh concrete, and an activator to binder ratio of 0.5. In this study constant activator to binder ratio was maintained for all the samples. The activator/binder ratio was selected in order to achieve a desired workability and to replicate mix designs adopted in previous studies [35] evaluating fly ash geopolymer concretes using materials from the same sources used here. Cylindrical reinforced concrete specimens, 15 cm height by 7 cm diameter, were cast and cured at 80 °C for 72 h. A single carbon steel deformed rebar, 300 mm in length and 6 mm diameter, was sand blasted and placed at the center of each cylinder mold prior to casting of the concrete. The elemental composition of the 1018 carbon steel rods was: carbon 0.14–0.2 wt.%, manganese 0.6–0.9 wt.%, sulfur 0.05 wt.%, phosphorus 0.04 wt.%, and iron 98.81–99.26 wt.%.

### 2.3. Accelerated carbonation

After 72 h of curing, the concretes were subjected to accelerated carbonation in an environmental chamber at a temperature of 24 ± 5 °C and relative humidity of 65 ± 5%. A CO<sub>2</sub> gas tank was used to pass the gas inside the chamber and was tightly sealed to prevent leakage as shown in Fig. 1. The rate of CO<sub>2</sub> injection was 100 cm<sup>3</sup> STP/min, corresponding to a CO<sub>2</sub> concentration of 5.0 ± 0.3%.

Control samples were stored under the same environmental conditions (T = 24 ± 5 °C and RH 65 ± 5%) without supplying CO<sub>2</sub>, for the length of this experiment. It is important to note that the control specimens were not isolated from the CO<sub>2</sub> in the atmosphere, and therefore it is likely that some natural carbonation took place over the time of testing. In the context of the present study, the results obtained from the assessment of these specimens are treated as representing 'non-carbonated' reference samples for comparison purposes.

### 2.4. Characterization of carbonated specimens

#### 2.4.1. Physical characterization of carbonated concretes

The carbonation depth of the geopolymer concretes after 450 days of CO<sub>2</sub> exposure was evaluated using two pH indicators: phenolphthalein and alizarin yellow R. The phenolphthalein and alizarin yellow R indicators were each produced as a 1% solution in alcohol. The color transition of phenolphthalein begins to occur at a

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