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Effect of Concentration of Multiwalled Carbon Nanotubes on Mechanical Strength of Ultra-High Performance Nanocomposite Cementitious Materials

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***Abstract* - Ultra-high performance cementitious composites (UPHCC) with their unique composition, mechanical properties and high durability characteristics form the basis for the design and production of ultra-high-performance concretes and special concretes that are required for extreme constructions, super-tall structures, bridge connections and special repairs. Calcium silicate hydrate (CSH) gel formation, which occurs at nanoscale level, is the main factor for strength gain of cement concrete. If a kind of nonoscale bonding can be developed between the CSH and the nanosized foreign material, that holds the cement matrix much better than the CSH itself, then the strength of cement concrete can be enhanced from the new nanocomposite. Multiwalled Carbon nanotubes (MWCNT), with their extraordinary mechanical properties and very high aspect ratio, are becoming the most promising nano materials. In this research, the MWCNT concentrations of 0.05, 0.15, 0.25, 0.35 and 0.5% by weight of cementitious paste are added to the cementitious matrix and their effect on mechanical properties, namely, compressive strength and split tensile strength are studied. Test results show that both the strengths can be improved with the inclusion of low concentration of MWCNTs.**

Keywords— UHPCC, cement, nanocomposite, bond, mechanical strength, MWCNT

I. INTRODUCTION

Ultra-high performance cementitious composites (UPHCC) are the unique cement composites that are explicitly designed to develop and produce special concretes for specific structures and constructions. UHPCC, typically with very high strength and good flow properties, are produced by using cement along with supplementary cementitious materials (SCMs) and/or fine fillers, at very low water-binder (w/b) ratio [1]. Super plasticizers are generally required to meet the w/b and rheology criteria. In general, cementitious materials are brittle and have very low tensile strength and strain capacity. Steel reinforcing bars, generally macroscopic are included to provided tensile strength, toughness and ductility. Researchers, in the past few years, started testing discrete macro to micro fibers to control the growth of cracks in cementitious materials [2-7]. By using discrete fibers, the tensile strength is developed from large quantity of individual fibers rather than few steel bars [2], and hence use of discrete fibers result in more uniform distribution of stresses within the cementitious matrix. The development of new nanosized fibers with exceptional properties like carbon nanotubes (CNT) provided an opportunity for nanaosized reinforcement within concrete. CNT, being the strongest material so far, is thought to be an ideal discrete reinforcement for cement concrete [8].

CNTs are tubular structures made up of one or multiple layers of graphite sheets. Graphite is a flat sheet formed by layers of carbon atoms bonded in a hexagonal pattern. Types of CNTs include: single walled CNT (SWCNT), that are made of only one sheet, and multiwalled CNT (MWCNT), made of more than one sheet. SWCNT has a diameter of 1 to 3 nm, and multiwalled MWCNT has a diameter range between 10 to 100 nm. The length of both SWCNT and MWCNT range between 0.5µm to 50µm. CNTs have very high aspect ratios ranging from 500:1 to 2;500;000:1. By adopting suitable techniques, CNTs can be distributed widely and densely at the microscopic scale covering longer lengths. This property of CNT helps to bridge the cracks between cement compounds and restrict them from increasing further and essentially create a new generation of a crack-free material [9, 10]. Salvetat et al [11] and Yu et al. [12], have shown that CNT has an average modulus of elasticity of around 1 Tpa, a tensile strength of 60GPa (which is theoretically 100 times stronger than steel), and an ultimate strain of 12%. Despite being the strongest material known, it has considerable flexibility. Because of its very high aspect ratio, high strength, and flexibility, it is thought to have a great significance in the construction industry if it can be used as a reinforcing material. However, the extraordinary and superior mechanical properties of the CNTs alone do not ensure mechanically superior nanocomposite cementitious materials.

The properties of nanocomposite are strongly influenced by two main factors: i) dispersion of CNTs within the cementitious matrix and ii) bond strength between the matrix and surface of the CNTs. CNTs are strongly attracted to one another because of high Vander Waals forces and causes the CNTs to be more prone to agglomeration which finally result in rapid settelement of CNTs out of suspension. Therefore, CNTs should be properly treated with surfactants and mixed with an ultrasonic mixer before adding into cementitious matrix. Research has shown successful results in dispersing CNTs within aqueous solutions [13, 14] but only a few of these techniques can be applied to cementitious materials because of the delayed hydration of the cement paste caused by surfactants [15]. Ethanol and methyl cellulose have been used widely for dispersion purposes to ensure a better bonding between CNTs and cement compounds. It is also reported that a high-range polycarboxylate based water reducing admixture disperses the CNTs well and with minimum effect on hydration time of cement paste [16].

Studies related to mechanical properties, microstructural characteristics, shrinkage and durability of CNT-cementitious composites have been reported in the literature [15, 17-22]. Overall, it is reported that the mechanical strength of cementitious composites can be increased by including CNTs, and the achieved reinforcement effect depends on effective dirpersion and efficient distribution of the reinforcement throughout the matrix [19, 22, 23]. It is observed in the literature survey that, up to 1% of CNTs by weight of binder is added to cementitious composites [15, 17, 18, 20, 22, 24]. Higher concentrations of CNTs obstruct the uniform distribution and becomes ineffective. Li et al. [22], observed a 19%–25% increase in the compressive and flexural strength of cement pastes reinforced with 0.4–0.5% CNTs by weight of cement only when they were surface modified by carboxylation). The use of non-treated CNTs resulted in lower compressive strengths than the reference mix without CNTs. Moderate increase in the mechanical strength of CNT-cementitious composites were reported by [18, 25, 26]. Using varying dosages of non-treated CNTs (0.5%–1%), Kumar et al. [27] attained the highest mechanical strength for 0.5% of CNTs, at 15% (compressive strength) and 36% (flexural strength) higher than that of control mixture without CNTs. For higher CNT dosages, the dispersion was less effective. Konsta-Gdoutos et al. [10] elasticity modulus using only 0.05% of CNTs by weight of cement.

While considerable research on inclusion of CNTs in cement pastes has been done, little focus was given to their inclusion in ultra-high performance cementitious composites (UHPCC), containing other supplementary cementitious materials along with the cement. The UHPCC should possess high packing density and flowability at very low w/b ratio. In the present study, the compressive strength and splitting-tensile strength of Ultra-high performance cementitious composite made of Ordinary Portland cement and Micro silica (10% by weight of cement) at w/b of 0.2 and reinforced with multiwalled CNTs (MWCNT) were investigated. Micro silica is used as SCM due to its ability to fillup the micro pores in the cement matrix owing to its sub-micron size. Also use of micro silica in UHPCC is extensively agreed in the literature. Ultrasonic energy was applied for effective dispersion of MWCNTs in water. MWCNT concentrations of 0.05, 0.15, 0.25, 0.35 and 0.5% by weight of cementitious paste have been used. Cube and cylinder specimens were casted and tested after 28 days of curing.

Table 1 Chemical composition and physical properties of materials

Material	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %	SO_3 %	LOI %	Avg. Particle size - µm	Specific gravity	Fineness M^2/gm
OPC (C)	21.41	4.88	3.82	63.69	1.56	2.36	1.4	15	3.15	0.33
Micro silica (M)	92.9	1.2	0.74	0.02	1	0.1	1.8	0.15	2.2	15 (min)

II. EXPERIMENTAL SETUP

A. Materials

The materials used for the preparation of UHPCC are: Ordinary Portland cement (OPC) (53 grade) conforming to IS: 12269-1987 (R2004); microsilica (M) 920 supplied by Elkem Industries, Mumbai conforming to ASTM-C(1240-2000) in dry densified form; Poly carboxylic ether (PCE) based super plasticizer (SP) supplied by Hella infra, Mumbai, used as surfactant and also as water reducing admixture; multiwalled carbon nanotubes (MWCNT) supplied by iENT Tech, Inc., (Carbon Horse), Tamilnadu and potable water conforming to IS 456-2000 are used. The chemical composition and physical properties of the materials used in the study is presented in Table 1. Physical properties of MWCNT are shown in Table 2. Fig. 1 shows the TEM and SEM images of MWCNT.

Table 2 Physical properties of MWCTs

MWCNT	Description
Production method	Chemical Vapor Deposition (CVD)
Diameter	10 nm
Length	6 - 9 µm
Purity	>98%
Metal particles	<1%
Amorphous carbon	<1%
Specific surface area	250 - 300 m^2/g
Bulk density	0.10 - 0.06 g/cm^3

(a) (b)

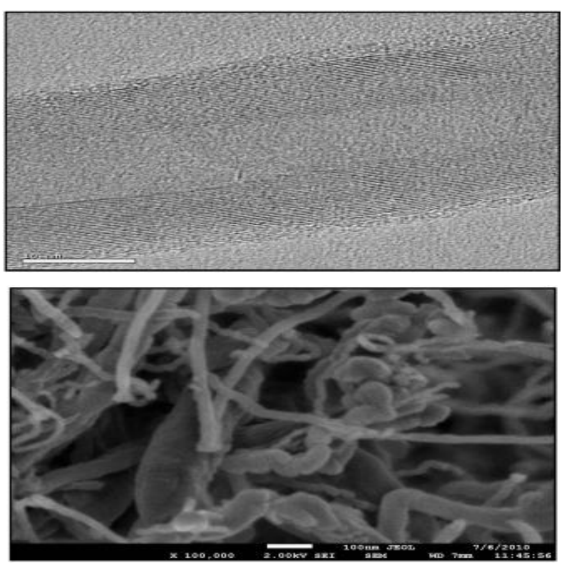

Fig. 1. TEM and SEM image of MWCNT a) MWCNT-1D structure of 10-15nm dia (TEM) b) MWCNT-1D entangled structure 6-9 µm (SEM)

B. Mixture constituents

Table 3 shows the different types of UHPCC mixes tested in the study. A constant w/b of 0.2 and PCE superplasticizer of 2% by weight of cementitious composite is used. MWCNTs concentration used are: 0.05%, 0.15%, 0.25%, 0.35% and 0.5%. Mixture designation CM is the control mix with OPC and Micro silica and without MWCNTs. For UHPCC mixes, the number after MWCNT indicates the concentration (% by weight of cement) of the MWCNTs in the mix.

Table 3 UHPCC Mix proportions and percentages of MWCNT

Mix ID	OPC %	Micro silica %	w/b ratio	MWCNT % OPC Wt.	Sonication time (min)
CM	90	10	0.2	0	60
MWCNT0.05	90	10	0.2	0.05	60
MWCNT0.15	90	10	0.2	0.15	60
MWCNT0.25	90	10	0.2	0.25	60
MWCNT0.35	90	10	0.2	0.35	60
MWCNT0.50	90	10	0.2	0.50	60

C. Mixing procedure

Calculated amounts of all OPC, Micro silica, Super plasticizer (SP) (also used as surfactant), MWCNTs and water were taken in separate containers. First SP was added to water and mixed properly. MWCNTs were gradually added to water mixed with SP with simultaneous stirring. Then the container with MWCNTs mixed with water and SP was placed in a sonicator for 60 minutes to obtain better dispersion of MWCNTs in the UHPCC paste. After completion of sonication, the water with SP and MWCNTs was gradually added to the mixture of OPC and microsilica (which was previously dry mixed for homogenation and de-agglomoration of micro silica particles) manually mixing with a scoop. The whole mixer was then mixed with a 5-speed electric planetary mixer till the mix was seen flowable. The mixing time lasted for about 5 minutes. After mixing, specimens for compression test and splitting-tensile strength test were casted and cured for 28 days.

D. Specimen preparation

As shown in table 3, one control sample and five different UHPCC- MWCNT samples were prepared. Three cubes and cylinders were cast for each mix ID. Because of the limited amount of MWCNTs, and also considering the capacity of the testing equipment, smaller size specimens were casted. Size of cube specimens was 50mm and cylindrical specimens was 50mm dia x 100mm length. All the specimens were kept in the molds at room temperature for 24 hrs. After demolding the specimen were kept in water for 27 days and cured. The specimens were then taken out of water, surface dried and then tested for compressive and splitting-tensile strengths at 28 days.

III. RESULTS AND DISCUSSION

Fig. 2 shows the compressive strength of the control mix without MWCNTs and UHPCC mixes with varying concentration of MWCNTs at the age of 28 days.

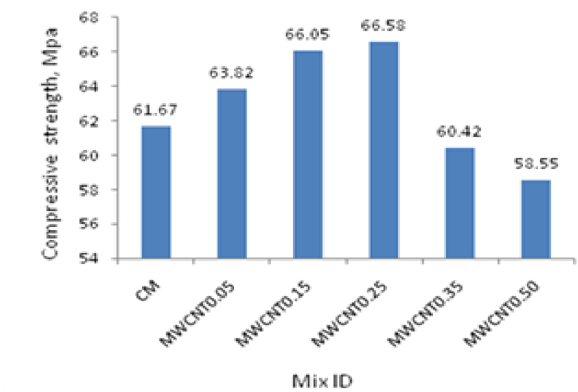

Fig. 2. Compressive strength of Control mix and UHPCC mixes w.r.t. MWCNT concentration (28 days)

From Fig. 2, it is observed that the maximum compressive strength of 66.58MPa occurred at a MWCNT concentration of 0.25%. Also the strength is almost same for 0.15% MWCNT. It is observed that the strength increased till 0.25% MWCNT and then decreased as the MWCNT increased. For both 0.35% and 0.5% MWCNT, the compressive strength is even less than that of control mix. The strength for 0.25% MWCNT is 108% when compared to that of control mix. This shows that the addition of smaller amounts of MWCNT has positive effect on the compressive strength of the UHPCC mixes. The decrease in compressive strength, as the MWCNT% increases beyond 0.25% may be due to inadequate dispersion of the high content of the MWCNT.

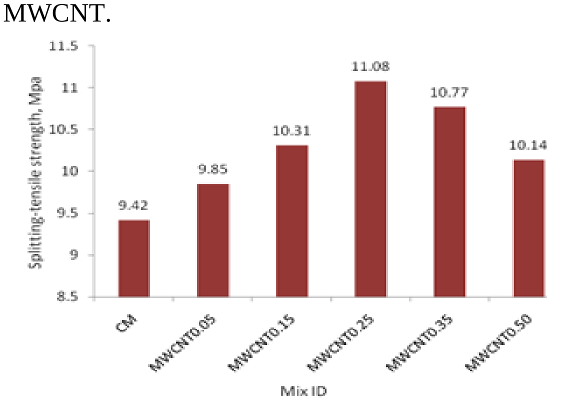

Fig. 3. Splitting-tensile strength of Control mix and UHPCC mixes w.r.t. MWCNT concentration (28 days)

From Fig. 3, it is observed that the highest splitting-tensile strength of 11.08MPa also occurred at a MWCNT concentration of 0.25%, which is 117% to that of the control mix. The same trend of compressive strength is also observed with the splitting-tensile strength. Hence, it can be said that, the optimum concentration of MWCNT is 0.25% by weight of UHPCC paste.

IV. CONCLUSIONS

In this paper, the effect of concentration of multiwalled carbon nanotubes (MWCNTs) on mechanical strength namely, compressive strength and splitting-tensile strength, of ultra-high performance nanocomposite cementitious materials is studied, and the following conclusions are drawn:

1. Addition of MWCNTs to the UHPC composites have a positive effect on both the compressive and splitting-tensile strength properties upto a concentration of 0.25% by weight of cementitious paste. Increase in MWCNT content beyond 0.25% led to the reduction in both strengths.
2. The compressive strength for UHPCC mixture is 108% and splitting-tensile strength is 117% compared to that of the control mix. This indicates that the MWCNTs are more effective in altering the tensile propreties of the UHPCC mixtures.
3. From the increased strength values, it can be understood that the dispersion of MWCNTs by combination of surfactant and sonication was productive, and hence both the techniques should be followed for proper and adeqaute dispresion of the MWCNTs.

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