Experimental Investigation of Mechanical Properties of Multi-Walled Carbon Nanotube Reinforced Cementitious Nano-Composites

A Dissertation

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By

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Certificate

I hereby certify that that the work being presented in the dissertation entitled **"Experimental investigation of mechanical properties of multi-walled carbon nanotube reinforced cementitious nano-composites"** in the partial fulfilment of the requirement of the award of the Degree of Master of Technology and submitted to the Department of Mechanical Engineering of Lovely Professional University, Phagwara, is an authentic record of my own work carried out under the supervision of Dr. Sumit Sharma, Assistant Professor, Department of Mechanical Engineering, Lovely Professional University. The matter embodied in this dissertation has not being submitted in part or full to any other university or institute for the award of any degree.

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This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

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The external viva-voce examination of the student was held on successfully …………………….

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ABSTRACT

This research was performed to investigate the uses of multi-walled carbon nanotubes (MWCNTs) when reinforced with Portland cement. The exceptional mechanical properties and geometrical characteristics, were the vibrant inspiration for this study. In this study, we joined this distinctive material (CNTs) with cement paste which is the most utilized man-made material. At the point when contrasted with other composite materials, a constrained measure of research has been directed on the CNTs/Cement composites.

To check the effect of MWCNTs on the mechanical properties of the cement composite, two distinctive mixtures of the MWCNTs/cement composite were prepared and tested. Distinctive groups had a fixed water cement ratio of 0.4, and different wt% of MWCNTs. The nanocomposites were prepared in the form of beam with scale of 127×12.5×5 mm. Three main mechanical properties: flexural strength, strain to failure and modulus of elasticity, were investigated at 14 and 21 days after casting of samples. The results of different nano composite groups were compared with the plain cement groups.

From mechanical testing we observed that, the MWCNTs increased the flexural strength of MWCNTs cement composites. 0.3% MWCNTs increased the flexural strength more than 0.2% MWCNTs cement composite at 21 days. Strain to failure of 0.2% MWCNTs cement composites was higher than 0.3% MWCNTs cement composites.

From this experimental study we concluded that the CNTs will be used as nano reinforcement, which will efficiently increase the mechanical properties of the cement composites.

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Introduction

1.1 Composite

A composite is defined as a structural material that consists two or more combined constituents that is combined at a macroscopic level and is not soluble in each other (Autar K. Kaw, 2006). A composite is a multiphase material that is synthetically prepared, as opposite to the one that take place or forms as you would expect. The each stages must be chemically different and parted by a different interphase. Composite materials are consist of three main functional constituents

a. Matrix (or primary phase)
b. Reinforcement (secondary phase)
c. Interphase

The matrix (or primary phase) fixes the fibers together freezing in the fiber orientation. The load acted to the composite are shifted at that moment to the fibers, the principle load bearing component, through the matrix, allowing the composite to resist compression, flexural, shear force and tensile loads. The capability of composite reinforcement with small fibers to support loads of any type is reliant on the occurrence of the matrix as the load-transfer medium, and the effectiveness of this load transfer is directly associated to the types of fiber/matrix bond. The matrix must distinct the fibers from individually so they can act as separate things. The matrix should also shield the supporting fibres from mechanical impairment and from environmental attack.

The reinforcement (secondary phase or dispersed phase) may be of nano, micro, or macro level. The different sizes of reinforcements help in the strengthening of the composite by different mechanisms. Generally, reinforcement is stiffer or harder in nature than the matrix. The degree of reinforcement or improvement of mechanical behavior are subject to strong attachment at the matrix and reinforcement interface.

1.2 Classification of the composites

Composites are classified on the basis of matrix phases as metal matrix composites, ceramic matrix composites, and polymer matrix composites. The classification according to the types of reinforcement are particulate composites, fibrous composites, and laminate composites, shown in the Figure 1.1.

1.3 Nano-Composite

The Nano-Composite material is an advanced invention having nano (one billionth of a meter) fillers spread in the matrix. Typically, the structure is a matrix-filler arrangement where the fillers like particles, fibers, or fragments surrounds and fixes together as distinct units in the matrix.

1.3.1 Significance of Nano-Composites

Nanoparticles and nano-layers have great surface-to-volume and aspect ratios and this makes them perfect for use in polymeric ingredients. Such arrangements combine the best properties of each component to possess enhanced mechanical and superconducting properties for advanced applications. The physical, chemical and biological properties of nanomaterials vary from the properties of distinct atoms and molecules or bulk matter. By making nanoparticles, it is possible to switch the essential properties of materials, such as their melting temperature, magnetic properties, charge capacity and even their color without altering the materials chemical arrangements.

Recent improvements in creating nanostructured materials with innovative properties have enthused exploration to create multifunctional engineering materials by designing arrangements at the nanometer scale. At nano level, some compounds makeover from inert to active, from an electrical insulator to conductors, from breakable to tough. They become stronger, lighter and more resistant. These renovated properties are what account for the infinite possible applications of nano-materials. Nanocomposites have prolonged much interest in recent times. Noteworthy efforts are in progress to control the nanostructures via innovative artificial methodologies. The properties of nanocomposite materials are subject to not only on the properties of their distinct elements but also on their morphology and interfacial appearances. By optimized manufacturing procedure and precise nano-sized second phase distribution, thermal stability and mechanical properties such as adhesion resistance, flexural strength, toughness, and hardness can be enriched.

1.3.2 Fiber reinforced nanocomposite

The composite which is formed by using fiber as reinforcement material is known as fiber reinforced composite. It consists of the three components (i) fiber as broken and distributes state, (ii) matrix in the continuous state and (iii) an interphase region. Natural fibers and synthetic fibers both are used as the reinforcement material in the composite.

1.3.3 Fiber reinforced cementitious nanocomposite

Concrete is the mixture of aggregate, sand, and cement, in which micro-cracks are develop at the early ages due to drying shrinkage and volume changes during solidifation. Basically the cracks are develop in the concrete because it is brittle in nature and it has weak in tensile strength. To improve this property of concrete the reinforcement of fiber is performed in concrete, which is known as fiber reinforced concrete (FRC). The reinforcement of fiber is performed with more or less randomly distribution of small fibers. Due to fiber reinforced the property of concrete will improve in all directions because fiber helps in transferring the loads to the internal micro-cracks. There is so many different types of fiber which is used as reinforcement are given by

a. Natural fibers
b. Synthetic fibers

1.4 Composition of proposed nanocomposite

The nanocomposite fabrication proposed in the present study combine it around two major constituents are as follows:

a. Carbon Nanotube
b. Cement

1.4.1 Carbon Nanotube

Carbon Nanotubes (CNTs) are allotropes of carbon which having cylinder-shaped nanostructure. It is discovered by Sumio Iijima (1991). It is divided into two main types such as single-walled carbon nanotube (SWCNTs) and multi-walled carbon nanotube (MWCNTs). A SWCNTs is a hollow cylinder of rolled graphene sheet, whereas MWCNTs is consist of two are more concentric tubes of rolled graphene sheet. There is a number of ways to roll graphene sheets, and the rolling up of the graphene lattice is characterized by the chirality (Grady, 2011). Three types of chirality are: central, axial, and planar, corresponding to three chiral elements, namely center, axis, and plane, and is defined as

$$\vec{C}_h = n\vec{a}_1 + m\vec{a}_2 \tag{1.1}$$

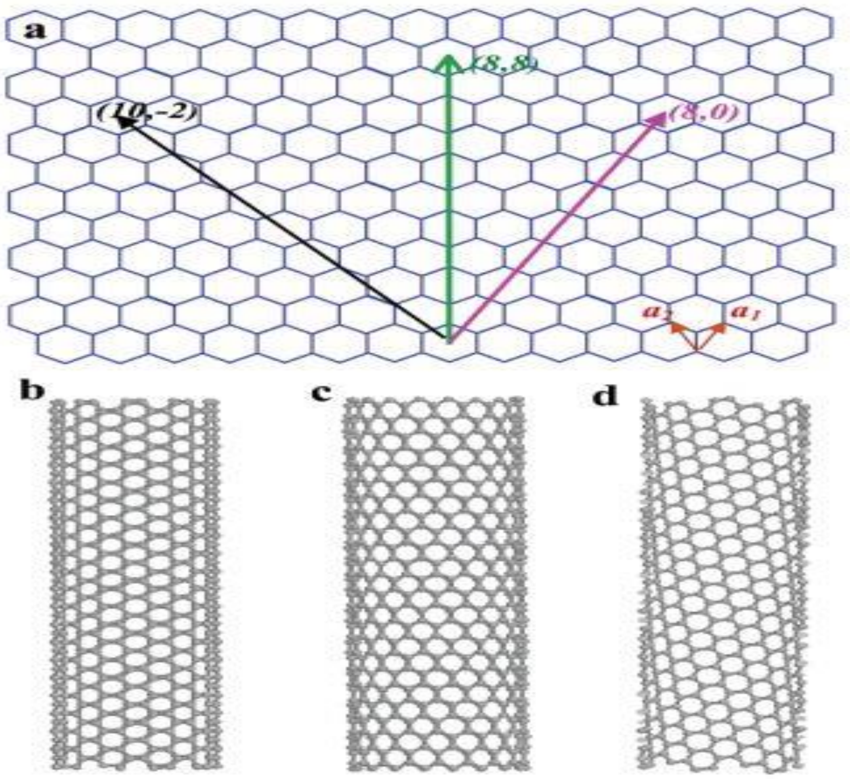

Figure 1.1, (a) schematic honeycomb structure, (b) arm chair, (c) zig zag, (d) chiral, tubes respectively. H Dai (2002)

Where n and m are integers which represent the digit of unit vectors $\overrightarrow{a_1}$ $\overrightarrow{a_2}$, along two directions in the hexagonal mesh of graphene as shown in Figure 1.1. A nanotube Constructed in this way is called an (n,m) nanotube. The diameter of a tube is represented by its (n,m) value, which is as

$$\mathrm{d} = \frac{a_{c=c}\sqrt{3(n^2 + nm + m^2)}}{\pi} \tag{1.2}$$

Where $a_{c=c}$ is the carbon=carbon bond length (1.42 Å). The inclination of hexagon of graphene sheet is categorized by chiral angle θ, which varies between 0˚ and 30˚ and is given by

$$\theta = tan^{-1}\left(\frac{\sqrt{3}m}{2n + m}\right) \tag{1.3}$$

1.4.1.1 Method to produce carbon nanotube

There is three major procedure for manufacturing of the carbon nanotubes such as arc discharge, visible light vaporization, and chemical vapor deposition.

1.4.2 Cement

Cement is a binder, an ingredient which used in construction that sets and strengthens and can fix added ingredients together. Cement is used as an element in the production of mortar and concrete to form a strong building material. It has divided into two main categories hydraulic and non-hydraulic cement. Hydraulic cement such as Portland cement which set and become adhesive due to a chemical reaction between dry elements and water whereas non-hydraulic cement not sets in wet condition but it sets and dries in the air by reacting carbon dioxide. Cement is the maximum used construction material in the world. It exhibits good fineness, soundness, setting time, strength and specific gravity, but it has poor tensile and flexural properties.

1.4.2.1 Portland cement

It is the common type of cement which is regularly used in the world. It is manufactured through heating of limestone and clay at 1450 ˚C in a kiln, the procedure is known as calcination, where Co_2 is released from $CaCO_3$ to form CaO, then mix with other material that contained within to form calcium silicate and further cementitious compound. The subsequent substance is “clinker”, powdered with gypsum to form “Ordinary Portland Cement”. It is basic constituent of concrete, mortar, and grout. It may be gray or white in color. The chemical composition of the Portland cement is given by

Table 1.1 Chemical composition of Portland cement (taken from wikipedia)

CONTENT	**PERCENTAGE (%)**
SiO_2	21.9
Al_2O_3	6.9
Fe_2O_3	3
CaO	63
MgO	2.5
SO_3	1.7

1.5 Applications

Fiber based nanocomposites are of increasing interest in the infrastructure sector because of their high compressive strength, modulus of elasticity, flexural strength and impact resistance.The main area of application, of the fiber based composite are given as

a. Aircraft parking, runway, and pavement
b. Tunnel lining and slope stabilization
c. Blast resistance structure
d. Dam and hydraulic structure
e. Wall, pipes, thin shell and manholes

1.6 Summary

This chapter discusses the basic definition and need of the nanocomposite materials. The chapter elaborates on the basic elements of these materials, the functions to be performed by them and answers the tedious question of using nanocomposite over conventional composites. The chapter also comments on the property and benefits of elements due to which they are preferred over other things of their category. Finally the origin of the problem and the potential applications of the proposed material are discussed.

Chapter 2
Literature review

2.1 Introduction

This segment shows the outline of work carried out by different researchers for improving the mechanical properties and other characteristics of fiber reinforced cementitious composite with cement as a matrix and carbon nano-tube as reinforcement.

Uygunoglu [3] prepared two different types of concrete specimen of 100 × 100 × 350 mm size, with and without steel fibers. The steel fibers are added in the ratio of 0, 0.2, 0.4, 0.6 and 0.8% by volume. The specimens were removed from mould after 24 hours and preserved in water until 7, 28, 56, 180 and 360 days. They studied the microstructure of the specimens by electron and optical microscopy for the 180 days specimen and observed that the flexural strength of steel fiber reinforced concrete (SFRC) improved with the concrete period and fiber volume fraction (V_f). It was also observed that the first crack development significantly decreased with an increase in V_f in all the concrete ages. Figure 2.1 shows the change in properties of cement after adding different amounts of fibers in it.

Su et al. [4] studied the mechanical properties of the ceramic fiber reinforced concrete (CRFRC) through a hydraulically-driven testing system and a split Hopkinson pressure bar system. They found that the quasi-static properties such as, compressive strength, splitting tensile strength and flexural strength of CRFRC increased with an increase in V_f. This can also be observed from Table 2.1.

Jiang et al. [5] predicted the mechanical characteristics of basalt fiber reinforced concrete using scanning electron microscope (SEM) and mercury intrusion porosimeter (MIP). They found that with the addition of basalt fibers there was an improvement in the tensile strength, flexural strength and toughness index but no changes were observed in the compressive strength. Comparing the plain concrete with the concrete reinforced with 12 mm basalt fiber, the compressive, splitting tensile and flexural strength improved by 0.18-4.68%, 14.08-24.34% and 6.30-9.58% respectively. As the basalt fiber length was expanded to 22 mm, the relating quality improved by 0.55-5.72%, 14.96-25.51% and 7.35-10.37%, separately and the MIP results indicated that the concrete containing basalt fiber possessed higher porosity.

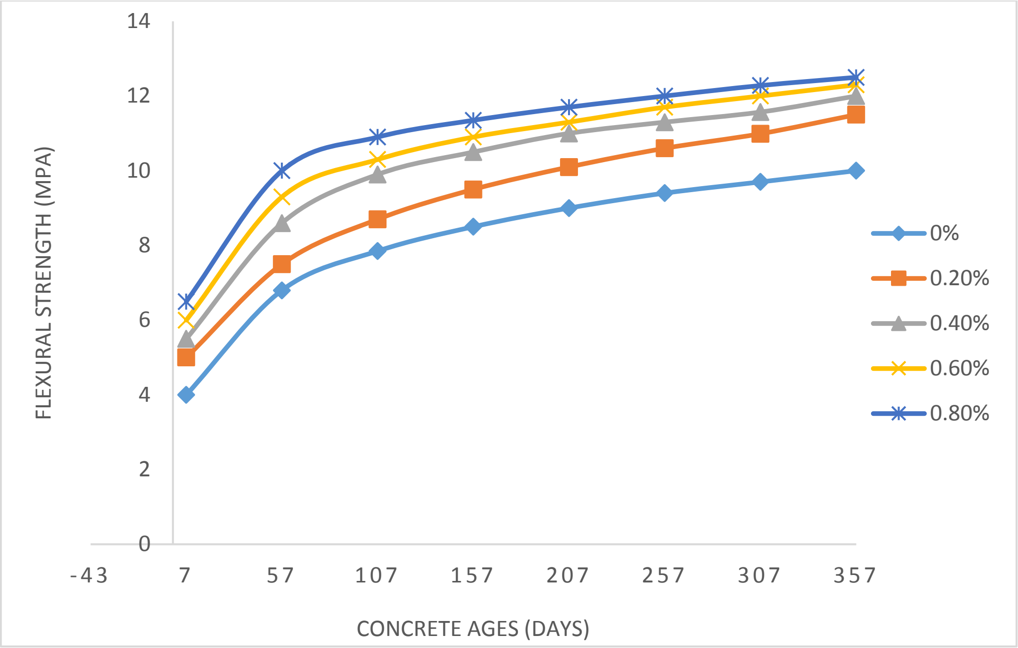

Figure 2.1. Flexural strength of SFRC vs. concrete age (Adapted from Uygunoglu [3])

Table 2.1. Quasi-static mechanical properties of CFRC.

Fiber Volume %	Compressive strength (MPa)	Splitting tensile strength (MPa)	Flexural (MPa)
0	56.6	3.99	7.9
0.1	60.8	4.25	7.7
0.2	64.4	4.05	8.3
0.3	65.1	4.33	8.9

Gul et al. [6] investigated the thermo-mechanical characteristics of raw perlite aggregate concrete on the accumulation of the hooked steel, wavy steel and polypropylene fiber. They prepared the specimen of 100% raw perlite concrete, and 0.25%, 0.75%, 1.25%, and 1.75% of fiber ratio and 350 kg/m^3 cement dosage. The samples were examined using the ASTM C-109, ASTM C-496, ASTM C-78, and ASTM C 1113-90. With growth in the steel fiber ratio, an increase in thermal conductivity, splitting tensile strength and flexural strength was observed whereas the compressive strength was found to decrease.

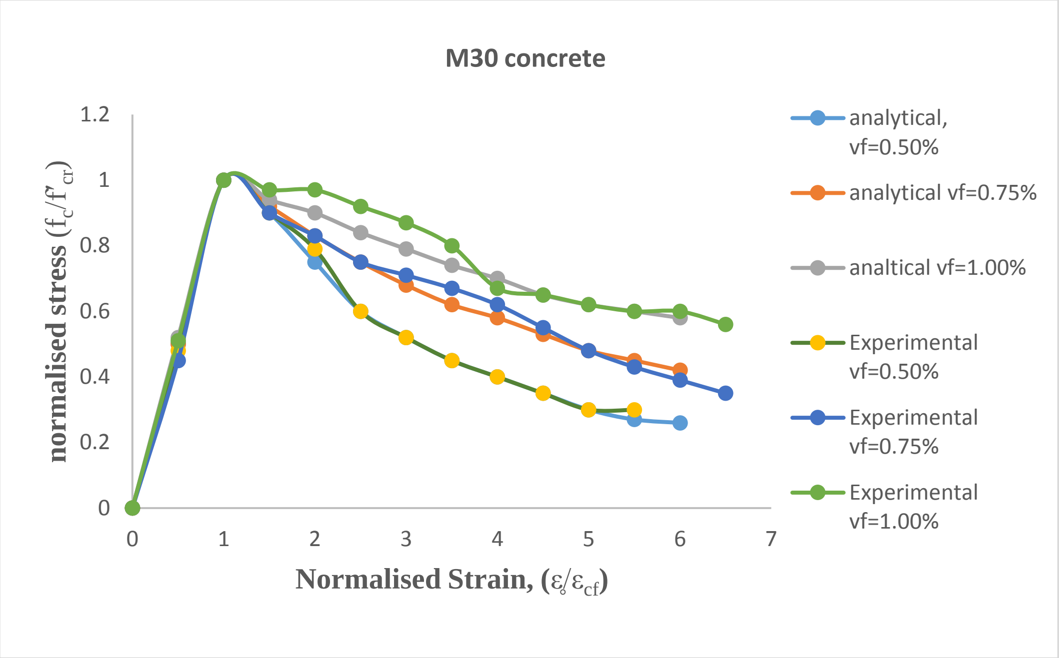

Figure 2.2. Analytical and experimental normalized stress-strain curves for steel fiber reinforced concrete (aspect ratio=82) (Adapted from Nataraja et al. [7])

Nataraja et al. [7] derived the stress-strain relationship for steel-fiber reinforced concrete under the compression test. They used the round crimped fibers with V_f of 0.5%, 0.75%, and 1.0% and two aspect ratios of 55 and 82. After preparing the specimen, the test was conducted under the ASTM C-39 standard in 3000 kN compression testing machine. A growth in the toughness and compressive strength of the composite was detected after the addition of crimped steel fiber to steel. Figure 2.2 show the changes in the stress-strain curve after the accumulation of the steel fibers.

Silva et al. [8] investigated the cracking mechanism in sisal fiber reinforced composites. The fiber was manufactured by cast hand lay-up process. The matrix was prepared with fractional cement unused by metakaolin and calcined leftover crumpled clay brick for improving the durability. The crack development was examined by means of a high-resolution image capturing procedure. The composite exhibited extraordinary modulus in the linear-elastic zone with elastic modulus between 30-34 GPa under flexural and direct tension test. The extraordinary energy engagement capacity of the developed composite system was

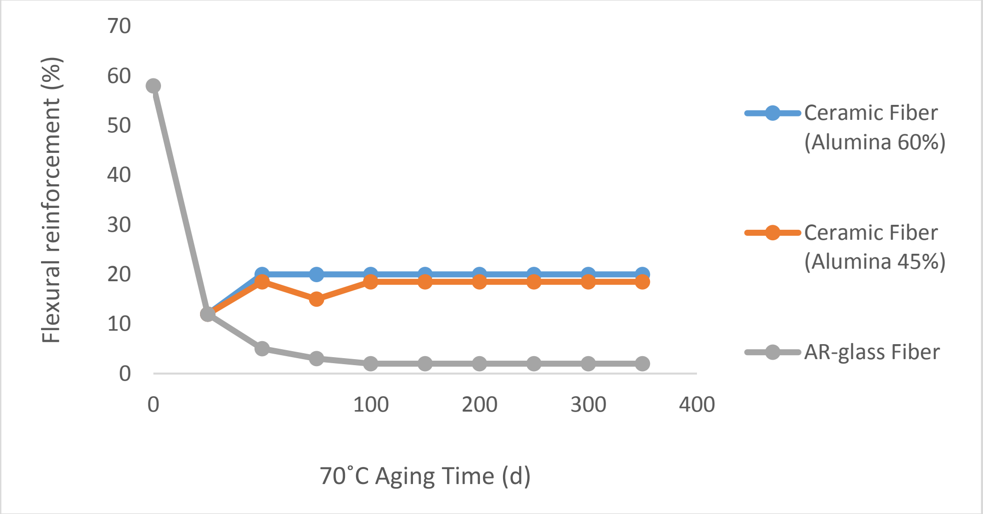

Figure 2.3. Change of the flexural reinforcement of fiber reinforced mortar with aging time (Adapted from Ma et al. [9])

replicated in high toughness values underneath tension and bending loads of approximately 45 and 22 kJ/m^2. Microstructural analysis designated that the sisal fibers remained capable to bridge and arrest the cracks within the tensile region of reaction leading to a high mechanical performance and energy absorption capacity.

Ma et al. [9] predicted the mechanical characteristics and strength of the ceramic fiber reinforced cement composites by the wet-hot accelerating method. The outcomes demonstrated that the flexural strength of concrete improved by accumulation of ceramic fibers, but the consequence of the flexural strength depended on numerous factors, including fiber length, fiber content and kinds of matrices, the durability of ceramic fiber in ordinary cement tested and was ample superior than that of alkali-resistant (AR) glass fiber. From Figure 2.3, it could be observed that the impact of AR fiber on the flexural reinforcement of concrete was much better at an early age but it decreased sharply with aging time and was almost negligible at 60 days.

Nagy et al. [10] calculated the thermal characteristics such as thermal conductivity, density and specific heat capacity of steel, plastic and glass fiber reinforced concrete. They used Humix 50 cold formed steel fibers, Concrix ES bi-component polyolefin macro plastic fibers and Aveeglass type 12 mm HP E-glass fibers for preparing the samples. Increasing the volume of steel fibers was found to have negligible effect on the thermal conductivity of the fiber reinforced concretes, since the changes in thermal conductivity were principally correlated to the density which was correlated to the total porosity. Fibers could bring air in the sample, and rise the total porosity, subsequent in lower density and thermal conductivity. This result was verified by measurement, simulation and correlation method.

Toutanji et al. [11] studied the behavior of carbon fiber reinforced composites in direct tension. For this the PAN based carbon fiber was taken and specimens were prepared by chopping fiber in 10mm length and dispersing them in cement matrix. The specimen was of 16 mm and 120 mm diameter and length respectively, and the fiber content was 0, 1, 1.5, 2 and 3% by volume. The test was conducted by cementitious composite axial tensile technique (CCATT). It was detected that the direct tensile property increased by 56% by the addition of PAN-based carbon fiber. From Figures 2.4 and 2.5, it could be inferred that the increase in tensile strength with the expansion of carbon fiber was nonlinear.

Mertol et al. [12] studied the flexural performance of light and deeply reinforced steel fiber concrete beam. For this, 20 different specimens of size 180 × 250 × 3500 mm were made and the reinforcement was done in the ratio of 0.2% to 2.5%. Four point bending test was performed on the beam specimen. For this the rollers were positioned on mutual sides of the beam and 300 kN load was applied by a hydraulic jack. The load-deflection behavior was examined and the ultimate load and service stiffness of beam were found to increment with the expansion of steel fibers.

Konsta-Gdoutos et al. [13] prepared the samples of CNT (short and long two types of MWCNTs) reinforced cement composites of size 20×20×80 mm. The fracture test was conducted using the three point bending test with the closed-loop servohydraulic testing machine. It was observed that the dispersion of small amount of MWCNTs, 0.025 and 0.08 wt.% of cement, increased the strength and stiffness of cementitious matrix and also the nanoporosity of the material was reduced with the addition of MWCNTs.

Mansur et al. [14] examined the mechanical characteristics of jute fiber reinforced cement composites. Different types of matrices were prepared in which cement-sand were present in the ratio of 1:0, 1:1 and 1:2. The length and V_f of fibers were 12, 18, 25, 38 mm and 1, 2, 3, 4% respectively. Three different types of specimen were prepared for tensile (150×38×13mm), compressive (50×100 mm cylindrical) and flexural strength (380×102×13mm) testing. The avery machine was used for compression and Instron machine for tensile and flexural testing. After the test, 97% increase in tensile strength and

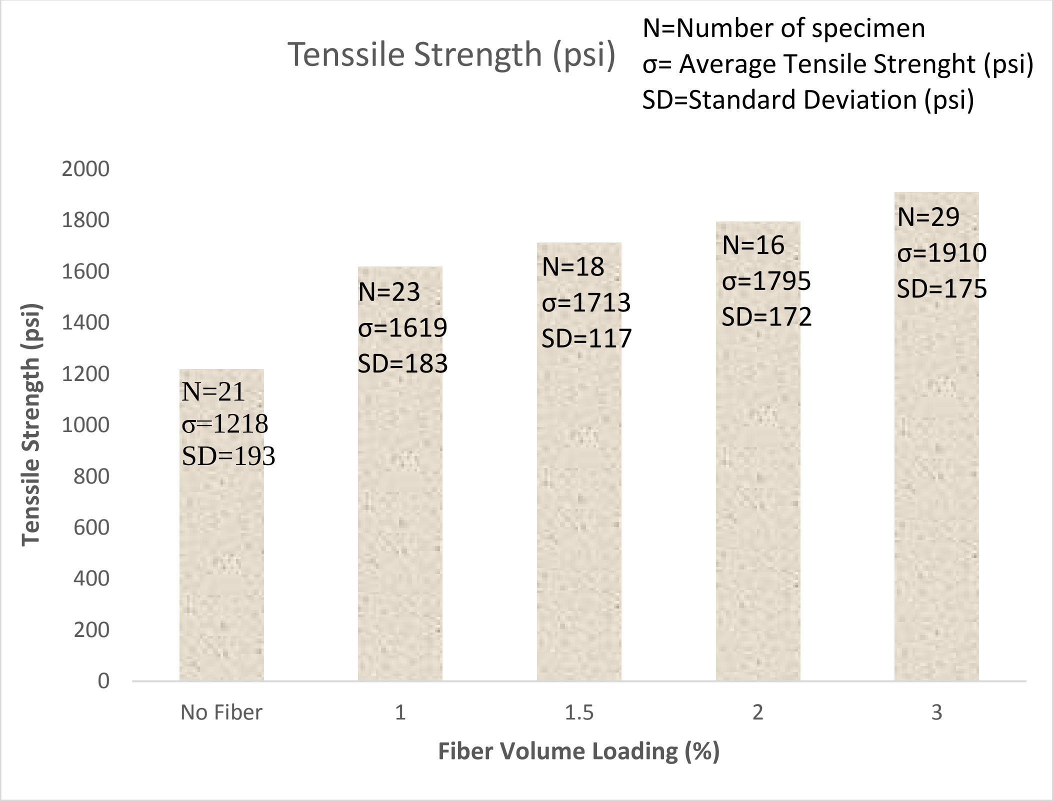

Figure 2.4. Effect of PAN-based carbon fibers on the tensile strength of cementitious composites (Adapted from Toutanji et al. [11])

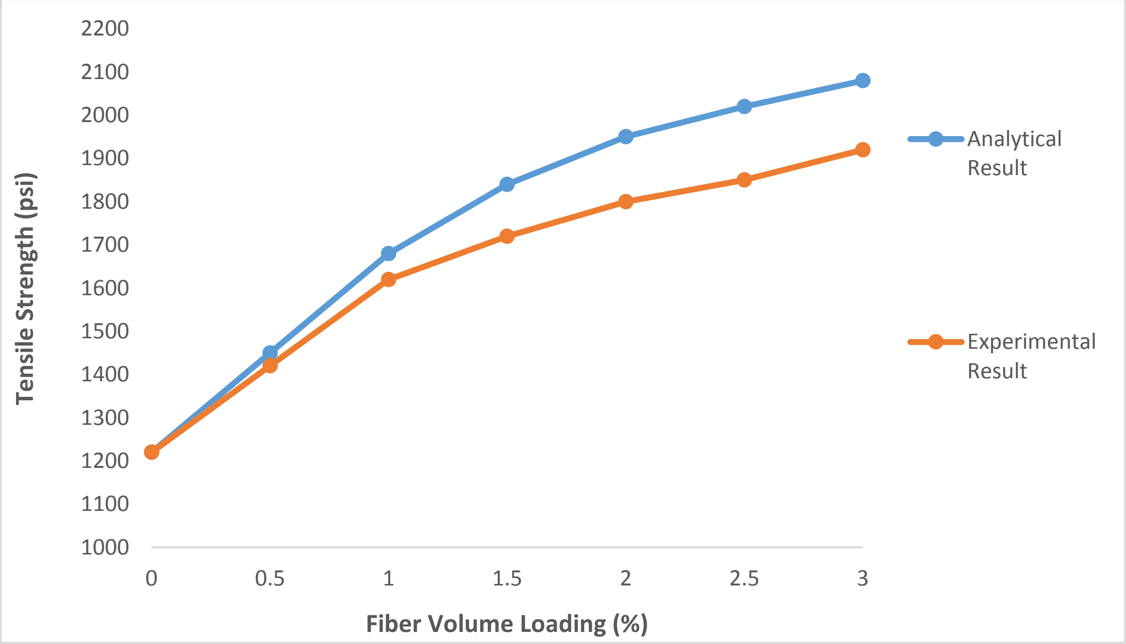

Figure 2.5. Tensile strength data for CFRC specimens with different fiber loading (Adapted from Toutanji et al. [11]).

60% increase in flexural strength was observed along with very little effect on the compressive strength.

Mansur et al. [15] prepared three different types of specimens of bamboo-mesh reinforced cement composite for conducting the tensile, flexural and impact strength tests. The test were performed on the Instron testing machine. The results showed that the reinforcement of bamboo mesh provides substantial ductility and durability to the cement and improved its tensile, flexural and impact strengths.

Kittl et al. [16] found the properties of copper fiber reinforced cement composite. For this, short ductile copper fiber was used. They prepared the specimens of size 55×20×4 mm with fiber content varying from 0-9% by weight. Portland cement having a specific surface of 305 m^2/kg was used as matrix. Three-point bending tests were conducted and it was witnessed that reinforcement of copper fiber improved the strength up to 50% in the moist state and up to 20% in the dry state.

Kim et al. [17] investigated the heat dependent mechanical properties of cement-CNT composites. Silica fume and poly-carboxylic acid centred superplasticizer was used as a dispersion agent for CNTs. Six different specimens of CNT-cement composites of size 50×50×50 mm were prepared in which the CNT was kept in the ratio 0.1 to 2.0 wt.% of cement. Silica fume and superplasticizer were added in 10.0 wt.% and 2.0 wt.% respectively. The electrical resistivity of the specimen was measured by digital multimeter by using 4-probe method. DC power supply of 3-20V was attached to the specimen for calculating the self-heating property of the specimen and K-type thermocouple was attached to the specimen for recording the surface temperature. The test for compressive strength was conducted according to ASTM C 109 on UTM at 3000 kN capacity. It was found that the heat generation capacity of the specimen was developed with a growth in the quantity of CNT and composites having CNT less than 0.6 wt% was suitable as a heating element. From Figure 2.6, it could be seen that the regularly heated CNT-0.3 and CNT-0.6 samples displayed a rise in the compressive strength levels in association to the non-heated CNT-0.3 and CNT-0.6 samples.

Siddique et al. [18] described the different properties of CNTs such as, electrical, mechanical, thermal and kinetic properties. The effect of CNTs on the properties of cement mortar was studied. It was concluded that the addition of CNTs along with fly ash in

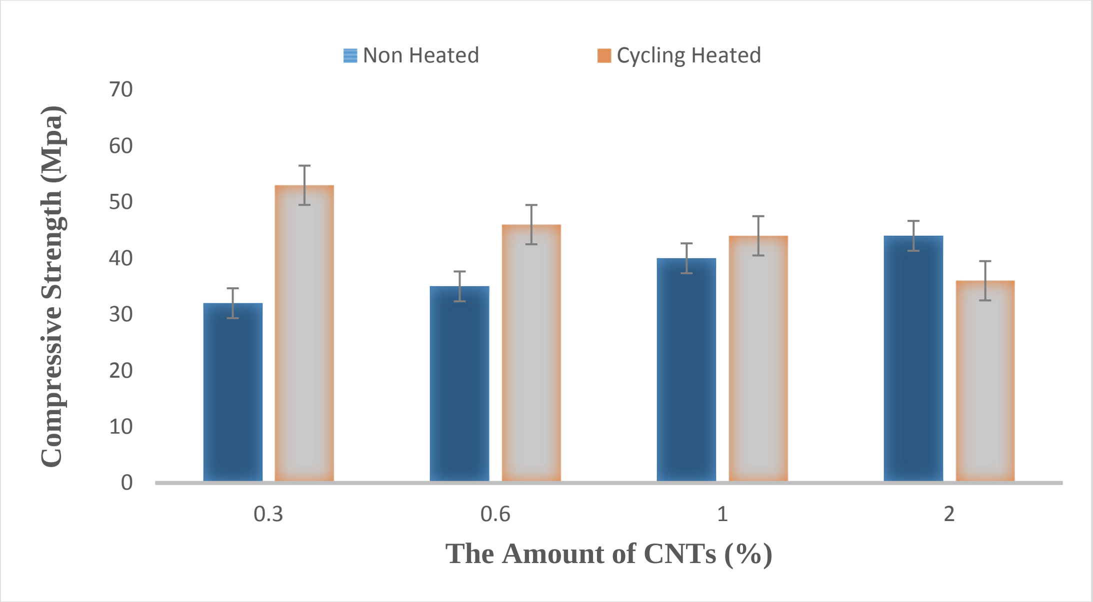

Figure 2.6. Compressive strength of CNT-embedded cementitious composite after cyclic self-heating (Adapted from Kim et al. [17]).

Cement, prompted topping off of the hydration products for example, calcium silicate hydrates and ettringite. The micrographs demonstrated great cooperation amongst CNT and the fly ash cement matrix. CNT gone about as a filler bringing about a denser microstructure and higher quality when contrasted with the reference fly ash mix without CNTs. The compressive strength of fly ash cement composite was found to increment with increment in CNT content and the most astounding quality was accomplished with CNT substance of 1% by weight.

Camacho et al. [19] predicted the mechanical characteristics and toughness of CNT-cement composites. Portland cement type EN 197-1 CEM I 52.5 R and multi-wall carbon nanotubes (MWCNT, BAYTUBES C 70P) were used for making the specimens in which the CNT percentage varied from 0-0.5% of cement mass and the plasticizer percentage from 0-2.2% of the cement mass. Prismatic specimens of size 4×4×16 cm^3 were prepared in relation to European standard UNE EN 196-1 for mechanical testing. Another specimen of size 80×55×20 mm^3 was prepared for the corrosion rate test. The accumulation of CNTs to Portland cement mortars was found to have negligible effect on the bending strength.

Ghaharpour et al. [20] studied the deposition of CNTs on cement by chemical vapor deposition (CVD) process and the effect of synthesis. Portland sulphate-resistant (SR) cement, iron nitrate salt ($Fe(NO_3)_3.9H_2O$) and ethanol were used as ingredient materials. For preparing the specimens, wet impregnation technique was used for synthesizing iron oxide nanoparticles and increasing its content in cement. For CNTs, CVD method was used in which acetylene was chosen as the main carbon supply due to its low decomposition temperature and Ar was used as carrier gas. After preparing the specimens, the testing was performed on MIRA3-TESCAN FE-SEM with a voltage of 5 kV equipped with an EDS analyzer operating at a voltage of 5 kV. The results showed that the interaction of iron oxide and cement at lower temperatures (700-800 ˚C) was strong enough to prevent agglomeration of MWCNTs. TEM investigations showed that the majority of carbon nanostructures on cement particles were MWCNTs, some of which had a bamboo structure. The average diameter and yield of CNTs was found to increase with increase in the reaction temperature, synthesis time and catalysts contents.

Tyson et al. [21] prepared five different specimens of 0%, 0.1% and 0.2% CNTs, 0.1% and 0.2% CNFs. Flexural tests were performed on these specimens. Figure 2.7 shows that the accumulation of CNFs and CNTs improved the ultimate dislocation up to 150% above plain cement paste, which was found to be very useful for infrastructure uses in which greater ductility and strain capacity to failure were desired. The average peak stress was increased to 82% for CNFs at 7 days and in other cases a decrease in strength was observed, which has been shown in Figure 2.8.

Li et al. [22] investigated the pressure sensitivity of CNT-cement composites. Two different types of specimen, one in which CNTs were treated with a mixture of H_2SO_4 and HNO_3 (SPCNTs), the other consisted of untreated CNTs (PCNTs). Electrical resistance test was conducted using the four probe method. From Figure 2.9 it could be seen that the electrical conductivity and field discharge of CNTs decreased because of the curing whereas the flexural strength of cement-based composites was found to improve due to the accumulation of cured CNTs as shown in Table 2.2.

Luo et al. [23] prepared he MWCNT reinforced ordinary Portland cement and determined the mechanical properties using the three point bending method (ASTM 399). It was observed that the accumulation of CNTs improved the flexural strength and the stress-intensity factor of the nanocomposites. The maximum increase in stress was near to 45%

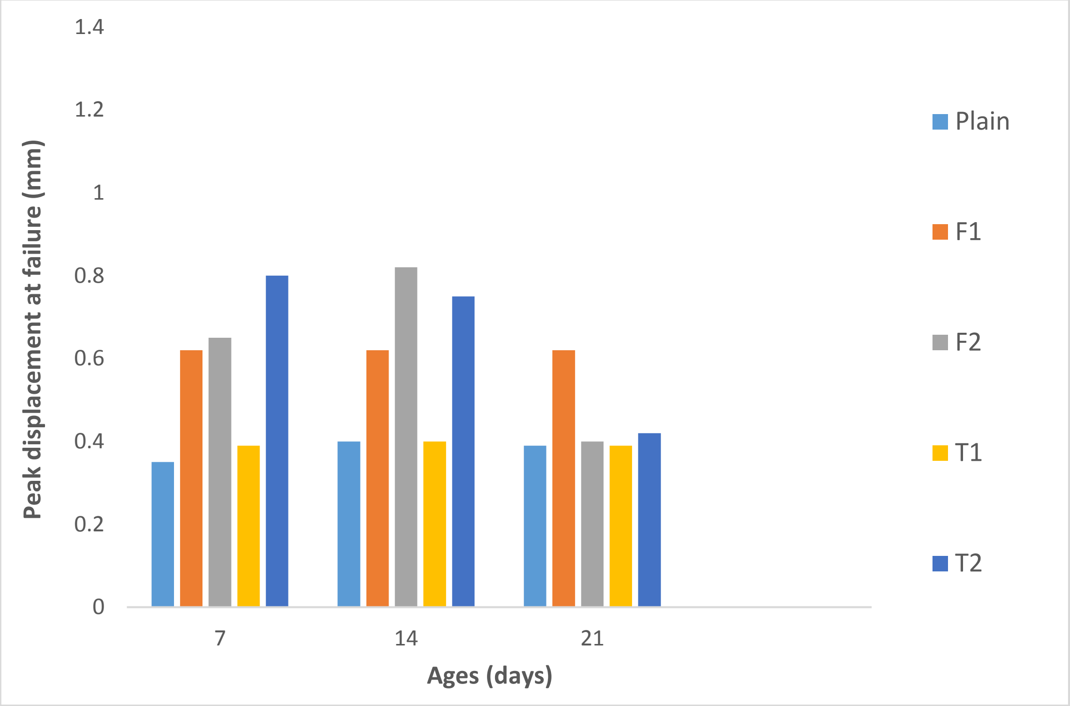

Figure 2.7. Effect of CNFs and MWCNTs on the strain capacity of cement paste (Adapted from Tyson et al. [21]).

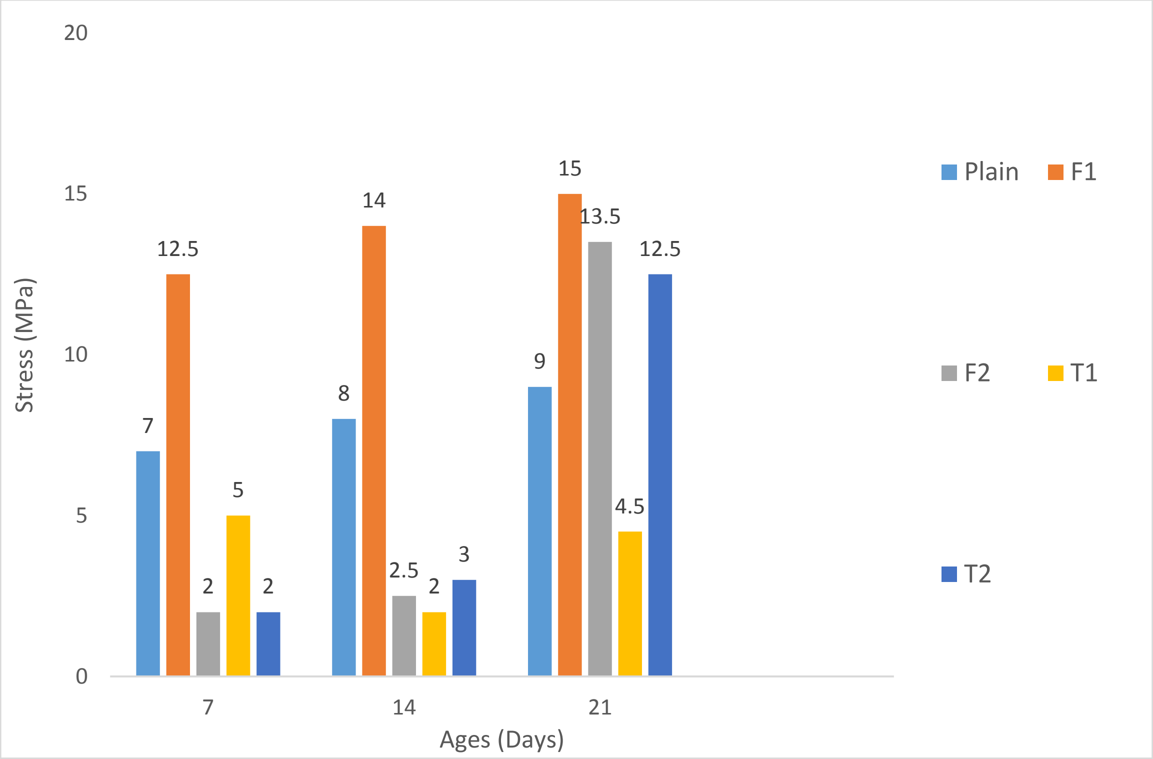

Figure 2.8. Effect of the CNFs and MWCNTs on the ultimate strength of cement paste (Adapted from Tyson et al. [21]).

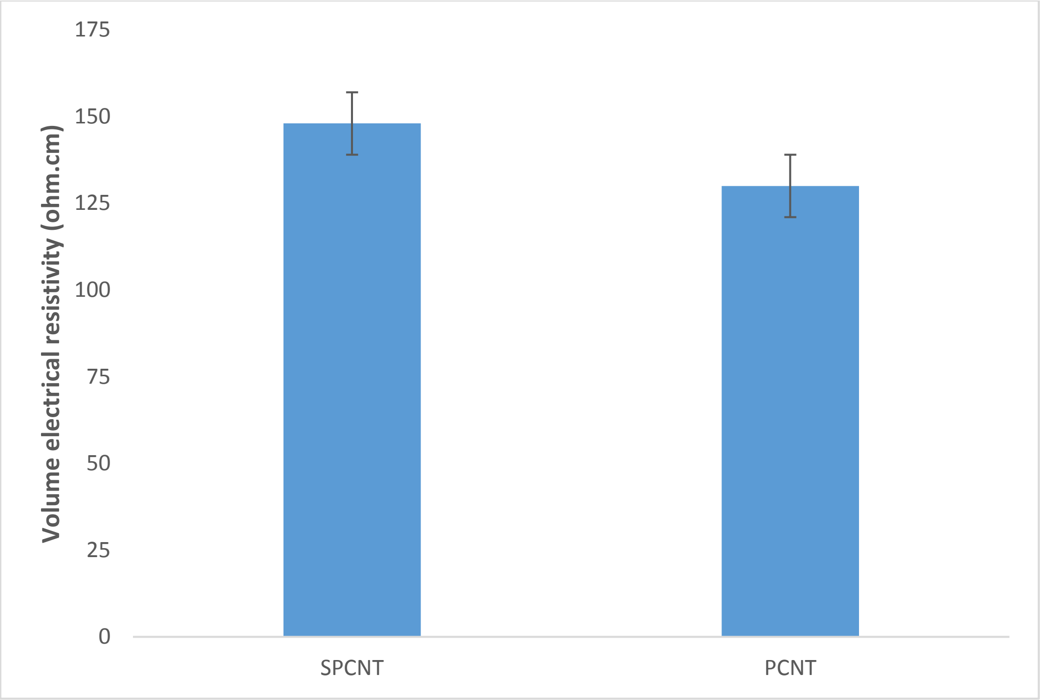

Figure 2.9. Volume electrical resistivity of different mixes after 28 days curing (Adapted from Li et al. [22]).

Table 2.2. Strength of different cement paste after 28 days curing.

Mix	Compressive strength (MPa)	Flexural strength (MPa)
PCNT	69.41±3.3%	9.56±3.6%
SPCNT	72.13±4.2%	9.97±2.7%

as for the straightforward the simple cement paste specimen. It was watched that the expansion in quality prompted unrivaled fiber connecting ability of scattered nanotubes upon the break start while bowing.

Li et al. [24] prepared cementitious composites by adding MWCNTs (treated with a mixture of HNO_3 and H_2SO_4) in cement. The mechanical properties of the recently framed composite were examined and the outcomes demonstrated that the treated nanotubes enhance the flexural quality, compressive quality and failure strain of cement matrix composites. The porosity and pore estimate circulation of the composite was resolved utilizing Mercury Intrusion Porosimeter It was watched that the expansion of CNTs brought about fining the pore estimate circulation and abatement in porosity. It was shown that CNTs act as bridges across cracks and voids.

Hu et al. [25] studied the effect of compressive strength and fracture toughness in CNT reinforced cement composite. MWCNTs, MWCNTs-COOH and Portland cement were used for making a cube of 70 mm size. Testing was performed using the three point bending method. The results showed that MWCNTs-COOH improved the breakage and compression properties of the composite compared to MWCNTs. With addition of 0.1 wt% of MWCNTs, the fracture energy and fracture toughness of the test specimen were found to increase by 26.2% and 11.4%, respectively. Whereas on the addition of 0.1 wt% MWCNTs-COOH, the fracture energy and fracture toughness of test specimen improved by 42.9% and 19.2%, respectively.

Al-Rub et al. [26] concentrated the impact of reinforcing the cement using different types of CNTs. Two different types of CNTs, short and long MWCNTs, having different aspect ratio were used as reinforcements. The results showed that the flexural quality of short MWCNT (with 0.2 wt.%) reinforced cement increased by 269% whereas the long MWCNT (with 0.1 wt.%) reinforced cement showed an increase of 65%, compared to the simple cement specimen at 28 days as shown in Figure 2.10. The ductility increased to 86% for short MWCNTs (with 0.1 wt.%) and 81% for long MWCNTs (with 0.2 wt.%), at 28 days. Table 3 summarizes some of the main studies based on fiber reinforced cement composites.

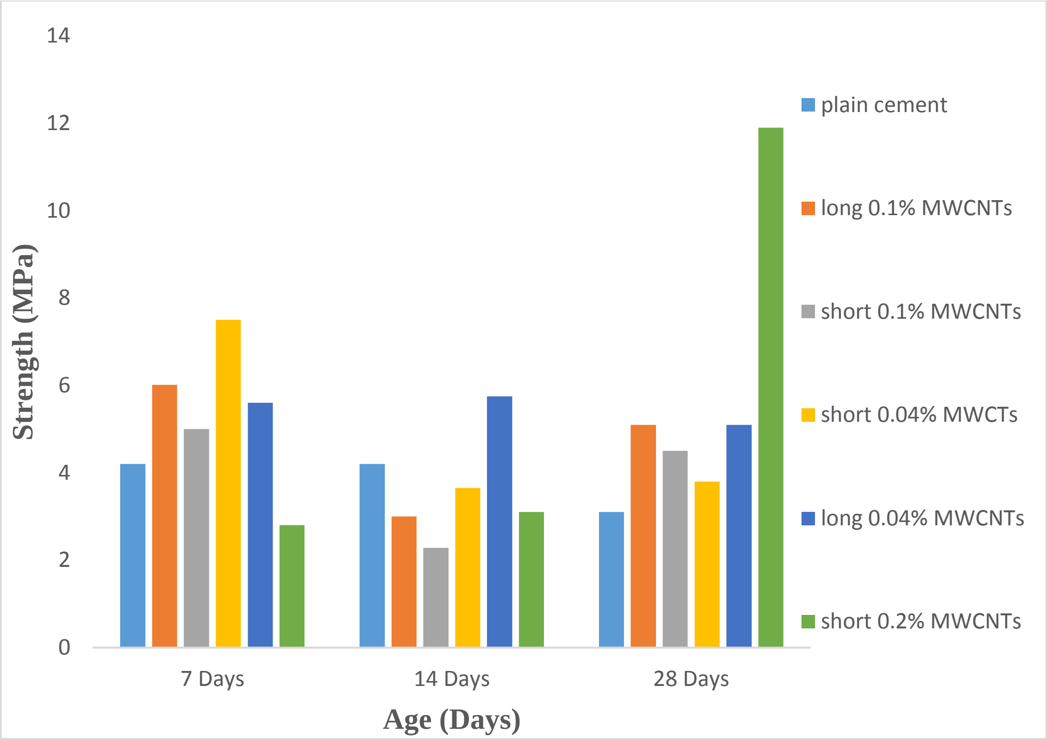

Figure 2.10. Average flexural strength results for different MWCNTs composite specimens (Adapted from Abu Al-Rub et al. [26]).

Table 2.3. Summary of important studies based on fiber reinforced cement composites.

References	**Approach used**	**Material used**	**Properties studies**	**Remark**
Kittl [16]	Experimental	Copper fiber and cement	Strength	Effect on mechanical properties
Li [24]	Experimental	HNO_3 and H_2SO_4 treated CNTs and cement	Flexural strength, compression strength, and failure strain	Effect on mechanical properties
Hu [25]	Experimental	MWCNTs, MWCNTs-COOH, and cement	Fracture and compression strength	Effect on mechanical properties
Abu Al-Rub [26]	Experimental	Long and Short MWCNTs and Portland cement	Flexural strength and strain capacity	Effect on mechanical properties

2.2 Objective of the study

The available literature has showed that several studies have been performed on fiber based nanocomposites mainly consist of carbon nanotubes as reinforcement and also various micro level composites having reinforcements like steel fiber, jute fiber, copper fiber, etc. The objective of the present study is to “investigation of mechanical properties of multi walled carbon nanotube reinforced cementitious nanocomposite”. The important topics to be taken up during the planned work are as follows

a. Manufacturing of nanocomposites with different compositions to obtain the most favourable composition (weight ratio) of multi walled carbon nanotube, cement, and superplasticizer resulting in maximum improvement in desired properties.

b. Assessment of mechanical properties and classification of nanocomposites to understand the correlation between structure and resulting properties.

Chapter 3

Materials and Methods

3.1 Introduction

This chapter is related to the materials and methods which are used in the experimental work. In this section we have briefly discussed about the materials which are used in this experimental study and the process which are used for making the nano-composites.

3.2 Materials

The materials which have been used in this experimental study are listed as follows:

a) Cement and superplasticizer
b) Carbon nanotube
c) water

3.2.1 Cement and Superplasticizer

The cement which is used in this experimental work is Portland Pozzolana cement (PPC), which shown in Figure 3.1. The commercial water reducing admixture Polycarboxylate, used as a superplasticizer.

3.2.2 Carbon Nanotube

The CNTs, used in this experiment are Multi-Walled Carbon Nanotubes (MWCNTs), provided by Nano Partech, which shown in Figure 3.2. The specification and physical properties of the MWCNTs are given in table 3.1.

3.2.3 Water-

Deionized water was used as the mixing water for all the groups, which was prepared by a RO water filtering methods and the pH value ranges from 5.5-6.0.

Figure 3.1 Sample of Portland cement

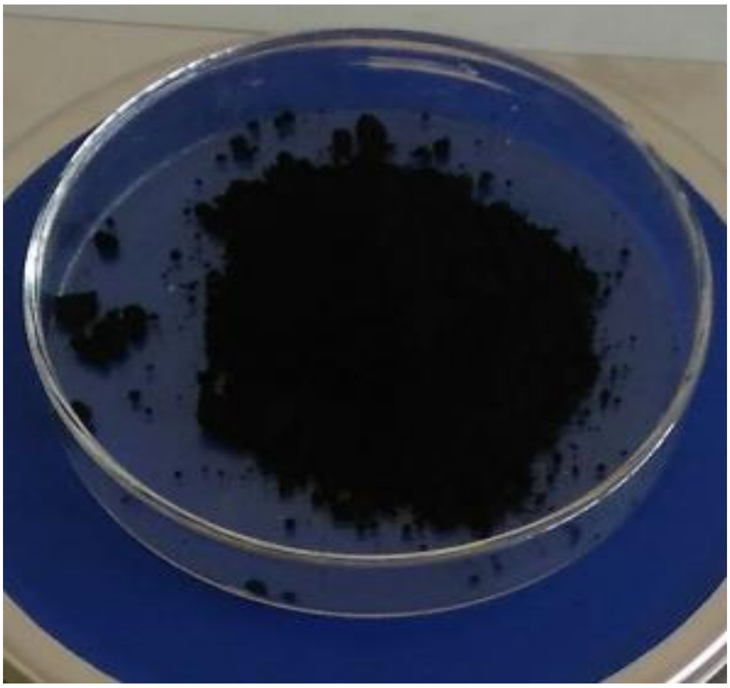

Figure 3.2 Sample of Multi-Walled Carbon Nanotube in plate.

Table 3.1 Physical properties of MWCNTs (Provided by Nanopar Tech)

Materials	Multi-Walled Carbon Nanotube
Diameter	10-20nm
Length	4-8µm
Purity	>99%
Average interlayer distance	0.34nm
Real density	1-2g/cm^3

3.3 Methodology

Three different groups of samples of MWCNTs reinforced cement composite at three different concentration of MWCNTs are 0, 0.2 and 0.3 wt% of the cement. All specimen had a water/cement ratio of 0.4 and superplasticizer of 0.1% by cement weight. Five samples are made for each group for calculation of the mechanical characteristics and testing of samples will be done at 14 and 21 days.

3.3.1 Preparation of Multi-walled Carbon Nanotubes Solution

The process of making of the nanocomposite cement mixture begins by weighing the required measure of MWCNTs by utilizing a precise weighing machine, shown in Figure 3.3. The required amount of the mixing water and surfactant and included with MWCNTs, into a water beaker. The mixing procedure was achieved by using an ultrasonic mixer from Labman scientific instrument, shown in Figure 3.4. The high frequency of the ultrasonic wave that exchanged through the water, with most extreme power that could achieved 50 watts and frequency of 40 kHz, will give the decent level of scattering of the MWCNTs fibers in water. Due to suitable ultrasonic waves and for a specific time of sonication, the high energy into the beaker will break the collections and bunches of MWCNTs by separating the Van-der Waals forces between the nano-fibers. The measure of energy and time of scattering is a basic component for accomplishing a decent scattering of MWCNTs, less energy and time couldn't ensure a decent scattering, while more energy and time would disintegrate and break the nano-fibers. The sonication time frame for diverse MWCNTs lengths fluctuated from 40 to 50 minutes.

Figure 3.3 Precise scale is used to weigh MWCNTs (courtesy NIT Jalandhar)

Figure 3.4 Ultrasonic wave mixer from Labman scientific instrument used for MWCNT mixing (courtesy NIT Jalandhar)

3.3.2 Mixing of Portland cement and Multi-walled Carbon Nanotube Solution

The required quantity of the sonicated MWCNTs solution was then filled in mixer jug of a different speed planetary kitchen mixer, shown in Figure 3.5. The mixer was begun with just MWCNTs solution, and after that cement powder was added continuously to the solution. Later on addition of cement powder, the blending time was an aggregate of 8 minutes.

3.3.3 Preparation of Nanocomposite

Instantly after blending is completed, the mixer of cement composite is transferred to the molds which was made by paper cardboard and covered by sheet of plastics, of cross-section 127×12.7×5 mm, as shown in Figure 3.6, which has been engaged on top of a vibrating table. The vibration throughout the moulding the cement paste, will help to easily fill the shape and decreasing the air voids and entangled air bubbles. This is crucial to get strong uniform cross-section of the material all through the length of mold, without having deficiencies because of air voids that would altogether influence the mechanical property of the composite.

The prepared models of cement composite were then permitted to air cured in the molds at room temperature for 7 days before removing from the molds, and after that it will removed from the molds.

3.4 Preparation of the Cement Composite Group

Three distinctive groups of the MWCNTs reinforced cement composite were made in the duration of this experiment. The first group was made of only cement (for reference) and other two groups includes MWCNTs as a reinforcement by changed mass of MWCNTs by weight of cement (0.2% and 0.3%). All samples with cement (reference) samples have a water/cement ratio of 0.4. An outline of the mixer preparation is shown in table 3.2. Five samples were made of each groups for every testing on 14 days and 21 days.

Figure 3.5 Different speed planetary kitchen mixer for preparing Nanocomposites

Figure 3.6 Image of mould.

Table 3.2 Outline of mixer preparation

Test Samples	**Superplasticizer (wt% of cement)**	**MWCNTs (wt% of cement)**	**Ultrasonication time (min)**
Plain Cement	0.1	--	--
MWCNT 0.2%	0.1	0.2	50
MWCNT 0.3%	0.1	0.3	50

Chapter 4
Results and Discussion

4.1 Experiments and Results

Three different testing were performed "flexural strength, strain to failure and modulus of elasticity" to check the mechanical properties of the nanocomposite samples. The three point bending machine (Dak Series 7200 Model No UTB9163) was used for testing, which was provided by CIPET Amritsar, shown in figure 4.1. The load cell applied during the test was 1 kN, having least count of 0.01 N and all testing was executed at a speed of 1 mm/Min.

Flexural Strength

Flexural strength may be defined as the stress, which stored in the body before it deforms plastically in fracture test. It can be calculated by three point bending test as shown in figure 4.2 in which the sample is bent up to the plastic deformation. This signifies the maximum stress gained by the sample at the time of deformation. Which is represented by σ. For the rectangular sample the flexural strength will be calculated by equation 4.1.

$$\sigma = \frac{3FL}{2bd^2} \tag{4.1}$$

Where F is the load at fracture point (N), L is the gap between supporting span, b is the girth of sample and d is the depth of sample. By using this formula the flexural strength of the fracture material will be calculated.

Figure 4.1 Three point bending machine used for testing (courtesy CIPET Amritsar)

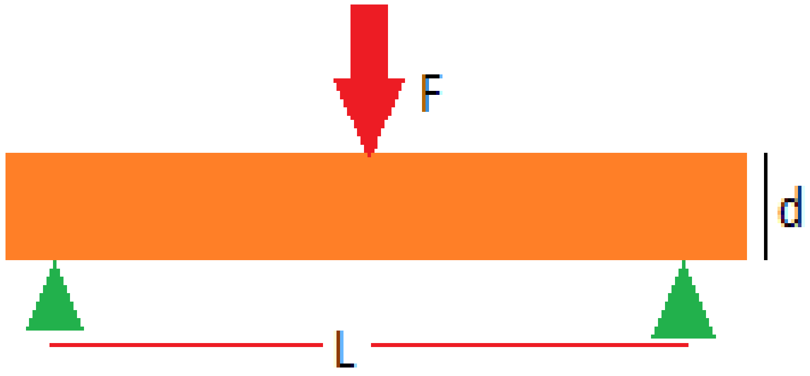

Figure 4.2 three point bending test methods

Figure 4.3 Before loading condition in 3 point bending test

Figure 4.4 Sample break due to applied load in 3 point bending test

Five samples of every groups have been tested for flexural strength on 14 and 21 days of sample preparation. Figure 4.3 and 4.4 shows, before and after the loading condition of nanocomposite samples in three point bending test. The average value of flexural strength for samples of each groups is listed in the table 4.1.

At the age of 14 days, the MWCNTs reinforced cement composite indicates a rise in the flexural strength in reference with the plain cement sample. The most elevated change in the flexural strength was found in the 0.2% MWCNTs samples with an expansion of 21.94% in reference with the plain cement sample. Whereas minute difference was found in the 0.3% MWCNTs with an expansion of 14.47% in the reference with the plain cement sample.

At 21 days, all the nanocomposite samples re-claimed their strength more than the 14 days. Though, 0.3% MWCNTs indicates the highest increment in the flexural strength, about 97.82% where as 0.2% MWCNTs showed, 34.64% increase in the flexural strength with reference to the plain cement samples.

Table 4.1 Average value of flexural strength

	Flexural Strength (MPa)	
	14 Days	**21 Days**
Plain Cement	6.7	7.8
MWCNTs 0.2%	8.17	10.502
MWCNTs 0.3%	7.67	15.43

For the evaluation of result, the flexural strength of different nanocomposite groups is shown in figure 4.5.

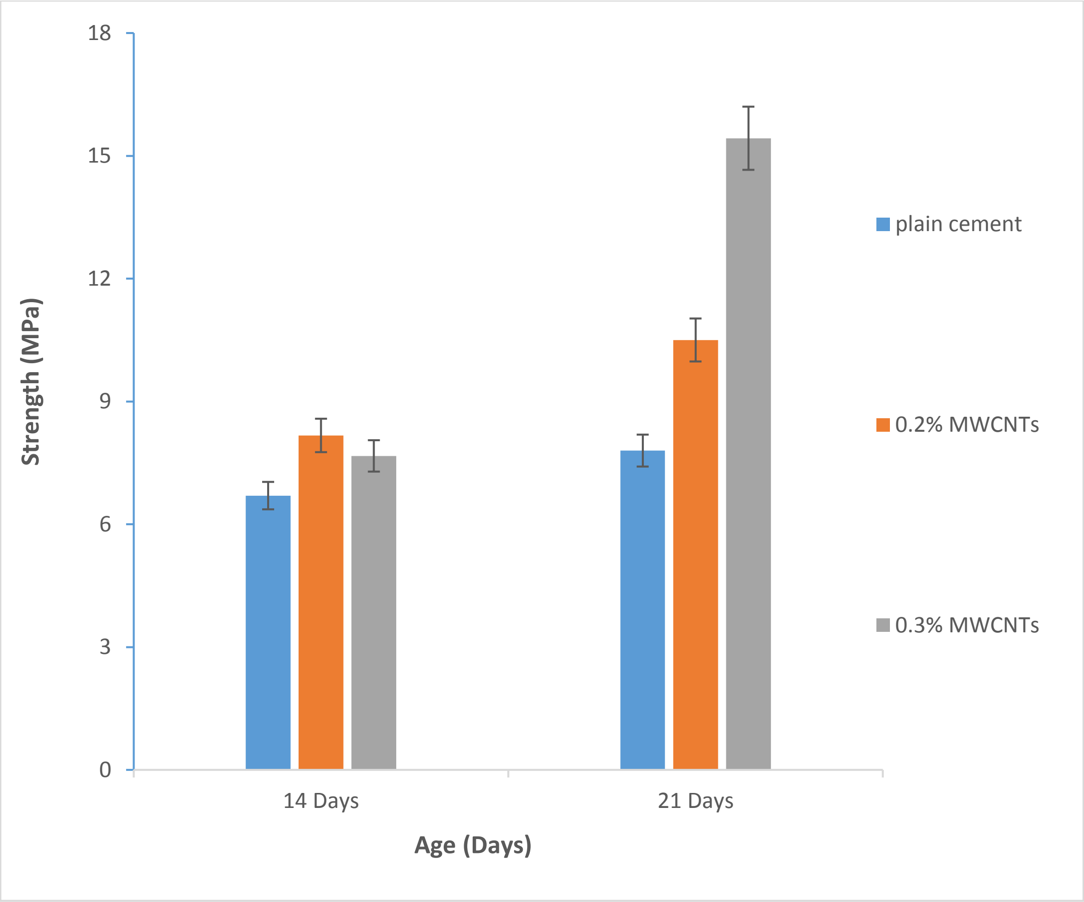

Figure 4.5 Average flexural strength of different nanocomposite groups

Strain to Failure

Strain may be defined as, it is magnitude of a deformation, equal to change in dimension of a deformed object divided by its original dimension, which is also known as ductility of the material. To perform this study, five samples of every groups have been tested for strain to failure on 14 and 21 days of sample preparation. The average value of strain to failure for samples of each groups is listed in the table 4.2

Table 4.2 Average value of strain to failure.

	Strain to Failure (%)	
	14 Days	**21 Days**
Cement	0.130	0.250
MWCNTs 0.2%	0.233	0.303
MWCNTs 0.3%	0.170	0.264

For the evaluation of result, strain to failure of different nanocomposite groups is shown in figure 4.6.

MWCNTs reinforced cement composite indicates a rise in the strain to failure with reference to the plain cement samples at 14 days. The eminent change in the strain to failure was found in the 0.2% MWCNTs and 0.3% MWCNTs samples with an expansion of 79.23% and 30.76% respectively in reference with the plain cement sample. At 21 days, the strain to failure value of the 0.2% MWCNTs and 0.3% MWCNTs indicates a minor increase of 21.2% and 5.6% respectively with reference to the cement samples.

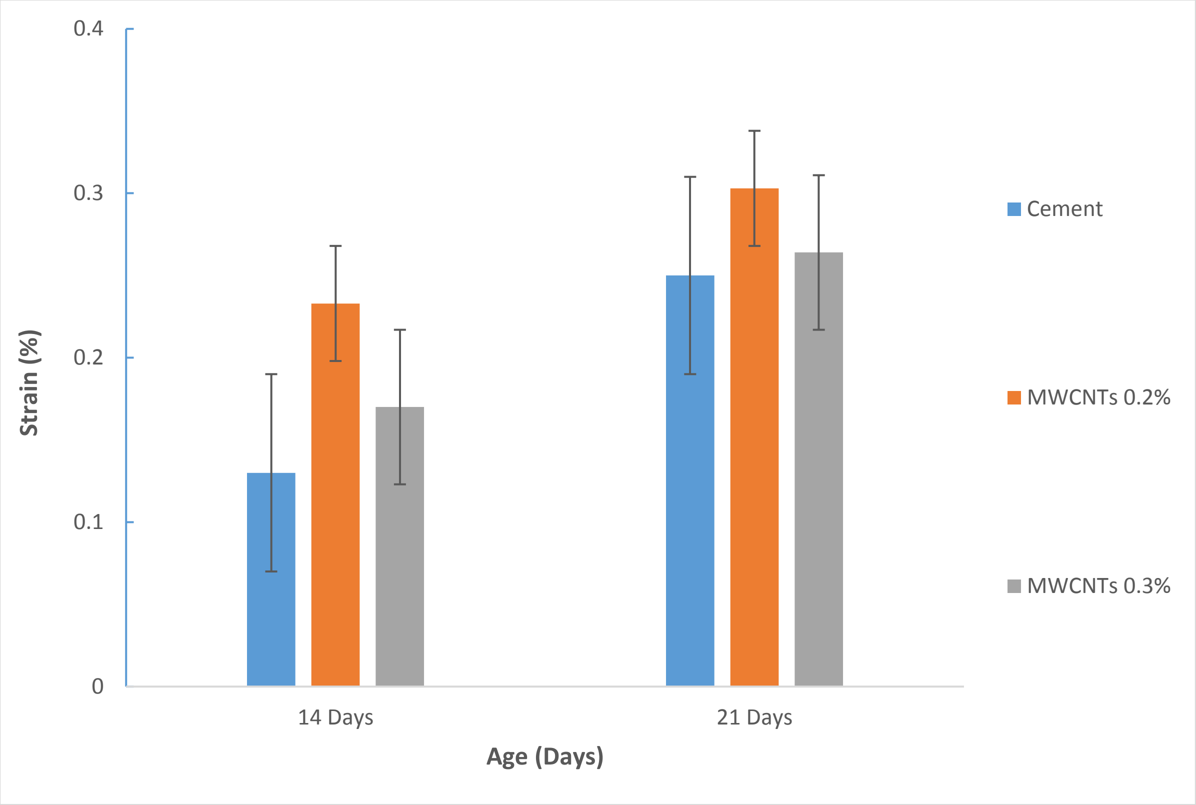

Figure 4.6 Average value of strain to failure of different nanocomposite groups

Modulus of Elasticity

Modulus of elasticity may be defined as, is a number that an object to being deform elastically when an external force is applied to it. Five samples of every groups have been tested for modulus of elasticity on 14 and 21 days of sample preparation. The value of average modulus of elasticity is shown in table 4.3.

For the evaluation of result, modulus of elasticity of different nanocomposite groups is shown in figure 4.7.

All nanocomposite samples have revealed an enlargement in modulus of elasticity. At 14 days, 0.3% MWCNTs has the maximum rise of 27.98% in modulus of elasticity in reference to cement samples. The 0.2% MWCNTs have also raised by 12.57% in modulus of elasticity with reference to the cement samples. Whereas on 21 days, 0.3% MWCNTs and 0.2% MWCNTs showed the raise of 20.91% and 16.79% respectively in the modulus of elasticity

Table 4.3 Average value of modulus of elasticity

	Modulus of Elasticity (GPa)	
	14 Days	**21 Days**
Cement	10.837	12.615
0.2% MWCNTs	12.2	14.734
0.3% MWCNTs	13.87	15.254

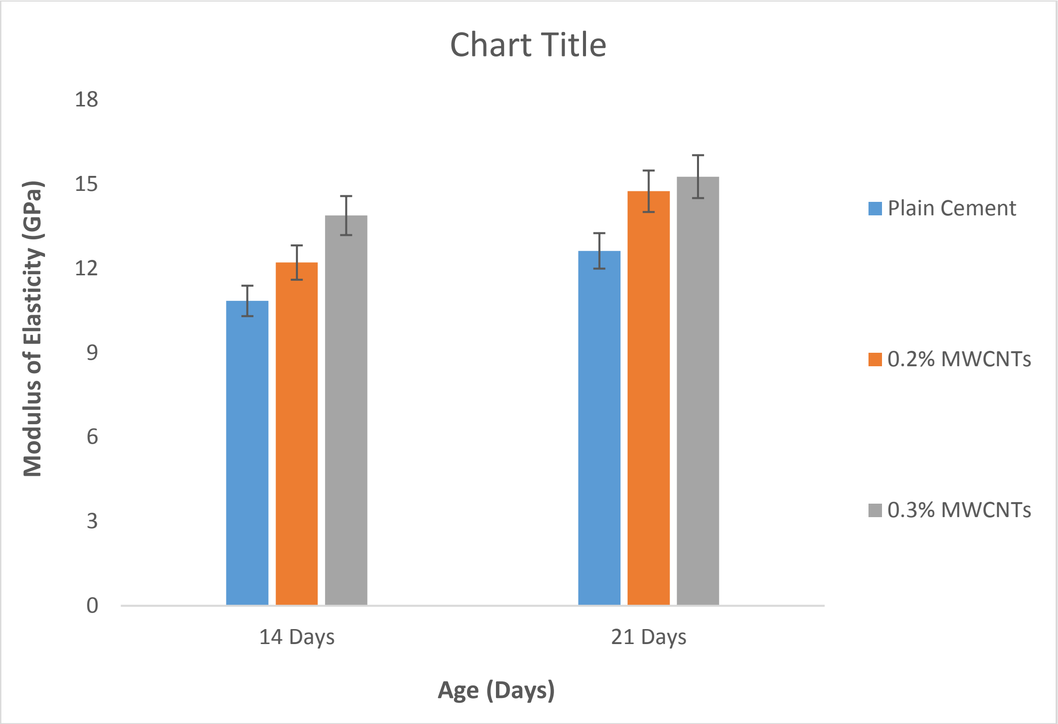

Figure 4.7 Average value of modulus of elasticity of different nanocomposite groups

4.2 Discussion

The results which are found in this experimental study, have revelled the dissimilarity in the mechanical properties between plain cement samples and MWCNTs reinforced cement samples. Figure 4.8 shows the stress strain diagram of the different samples of MWCNTs reinforced cement composite and plain cement samples at 14 days. It has been observed that, by using nano fibers in very small quantity, it may tremendously transforms the plain cement samples behavior.

For flexural strength, figure 4.5 shows that 0.3% MWCNTs have improved the flexural strength at 21 days as compared to the 0.2%MWCNTs. This behavior is equivalent to the prospects of that the nano fibers will needs high force to be pulled out from the matrix.

For strain to failure (which is also known as ductility), figure 4.6 shows that 0.2% MWCNTs nanocomposites has more strain to the 0.3% MWCNTs nanocomposites. This behavior was not as predicted, since it was supposed that the more fibers have more surface resistance, which would provide more ductility to the cement matrix. Though it may be because of the variation in dispersion of MWCNTs in cement matrix. For modulus of elasticity, figure 4.7 shows that somewhat improved perfection for the 0.3% MWCNTs nanocomposites over the 0.2% MWCNTs, at age of 21 days.

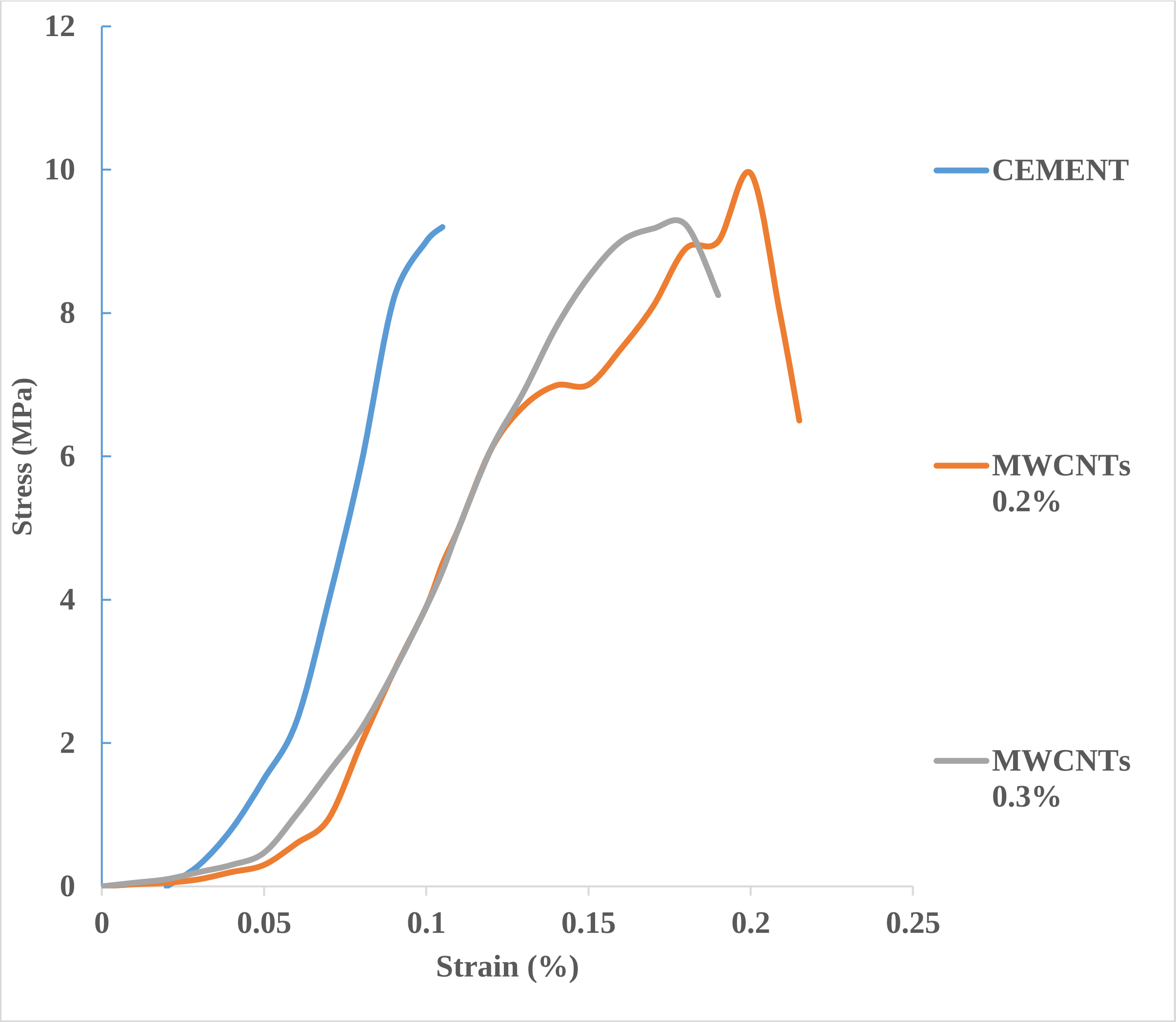

Figure 4.8 Stress strain diagram of the different samples of MWCNTs reinforced cement composite and plain cement samples at 14 days.

Chapter 5
Conclusion and Future Scope

5.1 Conclusion

In this study, ‘experimental investigation of mechanical properties of multi-walled carbon nanotube reinforced cementitious nano-composites’ have been performed. Two different wt% of MWCNTs have been used as reiforcement for the cement composites. By using 0%, 0.2% and 0.3%MWCNTs six differents groups of cement nanocomposites were casted and tested. The mechanical properties of MWCNTs cement composites were tested on three point bending test machine.

Three different mechanical properties have been tested: flexurel strength, strain to failure and modulus of elasticity, of the casted samples. From results it was observed that, 0.3% MWCNTs cement composite have more flexural strength as compare to 0.2% MWCNTs cement composite at 21 days, but 0.2% MWCNTs cement composite has more flexural strength at age of 14 days. For strain to failure, 0.2% MWCNTs cement composite has more strain to failure 0.3% MWCNTs cement composite at 14 days as well as 21 days. This will happen because of good dispersion of 0.2% MWCNTs as compare to 0.3% MWCNTs in cement composite.

5.2 Limitation

There is several limitation related to this experimental study which are listed as follows:

a. Dispersion and measurement of CNTs
b. Dispersion of CNTs in aqueous solution.
c. Measure the dispersion of CNTs in cement composite
d. Measurement of the final length of CNTs after ultrasonication process
e. Testing of small size nanocomposite samples

5.3 Future Work

The future works which are related to this experimental study are listed as follows:

a. In dispersion of CNTs in aqueous solution.
b. Dispersion amount of the CNTs in the cement composites.
c. Effect of curing methods on hydration and porosity.
d. Testing condition

Table 5.1 Average value of the mechanical properties of the cement nano-composites.

	MECHANICAL PROPERTIES					
	Flexural Strength (MPa)		**Strain to Failure (%)**		**Modulus of Elasticity (GPa)**	
	14 Days	21 Days	14 Days	21 Days	14 Days	21 Days
Plain Cement	6.7	7.8	0.13	0.25	10.837	12.615
0.2% MWCNTs	8.17	10.502	0.233	0.303	12.2	14.734
0.3% MWCNTs	7.67	15.43	0.17	0.264	13.87	15.254

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