

Role of Dispersion of Multiwalled Carbon Nanotubes on Compressive Strength of Cement Paste

Jyoti Bharj, Sarabjit Singh, Subhash Chander, Rabinder Singh

Abstract—The outstanding mechanical properties of Carbon nanotubes (CNTs) have generated great interest for their potential as reinforcements in high performance cementitious composites. The main challenge in research is the proper dispersion of carbon nanotubes in the cement matrix. The present work discusses the role of dispersion of multiwalled carbon nanotubes (MWCNTs) on the compressive strength characteristics of hydrated Portland IS 1489 cement paste. Cement-MWCNT composites with different mixing techniques were prepared by adding 0.2% (by weight) of MWCNTs to Portland IS 1489 cement. Rectangle specimens of size approximately 40mm × 40mm × 160mm were prepared and curing of samples was done for 7, 14, 28 and 35 days. An appreciable increase in compressive strength with both techniques; mixture of MWCNTs with cement in powder form and mixture of MWCNTs with cement in hydrated form 7 to 28 days of curing time for all the samples was observed.

Keywords—Carbon Nanotubes, Portland Cement, Composite, Compressive Strength.

I. INTRODUCTION

CEMENT is a construction material commonly used due to its low cost and high compressive strength. An improvement in basic mechanical properties and durability of materials based on cement is of high importance these days. Remarkable improvements in the electrical and mechanical properties of cement composite materials, when carbon nanotubes (CNTs) are used as fillers instead of other conventional materials, have been demonstrated by various research groups [1]–[3]. Water-cement ratio, porosity, bonding between cement and aggregates are some of the major factors that govern the strength of cement concrete. Carbon nanotubes showing outstanding mechanical properties have been the subject of many investigations as reinforcement for several composite applications. They are also highly flexible and capable of bending in circles and forming knots.

Different research groups have focused their studies on the effect of CNTs on the mechanical properties of these composites [4]–[9]. However, dispersion of CNTs in the cement matrix possesses the major problem when dealing with high performance cementitious composites. The strong van der Waals forces make it difficult to achieve desired level of

dispersion as CNTs tend to agglomerate and form bundles.

Different approaches for uniform dispersion of CNTs have been proposed so far. CNTs have been added to the cement as a powder using rotor mixing technique [10], [11] or as water dispersions [12]–[18]. Direct growth of CNTs on the cement particles [19], [20] was used as an alternative approach. Saez de Ibarra et al. [21] used gum Arabic as a dispersing agent to find slight gain in compressive strength and Young's modulus. Wansom et al. [22] investigated the electrical properties of CNT-cement nanocomposites using a polycarboxylate based superplasticizer and methylcellulose. More recently, in order to obtain homogenous dispersions of MWCNTs in water, Cwirzen et al. [23] used polyacrylic acid.

Different solvents (ethanol, toluene, chloroform) have been used for uniform dispersion of carbon nanostructures via sonication [24]–[26]. To ensure effective dispersion, acid treatment with a mixture of 98% H₂SO₄ and 66% HNO₃ was also suggested [27]. In present work the effect of dispersion of MWCNTs on compressive strength of MWCNT-Cement composite has been experimentally investigated for both MWCNT-Cement composite in powder form and MWCNT-Cement composite in hydrated form so as to determine the influence of dispersion of as-grown CNT with open flame synthesis using domestic LPG as the carbon feedstock.

II. EXPERIMENTAL PROCEDURE

A. Material Used

Commercial grade Portland cement (IS 1489) was used as the source material for all specimens. MWCNTs were prepared by an open Flame Synthesis method using domestic LPG as fuel and oxygen as oxidizer. The soot deposited on the substrate was collected from the substrate mounted on the test rig. Additional heating of soot for 15 min was done to remove traces of carbon from the soot.

Cross flow micro filtration technique was used to remove amorphous carbon, residual catalyst and other unwanted species. These impurities interfere with most of the desired properties of the CNTs. Filtration kit fitted with vacuum pump and Fluoropore filter paper (0.5 μm pore size) was used for the cleaning of CNTs from the deposited soot. Purification was done in four steps: first soot was dispersed in H₂O₂ and it was filtered, followed by DI water, Ethanol and SDS (Sodium Dodecyl Sulphate) treatments. Final sample was dried and characterization of the as grown CNT samples was done. Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Raman spectroscopy and X Ray Diffraction (XRD) techniques were used to get information about structures of the specimen synthesized. Tem images of

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the purified soot clearly indicated the presence of MWCNTs in diameter range of 8-20nm.

III. MIXING PROCEDURE

A. Aqueous Mixing Method

MWCNT-Cement paste with water/cement ratio of 0.4285 was prepared. 0.2% by weight of MWCNTs were added to cement. CNTs were gradually added to the water, and stirring was done at the same time. The beaker with CNT mixed water was then placed in a sonicator for 90 minutes for the uniform dispersion. The sonication of the CNTs in the beaker was achieved because of the vibration of the equipment. DI water with MWCNTs was gradually added to the cement and hand mixing was simultaneously done using a stirrer for 3 minutes. A mechanical mixer was also used for further homogeneous mixing. Specimens of size 40mm×40mm×160 mm, as shown in Fig. 4, were prepared using wooden moulds at the room temperature. After the 24 hours, specimens were taken out of the moulds and were placed in water for curing. The specimens were tested after curing for 7, 14, 28 and 35 days.



Fig. 1 Cement- MWCNTs & DI water paste



Fig. 2 CNT/Cement mixture in wooden mould



Fig. 3 Test specimen

B. Powder Mixing Method

A powder mixture of 350 grams of cement and 0.7 grams MWCNT was prepared. Then this mixture was added to 150 grams of DI water. Water/cement ratio of 0.4285 was taken as same as in case of first method. Carbon nanotubes were added in the amount of 0.2% by weight of cement. The mixture was kept for ultra-sonication to get uniform dispersion of CNTs in cement. The whole mixture was then further mixed by using a kitchen mixer for better mixing of CNTs and cement. Specimens of size 40mm×40mm×160mm, as shown in Fig. 3, were prepared to test for compressive strengths. Mixture was put in the wooden mould for 24 hours. It was then taken out and was put in water bath for curing. The specimens were tested after curing for 7, 14, 28 and 35 days.

C. Testing of Specimens

The compressive strength of the specimens was tested with Hydraulic Mechanical Testing System (MTS) compressive testing machine. Compression test machines perform tension or compressive tests on materials and products. The specimen was placed flat face horizontal and axial load was applied at a uniform rate till failure occurred



Fig. 4 Hydraulic Mechanical Testing System

IV. RESULTS AND DISCUSSION

The uniform dispersion of MWCNT is of high importance to achieve the desired level of reinforcement within the composite. However, due to van der Waals forces resulting from large surface area of MWCNT, they tend to adhere together and it becomes extremely difficult to separate them. Powder mixing of MWCNTs and cement was not found to be suitable for uniform dispersion. The compressive strength of cement MWCNT-Cement composite was found to be around

8.2% higher than those of pure cement specimen. Good quality of the CNT water dispersions significantly affected the mechanical properties of the composite materials. In aqueous mixing method sonicator was used for mixing the MWCNTs within DI water. Ultrasonic waves were transmitted into water and MWCNTs producing alternate expansion and compression. Microscopic bubbles were created by this pressure fluctuation. These bubbles increased in volume during negative pressure excursions and imploded viciously during the positive excursion. The collapsing of bubbles gives rise to huge number of shock waves, acoustic streaming, high pressure and extreme temperature. The total energy produced by the cumulative effect of this process is extremely high and capable of breaking agglomeration of MWCNTs and uniformly distribute the MWCNTs bundles across the DI water. An increase in compressive strength up to 22% was observed when CNTs were dispersed in DI water with sonication as given in Table I.

TABLE I
COMPRESSIVE STRENGTH (MPa)

Curing days	Pure cement Aqueous mixing without MWCNTs	MWCNTs, cement Powder form	MWCNTs, cement Aqueous form
7	12.53	13.5	15
14	14.47	15.65	17.37
28	17.05	18.5	20.08
35	17.20	18.63	20.20

V.CONCLUSION

The compressive strength of the cement composites containing Multiwalled Carbon Nanotubes have been investigated through experimental research. It was observed that the addition of carbon nanotubes to cement can enhance its compressive strength. This enhancement is due to the improvement of material microstructure. MWCNTs fill the fine pores and decrease the porosity of cement composites. Therefore, the newly formulated composites become much more compacted. Effective dispersion was achieved by applying ultrasonic energy to the mixture and no surfactant was used to disperse MWCNTs in DI water. The compressive strength of cement CNT composite with the blending of MWCNTs in aqueous form was found to be better than the compressive strength of the cement CNT composite when mixed in powder form. To achieve effective reinforcement, uniform dispersion is the key factor.

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