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# Enhancement of barrier properties of cement mortar with graphene nanoplatelet

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ABSTRACT

The transport properties of cement mortar with graphene nanoplatelet (GNP) are investigated experimentally in this study. GNP, a low cost carbon-based nano-sheet, was added to mortar at contents of 0, 2.5, 5.0 and 7.5%, by weight of cement. The water penetration depth, chloride diffusion coefficient and chloride migration were determined for cement mortar with GNP and compared with plain cement mortar specimens. Test results showed that the addition of 2.5% GNP can cause significant decrease of 64%, 70% and 31% for water penetration depth, chloride diffusion coefficient and chloride migration coefficients respectively. The reduced water and ions ingress can be partially attributed to a reduction in the critical pore diameter of about 30%. This refinement of the microstructure by the GNP is validated by the mercury intrusion porosimetry (MIP) results. The impermeable GNP also contributes to the reduced permeability due to the increased tortuosity against water and aggressive ions ingress.

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## 1. Introduction

Durability remains one of the most important properties of concrete, especially for structures exposed to aggressive environments or severe conditions [1–3]. The improvement in the durability performance of concrete has been widely accepted by the community as a means to reduce the life-cycle cost of an infrastructure especially in the maintenance during its service life [4]. The durability of concrete structures is strongly influenced by its transport properties to the harmful agents such as water, $CO_2$, chloride, etc [5]. Existing durability enhancement methods that are commonly used include lowering water–cement ratio with the aid of superplasticizers, adding supplementary cementitious materials and using chemical admixtures [6–8].

In recent years, there has been rising interest in the use of nano-particles in building materials to enhance mechanical performances and to create multifunctional capabilities [9–13]. These nano-particles can fill the voids in the cement paste, leading to lower porosity and higher strength [10–12]. The research on using nano-particles to improve the transport properties of cementitious composites is, however, limited. Ji [14] reported that the nano-$SiO_2$ can fill the voids in cement paste and thus reduce the water permeability of concrete. Gaitero et al. [15] used nano-$SiO_2$ to improve the resistance to calcium leaching of cement paste which can be attributed to the reduced porosity in the cement paste, increased length of the silicate chains and pozzolanic reaction of nano-$SiO_2$. At the same time, only few studies were carried out to study the transport properties of mortar and concrete, which are cement composites containing aggregates. Zhang and Li [16] reported that the addition of nano-$SiO_2$ and nano-$TiO_2$ can refine the pore structures and therefore reduce the permeability of concrete to chloride. Kong et al. [17] recently studied the influence of agglomeration of nano-$SiO_2$ on the performance of cement mortar and they reported that despite the existence of the weak zone between the cement paste and the agglomerates, and the higher porosity of agglomerates, the nano-$SiO_2$ can still be effective in blocking the ingress of chloride ions.

So far, nano-particles that have been introduced into cementitious materials include nano-$SiO_2$, nano-$TiO_2$, nano-$CaCO_3$, and nano-C such as carbon-nanotube and carbon-nanofiber [18]. Another form of carbon-based nano-material is the graphene nanoplatelet (GNP) which has been widely studied in polymer nanocomposites. GNP consists of several layers of graphene with a total thickness of less than 100 nm and a diameter of several micrometers (as shown in Fig. 1(a)). Jang and Zhamu [19] reported that GNP reinforced nanocomposites combine the benefits of good mechanical properties and impermeability. According to Compton et al. [20], the layered structure of GNP is effective in inhibiting the transport processes for gas and fluid in the host matrix due to increased tortuous paths. Another appealing advantage of GNP is the low cost when compared with other carbon nanoparticles, such as CNT or mono-layer graphene, whose large scale production is significantly more expensive [21,22]. Despite the numerous advantages of GNP and its wider adoption in polymer based composites, its potential has yet to be tapped upon in cement composites until recently [23–35]. The introduction of GNP into cement composites was found to increase the electrical conductivity by 2–3 orders of magnitude

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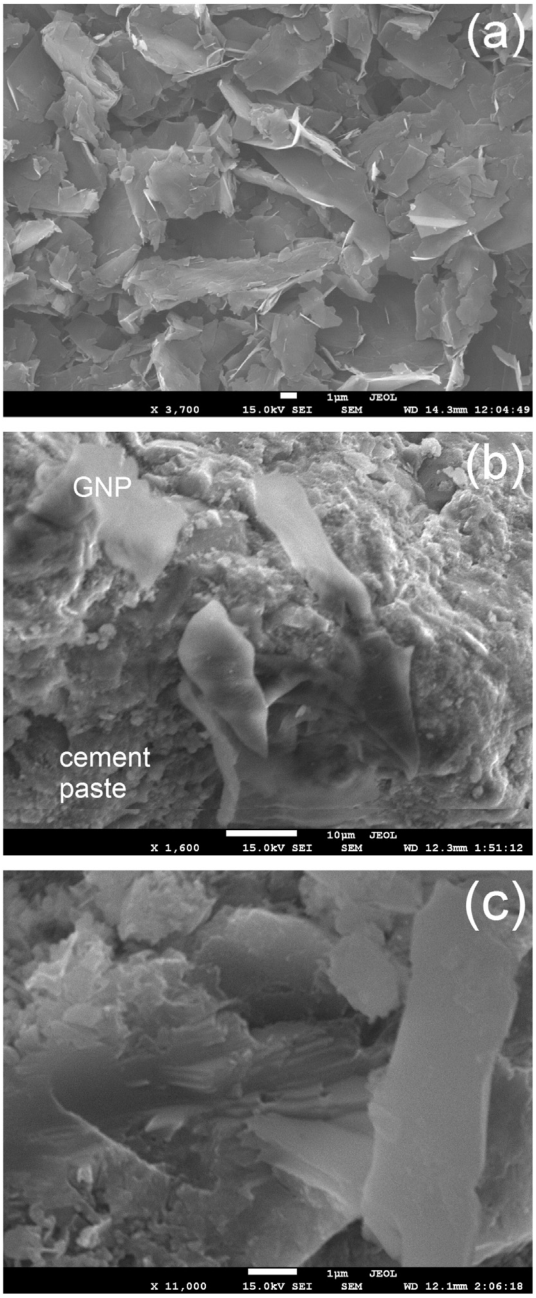


**Fig. 1.** SEM images of GNP A3775 (a) after sonication, (b) at the fractured mortar surface, and (c) clustering at 7.5% mortar.

and diminishes its sensitivity on the moisture content of the specimen [23]. This enhanced conductivity of mortar infused with GNP can also be exploited for creating self-(damage) sensing cement composites and simple closed-form solutions have been derived for predicting the damage in this conductive cement nanocomposite [24,25].

The first study on the transport properties of GNP nanocomposites in cement matrix medium is reported in this paper. Chloride diffusion and migration tests and water permeability tests were carried out according to the various test standards to investigate the main transport mechanisms of diffusion, migration and permeability respectively. The results for cement mortar with GNP are compared with plain mortar and the enhancement in the barrier properties brought about by the introduction of GNP is explained by the changes in pore structure, which are revealed via the mercury intrusion porosimetry (MIP) test, and the increase in tortuosity against water and chloride ion ingress, which are explained with analytical models. This work can shed light on the use of two-dimensional graphene as a highly effective barrier to enhance the barrier properties of cement composites.

## 2. Materials and methods

### 2.1. Materials

Ordinary Portland cement (OPC) was used in this study, with chemical composition shown in Table 1. The Blaine fineness of OPC is 393 $m^2$/kg. Natural sand with density of 2.65 (saturated surface dry) and fineness modulus of 2.95 was used. GNP was exfoliated from surface-enhanced expanded graphite flake (Grade A3775, Asbury Graphite Mills, Inc.). Its physical properties are shown in Table 2. The mix proportion for water: cement: sand was selected as 0.485:1:2.75, by weight according to ASTM C 109 [26]. GNP was added at contents of 0%, 2.5%, 5.0% and 7.5% by weight of the cement corresponding to 0%, 0.6%, 1.2% and 1.8% by volume of mortar, respectively.

The GNP particles will form precipitates in water which will remain as agglomerates when the suspension is added to mortar mixture and this will negate the benefits of nano-particle addition. A naphthalene sulfonate based superplasticizer (SP) (Darex Super 20, WR Grace) is used to disperse the agglomerates and stabilize the GNP particles in aqueous solution. The added SP could be absorbed at the GNP surface, surrounding them with negative charges and repel each other. This uniformly dispersed aqueous suspension of GNP with SP was mixed with cement and sand to create mortar with GNP. Different amounts of SP have been tested for the flowability of the cement mortar with GNP and the amount of SP is kept at a constant 50% of the weight of GNP in this study to ensure good consistency. In comparison with the recommended dosage of 400–2500 ml of SP to disperse 100 kg of OPC, SP is used at a higher dosage in this study to disperse the GNP which has a surface area 60 times (Table 2) larger than that of OPC. Even at 2.5% GNP, the total surface area of GNP is about 1.5 times of that of OPC. Thus, the use of 1500 ml SP for every 2.5% GNP lies within the expected range of 600–3750 ml SP based on surface area requirements for dispersion of GNP. With the above mentioned SP, all the mortar mixtures had similar flowability (with mini-slump diameter of 150 $\pm$ 10 mm).

### 2.2. Casting procedure

Prior to casting of mortar mixture, GNP was first ultra-sonicated with the aid of water and the SP. The desired amount of GNP was added to the mixing water together with dispersant and then hand-stirred for 1 minute. A high power ultra-sonication horn was used to disperse this suspension for 1 hour in a water bath to cool down the horn. According to previous literature [27], sonication technique is typically considered as a nondestructive process for the exfoliation of graphene which preserves the basal plane of GNP flakes. However, if the sonication time is excessively long (for example 5 to 10 hours as determined by Xia et al [28]), graphene can suffer from size reduction and damage. The efficiency of dispersing GNP without sonication in water, in water with SP, and the effect of sonication in water with SP can be observed via visual inspection (See Fig. 2.). In water, sedimentation of GNP at the bottom of the bottle was noticed after 2 hours. In contrast, GNP

**Table 1**
Chemical composition of OPC.

| Chemical composition | % |
|---|---|
| $SiO_2$ | 20.8 |
| $Al_2O_3$ | 4.6 |
| $Fe_2O_3$ | 2.8 |
| CaO | 65.4 |
| MgO | 1.3 |
| $SO_3$ | 2.2 |
| $Na_2O$ | 0.31 |
| $K_2O$ | 0.44 |

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