



Effect of reduced graphene oxide content on the microstructure and mechanical properties of graphene–geopolymer nanocomposites

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Abstract

Geopolymer nanocomposites with different rGO contents were synthesized by in situ reduction of graphene oxide under alkaline solutions. The effects of rGO contents on the microstructure and the mechanical properties of the rGO/geopolymer composites were characterized systematically. Results showed that GO could be partially reduced in alkaline geopolymeric solutions using relatively short synthesis periods of 15 min at loadings of 0–0.5 wt% at room temperature. The resulting rGO was evenly dispersed in the final amorphous composite structures. Measured fracture toughness values increased 61.5% with increasing rGO contents to 0.5 wt%, reaching 0.21 MPa m^{1/2}. The flexural strength of the rGO/GP reached a maximum value of 17.9 MPa with increasing rGO contents to 0.3 wt%. Strong interface bonding, crack deflection and propagation, rGO pull-out, wrapping and anchoring around geopolymer particles contributed to the improved fracture toughness and flexural strength.

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1. Introduction

Geopolymers have attracted considerable industrial attention as they offer a combination of low densities (700–1800 kg/m³), low costs, easy processing and are environmentally friendly materials [1–4]. This novel class of aluminosilicate material is easily synthesized under highly alkaline conditions through which alumino-silicate oxides reacts with alkali metal silicate solution activated by alkali metal hydroxide solution [3,4]. Previous research [5–8] has explored various kinds of geopolymer matrix composites reinforced with particulates and fibers, targeting methods of overcoming their disadvantages of low strength and susceptibility to brittle failure. Unfortunately, fiber reinforced

geopolymer slurries materials are quite difficult to cast; whereas particulates are difficult to disperse homogeneously, especially for nano-scale reinforcement e.g. MWCNTs [9]. Thus there is a continuing search for alternative methods of introducing high strength/modulus components that are also easy to disperse as means to improve geopolymer composite properties thereby permitting their use in more diverse potential applications such as sensor components, electromagnetic shielding and solid-state electrochemical parts [10–12].

To this end, graphene has inspired enormous interest because it meets the high strength/modulus needs mentioned just above while also offering outstanding, thermal and electrical properties [13–16]. Thus, it appears to offer properties desirable for making superior geopolymer composites providing a simple method can be developed that allows its easy and uniform dispersion [17–19]. Fortunately, graphene oxide (GO) also offers high specific surface area, ultrahigh strength and flexibility. Moreover, its multiple oxygen-functional groups permit facile dispersion in aqueous

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solvents [20]. One especially useful aspect of GO centers around the ease with which the oxygenated moieties are easily eliminated via thermal annealing and chemical treatment generating reduced graphene oxide (rGO) [21].

To date, a number of groups have explored the synthesis of rGO [22–26] reinforced composites finding quite promising properties. For example, Mehrali et al. [24] reported the fracture toughness of a calcium silicate/rGO composite increased by about 120% with 1 wt% rGO. Ramirez et al. [26] compared two types of Si_3N_4 composites containing different graphene sources. The toughness of rGO/ Si_3N_4 was much higher than that of graphene nanoplatelets/ Si_3N_4 sample reaching $10.4 \text{ MPa m}^{1/2}$ for 4.3 vol% rGO. Thus, rGO/nano-composite processing has attracted increasing interest.

In our previous work [27], we were pleased to be able to take advantage of the solubility of GO under the basic conditions and provided an effective way to synthesize the in situ reduced graphene oxide/geopolymer composites. We reported that the GO reduction was mainly attributed to conversion of $\text{C}=\text{O}$ to $\text{C}-\text{O}$ bonds and then elimination of residual oxygen-function groups under the alkaline conditions at 60°C for 72 h. But the effects of rGO contents on the microstructure and the mechanical properties of the rGO/geopolymer composites at room temperature were not investigated.

In this paper, explore various processing parameters in an attempt to optimize both the processing conditions that lead to effective formation of rGO but also with control of phase composition and microstructure of the rGO/GP composites. In addition, the effects of rGO content on the mechanical properties were investigated systematically.

2. Experimental

2.1. Materials

Starting materials were obtained from the following sources. GO was obtained from Nanjing XFNANO Materials Tech Co., Ltd., China (0.5–5 μm in diameter and 0.8–1.2 nm in thickness), KOH (85 wt%, Tianjin Guangfu Indus., China), silica sol (40 wt%, Jiangsu Xiagang, Indus, China) and kaolin (95%, Fengxian Reagent Factory, China, average particle size of 4.1 μm). The metakaolin powders were obtained from heat treated kaolin at 800°C for 2 h in air. The composition of geopolymer matrix used here was $\text{SiO}_2/\text{Al}_2\text{O}_3=4.0$, $\text{K}_2\text{O}/\text{SiO}_2=0.25$ and $\text{H}_2\text{O}/\text{K}_2\text{O}=10$ (mole ratio).

2.2. Composites fabrication

The geopolymeric solutions were prepared by mixing silica sol with KOH for 3 d with the help of magnetic stirring. GO dispersions were prepared with different contents (0, 1.25, 2.5, 7.5 and 12.5 mg/mL) by ultrasonically dispersing GO powders in distilled water for 3 h. Thereafter the obtained GO dispersion 100 mL was added to 1 mol geopolymeric solution and stir for 15 min to accomplish the simple in situ reduced process and obtain rGO/geopolymeric mixture at 25°C . The rGO samples were denoted as rGO0.5, rGO1, rGO3 and rGO5,

respectively. Here, the ratios of GO powders/metakaolin were 0, 0.05, 0.1, 0.3 and 0.5 wt%, respectively.

Thereafter, the rGO/geopolymer composite slurry was prepared by adding the metakaolin powders and mixing for 45 min using a high-shear mixer and ultrasonication. Finally, the slurry was cast into a plastic container and cured at 60°C for 7 d. The reduced graphene oxide/geopolymer (rGO/GP) samples with different rGO contents were abbreviated as rGO/GP0, rGO/GP0.5, rGO/GP1, rGO/GP3 and rGO/GP5, respectively.

2.3. Characterization

Fourier-transform infrared (FT-IR) spectra of GO and rGO were obtained on a Nicolet Nexus 6700 Fourier transform infrared spectrometer using ATR measurement mode. Samples of rGO for characterization were obtained by centrifuging rGO/geopolymer solutions, followed by washing with distilled water and drying in vacuum at 25°C . The size and thickness of the reduced graphene oxide (deposited on silicon substrates) were determined by atomic force microscopy (AFM, Bruker Dimension ICON-Pt). Raman spectra were conducted on a confocal Raman spectroscopic system (Renishaw, In Via) using a 633 nm laser. The phase composition of the rGO/GP composites were examined by X-ray diffraction (XRD, Rigaku, RINT-2000) with $\text{Cu-K}\alpha$ radiation and a transmission electron microscope (TEM, TECNAI G2 FEG, 200 kV). The scanning electron microscope (SEM, FEI QUANTI 200F) was used to observe the fracture surface of the rGO/GP composites.

Flexural strength and Young's modulus measurements of rGO/GP composites were conducted on specimens ($4 \times 3 \times 25 \text{ mm}^3$) using a three-point-bending fixture on an Instron-5569 tester, with a span length of 20 mm at a crosshead speed of 0.5 mm/min. Single edge notched bending (SENB) test was preformed to study the fracture toughness on the specimens ($4 \times 2 \times 20 \text{ mm}^3$) with a span of 16 mm at a cross-head speed of 0.05 mm/min. Five specimens were used under each test condition.

3. Results and discussions

3.1. In-situ reduction of GO

Fig. 1 shows the images of GO dispersions and rGO/geopolymeric solutions with different concentrations before and after in situ reduction. The color of GO dispersions is yellow–brown and deeper with the increasing concentration before reduction (Fig. 1a–d). The solution becomes slightly black after 15 min in situ reaction (Fig. 1e–h). Since the color change is a visible characteristic for GO reduction [20], it proves that the geopolymeric solution plays an important role in reducing GO. Moreover, the solutions exhibit uniformity and stability, which are very suitable for the synthesis of the rGO/GP composites in our work. It is well known that the oxygen functional groups, attached on the basal planes and edges of GO sheets, significantly alter the van der Waals interactions between the GO sheets and therefore permit facile dispersion in aqueous solvents [28]. As illustrated in our previous studies [27], these functional groups of GO sheets can

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