Journal of Physics and Chemistry of Solids 103 (2017) 67–72

Contents lists available at ScienceDirect

Journal of Physics and Chemistry of Solids

journal homepage: www.elsevier.com/locate/jpcs

Synergistic effect of graphene-oxide-doping and microwave-curing on mechanical strength of cement

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ARTICLE INFO

Keywords:
Cement
Graphene oxide
Compressive strength
Microwave curing

ABSTRACT

In this communication, efficient reinforcement of cement matrix was obtained by graphene-oxide (GO) doping and curing treatments. The compressive strength of plain cement is 14.3 ± 0.2 MPa. When the cement contained 0.5 wt% GO, its strength reached 19.4 ± 0.9 MPa. The strength can be further enhanced by curing, which follows the sequence: Microwave-cured GO-cement > Microwave and water-cured GO-cement > Water-cured GO-cement > GO-cement without curing. The highest compressive strength (32.4 ± 0.7 MPa), which was achieved by combining GO-doping and microwave curing, is 126.6 ± 8.1% higher than that without GO-doping and microwave curing. This demonstrates a synergistic effect of GO doping and microwave-curing on the strength of cement composite materials. Furthermore, X-ray diffraction (XRD), Fourier transform Infrared Spectroscopy (FTIR), and field emission scanning electron microscope (FESEM) characterizations revealed that the combination of GO doping and microwave-curing remarkably accelerated cement hydration, leading to the regular and compact structure and thus a high compressive strength. This work provides a new way to improve the mechanical properties of cement composites.

1. Introduction

Cement is a main component of concrete with nano-structures and multi-phases. As an important engineering material, concrete possesses an excellent compressive strength. However, microcracks, which appear during the deformation processing of cementation matrices, keep growing and thus decrease compressive strength. Various types of fibers, such as steel fibers, glass fibers, and polypropylene fibers, are used to inhibit the cracks [1]. The fiber's mechanical properties and geometry determine how the cement will be strengthened [2]. Furthermore, carbon nanotubes have been explored to improve the mechanical properties of concrete. For example, the introduction of carbon nanotubes (CNTs) into cement could lead to an increase (up to 22%) in the strength of the hydrated cement paste [3]. The effects of CNTs on the hydration process of cement and the compression strength and density of mortars were also observed [4–6].

In the past 10 years, graphene has attracted the most attention due to its unique properties. It is the strongest and the stiffest material as well as the most stretchable crystal [7]. Those impressive mechanical natures stimulated interesting research in applying graphene as a superlative filler in reinforced composites [8,9]. Furthermore, graphene oxide (GO) can easily form composites with polymer and ceramic materials with chemical bonds, affecting cement hydration crystal shape and increasing its flexural and compressive strength [10–14]. GO can control the cracks in nanoscale instead of usual micro-size [15] and increase the interfacial strength of Calcium-Silicate-Hydrate (C-S-H) gels in cement [16,17]. Very recently, we found that GO could play a catalytic role to accelerate the hydration rate of cement [18] and the combination of graphene oxide (GO) sheets and single-walled carbon nanotubes (SWCNTs) exhibited an excellent co-effect on the mechanical properties of cement [19].

The mechanical properties of cement care are strongly dependent on curing processes. The most commonly used process method is water curing [20]. The water curing helps to fill the capillary voids via producing cementing compounds to increase the strength of the cement [21]. Furthermore, microwaves (MW), which act as high frequency electric fields, can heat any material that contains mobile electric charges. It was found that MW curing can improve the early age strength of cement [22].

It was demonstrated that MW can reduce GO to graphene that has a higher Young's module and better mechanical properties, because the oxygen functional groups of GO decompose at temperature of 210 °C or above [23–26]. Therefore, it is reasonable to expect that MW-caring would affect the interaction between GO and cement in GO-doped cement materials. However, so far, the effect of MW on GO-doped cement materials has not been explored. The objective of this research

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http://dx.doi.org/10.1016/j.jpcs.2016.12.009
Received 25 September 2016; Received in revised form 13 November 2016; Accepted 12 December 2016
Available online 13 December 2016
0022-3697/

is to investigate the synergistic-effect of graphene-oxide-doping and microwave-curing on mechanical strength of cement. Furthermore, it was demonstrated that the synergistic-effect can increase the compressive strength of cement by 126.6 ± 8.1%.

2. Material and methods

2.1. Graphene oxide preparation

Modified Hummers method was used to prepare the graphene oxide [27,28]. The process is described as follow: 3 g of potassium permanganate, 0.5 g of graphite, 0.5 g of sodium nitrate, and 25 ml of sulfuric acid are mixed at room temperature, followed by keeping at 35 ℃ (with a water-bath and stirring) for 5 h to form a thick paste. Then, 40 ml of deionized water was added into the thick paste with stirring. After it was heated at 90 ℃ for 30 min, 100 ml of deionized water and 10 ml of 30% H_2O_2 was added. Finally, the obtained graphite oxide sample was washed by deionized water for several times, followed by drying in a vacuum furnace at 50 ℃. To prepare graphene oxide solution, the dried graphite oxide was dispersed in deionized water, followed by ultrasonic treatment for 48 h to achieve the full exfoliation of graphene oxide sheets from graphene oxide.

2.2. Preparation and compressive strength evaluation of GO-cement samples

Portland cement type II (No.1124), which was used in this work, consists of 51% tricalcium silicate (C3S), 24% dicalcium silicate (C2S), 6% dicalcium aluminate (C3A), 11% tetracalcium aluminoferrite (C4AF), 2.9% MgO, 2.5% SO_3, 0.8% Ignition loss, and 1.8% CaO [22]. The polycarboxylate superplasticizer (PC), which was purchased from BASF Corporation, is Polyheed 1020 (density: 1.068 g cm^{-3} and boiling point: 100 °C). To prepare a GO-doped cement sample, the cement, sand, water, and polycarboxylate superplasticizer (PC) solution were mixed at the weight ratio of 1: 3: 0.3: 0.1. Then the GO (0.05 wt%, 0.1 wt%, or 0.5 wt%) was added into the mixture with stirring, followed by fitting into the mold (Note: the weight percent of GO is based on the total mass of the composite material). The thickness, width, and length of samples are 14, 14, and 28 in., respectively. All the samples were removed out from molds after 24 h. For those stored in the normal atmosphere environment are referred to the samples without curing. Water-cured samples are those stored in a container covered with wet sponges (saturated with water) until their compressive strength tests. Microwave-cured samples are those stored in the normal atmosphere environment for a period and then treated in a 900 W microwave oven (Emerson, Model MW9339SB) for 5 min before their compressive strength tests. Microwave and water-cured samples are those stored in a container covered with wet sponges for a period and then treated in the microwave oven. Three samples were prepared under each condition. The compressive strength of all samples were tested (using an Intro 2406 mechanical testing machine) for 5 different storing periods, namely 3, 5, 10, 14, and 28 days, respectively.

2.3. Characterization methods

Cement samples with and without GO were subjected to X-ray diffraction (XRD) measurements using a Scintag XDS-2000 powder diffract meter with Cu Kα (λ=1.5406 Å) radiation at 45 kV (voltage) and 35 mA (current). The step size is 0.03° and the scan rate is 1.0°/min. The structures of the samples were characterized by Hitachi-4700 field emission scanning electron microscope (FESEM). A Fourier Transform Infrared Spectroscopy (FTIR) was also used to evaluate the oxygen functional groups of GO and GO-doped cement before and after microwave curing using a FTIR spectrometer (Perkin Elmer, Spectrum One). The FTIR spectra were baselined without normalization. The compositions of GO and microwaved GO were determined by elemental analysis (the Control Equipment Corporation Model 240XA).

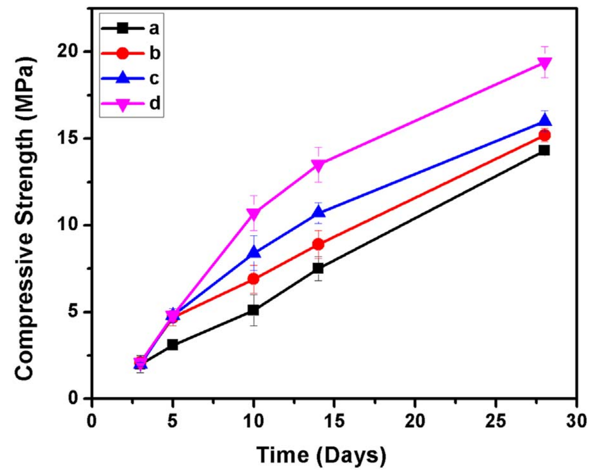

Fig. 1. Compressive strength of cement matrix without curing process with different GO contents: (a) Without GO, (b) 0.05 wt% GO, (c) 0.1 wt% GO, and (d) 0.5 wt% GO (The average values of compressive strengths were calculated considering 95% confidence interval with propagation of errors).

3. Results

The compressive strengths were examined for GO-doped cement samples. Fig. 1 shows that the compressive strength of cement without any curing process increased with increasing GO content. When GO content was 0.5 wt%, the compressive strength reached 19.4 ± 0.9 MPa after 28 days hydration, which is 35.7 ± 0.92% higher than that (14.3 ± 0.2 MPa) of the cement without GO additive. This indicates that GO sheets can remarkable enhance the mechanical property of cement. This happened because GO could regulate the hydration products as a template and accelerate the hydration process of cement as a catalyst. [13–22] More active sites could be provided by a higher GO content. This is supported by the following characterizations.

The effect of curing processes on mechanical properties of cement was evaluated. As shown in Fig. 2(a), one can see that the compressive strength of GO-free cement sample with water or microwave curing is about 2 MPa higher than that without any curing in 5-10 days of hydration. Furthermore, the cement sample with both microwave and water curing exhibited the highest compressive strength on the 28th day of hydration. However, the difference in the compressive strength disappeared for the samples with and without microwave curing on the 28th day of hydration. This is because that the microwave curing could improve only early age strength [5]. In contrast, the microwave and water curing processes can significantly increase the compressive strength for GO-doped cement with an order: Microwave-cured GO-cement > Microwave and Water-cured GO-cement > Water-cured GO-cement > GO-cement without curing (Fig. 2). Furthermore, the larger the GO content is, the greater increase in compressive strength can be obtained by microwave and water curing processes. When 0.5 wt% GO was introduced into cement, water and microwave curing processes increased its compressive strength to 23.1 ± 0.2 and 32.4 ± 0.7 MPa on 28th day, respectively, which are 19 ± 0.92 and 67 ± 1.14% higher than that without curing (Fig. 2(d)). This indicates the high co-effect of GO-doping and microwave-curing, leading 126.6 ± 8.1% increase for the compressive strength, namely, the strength of cement can increase from 14.3 ± 0.2 MPa (without GO doping and any curing) to 32.4 ± 0.7 MPa. Interestingly, the cement cured by both water and microwave shows lower compressive strength (27.6 ± 0.8 MPa) than that with only microwave curing. This may because the MW energy was partially absorbed by water, decreasing the MW effect on GO.

Fig. 3(a) shows the XRD patterns of GO. The interlayer spacing of