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Experimental study on compressive strength of cement-CNT composite paste

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The role of carbon nanotubes (CNTs) on the compressive strength characteristics of hydrated Portland IS 1489 cement paste has been studied. Standard specimens (40 mm × 40 mm × 160 mm) as per IS: 516-2004 were prepared by mixing 0.1% CNTs by weight to cement for determining the compressive strength of composites. The specimens were tested after 7, 14, 28 and 35 days of curing. Results show an increase in compressive strengths in Cement–CNTs composites having CNT content of 0.1% by weight of cement. The increase in compressive strengths with both techniques; mixture of CNTs with cement in powder form and mixture of CNTs with cement in hydrated form were 8.5% and 22%, respectively by holding the specimen for 28 days of curing. Increase in curing time by 7 days from 28 to 35 days did not bring any appreciable increase in compressive strength because of the complete absorption of all the ingredients into the mixture due to saturation.

Keywords: Portland cement, Carbon nanotube, Composite

1 Introduction

Portland cement (PC) has been used as a major construction material all over the world. The hydration of cement forming complex chemical compounds binds the aggregates together and provides strength to cement concrete. Water-cement ratio, porosity, bonding between cement and aggregates are some of the major factors that govern the strength of cement concrete. But chemical composition of cement remains the most important factor that affects its strength. CNTs are considered one of the most beneficial materials for nano-reinforcement. The unique mechanical, electrical and chemical properties of CNTs make them an attractive candidate for reinforcement of composite materials. It is due to the presence of carbon atoms with their tendency to make strong covalent bonds with other elements due to their electronegative character which produce stronger materials in form of composites. Thus, it becomes the strongest as well as flexible known material so far. So with the development of new nanosized materials in the form of tubes, fibers and particles have opened a new field for nano sized reinforcement within concrete[1-3].Two major challenges for using carbon nano structures (fibres, particles, tubes) to form cement concrete are the uniform dispersion and inefficient bonding in cement matrix, as reported by Makar *et al*[4]. and Groert [5]. Poor dispersion of CNTs leads to the formation of many defect sites in the nanocomposite and limits the efficiency of the CNTs in the matrix. Studies are still going on to counter these challenges[6]. The performance of concrete can be significantly improved by adding nanoparticles. The compressive strength of the cement paste is increased with the addition of nanotubes and decreased the number of mesopores as reported in earlier studies[7]. Different researchers have attempted to improve the uniform dispersion and bonding strength by various treatments. Saez de Ibarra *et al.* used gum Arabic as a dispersing agent to find slight gain in compressive strength and Young's modulus[8]. Wansom *et al*[9]. investigated the electrical properties of CNT-cement nanocomposites using a polycarboxylate based superplasticizer and methylcellulose. More recently, in order to obtain homogenous dispersions of CNTs in water, Cwirzen *et al*[10]. used polyacrylic acid polymers and sonication. The results showed a slight increase in compressive strength. It has been demonstrated that interfacial adhesion can be attributed to the interfacial chemical bonds and interaction between polar groups as hydroxyl or carboxyl on the surface of the reinforcing fiber and the active groups present in the matrix resin[11-13]. These chemical groups can originate strong chemical bonds between CNT and cementitious matrix, thus enhancing the reinforcement efficiency[14-16]. In the present study, the effect of compressive strength of cement paste as well as CNT-cement composite has been experimentally investigated for both CNT-cement composite in

powder form and CNT-cement composite in hydrated form so as to determine the influence of dispersion and functionalization on as-grown CNT with open flame synthesis using domestic LPG as the carbon feedstock obtained under controlled parameters of flow rate, substrate temperature, and exposure time.

2 Experimental Details

2.1 Materials used

Commercial grade Portland cement PC (IS: 1489) was used as the source material for all specimens. CNTs were prepared by an open Flame Synthesis method using domestic LPG as fuel and oxygen as an oxidizer. The carboneous soot (20 g) was collected from the substrate mounted on the test rig in small batches (0.5 to 1 g/min) during 5 h of continuous operation. Cross flow micro filtration technique was used to remove amorphous carbon and other unwanted species. The collected soot was heated fro about 15 min to remove traces of amorphous carbon. Finally, the samples were dried and characterizations of the as grown CNT samples were done. Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Raman spectroscopy and X ray diffraction (XRD) techniques were used to obtain information about CNT structures of the specimen synthesized. Figure 1 shows the TEM micrograph of the CNTs obtained from LPG. It is observed that CNTs were of multiwall in nature with diameter in the range 8-25 nm and lengths approximately in the range of 120 nm. SEM image of the purified sample is shown in Fig. 2. The surface morphology of the synthesized MWCNTs seems to be uniform. The SEM micrograph shows clear evidence of the formation of carbon nanotubes. Additionally, there are some small agglomerates which can be attributed to amorphous carbon present in the sample. In Fig. 3, the XRPD pattern of synthesized MWCNTs at LPG flow rate of 05.1 pm and O_2 flow rate of 41 pm is shown. The diffraction peaks were observed at 23.13°, 41.98° and 49°. The strong reflection peak at 23.13° confirmed that the MWCNTs were crystalline in nature as this peak arises due to interlayer stacking of graphene sheets. Using Scherrer equation, the approximate diameter of MWCNTs was calculated as 12.13 nm. As grown CNTs were characterized by Reneshaw Invia Raman Microscope with He-Ar laser (514 nm) with 50% laser strength. Figure 4 shows the results of Raman analysis showing G band at 1586.2 cm^{-1} and D band at 1351.4 cm^{-1}. The ratio between the D band and G band is an indicator of the quality of CNTs in a sample. (I_D/I_G) was calculated as 0.94 which indicated that highly ordered MWCNTs were present in this sample.

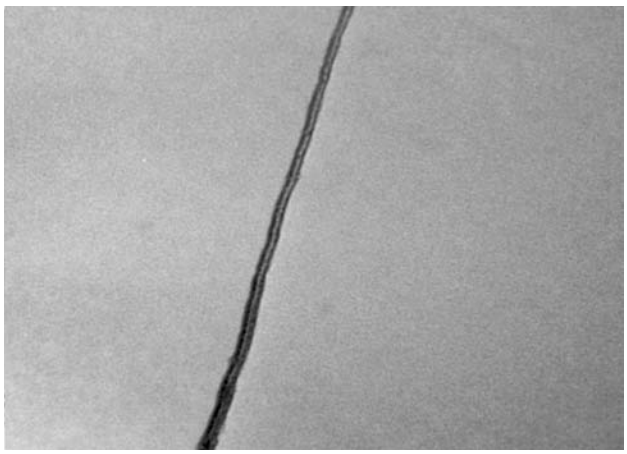

Fig 1 — TEM micrograph of synthesized MWCNTs

2.2 Specimen preparation

As per IS: 516-1959 (Reaffirmed 2004 Indian Methods of tests for strength of concrete),three specimens each of pure cement, Aqueous mixing

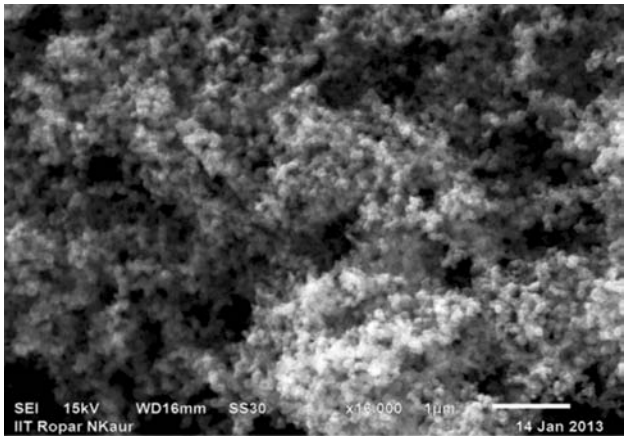

Fig. 2 — SEM image of purified MWCNTs

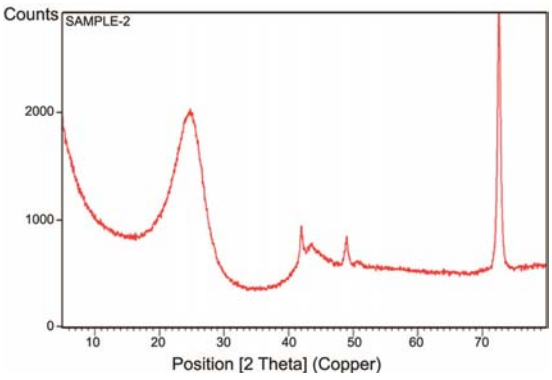

Fig. 3 — XRD spectra showing peaks of ordered MWCNTs

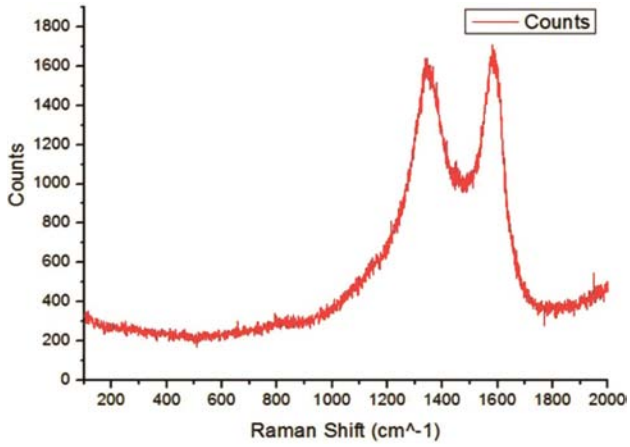

Fig. 4 — Raman spectra showing D band and G band peaks of MWCNTs

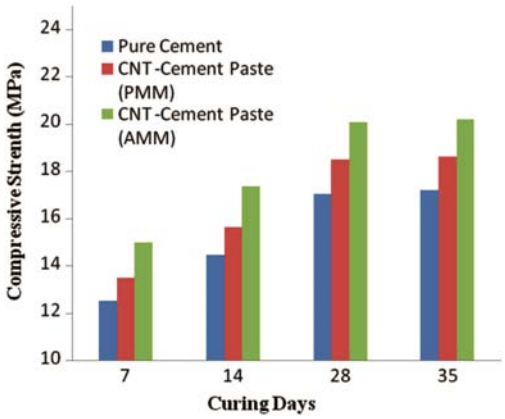

Fig. 5 — Comparison of the compressive strength of pure cement paste, CNT –cement paste (PMM) and CNT-cement paste (AMM)

method and Powder mixing method of size 40 mm×40 mm×160 mm each for 7, 14, 28 and 35 curing days were prepared.

2.2.1 Aqueous mixing method

In Aqueous mixing method (AMM), weight of PC was taken as 400 g to which 0.4 g of CNTs (0.1%) dispersed in 170 ml of deionized water (DI) was added to form the paste. The water/cement ratio was taken as 0.425. Mechanical stirring was simultaneously done for 15 min. For uniform dispersion, the mixture was then placed in a sonicator for about 90 min. (DI) water/CNTs mixture was gradually added to the cement and hand mixing was done using a stirrer for 3 min. For a homogeneous mixing, mechanical mixer at 1400 rpm was also used. No surfactant was used in the present work. Specimens of size 40 mm × 40 mm × 160 mm were prepared using wooden moulds at room temperature. The moulds were left to solidify for 24 h, and then specimens were taken out of the moulds and placed in water for curing. The specimens were tested after curing for 7, 14, 28 and 35 days.

2.2.2 Powder mixing method

In Powder mixing method (PMM), a powder mixture of 400 g of cement and 0.4 g (0.1%) CNT was prepared. Then 170 ml of DI water was added to this mixture.Water/cement ratio of 0.425 was taken same as in the case of AMM. 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 mechanical mixer for better mixing of CNTs and cement. Mixture was put in the wooden mould for 24 h. It was then taken out and put in water bath for curing. The specimens were tested after curing for 7, 14, 28 and 35 days and Fig. 5 shows the bar chart of the average readings taken.

3 Testing of Specimens

The compressive strength of the specimens was tested with Hydraulic Mechanical Testing System (MTS) by placing the specimen in the MTS compressive testing machine and applying axial load at a uniform rate till failure occurred.

4 Results and Discussion

The homogenous dispersion of CNT 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 CNT tend to adhere together and it becomes extremely difficult to separate them. Manual stirring for powder mixing of CNTs and cement was found to be less effective for uniform dispersion of nanotubes. This process was not capable of producing required energy to break the agglomeration of CNTs.

Therefore, the compressive strength of CNT-cement composite formed with powder mixing method was found to be less as compared to aqueous mixing method as shown in Fig. 5. Aqueous mixing of CNTs and DI water was done using sonicator. Ultrasonic waves were transmitted into water and CNTs causing alternate expansion and compression of the resultant mixture. 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 collapin of bubbles give 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 CNTs and distribute the CNTs bundles across the DI water uniformly. An appreciable increase in compressive strength for both the specimens (Powder mixing 8.5% and Aqueous mixing 22%) was observed. Lirisa *et al*[17]. and Campillo *et al*[11]. have also reported similar gain of 13% and 30% in the compressive strength of cement/CNT nanocomposites.

5 Conclusions

The focus of the present study is to investigate the compressive strength for PC, AMM and PMM without using any surfactant/chemical for dispersion of CNTs. The compressive strength of cement-CNT composite with the blending of CNTs in aqueous and powder form is better than the compressive strength of the pure cement. The dispersion of CNTs in aqueous form is better than the dispersion of CNTs in powder form. Uniform rate of increase of compressive strength takes place with the curing time from 7 to 28 days for all the samples. Increase in curing time from 28 days to 35 days did not bring any appreciable increase in compressive strength.

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