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The influence of rheological parameters of cement paste on the dispersion of carbon nanofibers and self-sensing performance

Hui Wang [a], Xiaojian Gao [a,b,*], Rui Wang [a]

[a] School of Civil Engineering, Harbin Institute of Technology, 150090 Harbin, China
[b] Key Lab of Structure Dynamic Behavior and Control (Harbin Institute of Technology), Ministry of Education, 150090 Harbin, China

HIGHLIGHTS

- Influence of cement paste fluidity and viscosity on CNFs dispersion was reported.
- Suitable fluidity and viscosity is favorable for dispersion of CNFs in cement paste.
- The better CNFs dispersion results in the improved piezoresistive performance.

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ABSTRACT

This paper aims to study the influence of rheological parameters of cement paste on the dispersion of carbon nanofibers (CNFs) and the subsequent electrical resistivity. Three groups of cement paste with water-cement ratio (w/c) of 0.4, 0.35, and 0.3 and three groups with w/c of 0.4 incorporating 10% silica fume (SF), 20% granulated blast-furnace slag (SL) and 0.4% carboxymethylcellulose sodium (CMC) were prepared. The CNFs were incorporated at 2.25% of binder volume. Different dosages of superplasticizer were added for each group, to obtain variable rheological performances of fresh paste. The average value and variability coefficient (C_v) of electrical resistivity for 16 specimens were determined for each mixture. Scanning electron microscopy (SEM) observation and piezoresistivity were tested on selected samples. Results showed that slump flow increased and the viscosity decreased with more addition of superplasticizer and higher w/c. For each group of cement paste, the slump flow of around 200 mm is most favorable for enhancing the dispersion of CNFs. The addition of CMC and SF improved the dispersion of CNFs, but SL had a disadvantageous influence. The lower C_v value and better dispersion of CNFs due to the rheological performance of cement paste led to the improved piezoresistivity.

1. Introduction

Concrete has been commonly used in different types of construction engineering due to its rich resources, good environmental adaptability, low cost and high strength. However, extensive damages of concrete structures happen all over the world. For the purpose of hazard mitigation, it is significant to monitor the damage of concrete structures, especially for concrete structures in ocean environment, frequent earthquake areas or other disaster areas. The conventional methods for damage sensing in concrete structures include fiber-optic sensors, strain gauge method and shape memory alloy method. Normally, these methods incur the high cost of installation and premature damage. Chen and Chung [1,2] pointed out that cement concrete modified with carbon fibers (CFs) can serve as sensors in concrete structures. The addition of CFs not only makes the concrete smart, but also makes the concrete a better structural material [3]. Improvement in concrete properties include the increased flexural strength, sensing effect, electromagnetic interference (EMI) shielding and the thermoelectric effect for melting ice [4–6].

CNFs are elegant engineered materials as they combine hollow cylinders of around 100 nm diameters and lengths of a few microns. The high specific strength, chemical resistance, electrical and thermal conductivity of carbon nanofibers make it attractive for use as reinforcement to develop superior cementitious composites [7–10]. In Hammel's [11] research, CNFs were expected to affect the nanoscale processes that control the hydration products of CNFs cement composites with better performance. When being compared with carbon nanotubes (CNTs), the CNFs present numerous exposed edge planes along the surface [12], which in turn

* Corresponding author at: School of Civil Engineering, Harbin Institute of Technology, 150090 Harbin, China.

E-mail address: gaoxj@hit.edu.cn (X. Gao).

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constitute potential sites for advantageous chemical or physical interaction. At the same time, CNFs can be produced at low cost, about 3–10 times cheaper than carbon nanotubes [8–10]. However, only few of researches have focused on the use of CNFs as self-sensing material in concrete structure [11–13].

Dispersing CNFs in the matrix is of fundamental importance for basic researches. The uniform distribution of CNFs has been shown to improve the mechanical strength, physical properties and conductive stability of specimen [14]. However, achieving effective dispersion in cement composites is a challenging task even though the CNFs can disperse well in liquid with the use of dispersant and ultrasound [14–16]. Several documents have focused on the use of dispersant and ultrasound to improve the dispersion of carbon fibers [15–20]. It was also reported that the homogeneity of cement paste may be destroyed by dispersant [20] and the structure of CNFs was likely to be damaged by ultrasound [21]. On the other hand, the rheological properties of cement paste as the matrix may have a great influence on the dispersion of CNFs. Adjusting the rheological properties of cement paste may be effective for CNFs dispersion without injuring the structure of CNFs and interfering with the hydration reaction of cement [20]. Unfortunately, few literatures in this field have been reported.

This paper aims to research the relationship between the rheological properties of cement paste and the CNFs dispersion. The dispersion effect was evaluated by the mean value and variable coefficient of electrical resistivity in one group of specimens. The piezoresistive effect was carried out for selected mixtures. Microstructure observation was determined by scanning electron microscopy (SEM).

2. Experimental

2.1. Raw materials

Carbon nanofiber used in this study is the PR-19-XT-LHT-OX fiber, with a density of 2.1 g/cm^3 supplied by the Pyrograf Products, Inc. The diameter of this fiber was 149 nm and the length was 19 μm resulting in an aspect ratio of 128.

The cement used was ordinary Portland cement with strength grade of 42.5 MPa according to Chinese standard GB175-2007 [22]. A commercially available, polycarboxylate-based, high-range water-reducing agent (SP) was used to adjust the flowability of fresh cement paste. Silica fume (SF) was used as a mineral admixture with specific surface area of 1.5×10^4 m^2/g and a SiO_2 content of more than 96% according to GB/T21236-2007 [23]. Slag powder (SL) was used as another mineral admixture with specific surface area of over 400 m^2/g according to GB/T-18046-2008 [24]. The particle size distribution and chemical composition of cementitious materials are shown in Tables 1 and 2. Carboxymethylcellulose sodium (CMC) was used as a dispersant agent for CNFs dispersion in cement paste [25]. As an odorless, tasteless and non-toxic white or yellowish powder, CMC is easily soluble in cold or hot water to become colloidal solution.

2.2. Mixing proportion and specimen preparation

To determine a suitable dosage of CNFs, five groups of mixtures were first prepared. For the first series of mixtures (CNFP-1), the weight ratio of cement: water: SP was kept constant (1:0.4:0.4%) while the dosage of CNF was increased from 0.75% to 6.5% by volume of cement. For the second series of mixtures (CNFP-2), the water to cement ratio (w/c) was kept as 0.4, the dosage of CNFs was increased from 0.75% to 3.125% by volume of cement and the dosage of SP was adjusted for controlling the slump flow of paste to around 200 mm. On the basis of CNFP-2 mixtures, 10% SF, 20% SL and 0.4% CMC was added to replace cement by mass respectively to prepare other three groups of mixtures (C-SF-0, C-SL-0 and C-CMC-0) and the SP addition for every group was kept constant.

Based on the above tests, the suitable dosage of CNFs was determined as 2.25% by volume of binder. Six groups of cement pastes were designed to research the effect of rheological properties on fiber dispersion as shown in Table 3. The first three groups represented three different w/c of 0.4 (C04), 0.35 (C035) and 0.3 (C03). When the w/c was kept as 0.4, 10% silica fume or 20% ground granulated blast- furnace slag were added to replace cement for group C-SF and C-SL respectively, and 0.4% carboxymethylcellulose sodium was added for group C-CMC. For every group of mixture, different dosages of SP were added to obtain cement pastes with different rheological properties.

For each mixture, 16 specimens of size $20 \times 20 \times 25$ mm were prepared and demoulded after 1 day curing at room temperature. For accelerating the experiment, the specimens were cured for another 3 days under temperature of 80 ± 2 °C and relative humidity of higher than 95%.

2.3. Testing methods

To determine the rheological properties of cement paste, the slump flow test was carried out by a steel cone with a bottom diameter of 60 mm, an upper diameter of 36 mm and a height of 60 mm (as shown in Fig. 1) according to Chinese Standard GB/T8077-2012 [26]. At the same time, the plastic viscosity of fresh cement paste was measured for each mixture by using a DV-II+ viscometer made by Brookfield company USA, as illustrated in Fig. 2.

Electrical resistivity measurement was performed on 16 specimens for every mixture by using the two-electrode method at a DC voltage of 0.3 V [27]. To reduce the polarization effect, speci-

Table 1
Particle size distribution of cementitious materials.

Particle size/μm	0.3	0.6	1	4	8	16	32	64
Cement	0	0.33	2.66	15.01	28.77	46.64	72.73	93.59
SL	0	0.85	3.51	19.63	35.01	58.85	84.9	97.9
SF	31.2	58.3	82.3	100	100	100	100	100

Table 2
Chemical composition of cementitious materials.

Types	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	SO_3	R_2O
Cement	20.86	5.47	3.94	1.73	62.23	2.66	0.48
SL	34.06	14.74	0.83	9.73	35.93	0.23	3.51
SF	96.2	0.7	0.6	0.5	0.4	0	1.3