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MICRO-STRUCTURE AND MECHANICAL PROPERTIES OF CARBON NANOTUBE/CEMENT NANOCOMPOSITES

Shama Parveen[1(*)], Sohel Rana[1], Raul Fangueiro[1,2]

[1]Fibrous Materials Research Group, School of Engineering, University of Minho, Guimarães, Portugal

[2]Department of Civil Engineering, University of Minho, Guimarães, Portugal

[(*)]*Email:* parveenshama2011@gmail.com

ABSTRACT

This work reports the development of carbon nanotube (CNT) reinforced cement nanocomposites with improved micro-structure and mechanical performance. Various types of CNT, such as single-walled and multi-walled (both pristine and acid functionalized) CNTs were dispersed in water with the help of magnetic stirring and ultrasonication using a biocompatible dispersant, Pluronic F-127 and, subsequently mixed with cement mixtures to prepare nanocomposite samples. A detailed dispersion study was performed in order to optimize the concentration of Pluronic to be used for achieving homogeneous dispersion and, the optimized CNT/water suspensions were used to fabricate CNT/cement nanocomposites. Micro-structure and mechanical properties of prepared nanocomposites were characterized and the experimental results have been analysed and discussed.

Keywords: carbon nanotube, cementitious composite, dispersion, micro-structure, mechanical properties

INTRODUCTION

Concrete is the most frequently used construction materials all over the world. However, its major drawback is the brittleness and susceptibility to crack formation and growth that reduces its durability (Allen, 2007). The concept of dispersing nanomaterials within concrete in order to develop crack free and durable construction materials has been realized in recent times (Chen, 2011). Carbon nanotubes (CNTs) are getting tremendous attention for this purpose in order to develop high performance and piezoresistive cementitious composites. Although CNTs possess exceptional physical and chemical properties, the successful transfer of these properties in to composite materials is strongly dependant on the state of CNT dispersion within matrix. Due to their strong agglomeration tendency, it is extremely challenging to obtain a homogeneous CNT dispersion, which is, however, a prerequisite for successful utilization of CNT in most of the applications including composite materials. The approach of dispersing CNTs directly within cement paste during mixing is not feasible, as the thickening of cement paste begins within a short period after addition of water (Gopalakrishnan, 2011). The mixing process using a Hobart mixer, commonly used to prepare mortar paste cannot ensure proper dispersion of CNT within cementitious matrix (Cota, 2012). To avoid this situation, the strategy commonly employed for mixing CNTs with cementitious matrices is to disperse these nanomaterials first in water, followed by mixing of nanomaterial/water dispersion with cement using a conventional mixer. Various physical and chemical techniques have been tried to prepare homogeneous aqueous dispersion of CNT such as ultrasonication, mechanical stirring, using surfactants, polymers, admixtures, through CNT functionalization, etc (Parveen, 2013). However, these dispersion routes should be carefully selected so that they do not interfere with the processing of cement nanocomposites. Many surfactants that are successfully used to disperse carbon nanomaterials in polymeric matrices have been reported to create problems in cement hydration, entrap air in the cement paste or react with the water reducing admixtures (Yazdanbakhsh, 2009). In the present study, high concentrations of various types of CNT (single walled and multi-walled, both pristine and carboxyl functionalized) were dispersed in water with the help of ultrasonication and mechanical stirring, using Pluronic F-127, which is a biocompatible dispersant. The effect of Pluronic concentration on the homogeneity and stability of CNT dispersion has been thoroughly investigated and the optimized CNT suspensions were used to prepare CNT/cement nanocomposites. The CNT/cement concrete samples were characterized for micro-structure and mechanical properties (compressive and flexural) and the results have been compared with neat concrete samples.

MATERIALS AND METHODS

Different types of CNTs (single walled or SWCNT, multi-walled or MWCNT, functionalized SWCNT or f-SWCNT and functionalized MWCNT or f-MWCNT) were purchased from Nanostructured & Amorphous Materials, Inc. (Houston, USA). The morphology of these CNT, as characterized by Scanning Electron Microscope (SEM) is shown in Fig. 1.

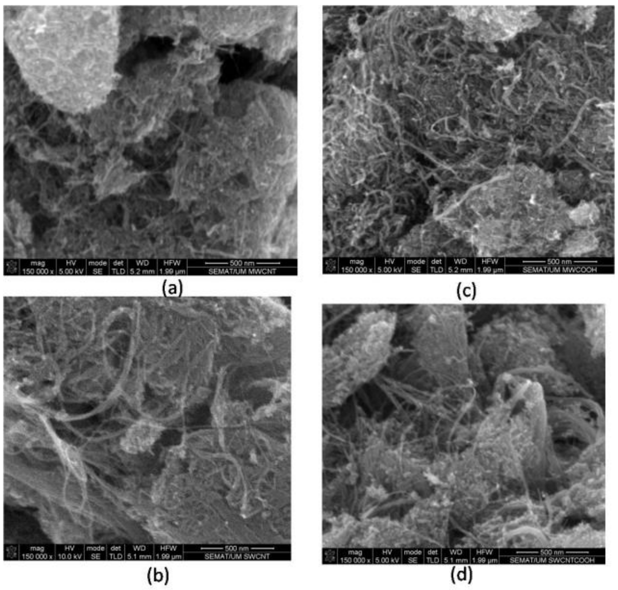

Fig. 1. SEM micrographs of CNT powder: (a) MWCNT, (b) f-MWCNT, (c) SWCNT and (d) f-MWCNT

It can be observed that these nanotubes are in highly agglomerated stage. However, as compared to pristine nanotubes, the functionalized CNT powder seems to be cleaner, due to removal of impurities owing to the acid treatment and the functionalized nanotubes are better separated in the powder. Suspensions of these different types of CNT in water were prepared through magnetic stirring of 15 minutes and subsequent ultrasonication for 1 hour. The freshly prepared CNT suspensions were characterized by Optical microscopy to investigate the dispersion homogeneity. Additionally, the suspensions were allowed to stand for several days and visually observed for any sedimentation.

Cement/CNT composite samples were prepared through mixing of prepared CNT suspensions with Ordinary Portland Cement and standardized sand in a Hobart mixer. A cement to water ratio of 0.5 was used in all samples. Consistence of prepared mortar pastes was evaluated using a flow table according to EN 1015-3 standard. The samples were then kept for 28 days for setting prior to mechanical testing. The details of prepared mortar samples and the flow values of the mortar pastes are provided in Table 1. The samples were tested for compressive and flexural testing according to BS EN 196-1:1995 standard.

Table 1. Details of CNT/cement samples

Sample No.	**CNT Type and %**	**Pluronic %**	**Defoamer: pluronic**	**Flow values (mm)**
1	-	-	-	189,187
2	0.1 MWCNT	5	1:2	183, 177
3	0.1 MWCNT	3	1:2	183, 184
4	0.08 MWCNT	3	1:2	190, 185
5	0.1 SWCNT	5	1:2	176, 175
6	0.1 SWCNT	3	1:2	177, 179
7	0.08 SWCNT	3	1:2	177,176
8	0.1 f-SWCNT	5	1:2	175,172
9	0.1% f-SWCNT	3	1:2	192,187
10	0.08% f-SWCNT	3	1:2	193,191
11	0.1% f-MWCNT	5	1:2	181,182
12	0.1% f-MWCNT	3	1:2	190,191
13	0.08% f-MWCNT	3	1:2	186,185

RESULTS AND DISCUSSION

The optical micrographs of freshly prepared aqueous suspensions of MWCNTs (pristine and functionalized) using pluronic are provided in Fig. 2. In this study, three different concentrations of MWCNTs (0.3 wt.%, 0.2 wt.% and 0.1 wt.%) have been used and they have been dispersed using different concentrations of pluronic (1%, 3% and 5%). The aim here was to study the quality of dispersion obtained in different cases and to optimize the concentrations of CNT and pluronic in order to achieve homogeneous dispersion.

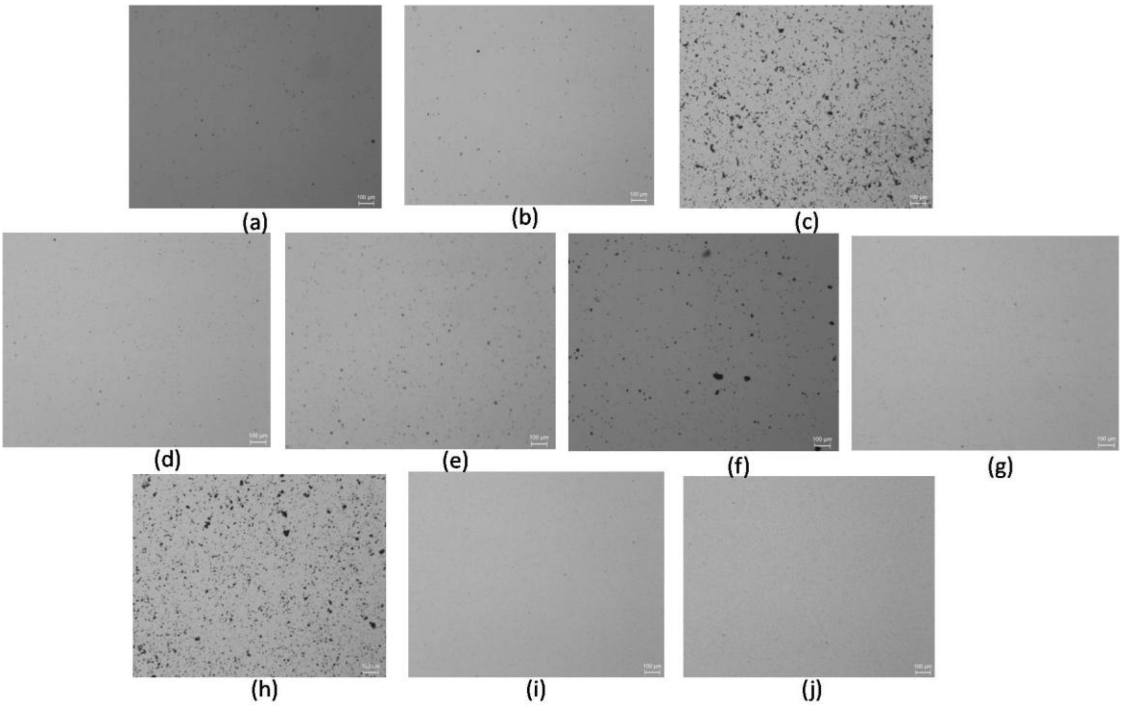

Fig. 2. Optical micrographs of CNT dispersion in water prepared using pluronic: (a) 0.1% MWCNT-1% Pluronic, (b) 0.1% MWCNT-3% Pluronic, (c) 0.2% MWCNT-3% Pluronic, (d) 0.2% MWCNT-5% Pluronic, (e) 0.3% MWCNT-5% Pluronic, (f) 0.1% MWCNT-COOH-1% Pluronic, (g) 0.1% MWCNT-COOH-3% Pluronic, (h) 0.2% MWCNT-COOH-3% Pluronic, (i) 0.2% MWCNT-COOH-5% Pluronic and (J)) 0.3% MWCNT-COOH-5% Pluronic

It can be clearly observed from these dispersion micrographs that both 0.1% MWCNT and MWCNT-COOH can be homogeneously dispersed in water using only 1% pluronic. These dispersions are very uniform and free from visible CNT agglomerates. On the other hand, the micrographs of 0.2% MWCNT (as well as MWCNT-COOH) dispersed using 3% pluronic shows visible CNT agglomerates. An increase in pluronic concentration to 5% helped to reduce these agglomerates and led to good quality dispersion. It can also be observed that 0.3% MWCNT and MWCNTs-COOH can be uniformly dispersed using pluronic concentration of 5%.

Similar observations were also made in case of SWCNTs and SWCNTs-COOH, as shown in Fig. 3. 0.1% SWCNTs, both pristine and functionalized, can be dispersed well using 1% of pluronic and homogeneous suspensions of 0.2% SWCNTs and SWCNTs-COOH can be prepared using 5% pluronic. However, the dispersions of 0.3% SWCNTs using 5% pluronic showed some visible CNT clusters (Fig. 3e). On the contrary, when 0.3% functionalized SWCNTs were dispersed using the same concentration of pluronic (5%), the dispersion was quite uniform and free from agglomerates, indicating the positive influence of functionalization on dispersion.

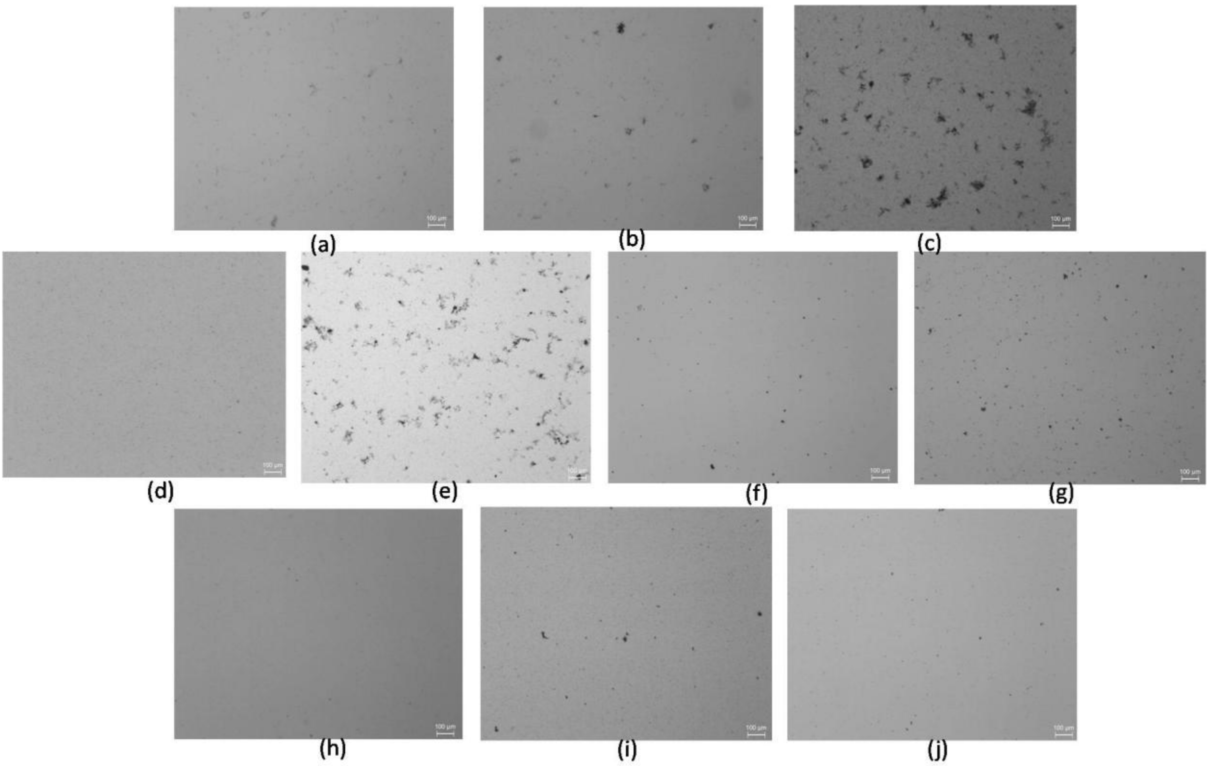

Fig. 3. Optical micrographs of CNT dispersion in water prepared using pluronic: (a) 0.1% SWCNT-1% Pluronic, (b) 0.1% SWCNT-3% Pluronic, (c) 0.2% SWCNT-3% Pluronic, (d) 0.2% SWCNT-5% Pluronic, (e) 0.3% SWCNT-5% Pluronic, (f) 0.1% f-SWCNT-1% Pluronic, (g) 0.1% f-SWCNT-3% Pluronic, (h) 0.2% f-SWCNT-3% Pluronic, (i) 0.2% f-SWCNT-5% Pluronic and (J) 0.3% f-SWCNT-5% Pluronic

Moreover, the visual observation of the suspensions showed no noticeable sedimentation of CNT even after standing for several months (Fig. 4).

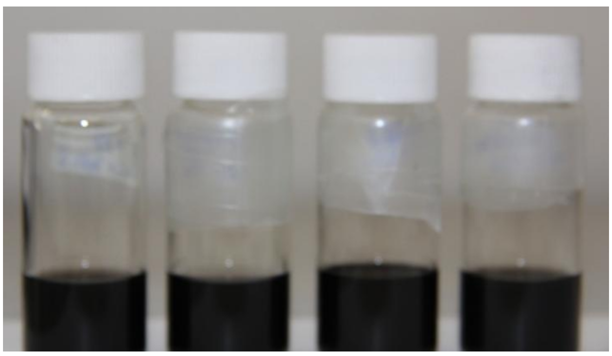

Fig. 4. 0.3% CNT/water suspensions prepared using 5% Pluronic after standing of 2 months

MICROSTRUCTURE AND MECHANICAL PROPERTIES

The initial results showed that the addition of CNT to cement mixes significantly improved the microstructure of cementitious composites leading lower porosity as compared to plain concrete samples. Also, there was considerable improvement in the compressive and flexural properties. The detailed testing and analysis are presently going on and the results will be discussed during the presentation.

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