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Effect of mixing methods on the electrical properties of cementitious composites incorporating different carbon-based materials



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HIGHLIGHTS

- Different mixing methods were proposed to evenly distribute carbon-based materials.
- Carbon nanotubes (CNT), graphene nanoplatelets (GNP), carbon fibers (CF) were used.
- Performance was assessed by electrical resistivity and compressive strength results.
- The best method for uniform distribution was mechanical mixing with shear effect.
- Longer CF was the best to tailor matrix properties among all carbon-based materials.

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ABSTRACT

Special attributes such as self-sensing ability could be included in conventional concrete-like materials, using carbon-based materials such as carbon nanotubes (CNT), graphene nanoplatelets (GNP) and carbon fibers (CF), which have recently come to the forefront due to their superior mechanical, thermal and electrical properties. However, their non-uniform distribution in cementitious systems stands in the way of taking full advantage of their benefits. To address this issue, an experimental study was undertaken, proposing several mixing methods to achieve uniform distribution. The performance of each method was assessed based on electrical resistivity (ER) and compressive strength measurements. Continuous reductions in ER measurements were observed with time, regardless of the mixing method and carbon-based materials used. Ultrasonication did not appear to be as useful as mechanical mixing with shear effect in terms of ER and compressive strength. Test results revealed that using longer CF (12 mm) in cementitious composites was the best way to tailor matrix properties in terms of electrical conductivity and compressive strength. CF was also easy to mix and has a relatively lower manufacturing cost than other electrically conductive carbon-based materials.

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1. Introduction

Portland cement concrete is the most frequently used construction material on earth, with yearly production of about 10 km³ [1]. Its widespread use is evident in residential, industrial, commercial, sports, agricultural and other applications, and its success as an efficient and adaptable building material has been proven well over the past century. However, its long-term performance in structures serving under severe environmental circumstances is

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becoming a subject of interest to many researchers. Mechanisms involved in reduced performance longevity are generally durability-based (e.g. alkali-silica reactivity, freeze-thaw susceptibility, rebar corrosion, sulfate attack etc.). Damage triggered and further exacerbated by the low durability characteristics of conventional concrete material necessitates maintenance and conservation or even full-scale reconstruction, leading to significant energy and raw material burdens. It is therefore critical to monitor the conditions of structures before they lose complete structural integrity [2].

This kind of monitoring is possible with the use of sensory components such as optical fibers, strain gauges, and piezoelectric strain sensors [3–5]. However, several papers in the current litera-

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ture have reported that these sensors have poor durability, low sensitivity and a poor survival rate [6,7]. Piezoresistive behavior of the cementitious matrix can also be used as a tool to monitor structural health. Piezoresistivity (the dependence of electrical resistivity on the applied strain [8]) is achievable in concrete by incorporating modifiers into the cementitious matrices and having the concrete material itself serve as a sensor. Modifiers that make piezoresistive behavior possible are naturally conductive materials (e.g. iron-based), carbon fibers, carbon nanotubes (CNT), carbon black, etc. Despite the wide availability of conductive materials that favor the electrical properties of cementitious matrices, carbon fibers are the most commonly used because they are cheaper than other conductive carbon-based materials and contribute to mechanical properties such as flexural strength, flexural toughness, tensile strength and ductility, and reduced drying shrinkage [9]. Given the fact that these materials perform differently in terms of conductivity and strain-sensing characteristics, it is hard to determine which one outperforms the other for a given structural application. However, it can be stated that these materials are generally at micro- or nano-scale, meaning that they have very large specific surface areas. Uniform distribution for the creation of an electrical network that can more easily capture or sense changes in applied strain in actual structures is therefore a challenge due to the agglomeration problem during mixing [10-12].

Several previous studies have made efforts to assess the dispersion quality of nano filaments in aqueous solutions [12–14], cement pastes [15], or cementitious composites [16,17] using a scanning electron microscope (SEM) and a transmission electron microscope (TEM). The downside of SEM and TEM methods is that they are solely dependent on visual observations to monitor good dispersion of nano filaments within a certain sample. There is also a high likelihood that the particles analyzed in these methods are small and therefore not representative of the whole sample. Other researchers have concentrated on the improvements in mechanical and electrical properties of cement pastes or mortars as an indication of the dispersion of electrically conductive materials. For example, Peyvandi et al. [18] employed the surface modification technique to facilitate the dispersion of graphite nanomaterials in aqueous media, and concluded that the addition of 0.13% of nanomaterial by weight of anhydrous cementitious materials improved the flexural strength of the cementitious matrix by up to 73%. The effects of silica fume use on the mechanical and electrical properties of cement/CNT composites were investigated by Kim et al. [19]. The study reported that when no silica fume was used in composite material, CNT tended to agglomerate, leading to insignificant effects on compressive strength and electrical resistance, while the small amounts of silica fume helped intermix some agglomerated CNT by mechanically breaking them into smaller sizes. In their study, Sanchez and Ince [16] added carbon nano fibers into silica fume/cement pastes and concluded that due to poor distribution of the fibers in the cementitious composites, no marked changes took place in the compressive and splitting tensile strengths of specimens. A different study by Li et al. [20] concluded that the compressive and flexural strengths of cement mortar can be increased by 19% and 25%, respectively, with chemically functionalized CNT with a concentration of 0.5% by weight of cement.

Based on the examples listed above, different studies have used a number of techniques for the proper distribution of a wide range of conductive materials. However, as there is no consensus on how to achieve this goal, the current experimental study was undertaken to address the issue. Electrically conductive materials from nano-scale (carbon nanotubes [CNT] and graphene nanoplatelets [GNP]) to micro-scale (carbon fibers [CF]) were used in composite material to distribute these materials in a way that would provide the highest possible electrical conductivity. This feature is vital for efficient self-sensing capability; a number of different mixing methods were therefore used, taking into account the wide literature review and laboratory experience of the authors. In addition to electrical property measurements, compressive strength results were recorded to compare mixing methods and determine which one was superior.

2. Materials and mixture proportions

2.1. Materials

The three conductive materials employed during the study were multi-walled carbon nanotubes (CNT), industrial graphene nanoplatelets (GNP) and chopped carbon fibers with 6 and 12 mm lengths and aspect ratios of 800 and 1600 (CF6 and CF12, respectively). Chemical and physical properties of CNT and GNP are provided in Table 1 and in the SEM photos in Fig. 1. CF had a tensile strength of 4200 MPa, elastic modulus of 240 GPa, elongation of 1.8%, density of nearly 1.7-2.0 g/cm³ and diameter of 7.5 µm. SEM photos of CF are also included in Fig. 1. In addition to the electrically conductive materials listed above, all mixtures produced in this study consisted of common ingredients such as CEM I 42.5R ordinary Portland cement (PC) (similar to ASTM Type I), Class-F fly ash (FA), fine silica sand with a maximum aggregate size of 0.4 mm, specific gravity of 2.60 and water absorption capacity of 0.3%, potable mixing water and polycarboxylate-ether-based high range water reducing admixture (HRWRA). In order to enhance the distribution of conductive materials, silica fume (SF) and nano-silica (NS), nano-calcite (NC) and a methylcellulose-based dispersion agent were also used in some of the mixing methods. The chemical and physical properties of PC, FA, SF, NS and NC are provided in Table 2. Particle size distributions of PC, SF, FA, and silica sand are provided in Fig. 2.

2.2. Mixture proportions

Several mixing methods were used to achieve better distribution of electrically conductive carbon-based materials and obtain the best self-sensing capability in cementitious composites. During the preparation of mixtures, water to cementitious materials (PC + FA) ratio (W/CM) and fly ash to Portland cement ratio (FA/ PC) were kept constant at 0.27 and 1.2, respectively. For all mixtures incorporating CNT and GNP, carbon-based material amount was constant (0.25% of total weight of cementitious materials). In mixtures incorporating CF, the amount of CF was 0.5% by volume of the total mixture, regardless of the length of fibers used. These utilization rates for different electrically conductive materials were decided based on previously conducted studies in literature [12,20–22]. For certain mixing methods SF, NS, NC and methylcellulose were used at 5%, 1%, 1% and 0.2% of the total cementitious materials, respectively, for the enhancement of dispersion quality.

3. Dispersion methods of electrically conductive materials

As mentioned above, the study used electrically conductive carbon-based materials both at nano- (CNT and GNP) and microscale (CF). In addition to the use of several conductive materials, different dispersion-enhancing materials were tested for certain mixing proportions as well. Given the significantly varying particle sizes of these materials, it was not possible to keep HRWRA amounts the same for all the mixing methods. Therefore, to ensure consistency of different mixtures, mini slump tests were performed and the quantity of HRWRA was selected based on similar flow deformation levels. In the mini-slump flow spread test, a truncated cone mold (diameter of 92 mm at the bottom, 44 mm at the top, with a height of 76 mm) was placed on a smooth plate, filled with mortar, and lifted upward. The slump flow deformation was defined as the dimension of the spread when the mortar stopped

Table 1						
Physical and chemical	properties	of C	INT	and	GNP.	

Physical properties CNT GNP Diameter (nm) ~10-30 ~5 Length (µm) ~10-30 - Thickness (nm) - ~50-100 Surface area (m²/g) >200 ~13 Chemical properties - ~96-99% Oxygen content - ~1%			
$\begin{array}{cccc} Diameter (nm) & ~10-30 & ~5\\ Length (\mum) & ~10-30 & -\\ Thickness (nm) & - & ~50-100\\ Surface area (m^2/g) & >200 & ~13\\ \hline \ \ \ \ \ \ \ \ \ \ \ \ \$	Physical properties	CNT	GNP
Chemical propertiesPurity>90%Oxygen content1%	Diameter (nm) Length (μm) Thickness (nm) Surface area (m²/g)	~10-30 ~10-30 - >200	~5 - ~50-100 ~13
	Chemical properties Purity Oxygen content	>90% -	~96-99% ~1%

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