



Investigation on dispersion of graphene oxide in cement composite using different surfactant treatments

Samuel Chuah^a, Wengui Li^{a,b,*}, Shu Jian Chen^a, Jay G. Sanjayan^c, Wen Hui Duan^{a,*}

^a Department of Civil Engineering, Monash University, Clayton, VIC 3800, Australia

^b Centre for Built Infrastructure Research, School of Civil and Environmental Engineering, University of Technology Sydney, Sydney, NSW 2007, Australia

^c Centre for Sustainable Infrastructure, Faculty of Science Engineering & Technology, Swinburne University of Technology, Hawthorn, VIC 3122, Australia

HIGHLIGHTS

- The dispersion of GO in water is independent of the sonication degree with only small amount of GO reagglomeration.
- High alkalinity and calcium ions are key factors inducing the agglomeration of GO in cement system.
- Polycarboxylate-based superplasticisers exhibits the most promising results to disperse GO in cement alkaline environment.
- ADVA 210 can disperse GO better in the pore solution environment, which was preferable to preparing GO-ADVA 210 suspension.
- The inclusion of 0.03% GO protected by cement weight increased the GO-cement composite flexural strength up to 67%.

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ABSTRACT

Graphene oxide (GO) is a novel class of two-dimensional nanoscale sheet material due to its excellent dispersibility in water, high aspect ratio and good intrinsic strengths. In order to obtain a well-distributed GO-reinforced cement composites, the dispersion of GO in water, alkali and several ionic species are investigated with the aid of UV–vis spectroscopy. High alkalinity and calcium ions are key factors inducing the agglomeration of GO in cement system. Dispersion of GO in simulated pore solution is the culmination of the alkali and salt experiments. Agglomeration of GO occurred when GO contacted with the simulated pore solution, highlighting the necessity to protect GO against such aggressive media. The test on surfactant compatibility was then carried out to ensure GO was effectively dispersed in polycarboxylate, air-entrainment and Gum Arabic admixtures within the pore solution. Polycarboxylate-based superplasticisers gave the most promising results to disperse GO in cement alkaline environment. Flexural experiments was performed to highlight the importance of fabrication protocol on the mechanical properties of GO-cement composites. The result shows that the amount of 0.03% GO by weight of cement can increase the flexural strength of GO-cement composite up to 67%.

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1. Introduction

Cement composite is an extensively used material for building and construction. However, it has limited structural applications due to poor tensile strength and flexural capacity. These weaknesses in tensile and flexural strength can be associated with pre-existing porous nature of cement composite. However, the development of cracks at the nanoscale can be regulated with

the aid of nanomaterials. Nanomaterials such as nanoparticle, nanofiber and carbon nanotube have been experimented on cement composite, but problem in dispersion remains the common denominator inhibiting the application of nanomaterials. As research into 2D graphene oxide (GO) begins in earnest, it is vital to understand the intrinsic properties of GO nanosheet, specifically its dispersion and compatibility with other components in heterogeneous cement composite [1].

Great strides have been taken to disperse carbon-based nanomaterials for composite reinforcement [2], electronic devices [3] and medical purposes [4]. In the context of cement composite, carbon nanomaterials possess promising potential owing to the superlative elastic moduli approaching 1.0 TPa and tensile strength of 11–63 GPa [5]. However, nanomaterials tend to form clumps

* Corresponding authors at: Centre for Built Infrastructure Research, School of Civil and Environmental Engineering, University of Technology Sydney, Sydney, NSW 2007, Australia (W. Li).

E-mail addresses: wengui.li@uts.edu.au (W. Li), wenhui.duan@monash.edu (W.H. Duan).

because of the strong van der Waals forces of attraction, and the formation of agglomerates within the cement paste is undesirable. Cement paste containing agglomerates usually experiences sudden failure because of the increased stress concentration [6]. Attempts have been made to disperse nanomaterials with the aid of chemical functionalization, surfactants, mechanical mixing or combinations of methods. Maintaining the dispersion of nanomaterials in cement paste requires chemical admixtures. For example, CNT was found to disperse well with polycarboxylate-based superplasticisers [7–9]. Nevertheless, the successful use of surfactants to disperse CNTs in cement is not just restricted to polycarboxylates, with other previous researchers reporting the use of admixtures such as Gum Arabic [10,11] and sodium dodecyl sulfate [12] can also adequately disperse carbon nanomaterials in cement paste.

GO is a two dimensional carbon allotrope with sheet morphology bonded to oxygen functional groups. This functionalization procedure helps making three-dimensional graphite into GO nanosheet. As a graphene derivative, GO consists of a hexagonal carbon network bearing hydroxyl, epoxide, carboxyl and carbonyl functional groups. These oxygen-containing functional groups render GO sheets hydrophilic, facilitating the dispersion of GO in water [13]. The application of mild ultrasonication input and oxygen-bearing functional groups is vital to the GO dispersion in water. Although GO has been dispersed in both aqueous and organic solvents [14,15], the study on dispersion within the field of cement composites is still very limited. The interactions of GO with cement pore solution rich in ions especially at early age are necessary to be investigated.

Since the study on the dispersion of nanomaterials in a multi-phase composite is complex, it is necessary to systematically isolate the factors influencing dispersion of GO. This study aims to shed light on the dispersive behaviour of GO subjected to alkaline and rich ionic environments. Various surfactants and chemical admixtures were used to maintain the separation of individual GO nanosheet. Compatibility issues between GO and commercially available admixtures was investigated. Simulated pore solution was used to analyze the threshold concentration for agglomeration of GO to occur. The UV–visible (UV–vis) spectroscopy was a key instrument for verifying the presence of GO and, more importantly, the degree of dispersion of GO. The suitable chemical admixture was then conformed by correlating dispersion and flexural strength of GO–cement composite.

2. Experimental methods

2.1. Raw materials

GO sheet with a concentration of 4 mg/mL was purchased from Graphenea (Spain). The chemical property of the GO product is shown in Table 1. Identification of the GO nanosheet was performed with Raman spectroscopy in Fig. 1. The Raman spectrum indicates a D-band at 1350 cm^{-1} and a G-band at 1580 cm^{-1} . The in-phase vibration of the hexagonal carbon rings emits the G-band. Meanwhile, the prominent D-peak arises from the structural defects present on the carbon basal plane for the attachment of hydroxyl and epoxide groups on the carbon basal plane [16]. The chemicals used to investigate the dispersion of GO in aqueous solution were sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium chloride (NaCl_2) and calcium chloride (CaCl_2) respectively. The analytically graded salts were purchased from Sigma-Aldrich.

Table 1
Chemical composition of graphene oxide.

Elements	Carbon	Hydrogen	Nitrogen	Sulphur	Oxygen
Composition (%)	49–56	0–1	0–1	0–2	41–50

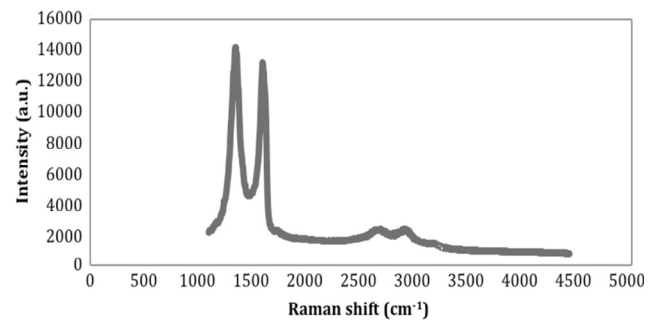


Fig. 1. Raman spectroscopy of GO obtained from Graphenea.

Commercially available surfactants such as Gum Arabic (from Sigma-Aldrich), Microair 905, Sika Viscocrete 6 and ADVA 210 (supplied by Grace Australia Pty. Ltd.) were used for dispersion analysis. The admixtures contained both active non-polar groups for adsorption on the surface of GO and polar groups to attach on cement particles/water in aqueous solution, thereby assisting in the dispersion of both cement particles and GO [17,18]. The cement used for the production of cement paste was provided by Cement Australia, West Footscray, and conforms to the requirements of Type I Portland Cement, as prescribed by the ASTM C150 (Standard Specification for Portland cement). The chemical composition of cement powder was determined by X-ray fluorescence as shown in Table 2.

2.2. GO suspensions and aqueous solutions preparation

To investigate the effect of alkalinity on the stability of GO dispersion, each mix of NaOH and KOH was prepared to have the pH values ranging from 7.0 to 13.5. Pellets of NaOH and KOH were added into distilled water by gradually increasing the mass. The corresponding pH of the solutions was measured using a glass electrode pH meter (Horiba, B-211). For the study on the GO solubility in the presence of ions, NaCl and CaCl_2 salts were each dissolved in separate containers, with the master batch concentrations set at 0.1 g/mL.

The simulated pore solution was prepared according to Ghods' recipe as displayed in Table 3 [19]. Then GO was added into the simulated pore solution until the final concentration was 0.5 mg/mL. This concentration was identical to the pore solution of cement paste containing 0.02% GO by weight of cement for a water-to-cement ratio of 0.4. The dispersion effectiveness of admixtures in aqueous solutions was investigated by preparing suspensions of GO and admixture in small quantities. In a small cylindrical container, 7 mL of GO and simulated pore solution were mixed with 1.0 mL of admixture. Then the suspension was mixed in a high-shear mixer at 4000 rpm for 2 min, according to the cement paste mixing process in ASTM C1738 (Standard practice for high-shear mixing of hydraulic cement pastes). Aliquots of the suspension were extracted and dropped into a cuvette for UV–vis spectroscopy.

2.3. UV–vis characterization of GO suspensions

UV–vis spectroscopy is a widely used method for characterising the dispersion of carbon nanomaterials [5]. Initially applied to investigate the dispersion of CNTs, UV–vis has also proved to be an invaluable tool when applied to GO. As an indirect method, UV–vis analyses the degree of dispersion of GO in a non-quantitative manner. Nevertheless, the underlying concept involves better dispersion corresponding to higher absorbance. A Shimadzu UV 4800 spectrophotometer with a wavelength range

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