



Hydration kinetics, pore structure, 3D network calcium silicate hydrate, and mechanical behavior of graphene oxide reinforced cement composites

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HIGHLIGHTS

- The effects of PC modified GO on the hydration kinetics, pore structure and C-S-H gels were systematically investigated by a combination of isothermal calorimeter, MIP, nitrogen isotherm adsorption analysis, XPS, and NMR.
- NMR results showed that the polymerization degree of C-S-H gels was increased with the incorporation of GO.
- A 3D network structure of GO modified C-S-H gels was originally proposed that GO intercalated into the interlayer space of C-S-H gels through ionic bonding with Ca^{2+} and filled in the gel pores.

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ABSTRACT

The effects of graphene oxide (GO) on the hydration kinetics, pore structure, mechanical properties, and the structure of calcium silicate hydrate (C-S-H) gels were systematically investigated by combinatorial techniques. GO can accelerate the cement hydration, refine the pore structure and increase the polymerization degree of C-S-H gels due to the nucleation effects of GO. The specific surface area of cement paste and the number of gel pores were reduced with the addition of GO. A 3D network structure of GO modified C-S-H gels was originally proposed based on the obtained results that GO could intercalate into the interlayer space of C-S-H gels through ionic bonding with Ca^{2+} and fill in the gel pores. A small fraction of GO (0.022 wt%) increased the 28 d compressive strength by 16.31%–25.60% at a various water to cement ratios, indicating that GO is a potential nano-reinforcing material for cement composites. The results would provide a well understanding of the reinforcing mechanisms of GO in cement composites and pave a pathway for the design of high-performance cement composites.

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1. Introduction

Concrete retains its popularity as the construction material in the field of engineering all over the world. However, the major drawback of cement-based materials is the brittle nature [1,2]. Many attempts have been developed to enhance the performance of cement composites by the introduction of reinforcement mate-

rials. In the last decades, a wide variation of fibers, including steel fiber, carbon fiber, and polymer fiber, have been used to overcome the brittle failure of cement composites [3–6]. However, since the size of these conventional fibers is usually at micrometer or millimeter scale, their reinforcing effects are confined to the macro- or mesoscales [7]. It is well established that the nature of calcium silicate hydrate (C-S-H) gels has controlling effects on the properties of cement composites because the nanocrystalline regions of C-S-H gels make up about 60% ~ 70% of the hydration products [8,9]. Therefore, it is essential to find a reinforcing material that could interact with C-S-H gels at the nanoscale to optimize the cement composites.

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The ongoing development of nanomaterials has provided opportunities to make it possible to reinforce cement composites at the nanoscale. The recent emergence of graphene, a flat monolayer of SP^2 bonded carbon atoms in a two-dimensional honeycomb lattice with high aspect ratio and exceptional mechanical and electrical properties, has attracted tremendous attention [10]. Graphene oxide (GO), a derivative of graphene, can be viewed as a layer of graphene with oxygen-containing functional groups grafted on its surface through oxidation reactions, which make GO hydrophilic and easily dispersed in water [10].

The large specific surface area and active functional groups make GO an attractive candidate to reinforce cement composites and much research has been carried out about it. The tensile strength was reported to be increased by 48% with the addition of 1.5 wt% GO in cement matrix [11]. With the addition of 0.02 wt% and 0.03 wt% GO, the compressive strength and indirect tensile strength were improved by about 13% and 41%, respectively [12]. In our earlier study, the introduction of as little as 0.022 wt% GO can increase the compressive strength, flexural strength, Young's modulus and flexural toughness by factors of 34.1%, 30.3%, 32.3% and 33.0%, respectively [13]. Aliakbar et al. [14] conducted a comprehensive study to evaluate the influence of GO concentration on the mechanical properties of cement mortar composites. Further investigation was performed by the research team to evaluate the influence of oxygen functional groups and defectiveness of graphene structures on the mechanical properties of graphene-cement mortar composites [15]. The results showed that with the optimum concentration of 0.1% GO that increased the tensile and compressive strength of the composite by 37.5% and 77.7%, respectively. An addition of 0.1% rGO prepared by 15 min reduction and 0.2% (wt%) hydrazine with a mild level of oxygen groups resulted in a maximum enhancement of 45.0% and 83.7%, respectively, in the 28-day tensile and compressive strengths. Slightly different results were reported by Li et al. [16] showing that the maximum flexural strength was achieved by GO dosage of 0.04% with the increase of 14.2% relative to that of the reference sample. Lu et al. [17] studied the effect of GO on the mechanical behavior of the Strain Hardening Cementitious Composites (SHCCs), the results indicated that 0.08 wt% GO leads to 24.8% increase in compressive strength, 37.7% increase in tensile strength, 80.6% increase in flexural strength and 105% increase in flexural toughness.

In addition to strength gain, the durability-related properties of cement composites can also be improved with the addition of GO. Chloride diffusion tests were carried out by Sanjayan et al. [18] to evaluate the effects of GO on the transport properties of cement composites. The results demonstrated that incorporation of a very low fraction of GO could effectively hinder the ingress of chloride ions. Another study of Sanjayan et al. [19] showed that the cement composites reinforced by GO exhibited higher freeze-thaw resistance compared to the control mix. Moreover, the addition of GO can bring certain smartness and multifunctions to cement-based materials. A cement matrix with a shielding effectiveness of 46 dB (>99% attenuation) had been successfully fabricated by incorporating 30 wt% GO along with an appropriate amount of ferrofluid [20].

Extensive research has been conducted to investigate the reinforcing mechanisms of GO in cement matrix, however, the role of GO on cement composites has not been clearly defined in the literature due to the controversial results.

It has been proposed that GO could provide nucleation sites for hydration products, resulting in a higher degree of cement hydration [21–23]. Gong et al. [23] reported that with the addition of 0.03 wt% GO, the nonevaporable water content and the calcium hydroxide content in cement paste were increased by 9% and 6%, respectively, suggesting that the addition of GO sheets can enhance

the degree of cement hydration. On the contrary, Wang et al. [24] observed that with the addition of 0.02 wt% GO, the cumulative hydration heat of cement and the rate of heat release decreased over 50% compared to the plain paste. A recent study of Ghazizadeh et al. [25] indicated that GO accelerated the hydration of alite only marginally due to GO aggregation in alite paste. Similar results were found by Li et al. [26] that GO aggregation occurred immediately after introduced into cement paste suspension. Therefore, it was GO aggregates rather than the dispersed GO nanosheets served as the nucleation sites of hydration products.

Another mechanism raised by many studies is the strong interfacial bonding between the functional groups of GO and C-S-H gels [27–30]. However, it is acknowledged by many groups that the functional groups of GO are not chemically stable in the high alkaline solution [31,32]. The findings of the study of Ghazizadeh et al. [25] showed that the hydrophilic functional groups present on the GO surface were reduced when exposed to high-pH electrolytes containing calcium cations. They make a conclusion that the idea of interfacial bonding between GO and C-S-H gels suggested in previous studies is probably invalid since GO reduces at high pH and loses the majority of functional groups. Therefore, further validation studies are needed to identify the stability of the functional groups of GO in the alkaline environment of cement paste.

Moreover, no consistent analyses on the influences of GO on the pore structure of cement paste can be found in the literature so far. Sharma et al. [21] reported that the total pore area, median pore diameter, and total porosity reduced with the incorporation of GO. Similar results were obtained by Long et al. [33] that the addition of GO reduced the total porosity and the threshold pore diameter compared to the reference mortar without GO. However, in the study of Sanjayan et al. [18], the addition of GO increased the number of small pores (10–15 nm) and the portion of capillary pores in the range of 100–1000 nm as well as the total porosity of cement mortar. The nitrogen adsorption analysis conducted by Pan et al. [27] showed that with the addition of GO, the pore volume of cement paste in the pore diameter of 1–80 nm was increased. Results also showed a significant increase in the volume of small gel pores (1–10 nm) and medium pores (1–45 nm) for the composites containing GO.

Despite the research enthusiasm on GO reinforced cement composites, the investigation of the effects of GO on the structure of C-S-H gels, the main constituent of hydrated Portland cement, is limited. In the paper of Hou et al. [30], the intrinsic interaction between GO and cement hydrates was investigated by a combinatorial approach of experiment and molecular dynamics simulation. The dynamics simulation results revealed that the functional hydroxyl groups in GO can enhance the bonding with C-S-H gels and increase the silicate chain length. However, Yang et al. [34] reported that the C-S-H structure of cement paste with GO was not observed to have undergone any change via analysis of ^{29}Si nuclear magnetic resonance (NMR).

The different conclusions derived from these investigations may be influenced by the different aggregate/dispersion states of GO. Therefore, more elaborate research is required to investigate the reinforcing mechanisms of the dispersed GO sheets in cement composites in further detail. In our research, GO was first modified by polycarboxylate superplasticizer (PC) to achieve uniform dispersion in cement pore solution. Then the mechanical strengths at different GO concentrations and at a various water to cement ratios were determined. Furthermore, the stability of the functional groups of GO in the high-pH solution was characterized by X-ray photoelectron spectroscopy (XPS). Moreover, the effects of the dispersed GO sheets on the hydration kinetics, pore structure, and C-S-H gels were systematically investigated by a combination of techniques, such as isothermal calorimeter, mercury intrusion porosimetry (MIP), nitrogen isotherm adsorption analysis, XPS,

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