



# The role of graphene oxide in limited long-term carbonation of cement-based matrix

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## HIGHLIGHTS

- Cementitious matrix with GO showed very limited long-term carbonation.
- GO enhanced microstructure and reduced porosity of cementitious composite.
- GO showed an interlocking effect of calcium and carbonate ions.

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## ABSTRACT

Carbonation leads to a drop in the alkalinity of cementitious materials because carbon dioxide mainly attacks calcium phases and converts them into carbonated phases. The carbonation rate depends on the ease with which carbon dioxide can proceed from outer to internal surfaces. In this study, graphene oxide (GO) is used to enhance the resistance of cement matrix against carbon dioxide attack. Graphene oxide was added to cement mix sufficient amount of water to produce cementitious matrix. Visual colour change, porosity and compressive strength tests were performed to investigate effect of the carbonation on cementitious materials. The results show very low carbonation depth in the graphene oxide cement mix. After 18 months, the plain cement mixture was nearly fully carbonated compared to only about 4 mm for the GO mix. This significant result can be attributed to the influence of interlocking of GO to different anionic and cationic ions.

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## 1. Introduction

Carbonation is an important cause of steel reinforcement corrosion which leads to the deterioration of reinforced concrete structures and shortens their service life. The reaction takes place in the aqueous media of cementitious matrix inside the pore water system at the surfaces in contact with the atmosphere [1]. Carbon dioxide diffuses into cementitious materials and reacts with alkaline compounds/environment. Such a reaction causes a drop of pH of cementitious matrix from 12–13 to about 9. Due to the pH decrease, the passivation layer around steel depletes and the corrosion rate can increase [2]. Although the pH drop of concrete is the most well-known effect of carbonation, long-term carbonation can cause dramatic reduction of compressive strength [2,3]. It has been widely reported that the carbonation rate of cement-based

materials depends mainly on the microstructure, which includes hydration products (calcium hydroxide, calcium silicate hydrate, alkaline oxides, etc.), and the pore structure (pore size distribution, total porosity, pore saturation, etc. [3–5]. Hence, it is crucial to understand the role of the hydration products and pore structure to find a way to control the carbonation process. In this study, graphene oxide (GO) has been used to enhance hydration of cementitious material in order to make it more durable and resistant to carbonation. The role of GO will be seen into two concepts: the transport of CO<sub>2</sub> molecules into cementitious matrix through pore network and the reaction of these molecules with Ca(OH)<sub>2</sub> and other hydration products.

Recently, GO has attracted research interest due to its remarkable physical, electric and mechanical properties [6,7]. It has been reported that GO has the ability to integrate with other functional materials at a molecular/nanometre scale to form functional GO-based hybrid materials. The reason behind GO's functional characteristics is the oxygen-containing functional groups which make GO able to enhance the chemical and physical properties of

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the host material [7–9]. Investigation of the impact of GO addition on cement matrix microstructure can assist researchers to understand the transport behaviour of CO<sub>2</sub> in cementitious materials containing GO. In addition to the influence of GO on the microstructure of cement matrix, researchers have reported its ability to interlock different species such as a large molecule of carbon dioxide [10]. GO sheets have various collation states, starting from a monolayer up to the interlocking of many sheets to form films with different thicknesses. Furthermore, GO sheet has a high folding tendency due to its high aspect ratio [11,12]. These properties of GO make it very efficient as an obstacle agent for molecule transport. Moreover, GO has high surface area; the estimated surface area of completely exfoliated GO is about 2630 m<sup>2</sup> g<sup>-1</sup> in which the surface chemistry and flat geometry is actively desirable [13,14]. Therefore, the use of GO seems promising as an effective agent in enhancing durability of cementitious materials.

## 2. Materials, specimen preparation

### 2.1. Characterization of graphene oxide

GO solution by a concentration of 4 mg/mL in water was supplied by Graphenea Company, Spain. Fig. 1 shows the X-ray diffraction (XRD) analysis of GO. XRD analysis shows an intense peak at 10°. When natural graphite flake is completely oxidized to GO, a new signified broad peak at 10° appears due to the increase in spacing between the basal planes from 0.34 nm to about 0.7 nm [15].

Fig. 2 shows the XPS analysis of GO. The C1s XPS analysis of GO shows the aromatic C–C bonds at BE = 283.9 eV and it represents 67.66%; the O1s band is at 529.9 eV and it represents 29.09%. Furthermore, there are very broad peak, starts from 284 to 290 eV, indicating a remarkable degree of oxidation besides the presence of various oxygen-lifting groups [16].

### 2.2. Specimen preparation

Same water to cement ratio was used in all mixes, the control mix without GO (CM) and the cement mix with GO (GM). Ordinary Portland cement supplied from cement Australia and natural Australian sand were used to fabricate test specimens. The dry contents were first mixed for 2 min in the electric mixer at a moderate speed. Then, GO and water were added to dry contents and stirred for 3 and 2 min at high and moderate speeds respectively. The fresh mixture was cast into cylindrical moulds of 100 mm diameter and 50 mm height and compacted to remove the entrapped air. After that, specimens were air-cured for 24 h in their mould. Finally, all specimens were demoulded and moist-cured at room temperature for an additional 28 days.

### 2.3. Carbonation of samples

Samples were placed in a carbonation chamber under specific conditions. CO<sub>2</sub> concentration was 5% with a relative humidity of 65% and the temperature of 21 °C. Periodically, samples were removed from the chamber and tested for carbonation progress. Following RILEM CPC-18 Measurement of hardened concrete carbonation.

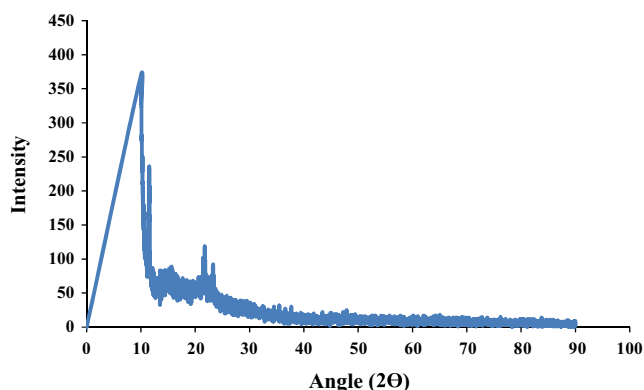


Fig. 1. XRD analysis of GO.

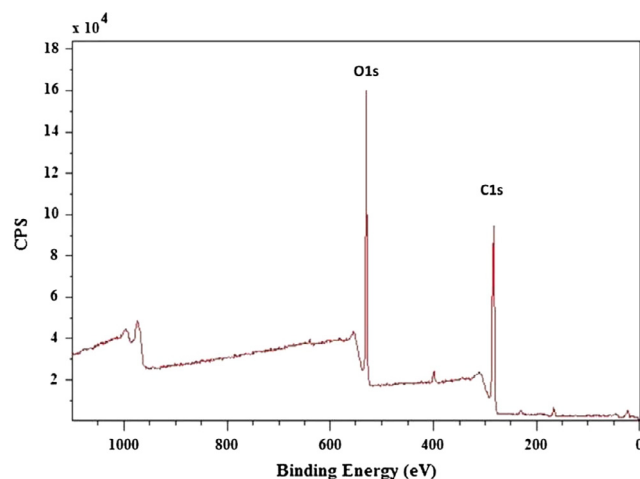


Fig. 2. XPS analyses of GO.

## 3. Test methods

### 3.1. Tests for monitoring carbonation progress

#### 3.1.1. Phenolphthalein colour change

Carbonation depth was tested using a solution of phenolphthalein indicator, by spraying the indicator on freshly exposed surfaces. The pink areas indicate the presence of alkaline in the cementitious materials with pH values in excess of 9; the colourless areas indicate lower levels of pH. All specimens were removed from the carbonation chamber at specific ages. The samples were cut into two halves and the freshly cut surfaces were tested using phenolphthalein indicator spray. The depth of carbonation measurement was carried out immediately after the broken surface was exposed to phenolphthalein indicator followed by a second reading taken after 24 h.

#### 3.1.2. MIP

This test method is used to determine the volume and the distribution of pores in GO cement matrix. AutoPore IV 9500 was used to conduct the test by applying different levels of pressure to a sample immersed in mercury. The test was conducted following the standard test method described by ASTM D4404-10. The test specimens were small pieces weighting about 1 g derived from each sample.

#### 3.1.3. Phase analysis

Samples from noncarbonated and carbonated specimens were prepared for XRD scanning as follows. Each sample was crushed to a fine powder; any aggregate particles were extracted and then the powder was sieved using 75-μm mesh. The powders were sealed in plastic bags and stored for the XRD scan. The scan angle was from 10 to 90°.

#### 3.1.4. Thermogravimetric analysis

The same powders used for XRD were used for thermogravimetric analysis (TGA) testing. The temperature rate and range were 10 °C/min and 10–900 °C respectively.

#### 3.1.5. BET isotherms analysis

The Burnauer-Emmett-Teller (BET) isotherm is used in this study to monitor the effect of carbonation on the pore structure of cement composites. Small portions weighting less than 1 g were subtracted from each mix at specific storage times in the carbonation chamber. Test samples were preconditioned prior to the

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