



Laboratory investigation of graphene oxide suspension as a surface sealer for cementitious mortars

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HIGHLIGHTS

- Graphene oxide (GO) was used as a new surface sealer for cementitious mortars.
- GO could form a dense layer physically and chemically bonded to mortar surface.
- GO effectively mitigated moisture loss and facilitated the hydration process.
- Surface treatment by GO led to a densified microstructure of cementitious mortars.
- Timing and curing condition influenced the sealing property of GO.

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ABSTRACT

This work investigated the influence of graphene oxide (GO) suspension as a surface sealer on the properties of two types of cementitious mortars (Portland cement mortar and high-volume fly ash (HVFA) mortar) under two curing conditions (wet curing and dry curing). Experimental results showed that GO surface treatment slightly improved compressive strength of wet cured HVFA mortar and dry cured cement mortar while it significantly reduced the water absorption of wet cured HVFA mortar and the gas permeability of both wet cured and dry cured cement mortars. Scanning electron microscopy (SEM) analysis suggested that the GO surface treatment facilitated the hydration process and densified the microstructure of both types of mortars. X-ray diffraction and Thermogravimetry results revealed the formation of hydroxyl grafted AFm phase at the interface of GO/HVFA mortar. Moreover, the GO surface treatment was found to decrease the carbonation of HVFA mortar but increase that of cement mortar.

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1. Introduction

Moisture loss is considered the main reason for the shrinkage of concrete, especially early-age shrinkage. Free water in the hydrating cementitious matrix has the tendency to evaporate out and cause plastic shrinkage and drying shrinkage. In addition, during the cement hydration process, free water is gradually bonded via chemical and physical mechanisms [1], causing autogenous shrinkage. The shrinkage of concrete can induce localized tensile stress if the cementitious matrix is restrained. Cracks can develop when the tensile stress exceeds the local tensile strength. Cracks provide an invasion path for deleterious substances (e.g. water, Cl^- , SO_4^{2-} and

CO_2), which can consequently result in the premature deterioration of concrete structures [2].

Previous studies indicated that internal curing and external curing are two common approaches used for mitigating moisture loss of concrete. For internal curing of concrete, pre-saturated lightweight aggregates (LWA) and super-absorbent polymers (SAP) are two popular materials used [3–6]. They are supposed to be uniformly distributed in the concrete mixture and act as internal water reservoirs to provide curing water to the cement paste and thus promote cement hydration. Furthermore, water-saturated normal-weight aggregates (NWA), wood-derived products, and recycled aggregates also exhibited remarkable role of reserving and releasing water [7–9]. Water-soluble chemicals such as Shrinkage Reducing Admixtures (SRA) showed a potential of internal sealing [10]. In the case of external curing, water curing and sealed curing showed great efficiency in keeping the moisture level

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inside the concrete. Common ways of water curing are water ponding, water spraying, fog misting and saturated coverings [11,12]. The materials often used for sealed curing are water-proof paper, plastic sheeting, and curing membranes [12–15]. Poor construction practices can induce high risk of moisture loss from the concrete and effective solutions (e.g., spraying a graphene oxide suspension as curing compound) are thus highly desirable to mitigate this risk before it occurs. Different from internal curing, this type of measures to mitigate early-age shrinkage-reduced cracking of concrete post construction is rarely explored.

Recently, graphene oxide (GO), has been employed as nano-reinforcement in cementitious mortars or concretes [16]. GO presents a template effect on the hydration process of cementitious binder and regulates the formation of hydration products [16]. The flower-like structure of the hydration products of cement mortar suggested several advantages of GO, including: high values of tensile strength, high aspect ratio (thin layered structure) and surface area (contacting area) [17,18]. High aspect ratio and large contacting area offer GO a great potential to be a good surface sealer for cementitious materials. In this context, the oxygen functional groups, carboxyl and hydroxyl [19], on the GO sheet can actively interact with the surface of cementitious materials and also can significantly improve its dispersion in water [20]. Compared with ordinary graphene sheet, GO could be more evenly distributed in the cementitious matrix and thus effectively facilitate the hydration process of cementitious binder. In addition, a previous study reported that carboxyl acid groups and hydroxyl groups could react with hydration products of cementitious materials and form covalent bonds [21]. Therefore, GO would not only be physically attached but also be chemically bonded with the cementitious substances.

This work presents the application of an aqueous suspension of GO as an alternative approach to external curing of cementitious composites. The experiments have been designed to investigate the influence of GO on the microstructure of cementitious mortars and the possible chemical reaction products produced at the interfacial zone of GO and cementitious mortars. Relative to the concrete dry cured in the first 24 or 48 h after casting, the concrete with GO suspension sprayed in the early stage may have a significantly decreased risk of cracking due to early-age shrinkage. The primary objective is to provide preliminary information on the promising application of GO as a surface sealer in the relatively early stage of concrete curing.

2. Experimental

2.1. Materials

The analytical grade chemicals: H_2SO_4 (98%), KMnO_4 , NaNO_3 , and H_2O_2 (30%) were used without further purification. Graphite, with an average particle size of 44 μm , an ASTM specification C150 [22] Type I/II Portland cement and a class C fly ash were used in this study as received. The fly ash was obtained from a coal-fired power plant at Boardman, OR, USA. The chemical composition of portland cement and fly ash have been reported elsewhere [23]. The fine aggregate used was multi-purpose siliceous sand and a high-range water-reducer was adopted. Before proportioning and admixing, the aggregate was prewetted and taken to a saturated surface dry (SSD) condition.

2.2. Preparation of graphene oxide (GO) suspension

Graphene oxide was synthesized following a procedure as reported elsewhere [16,24–26]. Three concentrations of aqueous GO suspensions were prepared under the ultrasonication: 0.25 g/L, 0.5 g/L and 1 g/L.

2.3. Preparation of mortar specimens

Two types of mortar specimens, i.e., the ordinary cement mortar and high volume fly ash (HVFA) mortar were investigated in this study. They were prepared with a constant sand-to-binder mass ratio of 1.5 and a constant water-to-binder mass ratio of 0.3. The replacement ratio of fly ash in HVFA mortar is 60%. Typically,

the sands were first mixed with binder (cement or cement and fly ash) in a mixer for 60 s. Then water was added and mixed for 120 s. Afterwards, the water reducer was added into the mixture and mixed continuously for about 5 mins.

After mixing, the fresh mortar mixture was cast into the 50 mm \times 100 mm cylindrical molds and carefully compacted to minimize the amount of entrapped air. All the specimens were demolded after curing under room temperature (RT) and relative humidity (RH) of 20 \pm 5% for 24 h.

2.4. Surface treatment

After demolding, all the mortar specimens were divided into four groups. The first two groups had surface treatment by GO suspension of different concentrations immediately. 5 g of the GO suspension was sprayed onto the whole surface area of all the mortar samples by a sprayer. Half of them were placed in a container with a temperature of 23 \pm 1 $^\circ\text{C}$ and RH of 95% (wet curing condition), while the other half were placed under normal lab condition (dry curing condition). The remaining two groups had the same surface treatment after being cured in the same container for another 24 h. And they were also placed under those two conditions, respectively. The first two groups were then cured continuously for 27 additional days, and the remaining two groups were then cured for 26 additional days. For all the four groups, three additional mortar specimens were prepared with surface treatment by water for a comparison purpose. Another two groups of control specimens without surface treatment were also prepared.

Table 1 gives the detailed information on sample grouping. All the mortar specimens with surface treatments were denoted as “type of mortar, timing of surface treatment, concentration of GO suspension-curing condition (wet or dry)”. Control specimens were denoted as “type of mortar, c-curing condition (wet or dry)”.

2.5. Compressive strength test

The compressive strength test was carried out in accordance with ASTM C39 [27] and reported in SI units of MPa.

2.6. Water absorption test

The water absorption test was conducted in accordance with ASTM C1585 [28]. All the samples for water absorption test and gas permeability test (below) were cylindrical disk samples with a 50 mm diameter and 10 mm thickness. To eliminate the potential difference of relative humidity between samples with and without surface treatment, they were all oven-dried at 105 $^\circ\text{C}$ under vacuum for 24 h before subjecting to the test.

2.7. Gas permeability test

Gas permeability test was performed following the procedure described in a previous study [29]. Before the test, all the samples also need to be oven-dried at 105 $^\circ\text{C}$ under vacuum for 24 h. The initial weight of the whole setup was measured at the beginning of test. The values of mass variation versus time due to the evaporation of methanol liquid were recorded every 30 mins up to 4 hours.

2.8. Characterizations

For morphology analysis, a strip of cross-sectional sample representing changes in microstructural properties was cut from the middle of the mortar specimens along its diameter. After being coated with gold and vacuumed, the slice samples were then subjected to scanning electron microscopy (SEM) to examine its localized morphology at microscopic level. The beam conditions used were as follows: 15 kV accelerating voltage, 50nA beam current, and focused beam diameter.

X-ray powder diffraction (XRD) and thermogravimetric analysis (TGA) were carried out to check the potential new chemical products produced at the interfacial zone of GO and mortar substrates. XRD was performed on a Siemens D5000 diffractometer using Cu K α radiation in the 2 θ region between 2 $^\circ$ and 60 $^\circ$. TGA was conducted using Mettler Toledo's TGA/DSC1 Thermogravimetric Analyzer under nitrogen atmosphere at a heating rate of 10 $^\circ\text{C}/\text{min}$ from room temperature up to 1000 $^\circ\text{C}$.

3. Results and discussions

3.1. Compressive strength

The results of the compressive strength of wet cured and dry cured cement mortar specimens at 28-day curing age are shown in Fig. 1, where each value was averaged from three individual tests. Relative to wet cured control specimens, wet cured cement mortar specimens with surface treatment showed lower compressive strength except for the specimens with early surface treat-

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