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Optimum carbon nanotubes' content for improving flexural and compressive strength of cement paste

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HIGHLIGHTS

- 0.25 wt% CNTs is the optimum weight fraction in terms of achieving maximum strength at a reasonable cost.
- CNTs were embedded within the cement hydration products.
- Initial and final setting times increase with an increase in the CNTs' content.
- Scanning voltages up to 5 kV would help in differentiating CNTs from the other cement particles.
- CNTs' penetrated calcium hydroxide (C–H) crystals' surfaces.

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ABSTRACT

This study investigated the effect of multi-walled carbon nanotubes' (MWCNTs) weight fraction on the setting time and mechanical properties of cementitious composites. Different cement mixes containing CNT-to-cement weight fractions of 0.03, 0.08, 0.15, 0.25, 0.35 and 0.5 wt% were prepared in addition to the control mix. The initial and final setting times of the fresh pastes were measured on the cast day and the flexural and compressive strengths of the hardened samples were determined after 28 days of moist curing. The fractured surfaces of the samples were then examined using a scanning electron microscope (SEM). The results showed that the 0.25 wt% CNTs is the optimum weight fraction in terms of achieving maximum strength at a reasonable cost. Batches with lower CNTs' contents than 0.25 wt% demonstrated lower flexural and compressive strengths, whereas batches with higher CNTs' contents than 0.25 wt% produced similar or slightly higher strengths. Analysis of variance (ANOVA) confirmed that increasing CNTs' concentration above 0.25 wt% will not have a significant effect on the compressive and flexural strengths. Investigations of the microstructure, which was carried out using SEM, showed good dispersions of the nanofilaments within the cement matrix. Spots of agglomerations were noticed in batches containing 0.25, 0.35 and 0.5 wt%. SEM images have also indicated that CNTs were embedded within the cement hydration products. The study findings were useful for determining the CNTs' content required to achieve both optimum dispersion and maximum strength enhancement of cementitious composites.

1. Introduction

Carbon nanotubes (CNTs) extraordinary strength and stiffness properties intrigued the interest of many researchers to mix them with cement paste and mortars in order to enhance their mechanical properties. Many research studies attributed the effects of CNTs' addition on the mechanical performance of cement materials to the weight fraction of the nanofilaments [1–11]. The use of CNT-to-cement weight fractions varying from 0.02 to 1 wt% was reported in the literature. Unfortunately, the reported findings could not be used to conclude a consistent weight fraction that provides the most optimum cement composites' mechanical properties. This may be related to the different choices of CNTs' type and mixing techniques, which in return affect the dispersion quality. Konsta-Gdoutos et al. [12] investigated the effect of dispersed CNTs on the mechanical properties of cement paste. They tested short CNTs-to-cement weight fractions of 0.048, 0.08 and 0.10 wt% and long CNTs-to-cement weight fractions of 0.025, 0.048 and 0.08 wt

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%. Their results indicated the highest strength gains in short CNTs-to-cement weight fraction of 0.08 wt% and in long CNTs-to-cement weight fraction of 0.048 wt%. Luo et al. [13] investigated the effect of adding 0.1, 0.5 and 1.0 wt% long CNTs' with silica fume on cement paste properties. On the 28th day, their results showed that the highest improvements in the flexural strength and the stress-intensity factor were obtained by incorporating 0.5 wt% CNTs into the cement mix. Kumar et al. [14] discussed the effect of using 0.5, 0.75 and 1 wt% long CNTs-to-cement weight fractions on the strength properties of cement paste. The compressive and tensile strengths results showed that the mix containing 0.5 wt% CNTs was the only one to gain strength in comparison with control batch. Using CNTs' content higher than 0.5 wt% resulted in a decreased strength. The authors [14] related the reason for this behavior to the formation of CNTs' clusters that resulted in a weaker bonding between CNTs and cement compounds. Nasibulina et al. [15] studied the effect of the addition of 0.02, 0.03, 0.05 and 0.09 wt% long functionalized CNTs on the compressive strength of the cement paste. The obtained results showed that the highest strength gain was attained in the 0.03 wt% batch, whereas the lowest gain was attained in the 0.09 wt% batch. Wang et al. [5] incorporated 0.05, 0.08, 0.1, 0.12 and 0.15 wt% CNTs into Portland cement pastes to investigate their effect on flexural toughness. The results showed a large improvement in the batches prepared using 0.08, 0.1 and 0.12 wt% CNTs' content compared to the batches of lower (i.e. 0.05 wt%) and higher (i.e. 0.15 wt%) CNTs' content. Yazdani and Mohanam [16] studied the effect of using short CNTs and long carbon nanofibers (CNFs) weight fractions on the flexural and compressive strengths of mortars. Nanofilaments dosages of 0.1 and 0.2 wt% were tested at various water-to-cement ratios. Compared to plain mortar samples, their results showed increments in the compressive strengths of about 150% for CNTs' samples and 200% for CNFs' samples. The results also showed an increase in the flexural strength of about 50% for both CNTs' and CNFs' samples compared to the plain mortar samples. The gains in the batches with 0.1 wt% CNTs were significantly higher than those in the batch with 0.2 wt% CNTs. Recently, Xu et al. [17] investigated the mechanical properties of cement paste using CNTs' weight fractions of 0.025, 0.05, 0.1 and 0.2 wt%. The strength test results, which were supported by microstructural analysis, showed that both compressive and flexural strengths were slightly improved with the incremental increase in the nanofilaments content.

It could be concluded from the results reported in the literature that cement batches containing higher CNTs' content had strength reductions compared to those blended with a relatively lower CNTs' content. The researchers attributed the reason of this strength reduction to the lower dispersion quality associated with incorporating high CNTs' weight fraction into the cement mix. A recent study by the authors showed that a mixing procedure comprising of a 30-min CNTs/water sonication and a 60-min CNTs' solution/cement mixing could improve the quality of CNTs' dispersion as well as the flexural strength when applied to cement batches with high CNTs' contents, such as 0.25 wt% [18]. In this study, the effect of using a wide range of long CNTs' weight fractions on the flexural and compressive strength was investigated using a relatively long mixing duration of 60 min. The microstructure of the batches was then investigated using a scanning electron microscope (SEM).

2. Testing methodology

2.1. Testing matrix

Table 1 shows the properties of the prepared batches, including the CNTs' weight fractions and the surfactant's percentages. The testing methodology started by preparing the experiment samples. Then, their flexural and compressive strengths were tested using the three-point bending and the cubic uniaxial tests, respectively. After that, the fractured samples microstructures were explored using SEM to examine the dispersion of the nanofilaments within the cement compounds.

2.2. Materials and equipment

The cement used in preparing the test samples was Portland cement, CEM I, Class 42.5 R complying with EN 197-1. Multi-walled carbon nanotubes (MWCNTs) produced using the catalytic chemical vapor deposition (CVD) process and provided by US Research Nanomaterials were used. The physical properties of the CNTs are shown in Table 2. A commercial polycarboxylate water reducing admixture, named ADVA Cast 575, provided by GRACE Products was used as a surfactant to help improve the dispersion of the CNT within the aqueous solution. The equipment used in preparing and testing the batches included a planetary laboratory mixer supplied by Hobart, an ultrasonic wave mixer, VCX 750 model, provided by Sonics and Materials Inc., a strength testing machine, of a model name ADVANTEST 9, provided by CONTROLS Inc., a scanning electron microscope (SEM), of a model name Nova NanoSEM, supplied by FEI, $40 \times 40 \times 160$ mm steel molds for flexural testing, and $50 \times 50 \times 50$ mm steel molds for compression testing (Fig. 1).

2.3. Mixing and casting

For each batch, 2800 g of cement and 1120 g of water were used to maintain a water-to-cement ratio (w/c) of 0.4. A surfactant-to-CNT content of 4:1 was fixed in all batches and added to the weight of water. The choice of the surfactant amount is based on previous studies that reported the optimum amount for acceptable CNTs dispersion in the solution [12,19]. First, the temperature of the mixing water was set at 23 °C and the CNTs were placed in. After that, the surfactant was added and the sonication process was carried out for 30 min. During the sonication process, an amplitude of 20% was preserved to prevent excessive breakage of the nanofilaments particles. Then, the cement mixer was operated at a low speed of 140 rpm and the cement was added to the water/CNT solution in five increments. This process was accomplished in 10 min. After that, the mixing was continued for another 50 min at a medium speed of 285 rpm. Then, the mixer was stopped and both fresh paste and strength test samples were poured and compacted into the molds as per ASTM C109 and ASTM C348 standards [20,21]. During casting, it was observed that the workability of the batches containing higher CNTs' contents than 0.15 wt% is higher than those of less CNTs' content. Also, it was noted that bleeding is occurring in some samples. However, a relationship between the bleeding and the strength gains of the samples was not observed. In most cases the strengths of the samples encountered bleeding were similar to those encountered no bleeding. Furthermore, it was noted that agglomerations of CNTs on the surface of the mix have continued to increase for higher CNTs' weight fractions (Fig. 2a). At the de-molding time (i.e. 24 h later), a larger amount of CNTs could be observed on the surface (Fig. 2b). Finally, the samples were installed in a curing water tank for 28 days.

2.4. Setting time test

The initial and final setting times of all batches were determined according to ASTM C191 [22]. The test was performed using a manual Vicat needle test (Fig. 3). Three samples were prepared from each batch. At each sample, six indentations were taken. First, the samples were allowed to set in a moist cabinet for 30 min after