INVESTIGATION ON THE PROPERTIES OF CEMENT MORTAR MODIFIED WITH MICRO AND NANO CARBON FIBERS

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CERTIFICATE

Certified that the thesis **"INVESTIGATION ON THE PROPERTIES OF CEMENT MORTAR MODIFIED WITH MICRO AND NANO CARBON FIBERS"** which is submitted by **Mr. Ajitpal Singh**, in partial fulfillment of the requirements for the award of the degree of Masters of Engineering in the **Department of Civil Engineering (CED),** Thapar University, Patiala, is a record of the candidate's own independent and original research work carried out by him under her supervision and guidance. The matter embodied in this thesis has not been submitted in part or full to any other University or Institute for the award of any degree.

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ABSTRACT

With the introduction of nanotechnology, the material properties of the concrete industry, allow engineer and architect the ability to use various materials in structure applications that were once impossible. Nanotechnology (Nano carbon fiber and Nano tube) creates new possibilities to improve material properties for civil construction and special focus in this area is put on Portland cement and gypsum. Carbon fiber (Microscale to Nanoscale) is typically a highly effective pozzilonic material, normally consisting of very fine vitreous particles approximately 1000 times smaller than overage cement particles. The use of nano-structured materials in mortar can add many benefits that are directly related to durability of these materials, beside the merit that it is possible to reduce the quantities of cement in the composite.

The present research work mainly focuses on study of the characteristics of mortar with different replacement levels of carbon micro fiber and carbon nano fiber of varying particle size in the mortar. Different carbon fiber replacement level incorporated in the study are 0.025%, 0.050%, 0.075% and 0.1% by mass of total cementations material content. The effects of different carbon fiber (from micro to nano particle size) on the compressive strength, split strength, flexural strength, bulk density, water absorption, weight loss of mortars upto 28 days and other properties are determined. SEM & XRD analysis is done to understand the micro-structural analysis to correlate the mechanical properties mainly compressive strength. On the basis of the results, relevant conclusions are drawn.

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CHAPTER – 1
INTRODUCTION

1.1 GENERAL

The introduction of nanotechnology represent a revolution that is allowing for the development of high performance and long lasting products and processes within on ideal context of sustainable development. It will affect almost every aspect of one's life (IWGN, 1999). In comparison to other technologies, nanotechnology is mush less well defined and well structured. Nanotechnology is on emerging field by science related to the understanding & control of matter at the nanoscale i.e. dimension between approx 1 and 100 nm. Nanotechnology encompasses nanoscale science, engineering and technology that involve imaging, measuring, modeling and manipulating matter at this length scale.

Just how small is nano? 'Nano' means 1 billionth or 10^{-9} . Therefore, 1 nm is 1-billionth of a meter:

- A sheet of paper is about 100,000 nm thick.
- A strand of human DNA is 25 nm in diameter.
- There are 25,400,00 nm in 1 inch.
- A human hair is approx 80,000 nm wide.
- On a comparative scale, if the diameter of a marble was 1 nm, then diameter of the earth would be 1 m.

There are mainly two ways to approach the nonoscale (i) "top down" approach (ii) "Bottom up" approach or molecular nanotechnology or molecular manufacturing. In which materials are engineered from atoms or molecular components through a process of assembly (Figure 1.1).

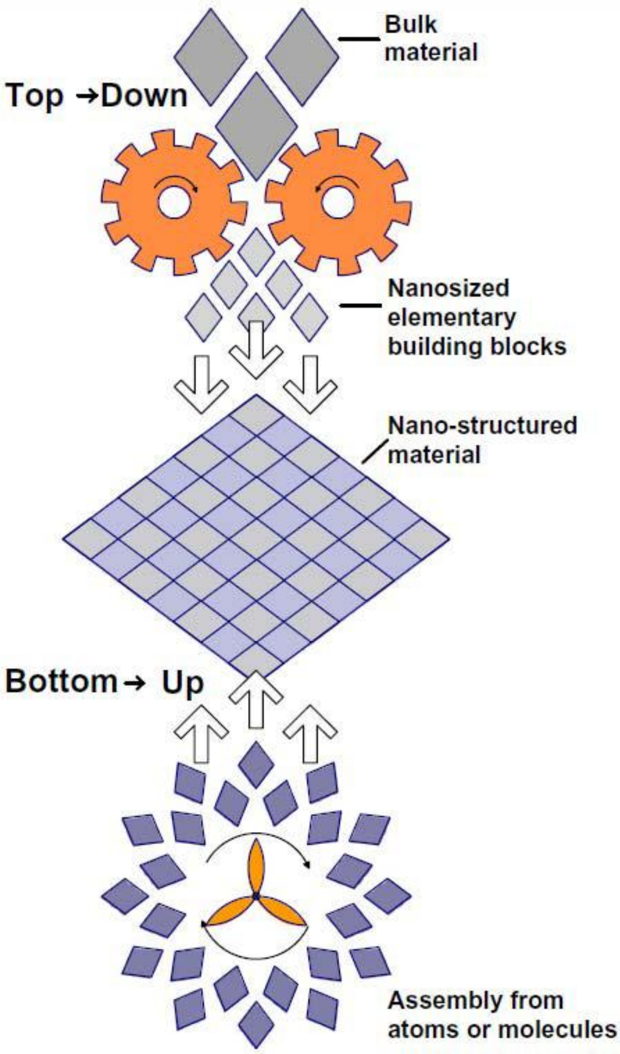


Figure 1.1: The top-down and bottom-up approaches in nanotechnology (*1*) (Sanchez and Sobolev, 2010).

Thus, the basic concept behind nano modification of materials is that of bottom up engineering, starting with engineered modification to the molecular structure with an aim to affect the bulk properties of the material.

Concrete, the most ubiquitous material in the world is a nanostructured, multiphase composite material that ages over time (Sanchez and Sobolev, 2010). It is composed of on amorphous phase, nanometer to micrometer size crystals and bound water. The properties of concrete exist in and the degradation mechanism occur across, multiple length scales (nano to micro to macro), when the properties of each scale derive from those of the next smaller scale (Figure 1.2).

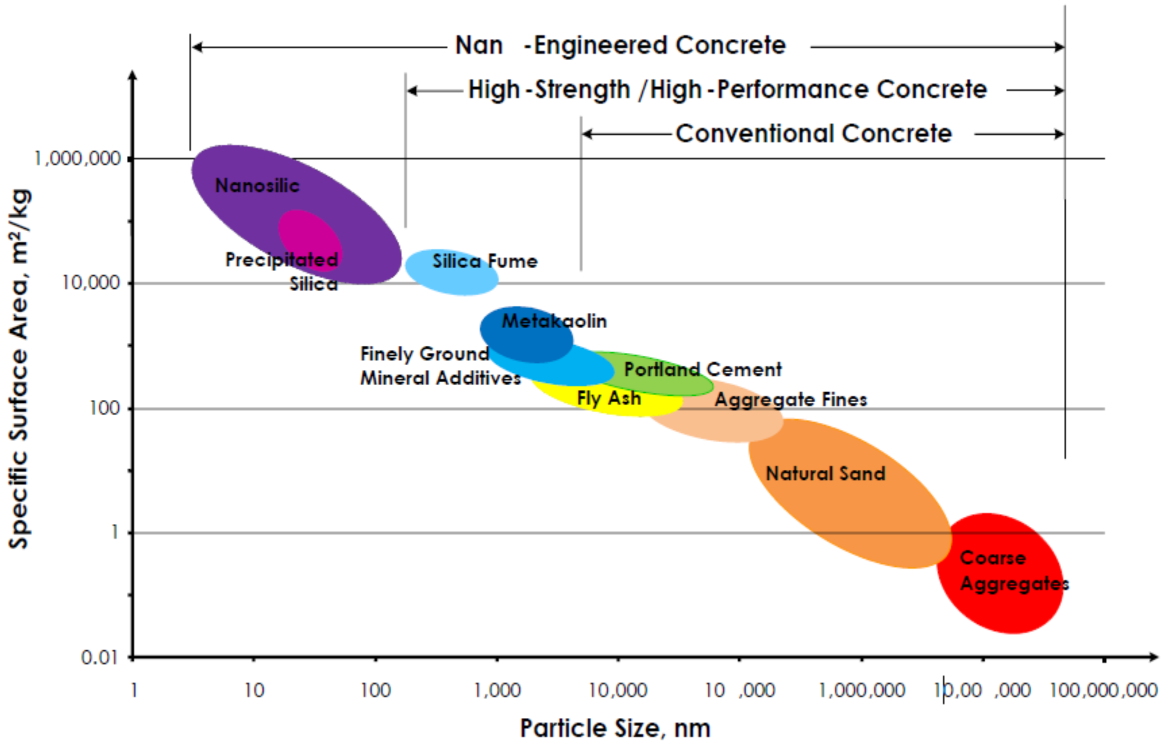


Figure 1.2: Particle size and specific surface area related to concrete materials
(adapted from Sobolev, 2005).

Nano engineering of concrete can take place in one or more of the three location such as (a) in the solid phases (b) in the liquid phase (c) at the interfaces between liquid – solid and solid – solid (Garhoczi, 2009).

The mechanical behavior of the concrete material depends to a great extent on structural elements that are effective on a micro and nanoscale. The size of the calcium silicate hydrate (C-S-H) phase, the primary component responsible for strength and other properties in cementations system, falls in the few nanometers range (Taylor, 1997). The structure of C-S-H is much like clay, with thin layers of solids separated by gel pores filled with interlayer and absorbed water (Mehta, 1986).

This has significant impact on the performance of concrete, because the structure is sensitive to moisture movement, at times resulting in shrinkage and consequent cracking if accommodation in element sizes are not made (Jennings et. al. 2007). Hence nanotechnology may have the potential to engineer concrete with superior properties through the optimization of material behaviour and required performance to significantly improve mechanical performance, durability and sustainability.

The development of nanotechnology based concrete material require a multidiscipline approach. Consisting of team of concrete material experts; Civil Engineers, chemists, physicists and material scientists. Porro et al. (2010) presented an overview of have nanotechnology could be applied to concrete technology, emphasizing the multidiscipline approach required for successful breakthrough leading to ultra high performance materials and new multiscale models that enable the prediction of balk materials properties from compassion parameters. Grove et al. (2010) identified opportunities for nanotechnology leading to a new concrete products and materials and also for improving the sustainable and reducing the environmental footprint of concrete based material in the future. Finally Brigisson et al. (2010) identified the following key breakthroughs in concrete technology that are most likely to result from the use of nanotechnology.

- Development of high performance cement and concrete materials as measured by their mechanical and durable properties.
- Development of sustainable concrete material and structure through engineering for different adverse environment, reducing energy consumption during cement production and enhancing safety.
- Development of intelligent concrete material through the integration of nanotechnology based self-sensing and self-powdered materials and cyber infrastructured technologies.
- Development of novel concrete materials through nanotechnology based innovative processing of cement and cement paste.
- Development of fundamental multiscale model (s) for concrete through advanced characterization and modeling of concrete at the nano - micro, and micro scales.

The development of fibers at the nanoscale has opened a new field of research within concrete. Research by various authors of this work on reinforcing cementations material using nanofibers such as multiwall carbon nano tube have shown that the flexural strength and stiffness of cementation matrices can be significantly increased by adding very low concentration of homogeneral dispersed carbon nano tubes (as little as 0.025%/0.050% by wt of cement). Recently in the field of fiber reinforced cement there has been much enthusiasm for the development of hybrid fiber system where two or more types of fiber are combined.

1.2 CEMENT

Cement is a binder that sets and hardens independently as well as binds other material together. Cement used in construction is characterized as hydraulic or non-hydraulic. Hydraulic cement (eg. Portland cement) harden because of hydration, chemical reaction that occurs independently of the mixture's water content. Hydraulic cement can harden even underwater or when control exposed to wet weather. Non hydraulic cement (eg. Gypsum plaster) must be kept dry in order to retain their strength. The most important use of cement is the production of mortar & concrete – the bonding pf natural or artificial aggregates to form a strong building material that is durable in the form of normal environmental effects. Cement set or cures when mixed with water causes a series of hydration chemical reaction .However keeping moisture content in cement during curing increases both speed of curing and its strength.

1.3. NANOTECHNOLOGY IN CONCRETE

The nanoscience and nanoengineering of concrete are common terms that have come into utilization. This describes two main approaches of application of nanotechnology in concrete researcher (Scrivener et.al. 2008, Scrivener, 2009). Nanotechnology creates new possibilities to control and improve material properties for civil infrastructure.

Nanoscience is the study of atom, molecules and objects whose size is on the nanometer scale (1-100 nanometer). Nanoscience deals with the measurement & characteristics of the nano and micro scale structure of cement based materials to broader understanding of how this structure affects macro scale properties and performance through the use of advanced technologies and molecular level modeling. These modeling helps in characteristics the various properties of concrete.

Nano engineering is a quickly emerging field. It is the practice of engineering on the nanoscale which encompasses the technique of manipulation of structure at the nanometer scale to develop new generation of customized, multidimensional, multifunctional, cementious composites with dynamic physical and mechanical performance as well a durable, potentially having a range of properties such as low electrical resisting, self sensing capabilities, self cleaning, self healing, self compelling, high performance, high durability , high ductility and control of cracks .

The construction industry was the only industry to identify nanotechnology as a promising emerging technology in the UK Delphi survey in the early 1990s. The importance of nanotechnology was also highlighted in foresight reports of Swedish and UK construction. Furthermore, ready mix concrete and concrete products were identified as among the top 40 industrial sectors likely to be influenced by nanotechnology in 10-15 years. However, construction has lagged behind other industrial sectors, such as automobile, chemicals, electronics and biotech sectors, where nanotechnology R&D has attracted significant interest and investment from large industrial corporations and venture capitalists. Recognising the huge potential and importance of nanotechnology to the construction industry, the European Commission in late 2002 approved funding for the Growth Project GMA1-2002-72160 “NANOCONEX” - Towards the setting up of a Network of Excellence in Nanotechnology in Construction. One of main tasks of the 12-monthproject was to produce a state-of-the-art report of nanotechnology in construction. The main objective of the state-of-the-art report was to evaluate current development, awareness and future potential of nanotechnology in the broad area of construction and the built environment. The report was prepared in two parts. The first part was based on results of an email survey of professionals and leading researchers in the construction industry. The second part was a desk study of worldwide nano-related activities and development relevant to construction and the built environment by analysing existing reports, publications and information available on the Internet. As the results in Table 2.6 show, the majority of the respondents are from Universities and Research establishments, and only 11% are from a large number of construction sectors other than materials and equipment supplier (including: contracting, design, survey, service, project management, etc.). The results of the survey of construction professionals and leading researchers on their involvement in nanorelated research and applications in construction are presented in Tables 1.1 and 1.2 respectively.

Table 1.1: Distribution of the survey respondents in various sectors related to construction

Core business sectors	Number of respondents	% of total respondents
University	28	34%
Research establishment	26	31%
Industry – materials and equipment supplied	15	18%
Industry – all sectors except the above	9	11%
Others not yet related to construction	5	6%

Table 1.2: Awareness of nano-related applications in construction

Awareness of nano-related research and applications	% of those responded	
	Academics/ researchers	Industrial personnel
Understanding phenomena (e.g. cement hydration) at nanoscale	82	58
Nano particles, fillers and admixtures	80	37
Nanostructure modified materials (e.g. steel, cement, composites)	73	26
New functional and structural materials	61	26
Surface/interface assessment, engineering	55	21
Special coatings, paints and thin films	45	21
Integrated structural monitoring and diagnostic systems	39	11
Self-repairing and smart materials	31	11
New thermal and insulation materials	20	11
Intelligent construction tools, control devices/systems	22	11
Energy applications for buildings – new fuel cells and solar cells	24	0
Biomimic and hybrid materials	20	0

Table 1.3: Involvement in nano-related activities by the respondents

Involvement in nano-related activities which are potentially applicable to construction	% of those responded	
	Academics/ researchers	Industrial personnel
High performance structural materials	80	37
Understanding phenomena at nanoscale	69	21
Multifunctional materials/components	40	11
Modelling/Simulation of nanostructures	38	5
Nanoscale techniques/instruments	31	11
Smart materials and intelligent systems	29	16

The results in Table 1.1 appeared to show that understanding phenomena at nanoscale and nano particles, filler and admixtures were the two activities/applications in construction best known to the respondents. The awareness of nano-related activities was generally much higher among the academics/researchers than the industrial personnel. Furthermore, the areas of activities which the respondents were more aware of are those of the materials development and fundamental research nature. The results in Table 1.2 seemed to be consistent with the findings of Table 1.3, *i.e.* the major activities of the respondents are in the areas of High performance structural materials and Understanding phenomena at nanoscale. It is noted that except for a limited involvement in the area of High performance structural materials, the industrial personnel have little involvement in nano-related activities. Another important finding coming out of the survey was the perception/attitude of the construction industry on nanotechnology developments and potential applications in constmctinn. Nanotechnology was, if anything, perceived as expensive and too complex to explain to clients who want a structure built as soon and as cheaply as possible. This contrasts with the very rapid growth in awareness of and interest in nanotechnology development in many other industrial sectors, such as chemicals, automobiles and energy, etc.

- *High performance structural materials* – nanostructure modification of steel/metals, ceramics/glass, polymers, cement/concrete, composites through process control or using nanoparticles, nanotubes and nano-admixtures.

- *Understanding phenomena at nanoscale* – nanostructure and macro-properties relationship, *(e.g.* hydration, shrinkage, deterioration, etc.) using new techniques.
- *Functional thin films/coatings* - much enhanced performance in, *e.g.*, optical, thermal, durable, abrasion resistant, self-cleaning, anti-graffiti, etc.
- *New sensors, devices and smart structures* – enhanced monitoring of structural and environmental conditions, and self-actuating capability, though this tended to be at micro rather than macro-level.
- *Sustainable energy, environment applications* - new fuel cells, energy-efficient lightings, special insulating and glazing, pollutants cleaner, etc.

1.4 CARBON MICROFIBER AND CARBON NANOFIBER

Carbon nano fiber are discontinues highly graphic, highly compatible with most polymer processing techniques and can be dispersed in an isotropic mode. They have excellent mechanical properties, high electrical conducting and high thermal conductivity which can be imported to a wide range of matrices including thermoplastics, thermostat, elastomer, ceramic and metals. Carbon nano fibers also have a unique surface state which facilitates functionalizing and other surface modification technique to tailor the nano fiber to the host polymer or application.

Carbon nano fibers are hydrophobic and will consequently not disperse well within water .Surfactants such as Triton –X100 and polyisobutylane have proven to be helpful in dispersing and maintaining the CNF in a stable suspension.

1.5 DISCUSSION ON COMMONLY USED NANO MATERIAL IN CONCRETE

The following areas have a significant potential for applications in construction and the built environment. Some of them are already being developed by industrial companies, and several products already have sales. This area is perhaps the most commercially developed and the use of these nanoscale materials has been the basis for many current and potential applications in various industrial sectors. There have been a large number of companies producing many different types of nano particulate/fiber materials, such as carbon nanotubes, nanoclay, metallic and non-metallic oxides, nano silica etc. For the construction sector in particular, nanoparticles, carbon nanotubes and nano fibers offer a potential for developing much stronger, tougher and more durable structural materials, as well as new functional materials/coatings and devices/systems with much enhanced performance for use in construction and the built environment.

The results of the survey and the desk study appear to indicate that nanotechnology R&D in the broad area of construction and the built environment lags behind other industrial sectors, and has been driven largely by governments, foresight institutes and academics/researchers working in the field. Nanotechnology application in construction is still a very small, fragmented pursuit and unknown outside the scientific circle. However, limited commercial activities have started to emerge and some nano-based materials/ products are now used or ready to be adopted by the industry, and many others are coming to the market. New products/processes are developed to fulfil a need and although this may not be specifically for construction, there are many opportunities for transferring technology. There appears to be a lack of awareness and negative perceptions of nanotechnology among construction professionals. To rectify this negativity, integrated actions are needed for targeted R&D, for technology watch and knowledge transfer in construction industry.

1.6 USE OF CARBON MICRO FIBER AND CARBON NANO FIBER IN CONCRETE

Carbon micro fiber and carbon nano fibers provide enormous opportunities to engineers the property of material by working atomic or molecular level. The uses of above new material in concrete are mentioned as under:

- To develop high performance, multifunctional ideal (high strength, ductile, crack free, durable) construction material, carbon nano fiber show promising role to modify/enhance the characteristics of the conventional construction materials such as concrete and steel.
- It is also brought out that a crack free durable concrete is possible if certain issues such as uniform distribution of CNF/CNT in composite and bond behaviour of CNF/CNT modified concrete can be addressed.
- It has been established in previous research that the flexural strength and stiffness of cementitious matrices can be significantly increase by adding low concentration of homogeneously dispersed carbon nano tubes (as little as 0.025% by weight of cement).

- Improvement on the mechanical properties of the matrix can be attributed to good bonding between CNF and the cement hydration product as well as ability of CNF to control cracking the nano scale by bridging nano cracks and pores.

1.7 RESEARCH SIGNIFICANCE

Cement based materials are complex materials consisting of several phase. In order to effectively reinforce and improve the response of cementitious material to loading, crack growth must be abated at the macro, micro and nano level. This study investigates the reinforcing effect of addition of CMF and CNF in cement mortars. The goal of the study is to evaluate the effect of ladder scale reinforcement in cement based materials using low concentrations of well dispersed carbon nano fibers and carbon micro fibers. Applications of nanotechnology in construction are mentioned as under:

- The construction industry was the only industry to identify nanotechnology as a promising emerging technology in the UK Dephi survey in the early 1990s.
- The importance of nanotechnology was also highlighted in foresight reports of Swedish and UK constructions.
- Furthermore ready mix concrete and concrete products were identified as among the top 40 industrial sectors likely to the influenced by nanotechnology in 10-15 years.
- However construction has lagged behind the other industrial sectors such as automobile, chemicals, electronics and biotech sectors. Where nano technology R&D has attracted significant interest and investment from large Industrial Corporation.
- Recognizing the huge potential, and importance of nanotechnology to the construction industry, the European commission in late 2002 approved for the growth project GHAI – 2002-72160 “NANOCONQ” towards The setting up of a network of excellence in technology in construction. One of the main task of the 12 month project was to produce a state of the art report of technology in construction.

- The main objective of the state of art report was to evaluate current development, awareness and future potential of nanotechnology in the broad area of construction and the built environment.
- The report was prepared in two parts. The first part was based on result of on email survey of professionals and leading researchers in the construction. The second part was desk study of worldwide nana-related activities and development relevant to construction by analyzing existing reports, publications and information available on the internet.

1.8 OBJECTIVE AND SCOPE OF PRESENT STUDY

Objective of this work is to investigate the effect on mechanical properties of cement mortar reinforced with varying proportion of CMF and CNF used as partial replacement of cement. The current brief research aims at providing cement mortars with ultra high performance to be used in various construction applications. Such as shortcrete in tunnels, marins structures, water treatment plant, retrofitting /major rehabilitations of infrastructure projects.

In order to achieve the objectives, various trial mixes of cement mortar were conducted varying the percentage of carbon nano fiber as well as carbon nano fiber dispersed into aqueous system as well as in stable suspension so as to mix properly with cement particles. Also many properties have been tested in order to achieve the aforesaid objectives, as fresh properties like consistency, setting times, physical characterizes and unit wt variations, hardened properties Such as compressive strength , weight loss, water absorption , split tensile strength, flexural strength, XRD and mirco structural analysis using scanning electron microscope (SEM). The results of these test were compared with that of the normal mortar mixture.

The use of nano-structured materials in mortar and concrete can add many benefits that are directly related to the durability of these materials, beside the fact that it is possible to reduce the quantities of cement in the composite mix.

- The scope of the present study is limited to the use of carbon micro fiber/nano fiber in the cement mortar by replacing some percentage of the cement with carbon micro fiber & carbon nano fibers.

- The properties of cement mortars (OPC grade 43) with carbon micro fiber as well as carbon nano fibers are experimentally studied.
- To investigate the effect of carbon nano fiber replacement level as well as carbon micro fiber on compressive strength, split tensile strength, flexural strength and durability aspect of cement mortar.
- To study the micro structure using SEM analysis as well as XRD analysis of phases concerning chemical compositions.

1.9 ORGANIZATION OF THE THESIS

The thesis comprises of 5 chapters. Each chapter has its own significance in the thesis.

Chapter 1: Represent the general introduction of the study background alongwith objective, scope, and limitation of the study.

Chapter 2: Covers a brief literature review variety of strength development of cement mortar containing carbon nano fiber, carbon micro fiber, carbon nano tubes etc. at early age and the adverse affects of addition of carbon fibers were reviewed as well. Limited information available in literature on effect of carbon nano fiber, carbon micro fiber on strength development of cement mortars and concrete was also reviewed.

Chapter 3: The detail of the experiment program materials used and its sources, proportioning of ingredients, specimen preparation and testing procedures are presented. This also discusses in details about the scanning electron microscope studies, XRD studies etc.

Chapter 4: Presents experiment results, analysis and relevant discussion.

Chapter 5: Summarizes test results and conclusion drawn based on findings of work, limitations and recommendation for further research are also presented.

CHAPTER – 2
LITERATURE REVIEW

2.1 GENERAL

In this chapter the research work concerning to various application and method used for investigation of use of carbon nano fiber and carbon micro fiber in cement mortar has been studied. This chapter gives a comprehensive review of the work carried out by various researchers in the field of strength development of cement mortar containing nano composite as carbon nano fiber, carbon micro fibers, carbon nanotubes etc. at early age and the adverse effect of over addition of carbon fibers were reviewed as well. Limited information available in literature on effect of carbon nano fiber, carbon nano fiber on strength development of cement mortar including durability & concrete were also reviewed.

2.2 REVIEW OF LATEST WORK IN THE FIELD

Pigeon et al (1996) stated that the frost durability of steel and carbon micro-fiber reinforced mortars was assessed. Tests of freezing and thawing and surface scaling were performed on the mortars. The effect of drying on the frost durability was also studied. The results show that the use of steel and particularly carbon micro fibers improve the frost and deicer salt scaling resistance of mortars. The improvement is however in part due to the "air entrainment" properties of the micro-fibers.

Key results obtained from the study can be discussed as under:

Table 2.1 gives the composition of the mortar mixtures. Four groups of five mixtures were made: the first group was prepared with 8% silica fume (at a fine aggregate to total binder ratio of 1), the second group with 15% silica fume (at a fine aggregate to total binder ratio of 1), the third group with 8% silica fume (at a fine aggregate to total binder ratio of 2), and a fourth group with 15% silica fume (at a fine aggregate to total binder ratio of 2). In each group, one mixture was made without fibers, one with 1% of steel fibers, one with 2% of steel fibers, one with 1% of carbon fibers, and one with 2% of carbon fibers. The steel fibers were 3 mm in length with a cross section of approximately 5 pm by 25 pm. The carbon fibers were 10 mm in length with a diameter of 18 nm. Both types of fibers have a strength of approximately 600 Mpa, but the elastic modulus of the carbon, at 30 GPa, is much lower than that of the steel (200 GPa). The relative density of the carbon is 1,65 (7,85 for the steel).

Table 2.1: Mix Characteristics

Mix Code	Water (Kg/m^3)	Cement (Kg/m^3)	Silica Fume (Kg/m^3)	Sand (Kg/m^3)	Sp[1] (L/m^3)	SF/B[2] (%)	S/B[2]	Fiber Content (%)	Unit Weight (Kg/m^3)
N-0-08-1	320	843	73	931	3.7	8	1	0.0	2169
St-1-08-1	318	838	73	917	6.0	8	1	1.0	2230
St-2-08-1	295	775	68	847	9.0	8	1	1.9	2140
C-1-08-1	312	819	71	895	9.6	8	1	1.0	2118
C-2-08-1	299	786	68	855	19.5	8	1	1.9	2050
N-0-15-1	324	789	140	933	4.7	15	1	0.0	2191
St-1-15-1	316	767	135	902	10.0	15	1	1.0	2200
St-2-15-1	301	729	129	863	1302	15	1	1.9	2170
C-1-15-1	625	770	136	909	12.1	15	1	1.0	2149
C-2-15-1	307	744	132	869	18.0	15	1	2.0	2080
N-0-08-2	242	635	55	1374	5.0	8	2	0.0	2310
St-1-08-2	218	574	50	1253	7.5	8	2	0.9	2172
St-2-08-2	205	539	46	1177	10.5	8	2	1.8	2113
C-1-08-2	220	579	51	1259	17.1	8	2	0.9	2134
C-2-08-2	211	555	48	1208	42.6	8	2	1.9	2074
N-0-15-2	236	573	101	1355	10.0	15	2	0.0	2265
St-1-15-2	225	545	96	1289	13.5	15	2	1.0	2160
St-2-15-2	201	488	86	1149	15.6	15	2	1.7	2066
C-1-15-2	220	535	94	1263	15.2	15	2	0.9	2136
C-2-15-2	212	516	92	1213	33.3	15	2	1.9	2080

[1]Sp: Superplasticizer; [2]SF: Silica Fume, B: Binder; [3]S: Sand Mix Code N: No fiber; St: Steel fiber; C: Carbon Code: Fiber type - Fiber percent -SF/B - S/B

Although, as expected, the results of the scaling tests performed on the micro-reinforced and on the non-reinforced mortars are relatively scattered (see Table 2.2), they indicate very clearly that, as a general rule, drying did not have a large influence. As just mentioned, the pore structure of pastes containing silica fume is little influenced by drying, because the capillary pores are very small and well distributed in the matrix. The results also indicate that the non-reinforced mortars generally suffered quite severe scaling (the mass of residues being larger in most cases than the

1 kg/m^2 limit), whereas the micro-reinforced mortars generally suffered little scaling. The rapid freezing and thawing cycle test results confirm the good frost durability of the micro-reinforced mortars. In the series of tests performed on the specimens not subjected to drying, the length change at the end of the tests is extremely large for all four non-reinforced mortars, but generally small for the micro-reinforced mortars, particularly those containing 2% fibers (steel or carbon) and 1% carbon fibers. This positive, but variable, influence of the fibers indicates that the increase in frost durability due to the fibers can not be related only to the lower air void spacing factor, and that the microfibers, by preventing crack propagation, can help to reduce the deterioration due to frost action.

Table 2.2: Surface Scaling Test Results

Mix Code	Spacing Factor (µm)	Scaled off Mass 14 Day Curing (Kg/m^2)	Scaled off Mass 90 Day Curing 23°C (Kg/m^2)	Scaled off Mass 90 Day Curing 38°C (Kg/m^2)
N-0-08-1	629	2.02	5.01	1.49
St-1-08-1	288	0.35	2.06	0.14
St-2-08-1	258	0.13	0.44	0.07
C-1-08-1	436	0.27	0.28	0.11
C-2-08-1	254	0.18	0.21	0.04
N-0-15-1	594	1.74	3.16	0.93
St-1-15-1	376	0.63	2.68	0.58
St-2-15-1	293	0.24	1.05	0.03
C-1-15-1	367	0.23	0.13	0.17
C-2-15-1	314	0.20	0.17	0.09
N-0-08-2	544	0.70	0.49	0.18
St-1-08-2	202	0.16	0.55	0.03
St-2-08-2	176	0.09	0.05	0.02
C-1-08-2	318	0.40	0.26	0.11
C-2-08-2	255	0.07	0.67	0.03
N-0-15-2	645	1.32	1.02	1.90
St-1-15-2	282	0.17	0.29	0.05
St-2-15-2	174	0.08	0.33	0.03
C-1-15-2	318	0.21	0.29	0.11
C-2-15-2	249	0.16	0.18	0.13

The use of steel and particularly carbon micro fibers can help to improve the frost and deicer salt scaling resistance of mortars, although part of this improvement is due to the "air entrainment" properties of these fibers. Because of this phenomenon, more tests will be needed to assess precisely the increase in durability due to the fibers.

Chung (1999) studied that degree of dispersion of short micro fibers in cement, as assessed by electrical resistivity measurement for the case of electrically conductive fibers at a volume fraction below the percolation threshold, is improved by the use of admixtures (namely, silica fume, acrylic particle dispersion, methylcellulose solution, and silane) and fiber surface treatment (such as ozone treatment). Acrylic particle dispersion is more effective than latex particle dispersion. Short fibers are used as admixtures in cement-based materials for the purpose of decreasing the drying shrinkage, increasing the flexural toughness, and, in some cases, increasing the flexural strength as well (Park and Lee 1993; Chen et al. 1997; Chen and Chung 1993; Toutanji et al. 1993; Banthia and Sheng 1996). In the case that the fibers are electrically conductive, the fibers may also provide nonstructural functions, such as self-sensing (for sensing the strain, damage, or temperature), self-heating (for deicing), and electromagnetic reflection (for electromagnetic interference shielding, i.e., EMI shielding) (Chung 2003). Although continuous fibers are more effective than short fibers as a reinforcement (Wen and Chung 1999), they are not amenable to incorporation in a concrete mix and they are relatively expensive. Low cost is critical to the practical viability of cement-based materials. Thus, this paper is focused on short fibers in cementbased materials.

Key results obtained from the study can be discussed as under:

Effect of Admixtures Used along with Fibers

Admixtures used along with the short fibers in the cement mixture can cause an increase or decrease in the degree of fiber dispersion. Admixtures include silica fume, polymer particles, waterbased dispersions, polymer water-based solutions, and silane.

Effect of Polymer Particle Dispersions

Due to the need for water in a cement mix, polymer particle dispersions based on water are more suitable than those based on other liquids. The polymers used in the dispersions are not soluble in the liquids used. As most polymers are not soluble in water, the choice of polymers for dispersions is wide compared to that of polymers for solutions.

At a carbon fiber (15 μm diameter; ~5 mm length) content of 0.35%, in the absence of silica fume, the electrical resistivity of the cement mortar is reduced from 1.59×10^6 Ω. cm (± 4.5%) to 0.58×10^6 Ω.cm (± 6.0%) upon addition of acrylic water-based dispersion (20% by mass of cement) (Table 2.2) (Cao and Chung 2001b). Latex and styrene acrylic water-based dispersions (also 20% by mass of cement) are less effective than acrylic dispersion, as they reduce the resistivity to 0.89×10^6 Ω. cm (± 7.7%) and 0.83×10^6 Ω. cm (± 5.1%), respectively (Table 2.3). As polymer particle dispersions are used in much larger amounts than methylcellulose, the cost of using the dispersions is relatively high.

Table 2.3 : Volume Electrical Resistivity and Tensile Strength of Cement Mortars Containing 0.35 Volume % Short Carbon Fibers (15 μm Diameter; ~5 mm Length): Silica Fume (SF) (Cao and Chung 2001b)

Organic admixture	Organic admixture amount (% by mass of cement)	Apparent water/ cement ratio	Resistivity (10^6 Ω. cm)		Tensile strength (MPa)	
			Without SF	With SF	Without SF	With SF
–	0	0.350[b]	1.59(±4.5%)	1.13(±3.9%)	1.45±0.11	1.04±0.11
Methylcellulose	0.4	0.350[b]	0.68(±2.8%)	0.31(±3.1%)	2.26±0.08	2.36±0.06
Acrylic	10[a]	0.297	0.67(±6.9%)	0.55(±5.3%)	2.15±0.08	2.26±0.07
Acrylic	15[a]	0.270	0.56(±4.3%)	0.49(±5.1%)	2.49±0.13	2.54±0.11
Acrylic	20[a]	0.244	0.58(±6.0%)	0.51(±3.1%)	2.56±0.20	2.46±0.09
Styrene Acrylic	10[a]	0.295	1.01(±4.9%)	0.93(±3.4%)	1.98±0.14	2.04±0.10
Styrene Acrylic	15[a]	0.267	0.96(±4.0%)	0.85(±5.2%)	2.07±0.20	2.28±0.15
Styrene Acrylic	20[a]	0.240	0.83(±5.1%)	0.77(±4.6%)	2.23±0.12	2.41±0.09
Latex	10[a]	0.298	0.66(±5.8%)	0.56(±4.7%)	2.02±0.15	2.19±0.13
Latex	15[a]	0.272	0.70(±9.3%)	0.63(±3.7%)	2.25±0.17	2.49±0.11
Latex	20[a]	0.246	0.89(±7.7%)	0.70(±6.1%)	2.08±0.20	2.40±0.08

[a]Including mass of water in the dispersion.
[b]Same as the true water/cement ratio.

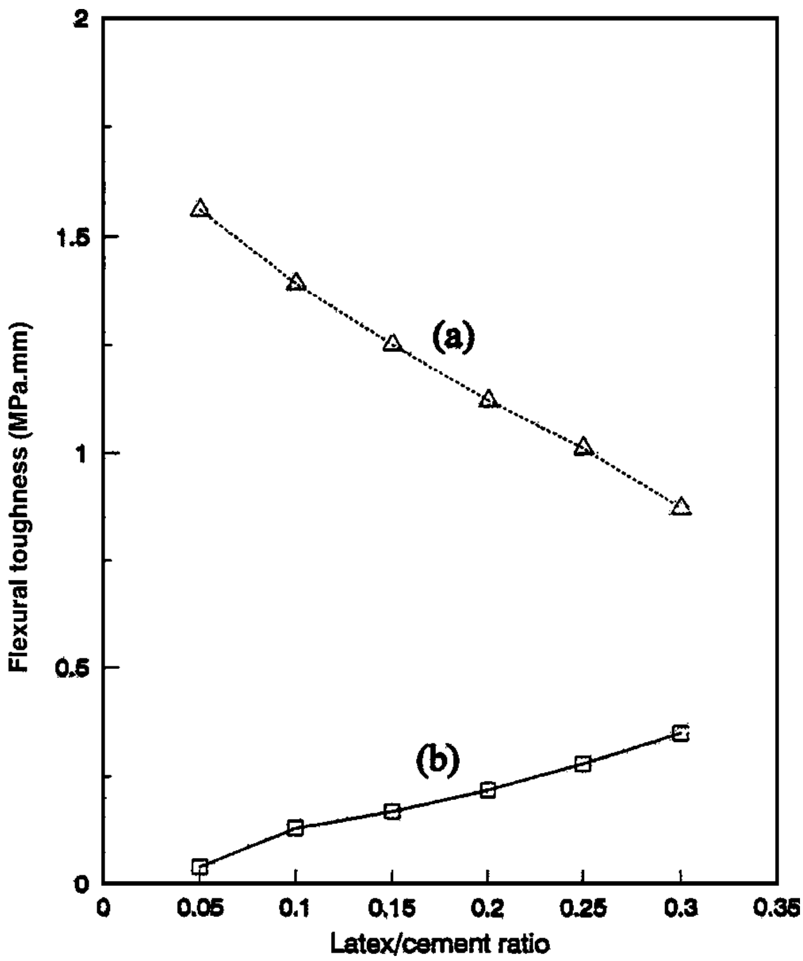


Figure 2.1: Effect of latex/cement ratio on flexural toughness when the cement paste contains: (a) 0.53 volume % carbon fibers; (b) no fibers

Chung (2000) stated that the review of cement-matrix composites containing short carbon fibers. These composites exhibit attractive tensile and flexural properties, low drying shrinkage, high specific heat, low thermal conductivity, high electrical conductivity, high corrosion resistance and weak thermoelectric behavior. Moreover, they facilitate the cathodic protection of steel reinforcement in concrete, and have the ability to sense their own strain, damage and temperature. Fiber surface treatment can improve numerous properties of the composites. Conventional carbon fibers of diameter 15 mm are more effective than 0.1 mm diameter carbon filaments as a reinforcement, but are much less effective for radio wave reflection (EMI shielding). Carbon fiber composites are superior to steel fiber composites for strain sensing, but are inferior to steel fiber composites in the thermoelectric behavior. Carbon fiber cement-matrix composites are structural materials that are gaining in importance quite rapidly due to the decrease in carbon fiber cost and the increasing demand of superior structural and functional properties. These composites contain short carbon fibers, typically 5 mm in length, as the short fibers can be used as an admixture in concrete (whereas continuous fibers cannot be simply added to the concrete mix) and short fibers are less expensive than continuous fibers. However, due to the weak bond between carbon fiber and the cement matrix, continuous fibers are much more effective than short fibers in reinforcing concrete. The silica fume is typically used along with a small amount (0.4% by weight of cement) of methylcellulose for helping the dispersion of the fibers and the workability of the mix. The improved structural properties rendered by carbon fiber addition pertain to the increased tensile and flexible strengths, the increased tensile ductility and flexural toughness, the enhanced impact resistance, the reduced drying shrinkage and the improved freeze-thaw durability.

Table 2.4: Properties of carbon fibers

Filament diameter	15 ± 3 μm
Tensile strength	690 MPa
Tensile modulus	48 GPa
Elongation at break	1.4%
Electrical resistivity	3.0×10^{-3} Ω cm
Specific gravity	1.6 g cm^{-3}
Carbon content	98 wt. %

Key results obtained from the study can be discussed as under:

This paper is a review of short carbon fiber reinforced cement-matrix composites, including concrete (with fine and coarse aggregates), mortar (with fine aggregate and coarse aggregate) and cement paste. Previous reviews are noted [96±101]. Table 2.4 shows the properties of the isotropic-pitch-based carbon fibers (15 mm in diameter, nominally 5 mm long) used by the author in the cement-matrix composites described below for the purpose of illustration. The properties relevant to the structural behavior of cement-matrix composites containing short carbon fibers are given in this section.

Table 2.5: Tensile ductility (%) of cement pastes with and without fibers (A: cement + water + water reducing agent 1 silica fume, A^+: A + methylcellulose + defoamer, A^+F: A^+ + as-received fibers, A^+O: A^+ + O_3-treated fibers, A^+K: A^+ + dichromate-treated fibers, A+S: A + silane-treated fibers)

Formulation	As-received silica fume	Silane-treated silica fume
A	0.020 ± 0.0004	0.020 ± 0.0004
A^+	0.023 ± 0.0004	0.021 ± 0.0004
A^+F	0.025 ± 0.0003	0.024 ± 0.0004
A^+O	0.026 ± 0.0003	0.027 ± 0.0004
A^+K	0.028 ± 0.0003	0.030 ± 0.0004
A^+S	0.031 ± 0.0004	0.034 ± 0.0004

Table 2.5 shows the tensile ductility. It is slightly increased by the addition of methylcellulose and defoamer, and is further increased by the further addition of fibers. The effectiveness of the fibers in increasing the ductility also increases in the above order. This trend applies whether the silica fume is as-received or silane-treated. For any of the formulations involving surface treated fibers, silane-treated silica fume gives higher ductility than as-received silica fume. The highest ductility is exhibited by cement paste with silane-treated silica fume and silane-treated fibers. The ductility is 39% higher than that of the cement paste with as-received silica fume and as-received fibers. It is 14% higher than that of the cement paste with as-received silica fume and silane-treated fibers.

Short carbon fiber cement-matrix composites exhibit attractive tensile and flexural properties, low drying shrinkage, high specific heat, low thermal conductivity, high electrical conductivity, high corrosion resistance and weak thermoelectric behavior. Moreover, they facilitate the cathodic protection of steel reinforcement in concrete, and have the ability to sense their own strain, damage and temperature.

Zhu et al (2004) presented the background information and current developments of nanotechnology in general. Then, the current activities and awareness of nanotechnology in the construction industry are examined by analysing results of a survey of construction professionals and leading researchers in the field. This is followed by results of a desk study of nanotechnology development and activities focussing on key areas relevant to construction and the built environment.

Examples of nanotechnology-enabled materials and products that are either on the market or ready to be adopted in the construction industry are provided. Finally, the future trend/potential and implications of nanotechnology development in construction are discussed. There are two ways to approach the nanoscale: shrinking from the top down, or growing from the bottom up. These two models are fundamentally different, both in the approach to creating structures and in the underlying science that will make them possible. The ‘top down’ approach entails reducing the size of the smallest structures towards the nanoscale by machining and etching techniques, whereas the ‘bottom up’ approach, often referred to as molecular nanotechnology, implies controlled or directed self-assembly of atoms and molecules to create structures. Nanoscience and nanotechnology inevitably cross the boundaries and bring together traditional sciences and technologies, such as chemistry, physics, the life sciences, materials and many engineering disciplines.

Key results obtained from the study can be discussed as under:

Funding of nanotechnology R & D

There has been an explosive growth of interest in nanotechnology over the last few years. The NNI in the US in 2000 was the catalyst to rapidly increasing funding and activities in this area. Global government spending on nanotechnology has more than doubled in the last 2-3 years, and has now grown to well over $2 billion a year. There is also substantial money flowing into nano-related research from multinational corporations and venture capital investments. Many of the world's largest companies, such as IBM, Intel, Motorola, Lucent, Boeing, Hitachi, Mitsubishi, NEC, Pfizer, Coming, Dow Chemical, 3M, General Motors, Ford, etc. have all had significant nanorelated research projects going on, or launched their own nanotech initiatives as graphically represented in Figure 2.2.

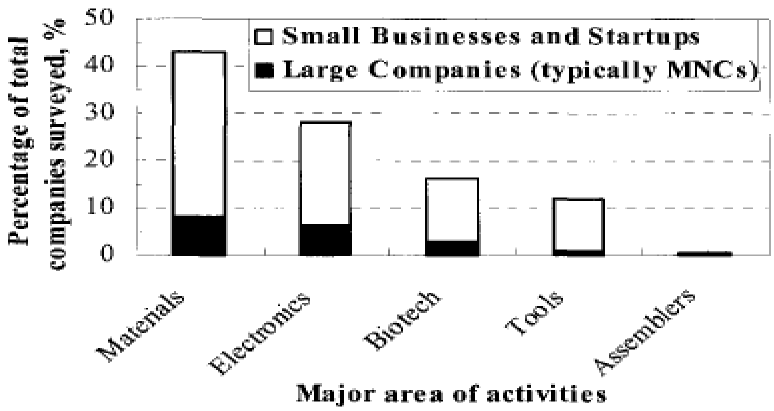


Figure 2.2: Profile of nanotech company focus

Lawler and Shah (2005) stated that to realize the potential of micro- and macrofiber blends in a concrete matrix, a concrete containing polyvinyl alcohol (PVA) or steel microfibers and steel macrofibers was designed using a mixture proportioning method that provides good workability in concretes containing microfibers. This procedure is based on an optimum paste volume fraction determined from the relationship between flow and paste content. The mechanical performance, water permeability of the cracked material, and shrinkage crack resistance of cast concrete were evaluated. In the hybrid concrete, the microfibers delayed the development of macrocracks and so the composite demonstrated greater strength and crack resistance than a similar matrix reinforced with macrofibers only. This influence was less pronounced than was observed with a mortar matrix in a previous study. This is explained by differences in the failure mechanism of the fibers: A stronger fiber-matrix bond resulting from a lower water-to-binder ratio caused the microfibers to break instead of pull out. Fibers are included in concrete to increase the strength and toughness of the material. The effectiveness of a particular type of fibers at performing this task is dependent on the material properties and geometry of the fibers. Including two or more types of fiber that make complementary and additive contributions to performance in a concrete mix is a method for maximizing the improvements possible through fiber reinforcement (Banthia and Sheng 1990; Bentur and Mindess 1990; Betterman et al. 1995; Mobasher and Li 1996; Qian and Stroeven 2000). One such blend, a combination of macrofiber (0.5 mm diameter) and microfiber (less than 0.022 mm diameter) has been demonstrated to be effective in a mortar matrix because these fibers influence crack growth at different stages of the failure process (Lawler et al. 2002a, 2003).

Key results obtained from the study can be discussed as under:

In addition to the hybrid-reinforced concrete, control mixes of unreinforced concrete and macrofiber reinforced concrete were cast (Table 2.6) Mixtures containing only microfibers were not examined because, as has been demonstrated elsewhere (Betterman et al. 1995), concrete reinforced with microfiber only is not expected to demonstrate anywhere near the postpeak toughing that can be achieved with macrofibers.

Table 2.6: Mixes Examined

Mix No.	V_p/V_{tot}	% fly ash	Fiber (%)		Superplasticizer (% by wt. binder)	Flow at casting (%)
			Macrofiber	Microfiber		
1	0.43	0	–	–	0.24	105.5
2	0.43	0	0.82	–	0.30	91
3	0.43	15	0.50	0.32 steel	0.77	72.5
4	0.39	15	0.50	0.29 PVA	0.73	93

Flexural Testing

Flexural mechanical properties of the mixtures were measured by three-point bending tests of the beams with a notch cut to one-third of the specimen height at the midspan, based on the procedure recommended by RILEM (1991). The tests were performed with a closed-loop servohydraulic testing machine with 89 kN capacity and an 89 kN load cell. The beams were 50 × 100 × 460 mm in size. The crack mouth opening displacement measured at the notch was used to control the test and was advanced at a rate of 0.15 mm/min.

Results

Flexure

Both the flexural strength and toughness of the composites can be determined from the flexural stress-displacement relations pictured in Fig. 2.3 for representative specimens. The stress at the development of the first macrocrack, i.e., where the curve makes a marked break from the prepeak linearity, is significantly increased by the addition of the microfibers. After an initial drop in load occurs, the PVA hybrid (containing 0.5% steel macrofiber and 0.29% PVA microfiber) exhibits deflection-hardening behavior, while the steel hybrid (containing 0.5% steel macrofiber and 0.32% steel microfiber) shows a drop in performance with a greater deformation that continued throughout the test. The macrofiber-reinforced concrete (containing 0.5% steel macrofiber) displays more prolonged deflection hardening and carries a greater stress than any of the hybrids after approximately 0.8 mm of displacement.

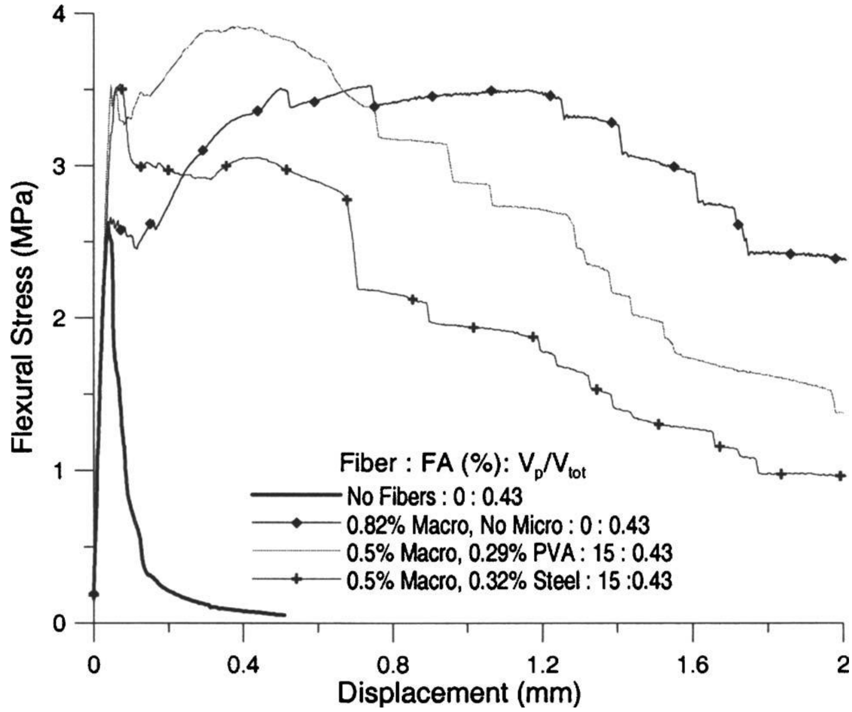


Figure 2.3: Stress versus midpoint displacement for concrete mixes

While the observed deflection hardening is likely influenced by the shear stresses inherent in flexural testing, the deflection-hardening process is significant because it demonstrates that a sequential mobilization of fibers, which does not typically occur in conventional fiber-reinforced concrete, is possible with these mixtures. Average properties for all beams are plotted in bar chart form in Figs. 2.4 and 2.5.

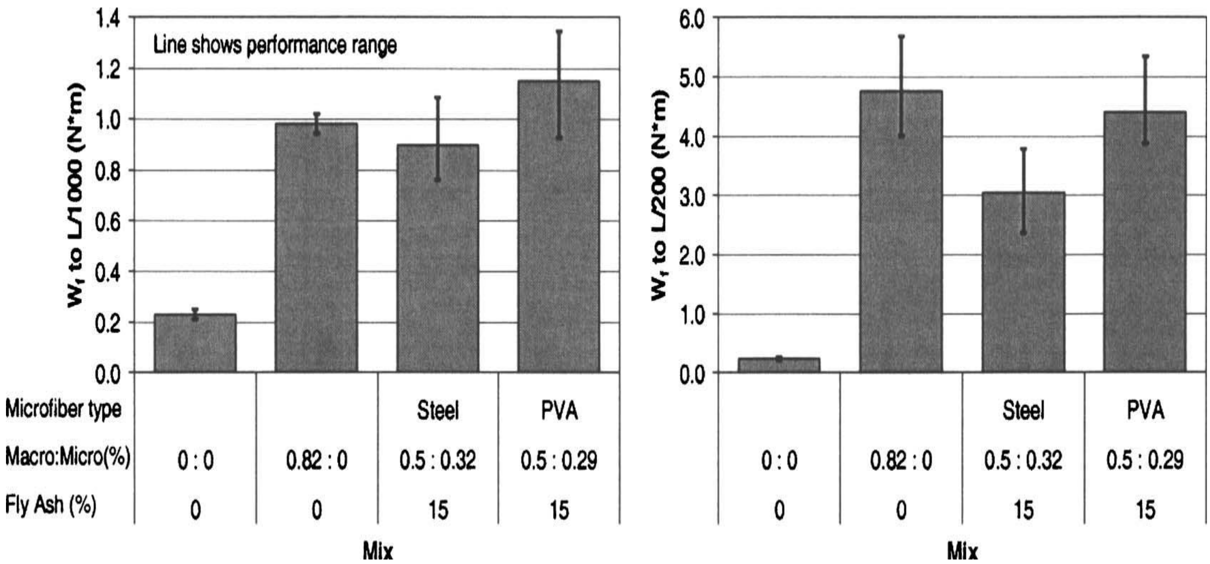


Figure 2.4: Toughness determined for small (0.4 mm) and large (2 mm) deformation

The line in the plot is not an error bar, but rather gives the maximum and minimum performance observed in that mixture. The work of fracture (W_f), i.e., the area under the curve, is calculated up to deformations of L/1,000 = 0.4 mm and L/200 = 2 mm, where L = span used for testing (Fig. 2.4). The PVA hybrid blend outperformed the macrofiber reinforcement at small displacements. However, when the full displacement is considered, the ability of macrofibers to bridge opening macrocracks is apparent and the superior improvement produced by these fibers in toughness is observed.

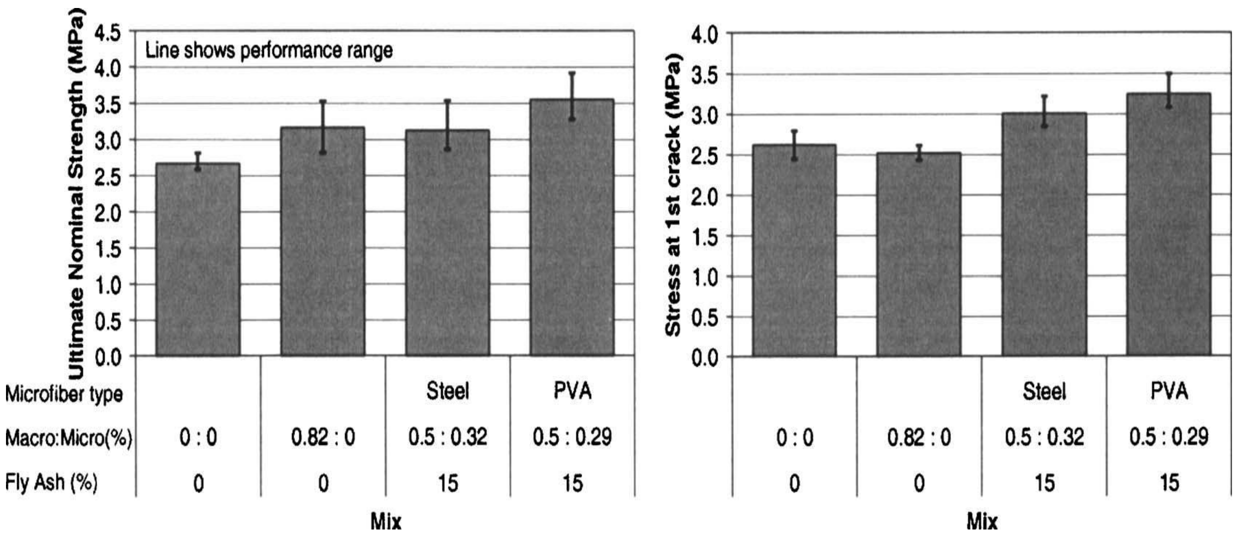


Figure 2.5: Ultimate strength and first crack stress

Micrographs obtained through scanning-electron microscopy of the fracture surfaces at 40X magnification are given in Fig. 2.6. The steel and PVA microfiber segments visible at the concrete surface are much shorter than similar exposed fibers in the mortar. This suggests that the critical length of the fibers, i.e., the minimum length of fiber over which the bond forces must act to cause the fiber to break, in the concrete matrix was lower as a result of a greater bond and so the fibers broke instead of pulling out. This increase in bond strength due to the lower w/b in the matrix caused more microfibers to break in the concretes than in the mortar. An increase in bond strength at the fiber-matrix interface has been linked to increases in the strength of the matrix (Naaman and Najm 1991) and to reductions in water content (Bentur 1989), both of which were present in the concrete compared to the mortar.

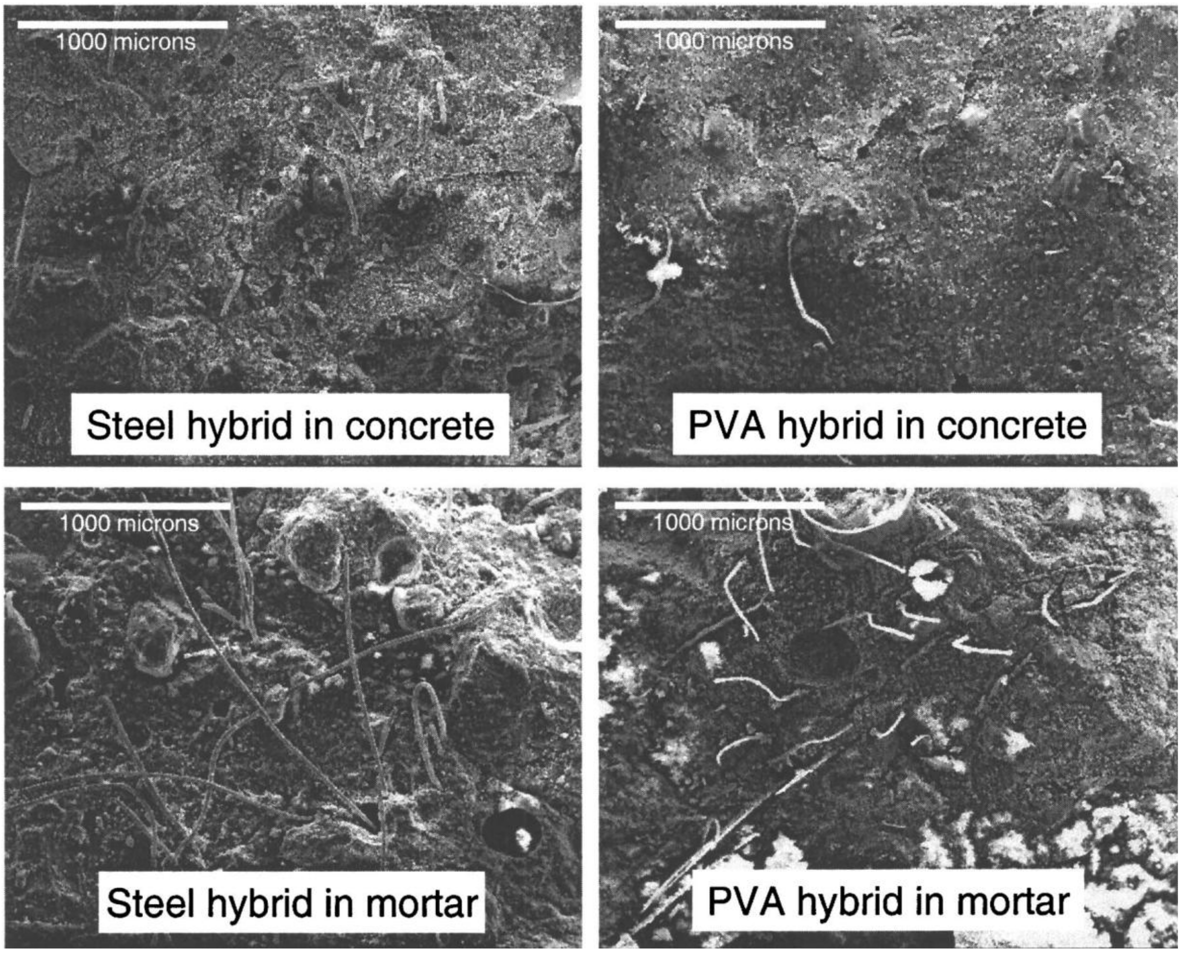


Figure 2.6: Scanning electron microscope micrographs of fracture surface showing fiber breakage in concrete and pullout in mortar (40X)

A workable blend of microfibers and macrofibers was achieved in concrete during this study. The flow of the wet mixture, mechanical performance, resistance to restrained ring shrinkage cracking and cracked permeability were all evaluated. In terms of ultimate strength, the steel hybrid mixture performance was comparable to the macrofiber while the PVA hybrid exceeded it by a significant margin. The toughness of the PVA hybrid was greater than that of the macrofiber mixture in flexural displacements less than 0.5 mm but not superior at larger deformations.

Konsta and Shah (2009) stated that due to their exceptional mechanical properties, carbon nanotubes (CNTs) are considered to be one of the most promising reinforcing materials for the next generation of high-performance nanocomposites. In this study, the reinforcing effect of highly dispersed multiwall carbon nanotubes (MWCNTs) in cement paste matrix has been investigated. The MWCNTs were effectively dispersed in the mixing water by using a simple, one step method utilizing ultrasonic energy and a commercially available surfactant. A detailed study on the effects of MWCNTs concentration and aspect ratio was conducted. The excellent reinforcing capabilities of the MWCNTs are demonstrated by the enhanced fracture resistance properties of the cementitious matrix. Additionally, nanoindentation results suggest that the use of MWCNTs can increase the amount of high stiffness C–S–H and decrease the porosity. Besides the benefits of the reinforcing effect, autogenous shrinkage test results indicate that MWCNTs can also have a beneficial effect on the early strain capacity of the cementitious matrix, improving this way the early age and long term durability of the cementitious nanocomposites. Advanced technological aspects of cement based materials have recently focused on developing high-performance cementitious composites, which exhibit high compressive strengths. Such composites however, exhibit also extremely brittle failure, low tensile capacity and appear sensitive to early age microcracking as a result of volumetric changes due to high autogenous shrinkage stresses. These characteristics of cement based materials are serious shortcomings that not only impose constrains in structural design, but also affect the long term durability of structures. To overcome the aforementioned disadvantages reinforcement of cementitious materials is typically provided at the millimeter and/or the micro scale using macrofibers and microfibers, respectively. Cementitious matrices however, exhibit flaws at the nanoscale, where traditional reinforcement is not effective. Carbon nanotubes (CNTs) present several distinct advantages as a reinforcing material for high strength/performance cementitious composites as compared to more traditional fibers. First, they exhibit significant greater strength and stiffness than conventional fibers, which should improve overall mechanical behavior. Second, their higher aspect ratio is expected to effectively arrest the nanocracks and demand significantly higher energy for crack propagation. Thirdly, provided that CNTs are uniformly dispersed, and due to their nanoscale diameter, fiber spacing is reduced. Few attempts have been made to add CNTs as reinforcement in cementitious matrices. Makar et al. investigated the reinforcing effect of 2.0 wt.% CNTs in cement using SEM and Vickers hardness measurements. The results obtained indicated that CNTs may affect the early hydration progress, producing higher hydration rates. Li et al. employed a carboxylation procedure to improve the bonding between 0.5 wt.% MWCNTs and cement matrix and obtained a 25% increase in flexural strength and a 19% increase in compressive strength.

Key results obtained from the study can be discussed as under:

Mechanical performance

Table 2.7: Properties of multiwall carbon nanotubes (MWCNTs)

	Aspect ratio	Diameter (nm)	Length (μm)	Purity (%)	Surface area (m^2/g)
Short	700	20-40	10-30	>95	110
Long	1600	20-40	10-100	>97	250-300

The characteristics property of available purified multiwall carbon nano tubes are represented in Table 2.7. The fracture mechanics test results of the average flexural strength of cement paste samples reinforced with short MWCNTs at amounts of 0.048 wt.%, 0.08 wt.% and 0.10 wt.% by weight of cement at the age of 3, 7 and 28 days are presented in Fig. 2.7. In all cases, the samples reinforced with MWCNTs exhibit higher flexural strength than plain cement paste. Samples reinforced with 0.08 wt.% short MWCNTs outperformed all other mixes, exhibiting the largest increase in flexural strength. Generally, the reinforcing effect of the MWCNTs mainly depends on their dispersion within the matrix, which leads to the reduction of the fiber spacing in the nanocomposite. It is observed that samples containing 0.10 wt.% MWCNTs exhibit consistently lower strength than the 0.08 wt.% mixes at all ages. It is possible that effective dispersion of short MWCNTs at a concentration higher than 0.08 wt.% cannot be achieved.

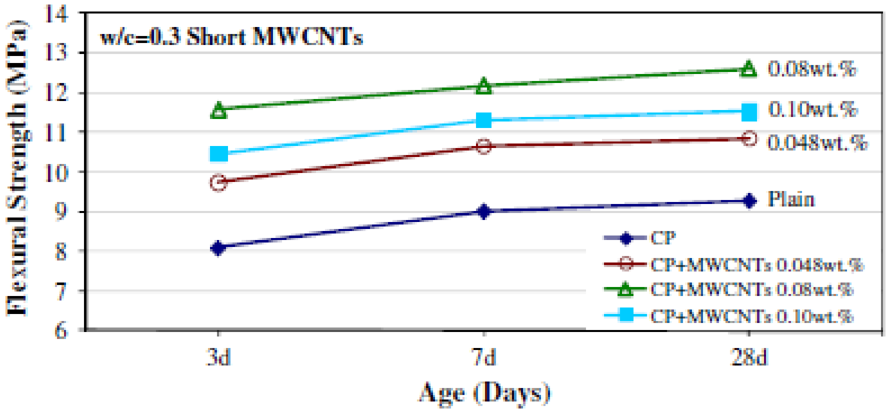


Figure 2.7: Effect of short MWCNTs concentration on the flexural strength of cement paste (w/c = 0.3)

Figure 2.8 shows the flexural strength results of specimens reinforced with 0.025 wt.%, 0.048 wt.% and 0.08 wt.% long MWCNTs. Similar to the specimens with short

MWCNTs, it was observed that in all cases, the samples reinforced with long MWCNTs show improved mechanical performance compared to the plain cement paste. However, contrary to the results obtained with the short MWCNTs, it is observed that samples reinforced with smaller amount of MWCNTs demonstrate higher flexural strength. These results are in good agreement with previous findings.

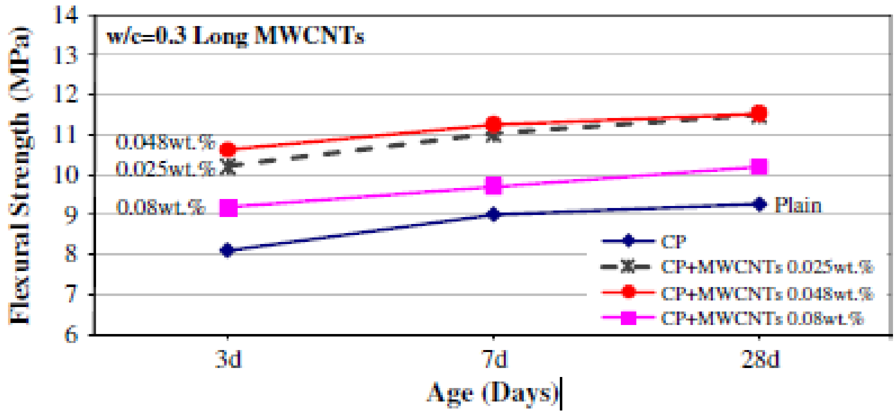


Figure 2.8: Effect of long MWCNTs concentration on the flexural strength of cement paste (w/c = 0.3)

The flexural strength of the specimens reinforced with MWCNTs shows an increase of 30–40% over plain cement specimens. This increase in the flexural strength seems to be the highest published so far with the lowest concentration of MWCNTs. Until now, the addition of the MWCNTs in cementitious matrices has resulted in either a decrease or smaller increase, up to 25%, of the flexural strength.

Therefore, the development of high-performance cementitious nanocomposites reinforced with multiwall carbon nanotubes was studied. It was found that small amounts of effectively dispersed MWCNTs (0.025–0.08 wt.% of cement) can significantly increase the strength and the stiffness of the cementitious matrix. In particular, lower amounts of long MWCNTs (0.025–0.048 wt.%) provide effective reinforcement, while higher amounts (close to 0.08 wt.%) of short MWCNTs are required to achieve the same level of reinforcement. It was also found that effectively dispersed MWCNTs provide a unique role in cement based materials.

Metaxa and Shah (2011) studied that have aimed at improving the low tensile strength, stiffness, and toughness of cementitious materials. This study also aims to show that all of these characteristics can be greatly improved by the addition of ladder scale reinforcement at the nano and micro scale. Carbon nano fibers (CNFs) as well as polyvinyl alcohol (PVA) micro fibers were used as reinforcement. The mechanical properties of the nanocomposites were investigated by fracture mechanics three-point bending test. The microstructure and the morphology of nanocomposite samples were studied using an ultra high resolution scanning electron microscope (SEM). The results clearly illustrate that the incorporation of micro fibers and nano fibers greatly improves the flexural strength, Young's modulus, and toughness of the cement matrix. In the work presented here, the characteristics of cement paste containing ladder scale reinforcement using carbon nano fibers (CNFs) and PVA micro fibers were investigated. Scanning electron microscopy (SEM) was employed to investigate the nanostructure of the nanocomposites.

Key results obtained from the study can be discussed as under:

Composites reinforced with CNFs

Table 2.8 presents the results of all mixtures tested at the age of 3, 7, and 28 days. Comparing the results of plain OPC with nanocomposites reinforced with CNFs, it is observed, that at all ages an increase in flexural strength, Young's modulus and toughness up to 40%, 75% and 35%, respectively, is achieved with the incorporation of CNFs. Typical load-CMOD curves of the 28 days response of plain cement paste and cement paste with CNFs are presented in Fig. 2.9. It is impressive that a concentration as low as 0.048wt% of cement CNFs can impose such a high increase in the Young's modulus. During the early stages of loading, nano fibers provide the material with the ability to carry higher load at the same CMOD. To better understand the effect of CNFs on the nanostructure of cement paste, SEM was employed. Figure 2.10 shows SEM images of the fracture surface of the nanocomposites at a scale of 500 nm (0.02 × 10–3 in). Initially, it is observed that mostly individual CNFs can be identified on the fracture surface. This indicates that good dispersion was achieved. It can also be seen that CNFs appear to be embedded into the hydration products, showing that good bonding between the nanofibers and the matrix was also achieved. Good bonding enables the load transfer between the matrix and the nano fibers which results to the improvement of the overall strength of the nanocomposite. A good illustration of CNFs acting as bridges between nanocracks and pores is also shown.

Table 2.8: Flexural Strength, Young's Modulus and Toughness of Cement Paste at 3, 7, and 28 days of Hydration

	3 days			7 days			28 days		
Mixture	**MOR, MPa (ksi)**	**E, GPA (ksi)**	**T, N ×mm (Ib×in×10^2)**	**MOR, MPa (ksi)**	**E, GPA (ksi)**	**T, N ×mm (Ib×in×10^2)**	**MOR, MPa (ksi)**	**E, GPA (ksi)**	**T, N ×mm (Ib×in×10^2)**
Plain OPC (CP)	3.9 (566)	5.6 (812)	1.8 (1.6)	4.9 (711)	7.4 (1073)	2.1 (1.9)	5.5 (798)	8.8 (1276)	2.3 (2.0)
CP + CNFs	5.6 (812)	9.9 (1436)	2.5 (2.2)	6.5 (943)	11.9 (1726)	2.7 (2.4)	7.2 (1044)	13.2 (1914)	2.8 (2.5)
CP + micro PVA	4.1 (595)	6.5 (943)	61.5 (54.4)	5.4 (783)	8.1 (1175)	66.2 (58.6)	5.8 (841)	8.9 (1291)	68.2 (60.4)
CP + CNFs +micro PVA	5.8 (841)	10.4 (1508)	61.8 (54.7)	6.6 (957)	12.7 (1842)	67.7 (59.9)	7.3 (1059)	14.6 (2118)	72.7 (64.3)

Note: MOR = flexural strength; E = Young's modulus; and T = toughness.

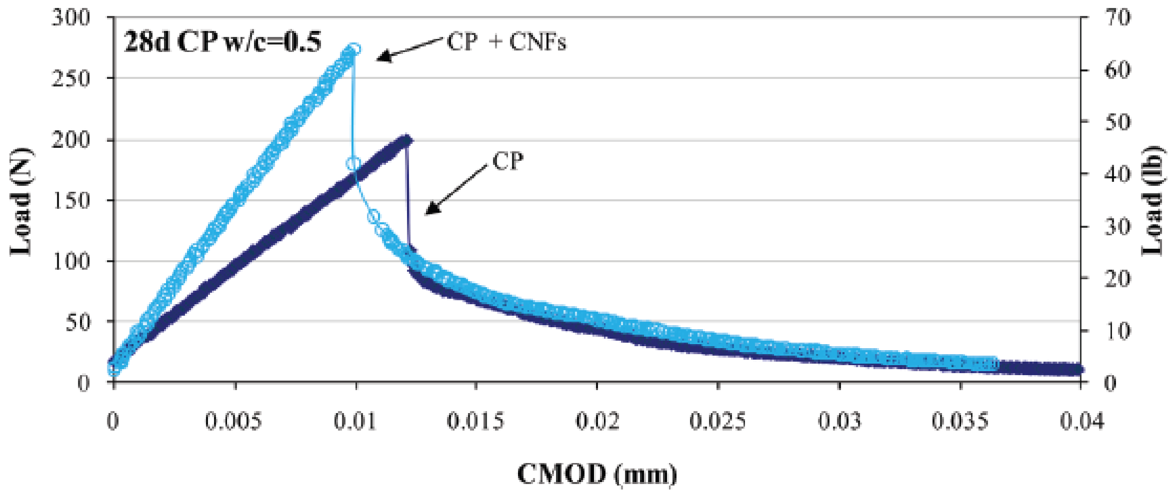


Figure 2.9: Typical load-CMOD curves of 28 days plain cement paste and cement paste reinforced with CNFs. (Note: 1 mm = 0.0394 in.)

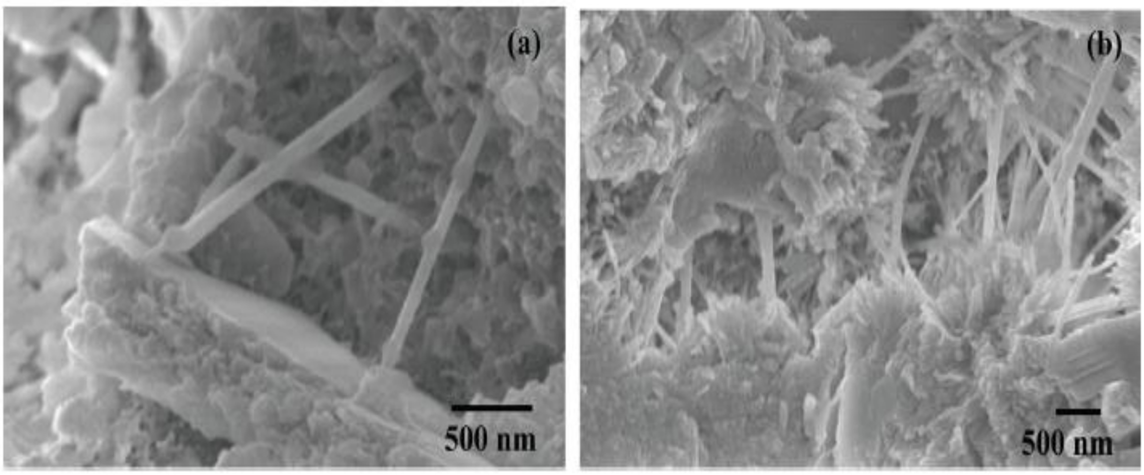


Figure 2.10: SEM images of fracture surface of cement nanocomposites reinforced with NCFs

Tyson et al (2011) presented on carbon nano tubes (CNTs) and carbon nano fibers (CNFs) are quickly becoming two of the most promising nanomaterials because of their unique mechanical properties. The size and aspect ratio of CNFs and CNTs mean that they can be distributed on a much finer scale than commonly used microreinforcing fibers. As a result, microcracks are interrupted much more quickly during propagation in a nanoreinforced matrix, producing much lower crack widths at the point of first contact between the moving crack front and the reinforcement. In this study, untreated CNTs and CNFs are added to cement matrix composites in concentrations of 0.1 and 0.2% by weight of cement. The nanofilaments are dispersed by using an ultrasonic mixer and then cast into molds. Each specimen is tested in a custom-made three-point flexural test fixture to record its mechanical properties; namely, the Young's modulus, flexural strength, ultimate strain capacity, and fracture toughness, at 7, 14, and 28 days.

Key results obtained from the study can be discussed as under:

Material

The CNFs have a diameter ranging from 60 to 150 nm, and a length of 30 to 100 μm. The CNFs have an aspect ratio (i.e., length to diameter) as high as 1,500. Table 2.9 summarizes the physical properties of the CNFs. The CNTs were MWCNTs (NC7000, Nanocyl, Sambreville Belgium) and were produced by a catalytic carbon vapor deposition (CCVD) process. They have a well-controlled diameter of 9.5 nm and an average length of 1.5 μm, with an aspect ratio of 150. The properties of the CNTs are also listed in Table 2.9. The CNTs and CNFs were used as received from the manufacturer without any surface functionalization. Previous research (Yazdanbakhsh et al. 2009) has shown that a high-range polycarboxylate-based water reducing admixture commercial superplasticizer (ADVA Cast 575, Grace Corporation, Cambridge, MA) works well to disperse the CNTs or CNFs with minimal effects on the hydration time.

Table 2.9: Physical Properties of CNTs and CNFs

	CNFs	CNTs
Diameter	60-150 nm	9.5 m
Length	30-100 μm	1.5 μm
Specific surface area	50-60 m^2/g	250-300 m^2/g
Purity	≥ 90%	≥ 90%

Preparation and Testing

Five batches of cement paste were produced. These included a reference sample of plain cement paste, two batches of cement paste with CNTs at 0.1 wt% and 0.2 wt% by weight of dry cement, and two batches with CNFs at 0.1 wt% and 0.2 wt%. Table 2.10 summarizes the composition of the five batches. These batches were labeled to indicate the components and their concentrations. The letter "F" represents CNFs, "T" represents CNTs, and "1" and "2" indicate filament dosages of 0.1 wt% and 0.2 wt%, respectively. All five batches had a water to cement ratio of 0.40, and the batches containing CNTs or CNFs had a surfactant (i.e., superplasticizer) to cement ratio of 0.005.

Table 2.10: Mix design of the test specimens

Test specimens	Water/cement ratio	CNFs:% weight of cement	CNTs:% weight of cement
Reference	0.4	0	0
F1	0.4	0.1	0
F2	0.4	0.2	0
T1	0.4	0	0.1
T2	0.4	0	0.2

Mechanical Properties

Fig. 2.11 shows the displacement at failure (i.e., peak displacement) for each batch at 7, 14, and 28 days. In Figs. 2.11–2.12, the bar represents the mean value, and the top and bottom error bars represent the third and first quartile, respectively. The addition of both CNTs and CNFs improves the peak displacement of cement paste. The greatest increase in peak displacement occurs with the addition of CNFs. A maximum increase of 150% was observed when using a concentration of 0.2 wt% CNFs. For the displacement at failure, both concentrations of CNFs outperformed CNTs. This is most likely attributed to the higher aspect ratios of the CNFs (i.e., approximately 1,000 for CNFs; 150 for CNTs), which makes CNFs more effective as reinforcements because of their larger interaction with the cement matrix. Another reason for the CNFs to outperform the CNTs is the enhanced dispersion achieved when using the CNFs. The average peak stress (i.e., average flexural strength) results are shown in Fig. 2.12. The largest increase of 82% is found at 7 days for CNFs. In many cases, the addition of CNTs shows a decrease in strength. The Young's modulus shows the same general trend as the strength. As shown in Fig. 2.13, the average modulus is less than the reference sample at both 7 and 14 days.

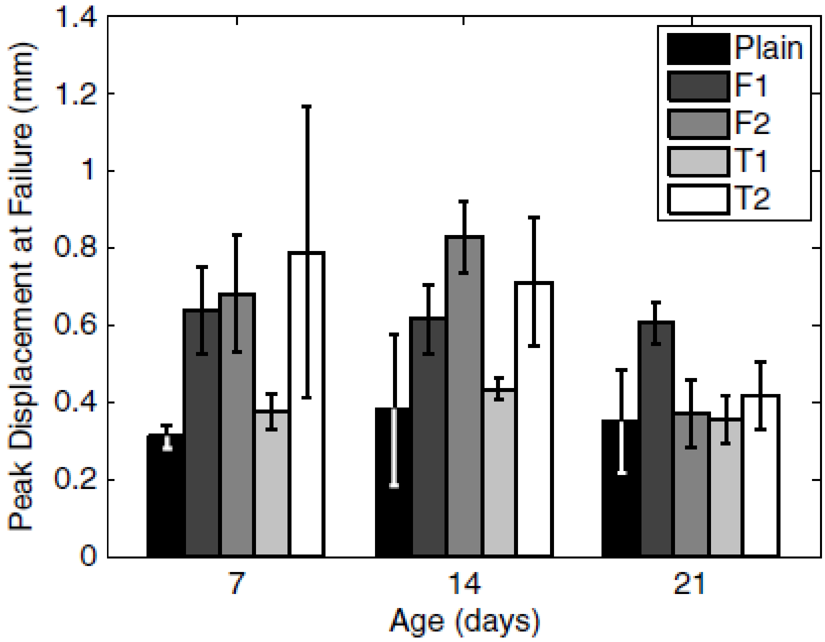


Figure 2.11: Effect of the CNFs and MWCNTs on the strain capacity of cement paste

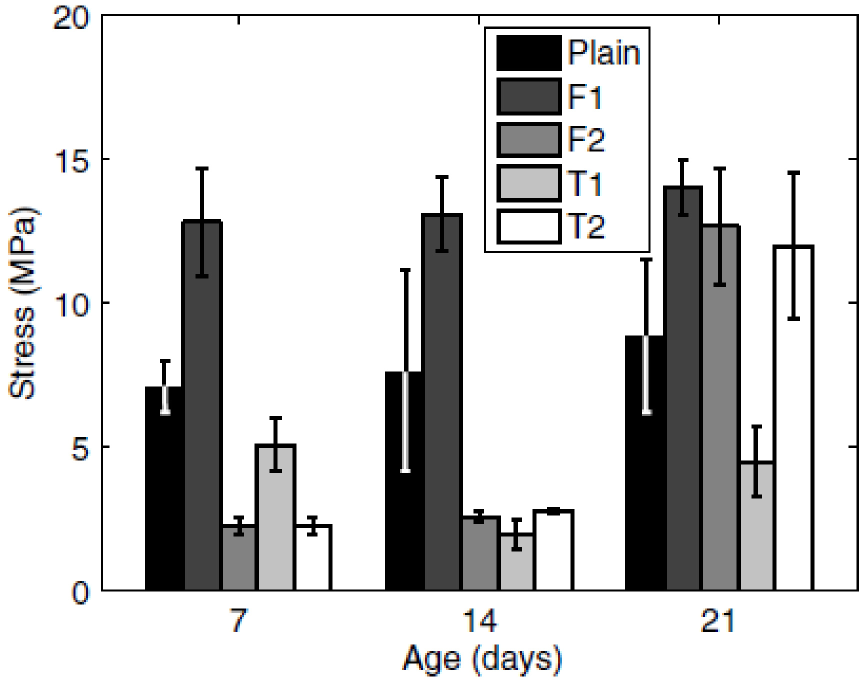


Figure 2.12: Effect of the CNFs and MWCNTs on the ultimate strength of cement paste

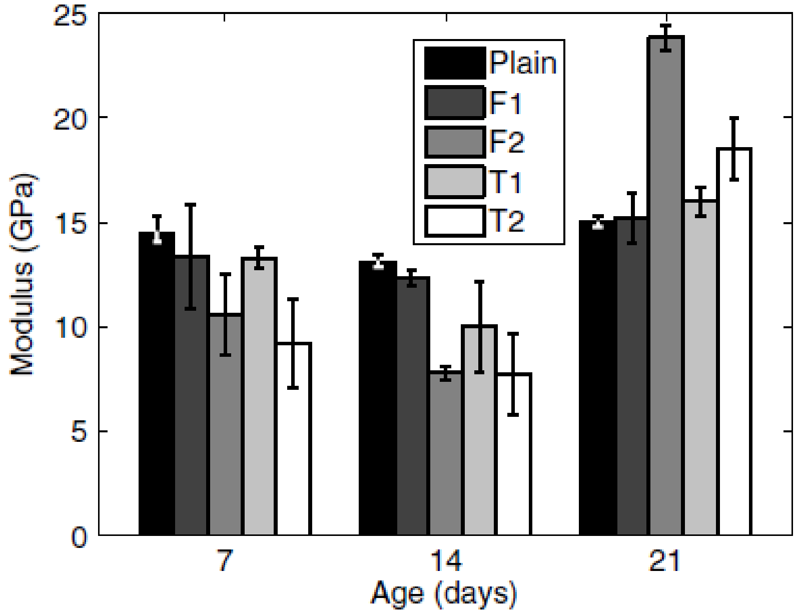


Figure 2.13: Effect of the CNFs and MWCNTs on the elastic modulus of cement paste

Kawashima and Shah (2013) stated is a summary paper on the work being done at the Center for Advanced Cement-Based Materials at Northwestern University on the modification of cement-based materials with nanoparticles, specifically nanoclays, calcium carbonate nanoparticles, and nanosilica. The rheological properties of clay-modified cement-based materials are investigated to understand the influence of nanoclays on thixotropy. The influence of the method of dispersion of calcium carbonate nanoparticles on rate of hydration, setting, and compressive strength are evaluated. And an in-depth study on the mechanisms underlying the influence of nanosilica on the compressive strength gain of fly ash–cement systems is discussed. The motivation behind these studies is that with proper processing techniques and fundamental understanding of the mechanisms underlying the effect of the nanoparticles, they can be used to enhance the fresh-state and hardened properties of cement-based materials for various applications. Nanoclays can increase the green strength of self-consolidating concrete for reduced formwork pressure and slipform paving. Calcium carbonate nanoparticles and nanosilica can offset the negative effects of fly ash on early-age properties to facilitate the development of a more environmentally friendly, high-volume fly ash concrete. Nanomodification is the manipulation of the structure at the nanoscale (less than 100 nm) to develop cement composites that exhibit enhanced or novel properties and functions. Carbon nanotubes

(CNTs) dispersed by ultrasonication can significantly improve the flexural strength of cement composites by controlling cracks at the nanoscale

Key results obtained from the study can be discussed as under:

Compressive strength

The compressive strength gain (1, 3, and 7 d) of 50 mm cube samples were compared for an OPC paste and 30% fly ash–cement pastes with and without a 5% nano$CaCO_3$ addition. All pastes had a w/b = 0.43. The results are shown in Fig. 2.14. At 3 and 7 d, the sonicated sample showed a greater improvement than the blended sample. However, neither reached the strength of the OPC sample. Work on modifying the sonication protocol to improve the stability of nano $CaCO_3$ suspensions is ongoing.

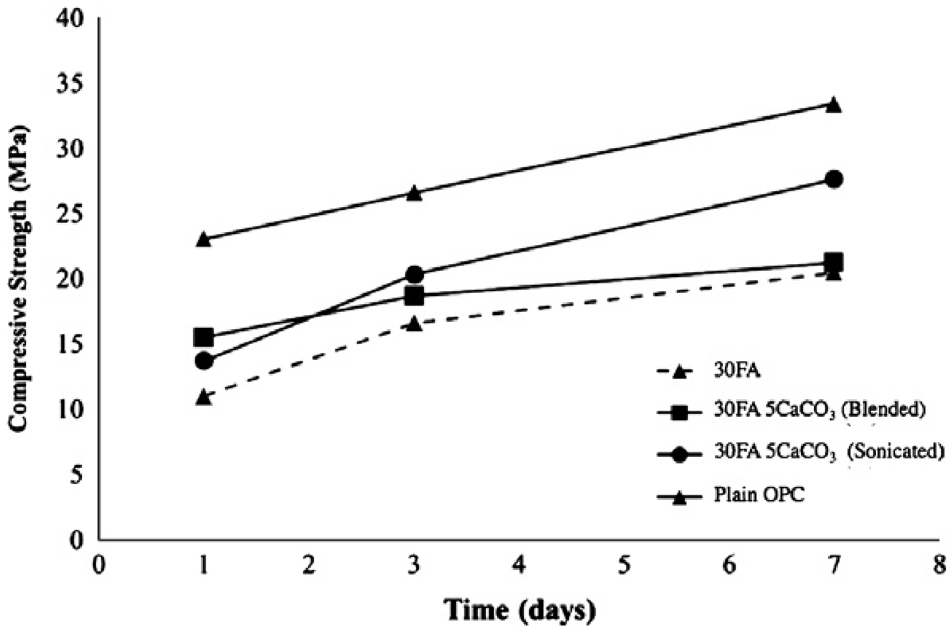


Figure 2.14: Compressive strength gain of 30% fly ash OPC pastes with and without a 5% nano$CaCO_3$ addition, prepared by blending or sonication, compared to plain OPC paste

Fresh properties

The effect of CNS dosage on the slump flow of fly ash–cement mortars is shown in Fig. 2.15. Mortars were prepared with a sand-to-binder ratio of 3 and a w/b = 0.5. River sand with a modulus of 2.8 was used. As expected, fluidity increased with fly ash replacement and decreased with CNS. The greater the amount of CNS, the greater the reduction in fluidity. It is also shown that the fluidity of CNS-added fly ash mortars with 40% and 60% of fly ash replacement are higher than that of 20% fly ash replaced mortar. This demonstrates that fly ash can help to increase the workability of CNS-added cement-based materials.

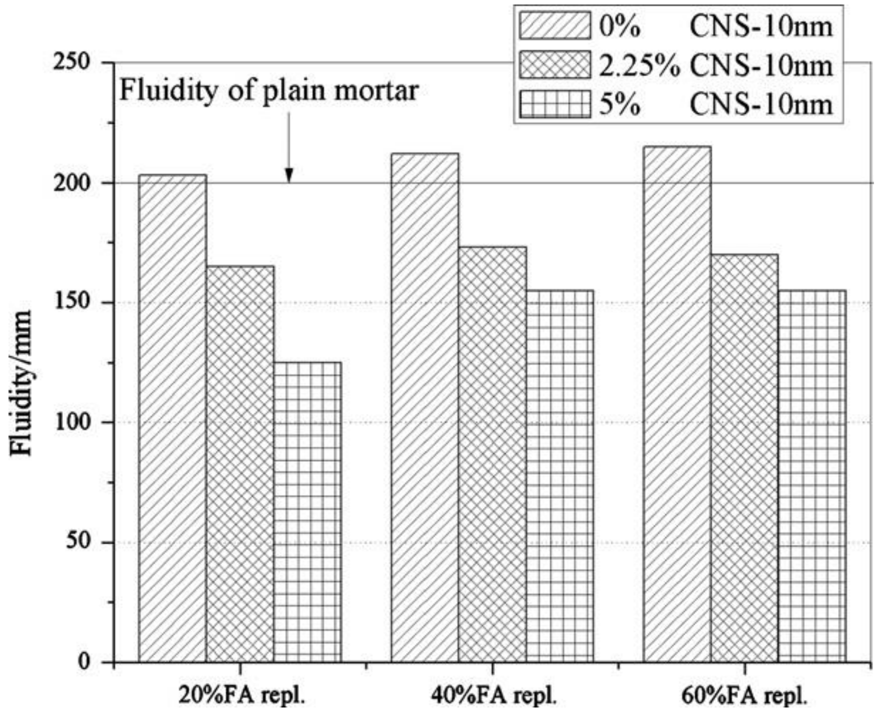


Figure 2.15: Influence of CNS on the slump flow of fly ash–cement mortar (w/b = 0.5, cement to sand ratio = 1:3)

Compressive strength

The compressive strength evolution of CNS-added cement–fly ash mortars is shown in Fig. 2.16. For the 40% fly ash–cement mortar, the addition of CNS significantly increased the strength gain early on – after 7 days, the compressive strength of 5% CNS added fly ash mortar was improved by more than 60%. However, the enhancing effect gradually decreased over time and after 3 months the compressive strength of CNS-added mortar was equal to (CNS-20 nm mortar) or less than (CNS-10 nm mortar) the control fly ash–cement mortar. This implies that nanoSiO_2 has no positive effect on the strength gain of fly ash replaced cement-based material at later ages. To determine why, the hydration and morphology of CNS-modified fly ash–cement systems were closely examined.

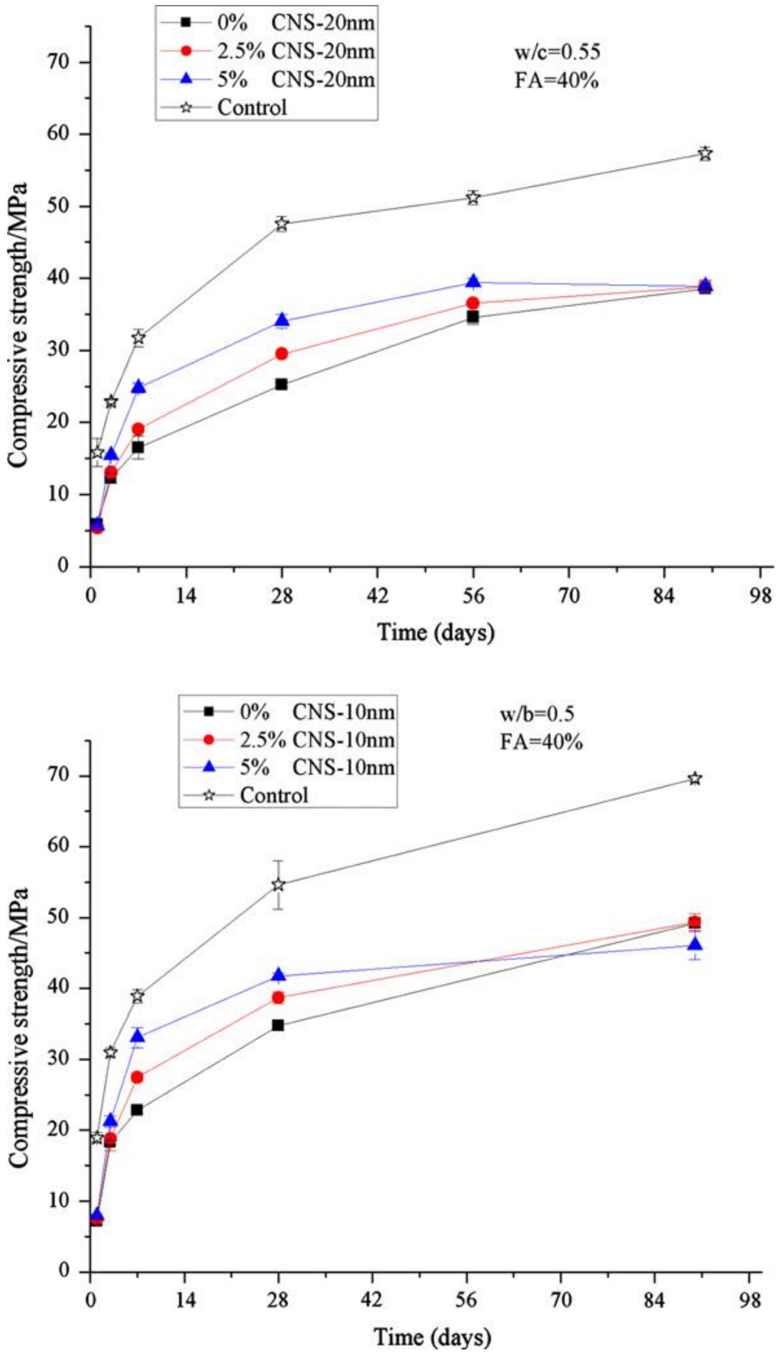


Figure 2.16: Effect of CNS on the compressive strength of fly ash mortar

Shear rheology results indicated that nanoclays have an immediate stiffening effect, governed by flocculation not water adsorption, but little influence over time. Similarly, clays have a significant effect on the instantaneous formwork response, and thereby maximum pressure, making them effective in reducing SCC formwork pressure.

Mo et al (2013) stated that Fiber research in concrete construction is an ongoing field and the use of carbon nanofibers (CNF) is examined. Fibers improve brittle materials such as concrete by enhancing tensile strength, ductility, toughness, and conductivity. Short-fiber composites are a class of strain sensor based on the concept of short electrically conducting fiber pull-out that accompanies slight and reversible crack opening. Despite the fact that nanotechnology is a relatively recent development in scientific research, the introduction of the concept is credited to Nobel Prize winner Richard Feynman from his 1959 lecture, "There's Plenty of Room at the Bottom". Feynman considered the possibility of direct manipulation of individual atoms as a powerful form of synthetic chemistry. Decades later, Feynman's concept morphed into the field of nanotechnology. Fibers arrest these cracks by forming bridges across them. With increasing tensile stress, a bond failure eventually occurs, and the fiber will pull out of the concrete allowing the crack to widen. Fig. 2.17 shows the bridging action of fibers across micro and macrocracks in concrete.

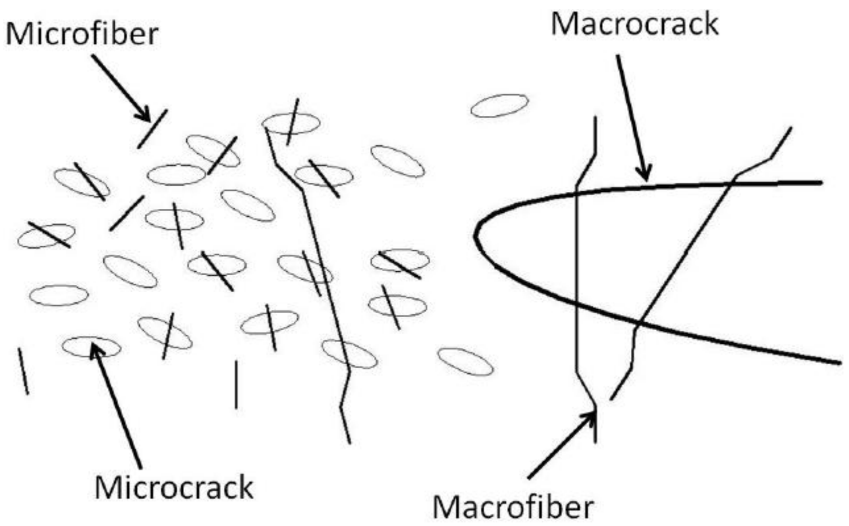


Figure 2.17: Bridging Action of Fibers Across Micro and Macrocracks

Key results obtained from the study can be discussed as under:

CNT and CNF dispersion

The majority of nanoreinforced composite research has been completed on polymers containing CNT or CNF. One of the main reasons for this is because uniform dispersion is difficult in cement-based materials. Well dispersed CNF results in uniform calcium-silicate-hydrate (CSH) gel formation, which improves the structural and electrical properties of the concrete. CNT and CNF are inherently hydrophobic and are attracted due to Van der Waals forces, causing the fibers to tend to agglomerate hindering their dispersion in solvants.

The CNF mixture is then slowly added to the mixer to gain a homogenous mix. The fresh concrete was used to create cylinders that were tested in compression. After the test, pieces of the cylinders were observed under a scanning electron microscope (SEM). The SEM showed significant CNF clumping in specimens made of normal CNF concrete and uniform distribution in SCC containing CNF, as shown in Figs. 2.18 and 2.19, respectively.

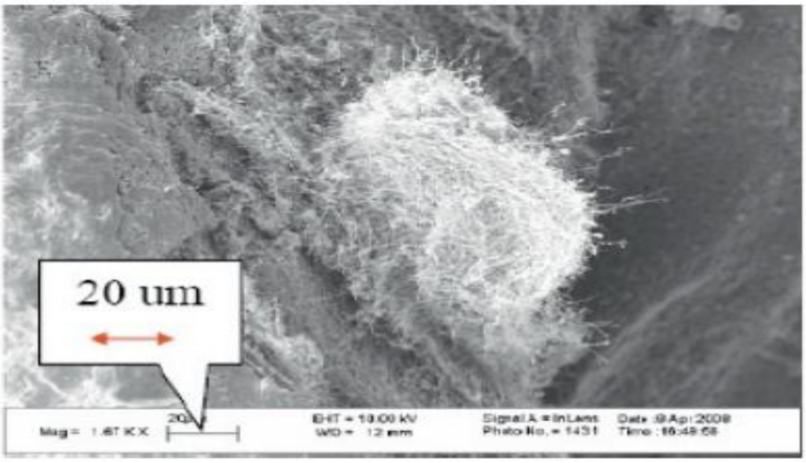


Figure 2.18: Scanning Electron Microscope Image of CNF Clump in Normal Cement (1670x Magnification)

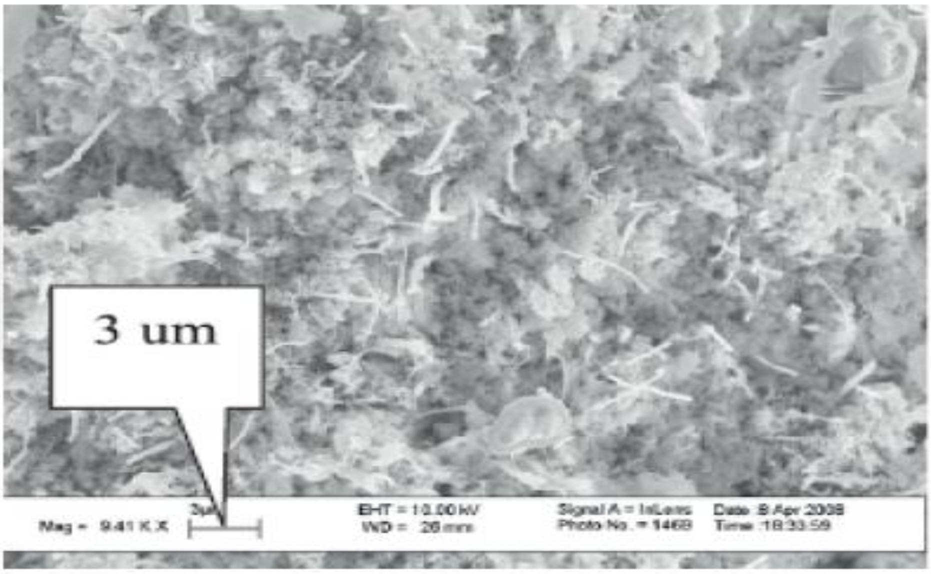


Figure 2.19: Scanning Electron Microscope Image of Well Dispersed CNF in a Uniform Self-Consolidating Cement (9410x Magnification)

CHAPTER – 3
EXPERIMENTAL PROGRAM

3.1 GENERAL

The experimental program of the research was designed to investigate the effect of addition of carbon micro fiber and carbon nano fiber in varying proportion as cement replacement in cement mortar. The effect on the mechanical and the micro structure property by the addition of varying proportions of CNF/CMF was studied. Cement mortar samples with cement sand ratio as 1:3 was prepared. CMF and CMF separately added in liquid form in various replacement of cement by weight as 0.025, 0.50, 0.075 and 0.1% respectively. Water/cement ratio was kept as 0.38.

3.2 MATERIAL USED

3.2.1 Ordinary Portland Cement

Vikram Premium Ordinary Portland Cement (43 grade) was used conforming to IS : 8112-1989 of BIS (reaffirmed 2005). Cement available in the local market is used in the research and cement used in all tests conducted is from the same batch. Physical properties are given in Table 3.1.

Table 3.1: Physical properties of Ordinary Portland cement 43 grade

Physical Property	Results obtained	IS 8112:1989
Normal Consistency	26%	–
Initial setting times (minutes)	110	Not less than 30
Final setting times (minutes)	235	Not less than 600
Fineness (Percentage)	1.5	10% max.
Specific gravity	3.12	3.15 max.
Compressive strength 3 days 7 days 28 days	 30.4 35.98 44.0	 Min. 23 N/mm^2 Min. 33 N/mm^2 Min. 43 N/mm^2

Figure 3.1: Ordinary Portland Cement 43 grade (Vikram Premium Week No. 20)

3.2.2 Fine Aggregate

Indian standard sand of three grades obtained from Tamil Nadu Ennore source was used throughout the research work. The size specification as well as technical details provided by supplier are shown in Table 3.2 and Table 3.3 were found compatible with the requirements of IS: 650-1991.

Table 3.2: Size of Indian Standard Sand

Sr. No.	Grade	Size specification of Indian standard sand Ennore
1.	Grade I	2mm to 1 mm
2.	Grade II	1mm to 0.5mm
3.	Grade III	0.5mm to 0.09mm

Table 3.3: Properties of Ennore Sand

Sr. No.	Properties	Ennore Sand used in Study
I.	**Physical Properties:**	
	Colour	Gayish white
	Specific gravity	2.64
	Absorption in 24 hours	0.30%
	Shape of grains	Sub angular
II	**Chemical Properties:**	
	Chemical analysis	
	SiO_2	99.30%
	Al_2O_3	–
	Fe_2O_3	0.10%
	CaO	–
	Loss on extraction with HCI	0.11%
	Loss of Ignition	–
III.	**Petrographic analysis:**	
	Quarts	97.40%
	Feldspar	2.50%
IV.	**Compressive strength:**	
	3 days curing	160 kg/sqcm
	7 days curing	220 kg/sqcm

Figure 3.2: Ennore Indian standard sand of three grades used in the study

3.2.3 Water

Ordinary potable water was used for both mixing and curing conforming to IS : 456-2000.

3.2.4 Carbon Nano Fiber and Carbon Micro Fiber

The two types of carbon fiber on different scale (nano to micro) were used in the study depending upon the particle size. They were procured from M/s Intelligent Material Private Ltd., Panchkula (India) – Nanoshel LLC (Wilmington DE) – USA. The samples are highly purity milled fiber with 99% with pure carbon fibers dried from temperature batch graphitization process. Nanoshel carbon nano fibers and carbon micro fibers are hydrophobic and will consequently not disperse well in water. Surfactants such as Triton – X100 and Polyisobutylane Succinimide (PIBSI) have proven to be helpful in dispersing and maintaining the CNFs in a stable suspension. The material properties of each carbon nano fiber and carbon micro fiber as furnished by supplier are given in Table 3.4. Fig. 3.3(a), (b) & (c) shows the details of CMF and CNF used in the study.

Table 3.4: Material properties of CNF and CMF as quoted in manufacture certificate of analysis

Sr. No.	Parameters	Nanoshel carbon nano fiber	Nanoshel carbon micro fiber	Remarks
1.	Carbon content (%)	99%	99%	Min. 95%
2.	Specific gravity	–	1.65	
3.	Electrical resistivity	0.00061 ohm-in	150 μΩ.m	
4.	Bulk density	2.1 gm/cm^3	–	
5.	Length	20-30 micro mtr.	90-110 micro mtr.	
6.	Dia	< 200 nm	< 8 -10 micro mtr.	
7.	Dispersant	Surfactants as Triton – X100	Surfactants as Triton – X100	Chemical and Physical properties are shown in appended certificates from manufacturer
8.	Colour	Black	Black	
9.	Specific Surface Area	> 18 m^2/gm.	–	
10.	Electrical Conductivity	> 100 s/cm.	–	
11.	PH value at 20°C	7 PH	–	
12.	Elongation to failure	20-30%	–	

Fig. 3.3(a): Dispersed Carbon Nano fiber in liquid form

Fig. 3.3(b): Carbon Micro fiber in powder form

Fig. 3.3(c): Pouring of dispersed CNF in experiment

3.3 SAMPLE PREPARATION AND PROPORTIONING OF INGREDIENTS

The proportioning of the cement mortar mix are blended in the ratio 1:3 as per IS: 4031 (Part 4) 1988. Carbon nano fiber in liquid form was properly mixed in the cement mortar. Required dispersion can be done by two methods.

3.3.1 Dispersion of CMF and CNF

(i) Dispersion into aqueous system (AM)

Surfactant such as Triton – X100 have proven to be helpful in dispersing and maintaining the CNF and CMF in a stable suspension.

(ii) Dispersion into organic solvent with ultrasonification

Ultrasonification is typically the preferred method to disperse CNF into low viscosity organic solvents. An ultrasonification time of 20 minutes at moderate intensity is recommended to maximize dispersion and minimize length reduction.

In this study, the CMF and CNF have been dispersed in water used to make cement mortar specimen. The CMF and CNF dispersed in AM system by making use of surfactant.

3.3.2 Samples for Mechanical Properties

Details of specimens used for testing of mechanical properties of compressive strength, split tensile strength and flexural strength given in Table 3.5 below:

Table 3.5: Specimen detail

Specimen	Specification (mm)	Test conducted
Mortar cubes	70.6mm × 70.6mm× 70.6mm	Compressive & split tensile strength
Mortar beam	40mm × 40mm × 160mm	Flexural beam

Details of samples retained for micro-structure analysis is given in Table 3.6 below:

Table 3.6: Samples for micro structural properties and chemical analysis

Specimen	Test conducted
Powder retained at respective days of curing	XRD
Broken piece of sample at respective days of curing	SEM

3.4 EXPERIMENTAL PROGRAMME AND METHODOLOGY

3.4.1 Mechanical Properties

Mechanical properties tests was performed to investigate the effect of incorporation of carbon nano fiber (Nanoshel) as well as carbon micro fiber (Nanoshel) individually in various batches. 0%, 0.025%, 0.050%, 0075% and 0.1% by cot of cement to be mixed cube mortar. In mechanical properties, compressive strength was investigated at the ages of 3 days, 7 days and 28 days, while split tensile strength and flexural strength was investigated at the ages of 7 days, 14 days and 28 days. Incase of compressive strength and split tensile strength the loading rate was taken on 70 kN/min and in case of flexural strength, the loading rate was taken on 2.65 kN/min. In addition to this bulk density and water absorption test were also conducted done at age of 28 days of curing for durability study.

The details of test matrix and nomenclature used for various additions of CMF and CNF are given in Tables 3.7 and 3.8 below:

Table 3.7: Details and nomenclature of cement mortar samples with CNF chemical

Details of addition of CNF in cement mortar by wt. of cement	Designed name for reference
0% carbon nano fiber/plain CM	CM_1 (Control)
0.025% carbon nano fiber	CNF M_2
0.050% carbon nano fiber	CNF M_3
0.075% carbon nano fiber	CNF M_4
0.1% carbon nano fiber	CNF M_5

Table 3.8: Details and nomenclature of cement mortar samples with CMF chemical

Details of addition of CMF in cement mortar by wt. of cement	Designed name for reference
0% carbon micro fiber/plain CM	CM_1 (Control)
0.025% carbon micro fiber	CMF M_2
0.050% carbon micro fiber	CMF M_3
0.075% carbon micro fiber	CMF M_4
0.1% carbon micro fiber	CMF M_5

3.4.2 Proportioning of Ingredients

The proportion of the cement sand is in ratio of 1:3. The amount of replacement in cement by CMF/CNF varies as percentages 0.025%, 0.050%, 0.075% and 0.1% by weight of cement respectively. The mix proportions of various mixes used in the study are designated as in Tables 3.9 & 3.10 for compressive strength, split tensile strength and flexural testing beam.

Table 3.9: Mix proportions for all mixtures prepared for compressive strength and split tensile strength (CMF/CNF)

Sr. No.	Designated Mix No.	Cement (gm)	Standard sand (gm)	Water (ml)	CMF/CNF (ml)	Remarks
1.	CM_1	200.00	600	76	Nil	0 % CNF
2.	CMF/CNF M_2	199.95	600	71	5	0.025 % CNF
3.	CMF/CNF M_3	199.90	600	66	10	0.050% CNF
4.	CMF/CNF M_4	199.85	600	61	15	0.075% CNF
5.	CMF/CNF M_5	199.80	600	56	20	0.1% CNF

Table 3.10: Mixing proportion for all mixtures prepared for flexural strength (CNF)

Sr. No.	Designated Mix No.	Cement (gm)	Standard sand (gm)	Water (ml)	CMF/CNF (ml)	Remarks
1.	CM_1	160.00	480	60.80	0	0 % CNF
2.	CMF/CNF M_2	159.96	480	56.80	4	0.025 % CNF
3.	CMF/CNF M_3	159.92	480	52.80	8	0.050% CNF
4.	CMF/CNF M_4	159.88	480	48..80	12	0.075% CNF
5.	CMF/CNF M_5	159.84	480	44.80	16	0.1% CNF

3.4.3 Chemical Composition and Micro Structure of Cement Mortars

The chemical compositions of the samples are investigated using X-ray diffractometer analysis (XRD) with Cu-radiation. In addition to this 30 kV and voltage of 40 mV was used with diffraction intensity in the range of 10-80°. The micro structure of the cement mortar samples are examined by using scanning electron microcopy (SEM) analysis which is used to identify the changes which occur in the microstructure including the formation and deformation of phases in the cement mortar sample.

3.4.4 Flow Chart

Flow chart of experimental programme of the research work is shown in Figure 3.4 below.

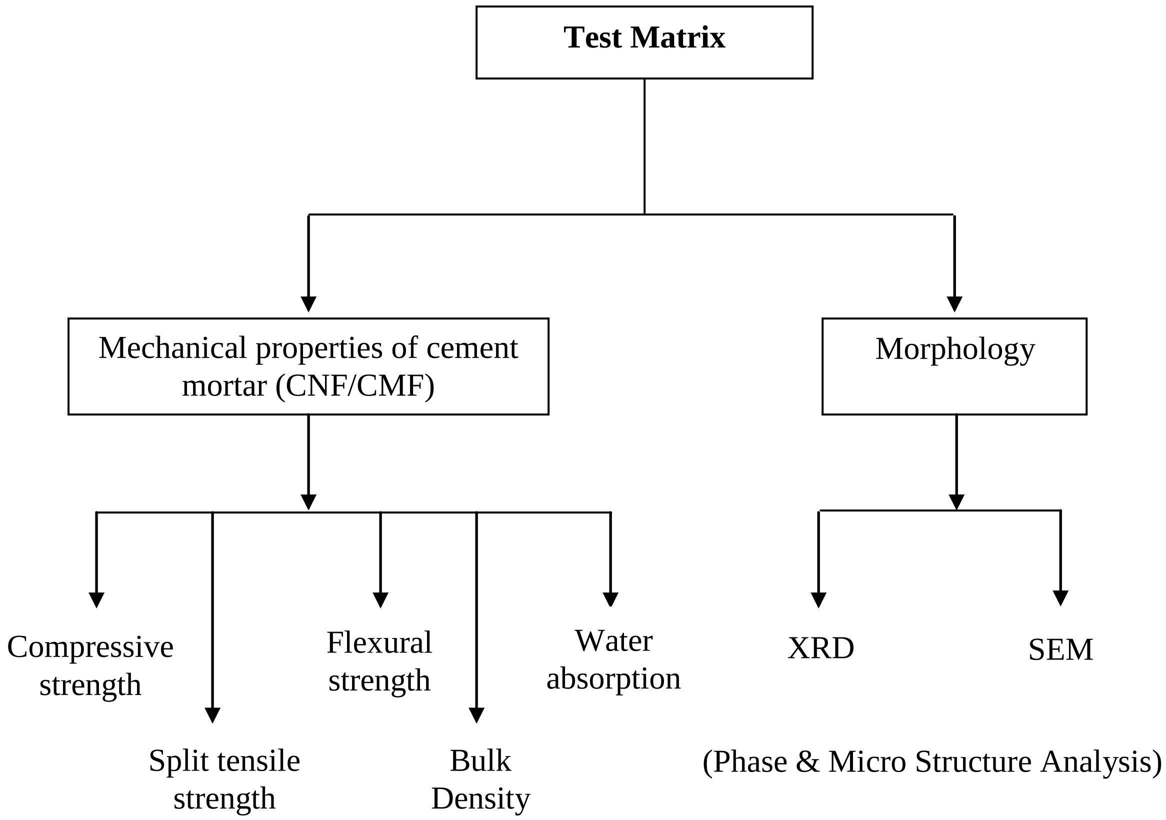


Figure 3.4: Flow Chart

3.4.5 Casting of Specimen

All specimen were cast according to the mix proportions in section discussed. For these mix proportions required quantities of material were weighed. Standard cubes of testing 70.6mm × 70.6mm × 70.6mm were used for compressive strength & split tensile strength. In addition to that size of 40mm × 40mm × 160mm were used for flexural strength as per provision of ASTM standard. The mixing procedure adopted as follows:

- Before casting the mould should be properly cleaned and oiled. The screw should be tightened properly in the perfect dimension before casting. Care should be taken that no gap should be left in the mould so that mortar start coming out.
- The three grade of standard sand used should be weighed with perfect accuracy alongwith cement.
- Clean appliances were used for mixing and portable was used in preparing the cubes & beams.

- The cement and standard sand were dry mixed in a tray for about 10 minutes. A uniform mix was obtained.
- The material for each mix 18 cubes (70.6mm × 70.6mm × 70.6mm), 9 for compressive strength for 3, 7 and 28 days and 9 for split tensile strength for 7, 14 and 28 days were weighed. For the same mix 9 beams (40mm × 40mm × 160mm) for flexural strength for 7, 14 and 28 days were prepared for control mix as well as for 0.025%, 0.050%, 0.075% and 0.1% respectively CMF/CNF.
- The material for each batch was mixed separately and the quantity of cement, standard sand and water used was cement (200gm), sand (600gm) and water $\left(\frac{P}{4}+3\right)$ percentage of combined mass of cement and sand, where P is the percentage of water required to produce a paste of standard consistency determined as describe in IS: 1403 (Part 4) 1988.
- Place on a non porous plate, a mixture of cement and standard sand. Repeat the procedure by using CMF also.
- Dry material was mixed with a trowel for one minute and then with water as well as dispersed CMF and CNF in liquid form. Until the mixture was of uniform colour.
- The time of mixing was in any event be not less than 3 minutes. If time taken to obtain the uniform colour exceed 5 minutes than reject the mixture and entire operation was repeated with a fresh quantity of cement, sand, water and CMF/CNF.
- The casting immediately followed mixing the top surface of the specimen was scrapped to remove excess material and achieve smooth finish. The specimen were removed from moulds after 24 hours and cured in water till testing as per requirement of test. Figures 3.5(a) – 3.5(d) shows the various steps of casting procedure.

Figure 3.5: (a) Pouring of carbon fiber colloidal form during mixing of material

Figure 3.5: (b) Mixing of material for mortar cubes in mixture machine

Figure 3.5: (c) Mixing of material for mortar cubes

Figure 3.5: (d) Vibration of moulds filled by mortar in vibrating table

3.5 TEST CONDUCTED

3.5.1 Compressive Strength Test (IS: 4031: Part 6)

The compressive strength test is performed on cubes to determine compressive strength at various ages.

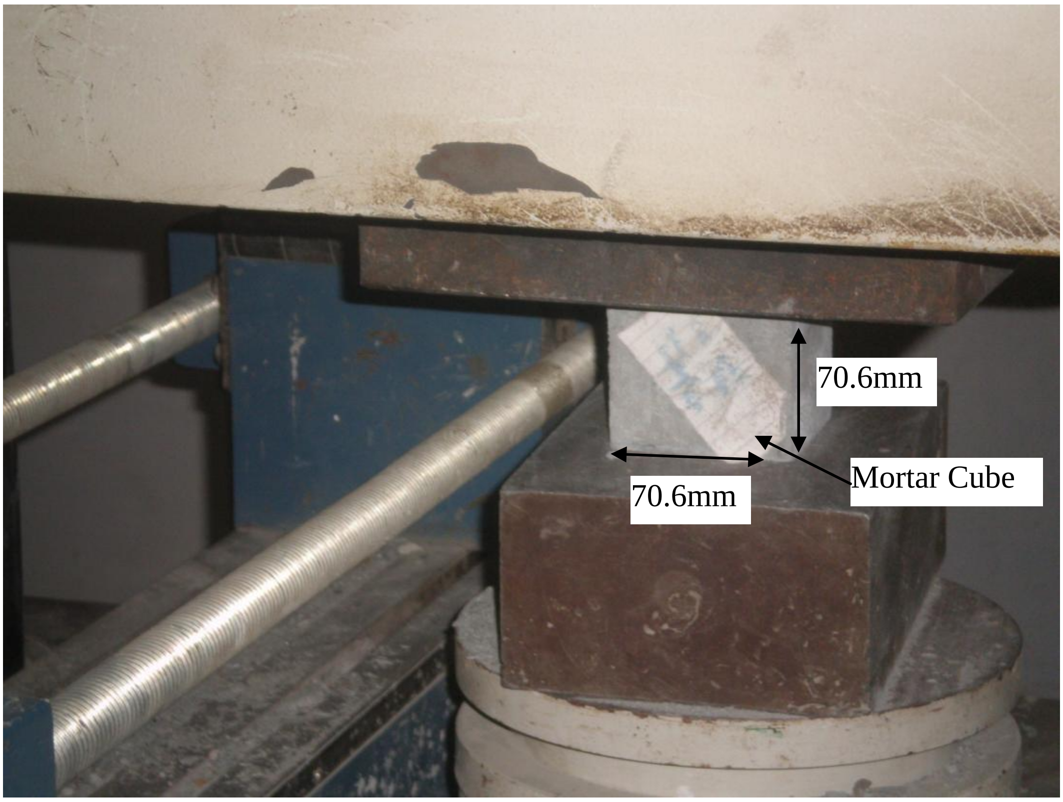


Figure 3.6: Compressive strength test on cube

Testing Machine: The testing equipment used was universal Testing Machine (Hung Ta Instrument Co. Ltd.) of 1000 kN capacity (Fig. 3.6). The rate of loading for compressive strength test is 70kN/min. The permissible error shall be not more than ± 2 percent of the maximum load. The testing machine is equipped with two steel bearing platens with hardened faces. One of the platens (preferably the one that normally will bear on the upper surface of the specimen) shall be fitted with a ball seating in the form of a portion of a sphere, the centre of which coincides with the central point of the face of the platen. The other compression platen was plain rigid bearing block. The bearing faces of both platens shall be at least as large as, and preferably larger than the nominal size of the specimen to which the load is applied.

Age at test

Compressive strength tests made at recognized ages of the test specimens, the most usual being 3, 7 and 28 days. Test at age of 56 days can also be performed. Where it may be necessary to obtain the early strengths, tests may be made at the ages of 24 hours ± ½ hour and 72 hours ± 2 hours. The ages shall be calculated from the time of the addition of water to the dry ingredients.

Number of specimens

At least three specimens, preferably from different composition, shall be made for testing at each selected ages of 3, 7 and 28 days.

Formula used

$$\text{Compressive Strength} = \frac{P \times 1000}{70.6 \times 70.6} \quad \text{N/mm}^2 \qquad \text{....(3.1)}$$

where

P = Load at fracture (kN) in compressive strength test

3.5.2 Flexural Strength Test

The flexural strength test is performed on 40mm × 40mm × 160mm mortar beams in two point loading to determine the flexural strength at various ages of curing of samples prepared by varying proportions of CMT/CNF in CM.

Testing Machine: The testing machine may be of any reliable type, of sufficient capacity for the tests and capable of applying the load at the rate specified which is 2.65 KN/min for 40mm × 40mm ×160mm size of mould. The permissible error shall be not more than ± 2 percent of the maximum load. The testing machine was equipped with two steel bearing platens with hardened faces. The specimen of beam sample was marked in the centre with offset of 20mm on both sides. The above specimen was installed and aligned over the ball seating in the form of a portion of a sphere, the centre of which coincides with the central point of the face of the platen. The arrangement of testing is shown in the Figure 3.7.

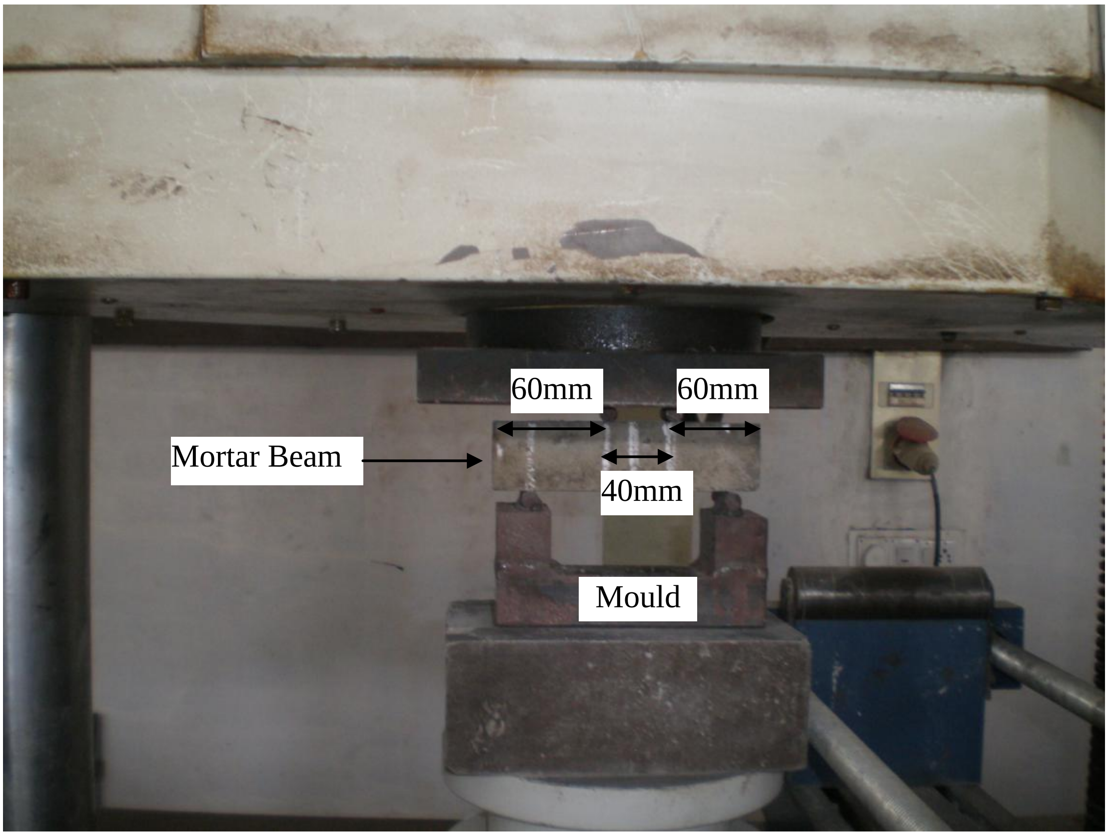


Figure 3.7: Beam under two point loading

Age at test

Test is conducted at 7, 14 and 28 days of curing of specimen. The ages shall be calculated from the time of the addition of water to the dry ingredients.

Number of specimens

At least three specimens, preferably from different composition, were prepared made for testing at each selected ages of 7, 14 and 28 days.

Formula used

f_t (Flexural strength/ Modulus of rupture) = $\frac{M}{Z}$

$$= \frac{(P \times x) \times 6}{bd^2}$$

$$f_t = \frac{(P \times x) \times 6 \times 1000}{bd^2} \text{ N/mm}^2 \qquad \text{....(3.2)}$$

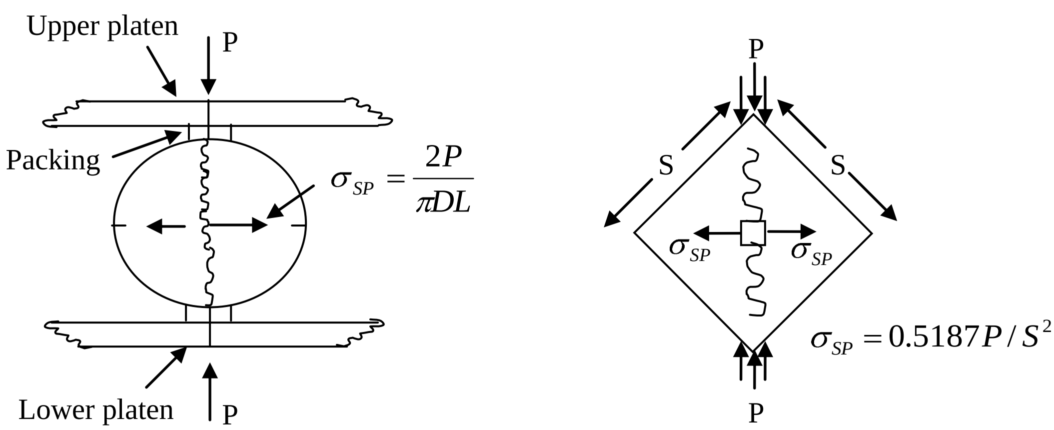
where

P = Load at which beam fails (kN)

3.5.3 Split Tensile Strength Test

The test consist of various methods two types of methods are discussed :

The split tests are well-known indirect tests used for determining the tensile strength of concrete, sometimes referred as the splitting tensile strength of concrete. The test consists of applying compressive line loads along the opposite generators of a concrete cylinder placed with its axis horizontal between the platens. Due to the applied line loading a fairly uniform tensile stress is induced over nearly two-third of the loaded diameter as obtained from an elastic analysis. The magnitude of this tensile stress (acting in a direction perpendicular to the line of action of applied compression) is given by $2P/\pi DL = 0.637P/DL$, where P is the applied load, and D and L are the diameter and length of the cylinder, respectively. Due to this tensile stress, the specimen fails finally by splitting along the loaded diameter and knowing P at failure, the tensile strength can be determined. The test can also be performed on cubes by splitting either (i) along its middle parallel to the edges by applying two opposite compressive forces through 15mm square bars of sufficient length or (ii) along one of the diagonal planes by applying compressive forces along the opposite. In the case of side-splitting of the cubes, as tensile strength is determined from $0.642P/S^2$ and in diagonal splitting it is determined from $0.5187\ P/S^2$, where P is the load at failure and S is side of the cube. In this case the test condition was the adopted as diagonal splitting as determined from $.5187\ P/S^2$ as shown in the Figure 3.8.



(a) Split strength testing of cylinder

(b) Split strength testing of cubes

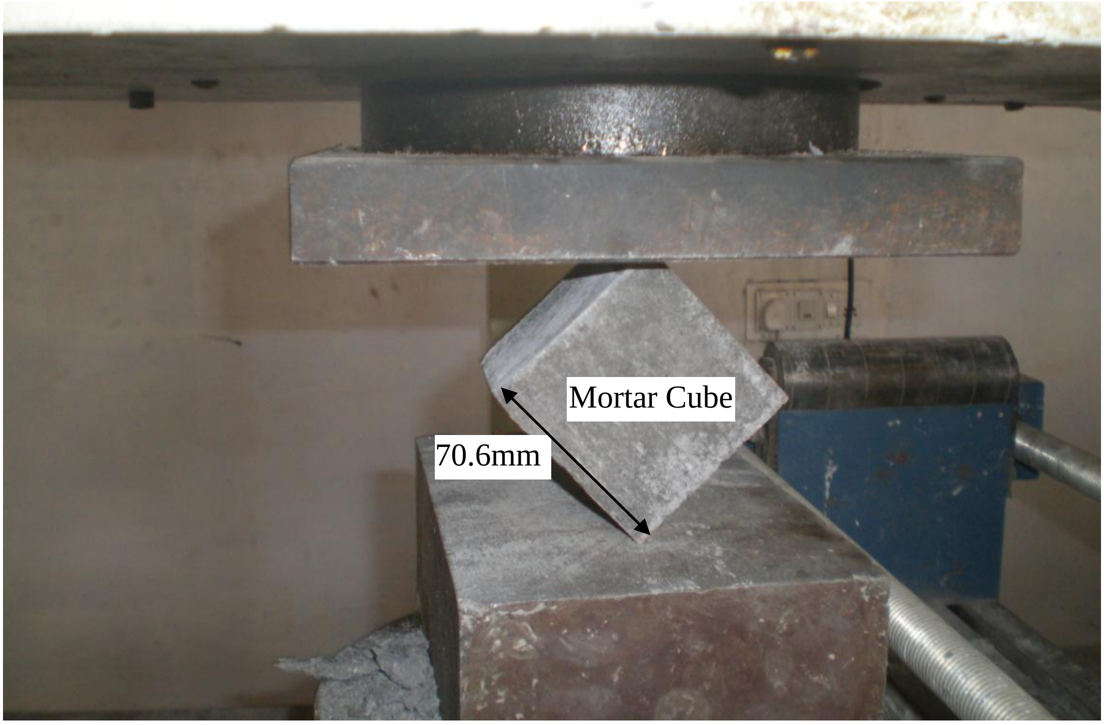


Figure 3.8: Cube under split tensile test

Age at test

Tests were conducted at recognized ages of the test specimens, the most usual being 7, 14 and 28 days. The ages shall be calculated from the time of the addition of water to the dry ingredients.

Number of specimens

At least three specimens, preferably from different composition, shall be made for testing at each selected ages of 7, 14 and 28 days.

Formula used

Split tensile strength = $\frac{0.5187P}{S^2}$

where

P = Load at fracture

S = Side of cube

3.6 CHARACTERIZATION TECHNIQUES

3.6.1 X-ray diffraction (XRD)

X-ray investigation in the powder form samples were characterized by X-ray diffractometer with CuK_α radiation for the identification of existing phases, crystal structure, lattice parameter of the crystalline solids. The sample is irradiated with monochromatic X-rays and the counters record the reflected radiation. The X-ray diffraction pattern were recorded using Bruker's diffractogram with CuK_α (λ=1.54Å) obtained from Cu target using an inbuilt Ni filter. The 2 values for XRD patterns, in the range of 10^o-90^o. The X-ray diffraction peaks were identified using Powder Diffraction Files (PDF) as shown in Figure 3.9.

Figure 3.9: XRD equipment used in the study [SAI Lab)

3.6.2 Scanning Electron Microscopy (SEM)

SEM is conducted to study the micro-structure properties of the samples. The samples which are cured for 3 days, 7 days and 28 days are used for the compressive strength test. This test is used to identify the changes which had occurred inside the micro-structure and also the formation and deformation of the phases as shown in Figure 3.10.

Figure 3.10: SEM equipment

3.7 CLOSING REMARKS

In this chapter, the experimental details of various tests and the methodology for investigating effect of carbon nano fiber and carbon micro fiber on mechanical, chemical composition and micro-structure properties have been discussed. In the following chapter the experimental results obtained is presented and discussed.

CHAPTER – 4

RESULTS, ANALYSIS AND DISCUSSIONS

4.1 GENERAL

This chapter represents details of the results and analysis of the experimental work carried out in the study by replacement of carbon micro fiber and carbon nano fiber in cement mortar for the various percentages by weight of cement and discussed. The results and analysis has been categorized into fresh cement mortar properties in control mix, hardened cement mortar property such as compressive strength, split tensile strength, flexural strength, bulk density & water absorption alongwith micro structural analysis using SEM as well as chemical compression with XRD in control as well as with carbon nano fiber/carbon micro fiber. Two different types of carbon fiber on various sizes as ‘Carbon nano fiber’ and ‘Carbon micro fiber’ were used in colloidal form as additive to CM.

4.2 COMPRESSIVE STRENGTH TEST RESULTS

In order to study the effect of addition of nanoshel carbon fiber in colloidal form in different proportions in cement mortar, as 0.025%, 0.050%, 0.075% and 0.1% by wt. of cement. Nanoshel carbon micro fiber is colloidal form in the same proportion as replacement in cement mortar by 0.025%, 0.050%, 0.075% and 0.1% by wt. of cement is carried out. All specimen of carbon (70.6 mm × 70.6mm × 70.6mm) are cured in water and tested at age of 3 days, 7 days and 28 days with our and with addition of CMF and CNF.

4.2.1 CMF Replacement

Perusal of Table 4.1 shows compressive strength of modified mortar at optimize use of 0.05% by weight of cement progressively increases by 9.05% in 3 days and 10.25% in 7 days and 10.49% in 28 days (age of curing) compared to control sample. However, compressive strength decreases at 0.1% of CNF by 10.46% in 3 days, 6.19% 7 days and 10.4% 28 days. The above characteristics are presented graphically in Figure 4.1 comparison of compressive strength.

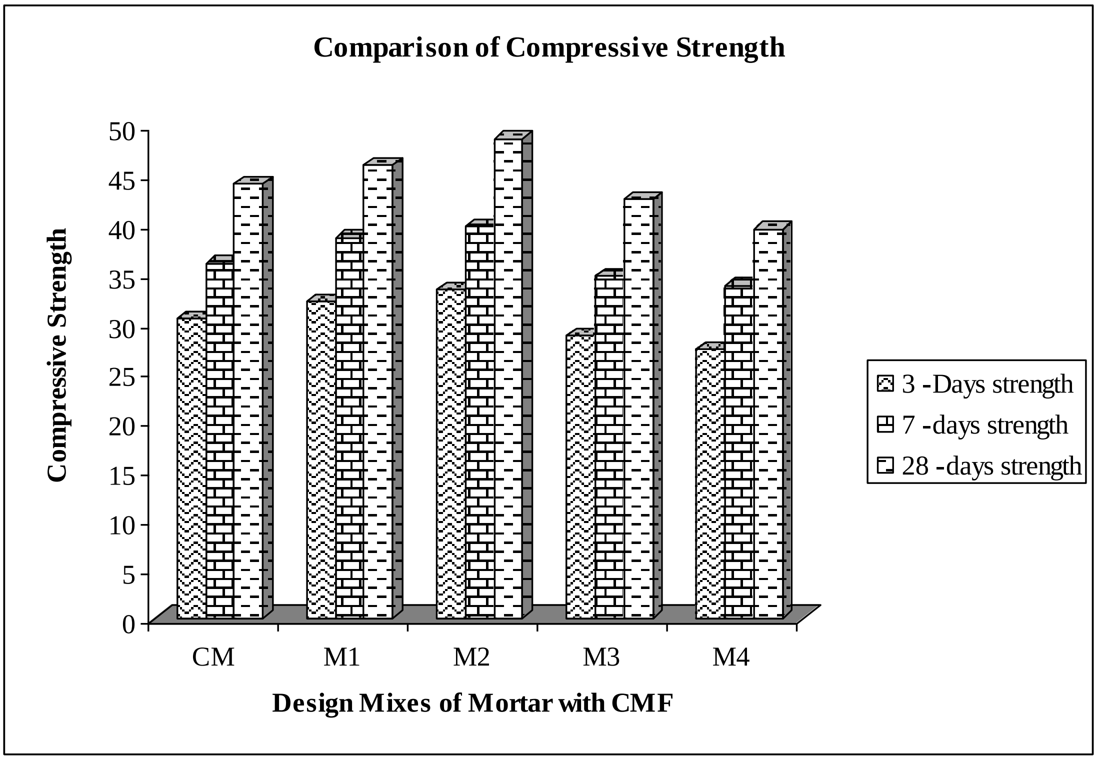
The trend of compressive strength results shows that there is a decrease in compressive strength for each mix after 0.075% of carbon micro fiber compare to control sample test results. Possibility of the variation may be due to the high water demand, segregation of carbon micro fiber at high dosages and improper mixing in cement matrix and no super plasticizer used.

Table 4.1: Compressive Strength of Cement Mortar in Control Mix & Cement Replacement with Carbon Micro Fiber at 3, 7 and 28days for Various Mixes

Sr. No.	Mix	Compressive Strength of Mortar (MPa)					
		3 -Days strength	% variation	7 -days strength	% variation	28 -days strength	% variation
1	CM	30.40	Reference	35.98	Reference	44.00	Reference
2	M1	32.04	+5.39	38.56	+7.17	45.94	+4.40
3	M2	33.28	+9.05	39.67	+10.25	48.61	+10.49
4	M3	28.67	–5.69	34.72	–3.5	42.44	–3.54
5	M4	27.22	–10.46	33.75	–6.19	39.42	–10.4

Note: (+) indicates increase and (–) indicate decrease in compressive strength

Figure – 4.1



4.2.2 CNF Replacement

Perusal of Table 4.2 shows compressive strength of modified mortar at optimize use of 0.05% by weight of cement progressively increases by 11.90% in 3 days and 15.64% in 7 days and 17.23% in 28 days (age of curing) compared to control sample. However, compressive strength decreases at 0.1% of CNF by 9.67% in 3 days, 12.35% in 7 days and 8.79% in 28 days. The above characteristics are presented graphically in Figure 4.2 comparison of compressive strength.

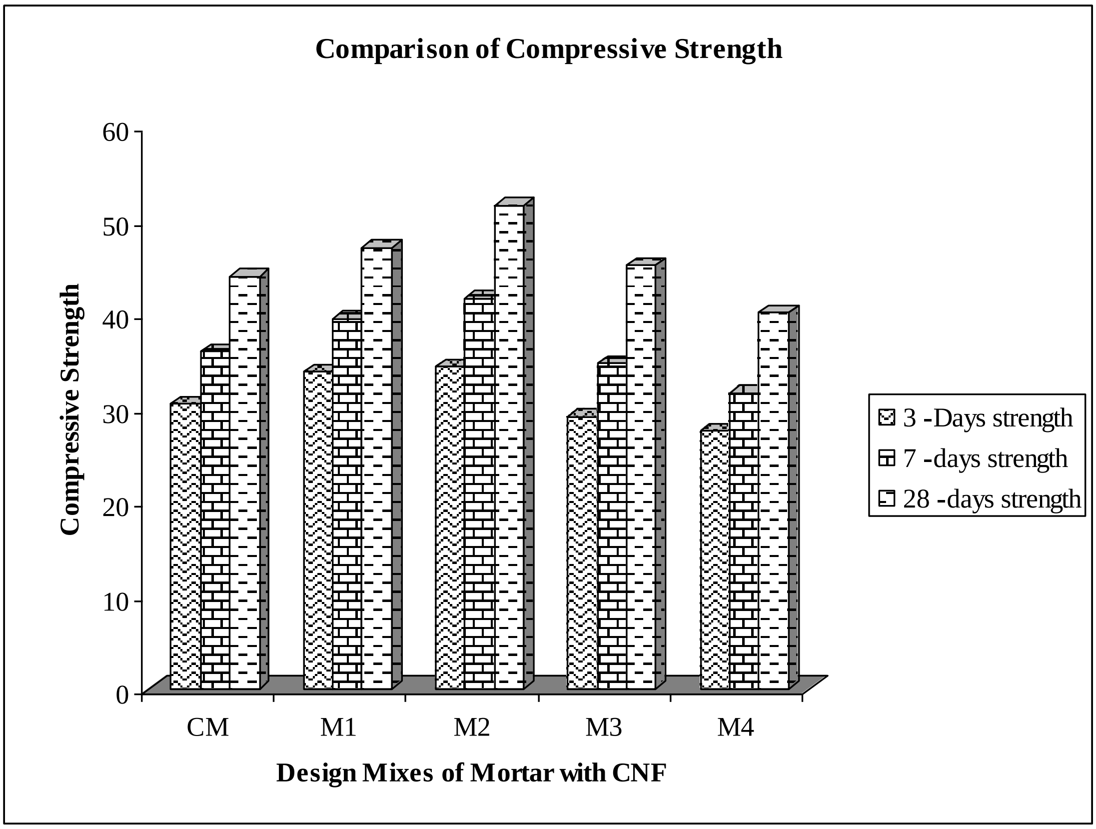
The trend of compressive strength results shows that there is a decrease in compressive strength for each mix after 0.075% of carbon micro fiber compare to control sample test results. Possibility of the variation may be due to the high water demand, segregation of carbon micro fiber at high dosages and improper mixing in cement matrix and no super plasticizer used. The reason behind this high result is due to small particle size and low percentage of colloidal carbon nano fiber which promote the pozzolonic reaction and fill the pores resulting in high compressive strength. The decrease of strength of various mixture at 0.075% and 0.1% (M_3 and M_4) could be attributed to the same reason as mentioned earlier.

Table 4.2 : Compressive Strength of Cement Mortar in Control Mix & Cement Replacement with Carbon Nano Fiber at 3, 7 and 28days for Various Mixes

Sr. No.	Mix	Compressive Strength of Mortar (MPa)					
		3 -Days strength	% variation	7 -days strength	% variation	28 -days strength	% variation
1	CM	30.40	Reference	35.98	Reference	44.00	Reference
2	M1	33.77	+11.08	39.49	+9.75	47.07	+6.81
3	M2	34.30	+11.90	41.61	+15.64	51.58	+17.23
4	M3	28.97	–4.70	34.71	–3.54	45.12	+2.53
5	M4	27.46	–9.67	31.53	–12.35	40.13	–8.79

Note: (+) indicates increase and (–) indicate decrease in compressive strength

Figure – 4.2



4.3 SPLIT TENSILE STRENGTH

4.3.1 CMF Replacement

Split tensile strength studies were carried out at the age of 7, 14 and 28 days. Test results are given below in Table 4.3.

As seen in the table, the test results indicated the addition of CMF at different percentages that the 0.05% replacement of cement displays better split strength test as compared to other replacement in all 7 days, 14 days and 28 days. Perusals of the split tensile strength in control mix and cement replacement with carbon micro fiber at all ages of curing depicts that there is increase of strength of 12.87% at 3 days, 24.42% at 14 days and 26.13% at 28 days. This means that increasing the percentage of carbon micro fiber is useful in increasing strength to a certain limit after which any increasing in the carbon micro fiber leads to decrease in the tensile strength.

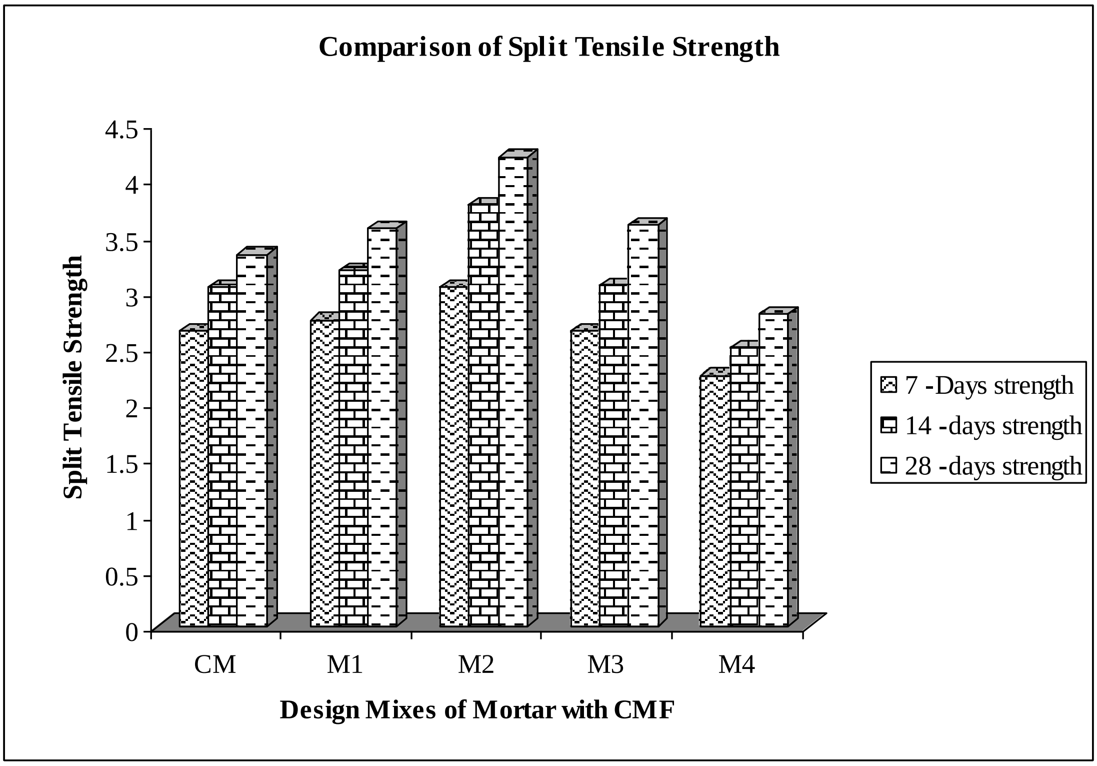
It can be concluded the small changes in the percentage of carbon micro fiber levels to dramatic changes in the split tensile strength which employ a message to researcher that a high level of quality control should be attend to control is changes.

Table 4.3: Split Tensile Strength of Cement Mortar in Control Mix & Cement Replacement with Carbon Micro Fiber at 7, 14 and 28days for Various Mixes

Sr. No.	Mix	Split Tensile Strength of Mortar (MPa)					
		7 -Days strength	% variation	14 -days strength	% variation	28 -days strength	% variation
1	CM	2.64	Reference	3.03	Reference	3.33	Reference
2	M1	2.74	+3.78	3.18	+4.95	3.56	+6.90
3	M2	3.03	+12.87	3.77	+24.42	4.20	+26.13
4	M3	2.64	0.00	3.05	–0.65	3.59	+7.80
5	M4	2.24	–15.15	2.49	–17.82	2.79	–16.21

Note: (+) indicates increase and (–) indicate decrease in split tensile strength

Figure – 4.3



4.3.2 CNF Replacement

Split tensile strength studies were carried out at the age of 7, 14 and 28 days. Test results are given below in Table 4.4.

As seen in the table, the test results indicated the addition of CNF at different percentages that the 0.05% replacement of cement displays better split strength test as compared to other replacement in all 7 days, 14 days and 28 days. Perusals of the split tensile strength in control mix and cement replacement with carbon micro fiber at all ages of curing depicts that there is increase of strength of 20.45% at 3 days, 31.35% at 14 days and 31.83% at 28 days. This means that increasing the percentage of carbon nano fiber is useful in increasing strength to a certain limit after which any increasing in the carbon nano fiber leads to decrease in the tensile strength.

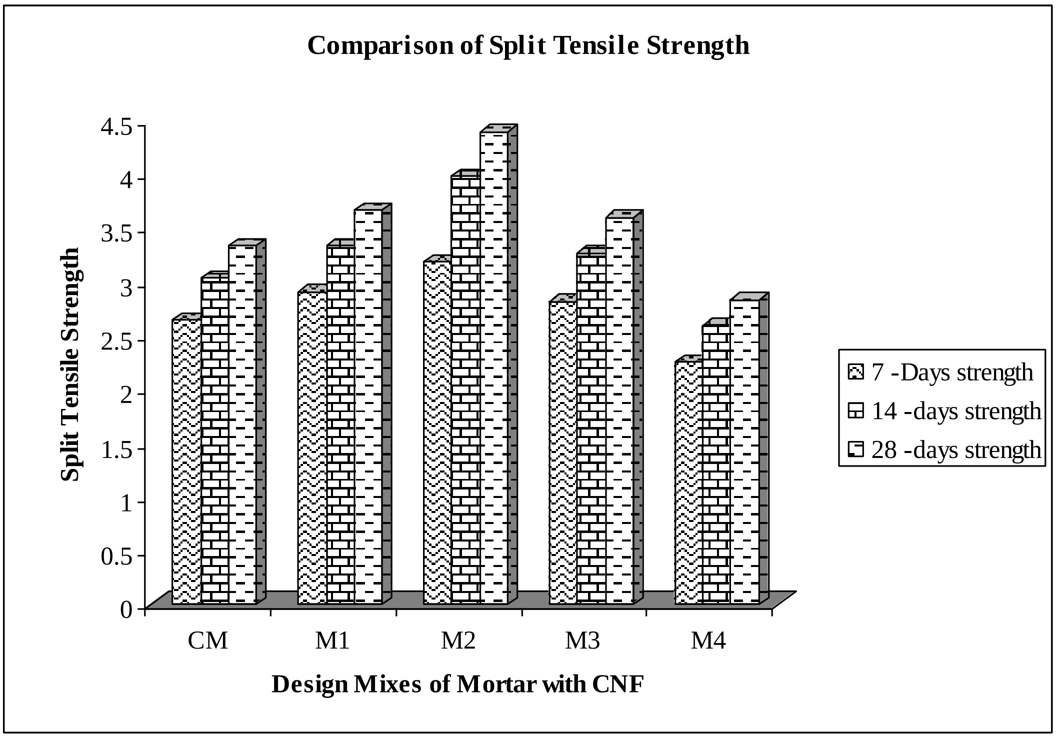
It can be concluded the small changes in the percentage of carbon nano fiber levels to dramatic changes in the split tensile strength which employ a message to researcher that a high level of quality control should be attend to control is changes.

Table 4.4: Split Tensile Strength of Cement Mortar in Control Mix & Cement Replacement with Carbon Nano Fiber at 7, 14 and 28days for Various Mixes

Sr. No.	Mix	Split Tensile Strength of Mortar (MPa)					
		7 -Days strength	% variation	14 -days strength	% variation	28 -days strength	% variation
1	CM	2.64	Reference	3.03	Reference	3.33	Reference
2	M1	2.90	+9.84	3.33	+9.90	3.66	+9.90
3	M2	3.18	+20.45	3.98	+31.35	4.39	+31.83
4	M3	2.81	+6.43	3.26	+7.50	3.59	+7.80
5	M4	2.25	–14.77	2.59	–14.52	2.83	–17.66

Note: (+) indicates increase and (–) indicate decrease in split tensile strength

Figure – 4.4



4.3.3 Comparative Performance of CMF and CNF

Perusals of the test results of CNF and CMF for all ages of curing in cement mortar on mechanical properties of matrix can be attributed to the good bonding between the CNFs and the cement hydration product as well as ability of CNF due to smaller size to control cracking the nano scale by bridging nano cracks and pores. The nano scale reinforcement was found to improve the split tensile strength at 20% more than the achieved by micro fibers.

4.4 FLEXURAL STRENGTH

Flexural strength of control sample of mortar mix CNF/CMF was carried out at the age of 7, 14 and 28 days. Test results are shown in Tables 4.5 & 4.6. As seen in quoted table, test result indicated the addition of CNF/CMF in cement mortar at different percentage. There is a increase of strength in CMF 0.05% at 7 days, 14 days and 28 days observed as 14.22%, 14.98% and 22.26% respectively, compare to strength at control mixes. In addition to that increase of strength in CNF 0.05% (M_2 mix) at 7 days, 14 days and 28 days observed as 17.49%, 18.93% and 25.88% respectively, compared to control mixes of mortar. However, after 0.075% and 0.1% decrease of strength was noted as compare to control mix samples. There is a decrease of flexural strength is mainly at 0.075% and 0.1% by weight of cement (CNF/CMF) due to improper dispersion of carbon fibers and more vides in cement matrix or lack of formation CSH gel.

Table 4.5: Flexural Strength of Cement Mortar in Control Mix & Cement Replacement with Carbon Micro Fiber at 7, 14 and 28days for Various Mixes

Sr. No.	Mix	Flexural Strength of Mortar (MPa)					
		7 -Days strength	% variation	14 -days strength	% variation	28 -days strength	% variation
1	CM	6.02	Reference	10.88	Reference	12.46	Reference
2	M1	6.29	+4.57	11.67	+7.26	14.34	+15.09
3	M2	6.87	+14.22	12.81	+17.74	14.98	+20.26
4	M3	5.85	–2.82	10.75	–1.20	12.24	–0.72
5	M4	4.83	–19.76	8.85	–18.65	10.69	–14.20

Note: (+) indicates increase and (–) indicate decrease in flexural strength

Figure – 4.5

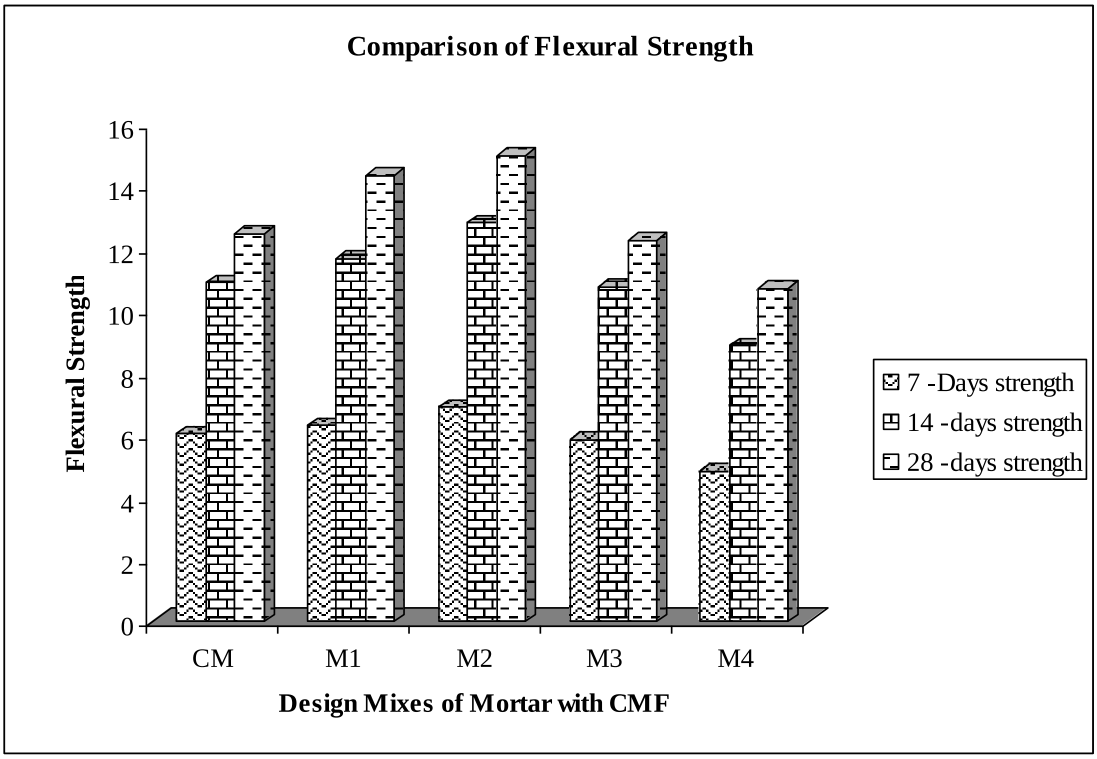
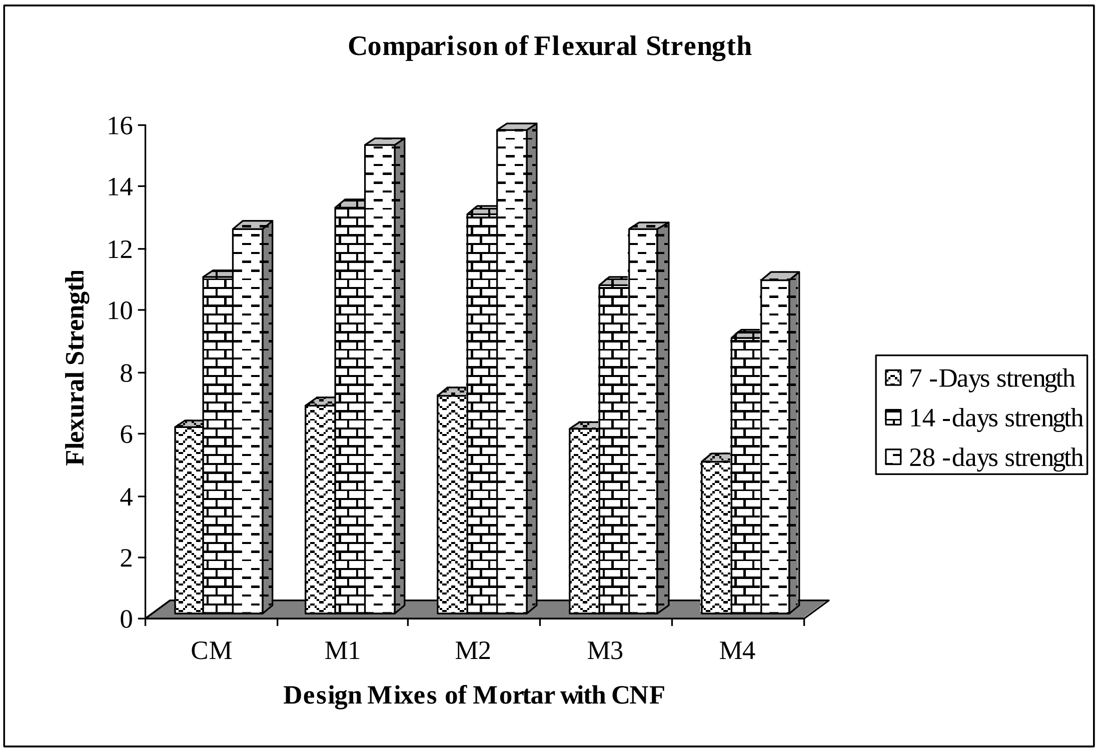


Table 4.6: Flexural Strength of Cement Mortar in Control Mix & Cement Replacement with Carbon Nano Fiber at 7, 14 and 28days for Various Mixes

Sr. No.	Mix	Flexural Strength of Mortar (MPa)					
		7 -Days strength	% variation	14 -days strength	% variation	28 -days strength	% variation
1	CM	6.02	Reference	10.88	Reference	12.46	Reference
2	M1	6.74	+11.97	13.17	+21.09	15.15	+21.09
3	M2	7.07	+17.49	12.94	+18.93	15.64	+25.58
4	M3	5.96	–0.99	10.65	–2.11	12.44	–0.16
5	M4	4.92	–0.18	8.92	–21.97	10.82	–13.10

Note: (+) indicates increase and (–) indicate decrease in flexural strength

Figure – 4.6



4.5 DURABILITY PROPERTIES

Water absorption test was conducted to access the durability property of the mortar mixes. The water absorption in bulk density is summarized in table 4.8 at 28 days. Water can penetrate in the form of liquid or paper within the capillary voids into the porous material. It can be seen with clarity from Tables 4.7 & 4.8 and Figs. 4.7 & 4.8 that the increase in the percentage amount of carbon nano fibers decreases the water absorption of mortar specimen. It can be the result of the enhancement impermeability mechanism of mortars due to super pozzolonic performance of carbon fiber particles. The fine particles block the channels connecting the capillary pores in the cement paste and generate is more homogeneous distribution of C-S-H gel resulting in less pore structure and permeable voids.

Table 4.7: Bulk Density and Water Absorption of Cement Mortar in Control Mix & Cement Replacement with Carbon Micro Fiber at 28days for Various Mixes

Sr. No.	Mix	Bulk Density gm/cc and Water Absorption %					
		Wt. of Mortar gm	% variation	Bulk Density	% variation	Water Absorption %	% variation
1	CM	826.00	Reference	2.35	Reference	1.87	Reference
2	M1	814.00	–1.45	2.31	–1.73	1.78	–4.81
3	M2	821.00	–0.61	2.33	–0.85	1.70	–9.09
4	M3	822.00	–0.48	2.34	–0.42	1.74	–6.95
5	M4	839.00	+1.57	2.38	+1.27	1.81	–3.20

Note: (+) indicates increase and (–) indicate decrease in bulk density and water absorption

Figure – 4.7(a)

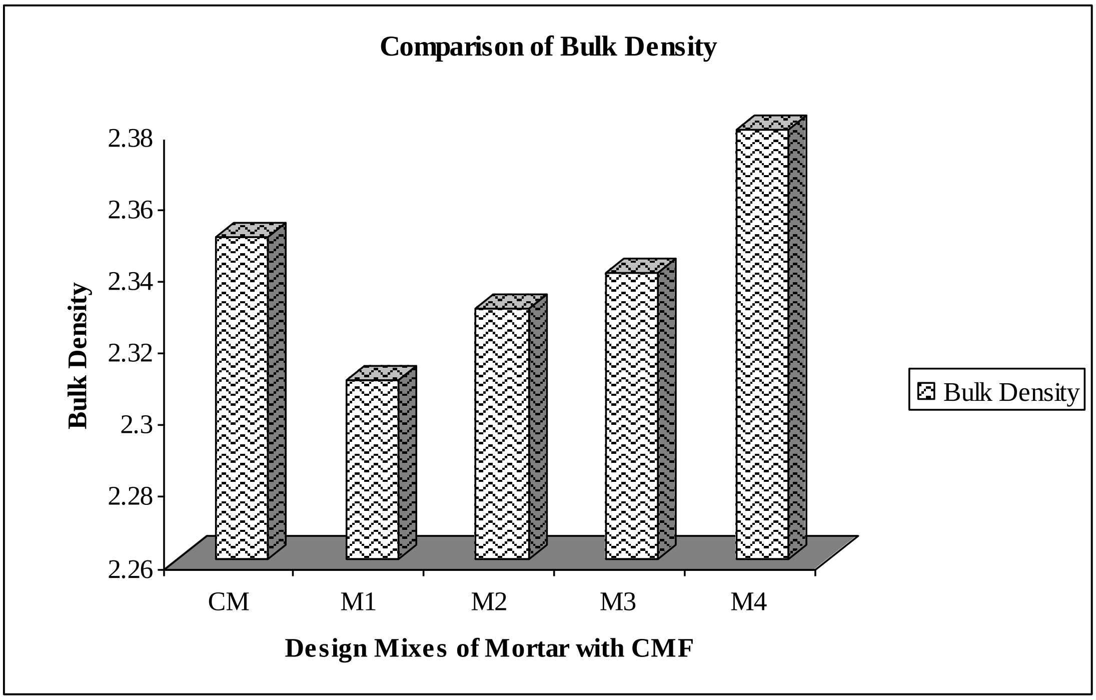


Figure – 4.7(b)

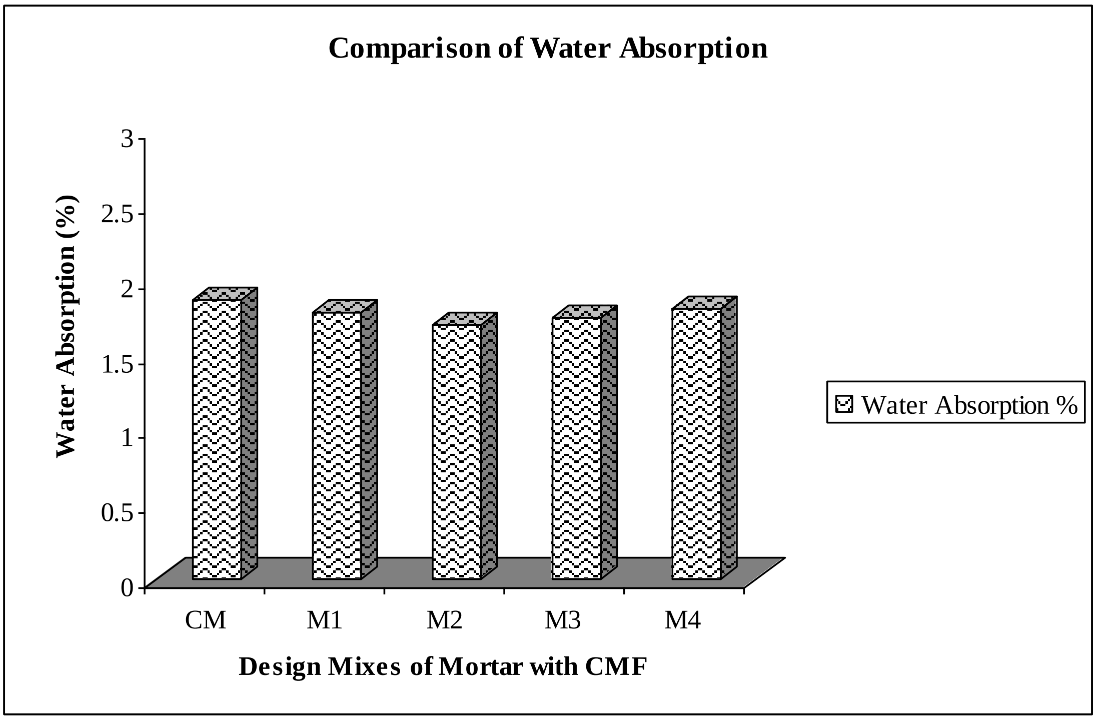


Table 4.8: Bulk Density and Water Absorption of Cement Mortar in Control Mix & Cement Replacement with Carbon Nano Fiber at 28days for Various Mixes

Sr. No.	Mix	Bulk Density gm/cc and Water Absorption %					
		Wt. of Mortar gm	% variation	Bulk Density	% variation	Water Absorption %	% variation
1	CM	826.00	Reference	2.35	Reference	1.87	Reference
2	M1	813.50	–1.51	2.31	–1.70	1.75	–6.41
3	M2	820.50	–0.73	2.33	–0.85	1.70	–9.09
4	M3	822.00	–0.48	2.34	–0.42	1.72	–8.02
5	M4	827.00	+0.12	2.35	0.00	1.76	–5.88

Note: (+) indicates increase and (–) indicate decrease in bulk density and water absorption

Figure – 4.8(a)

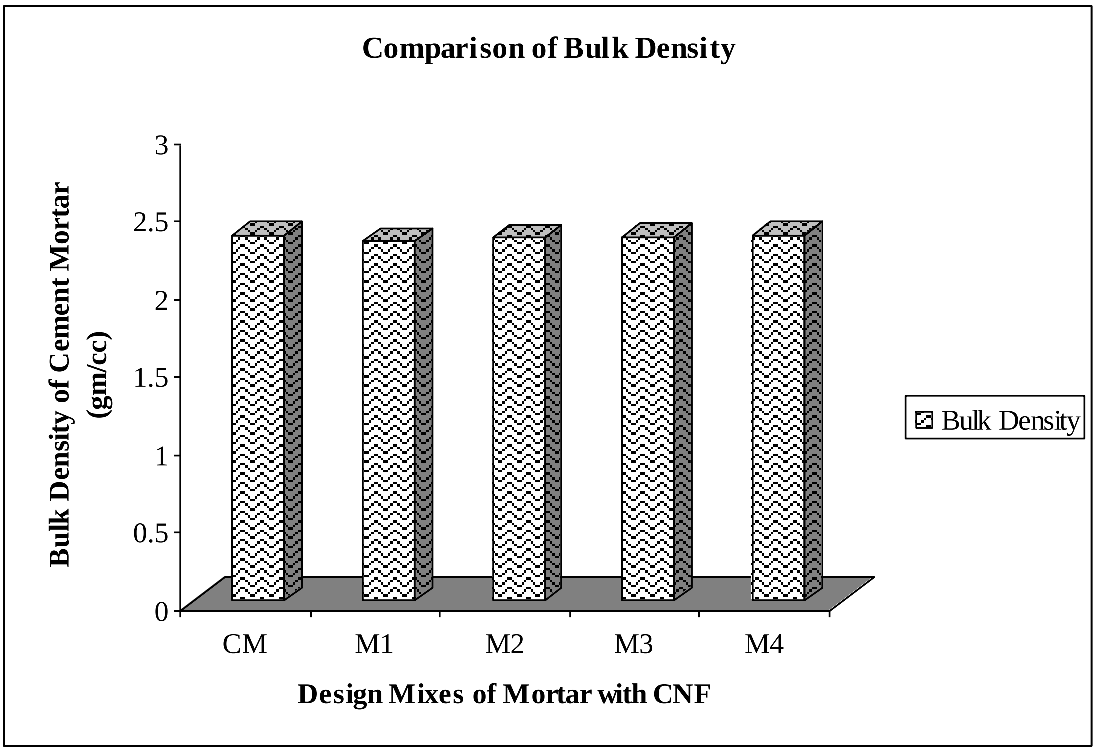
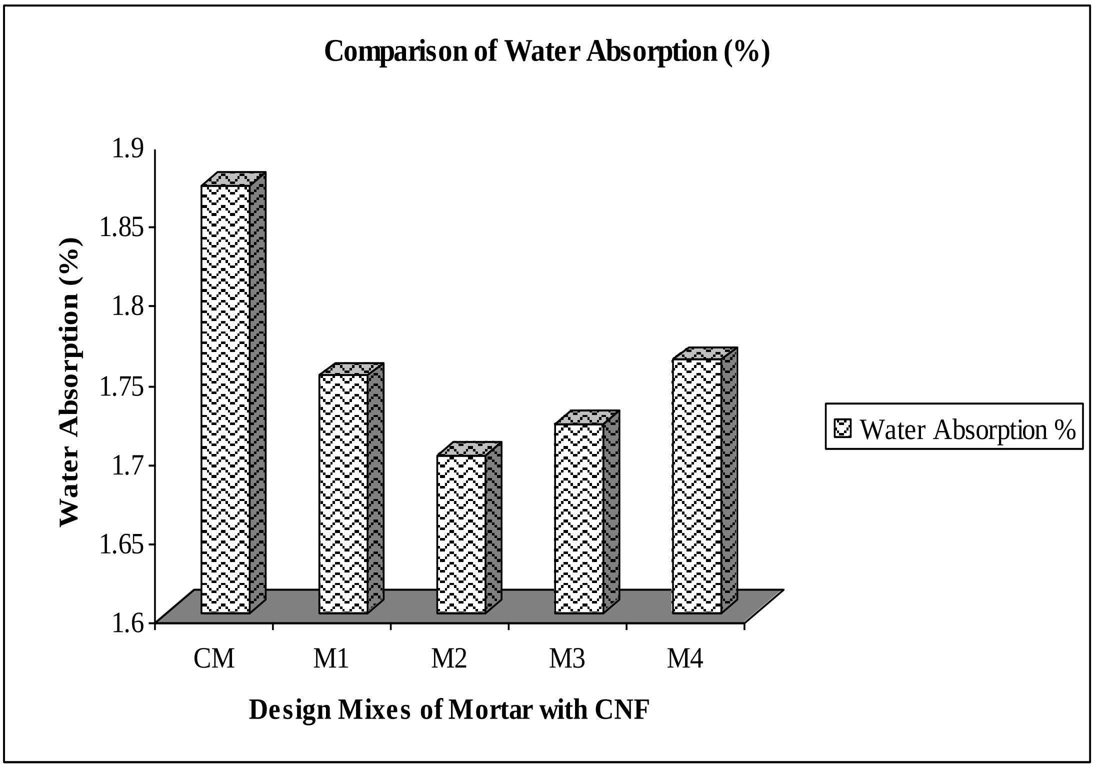
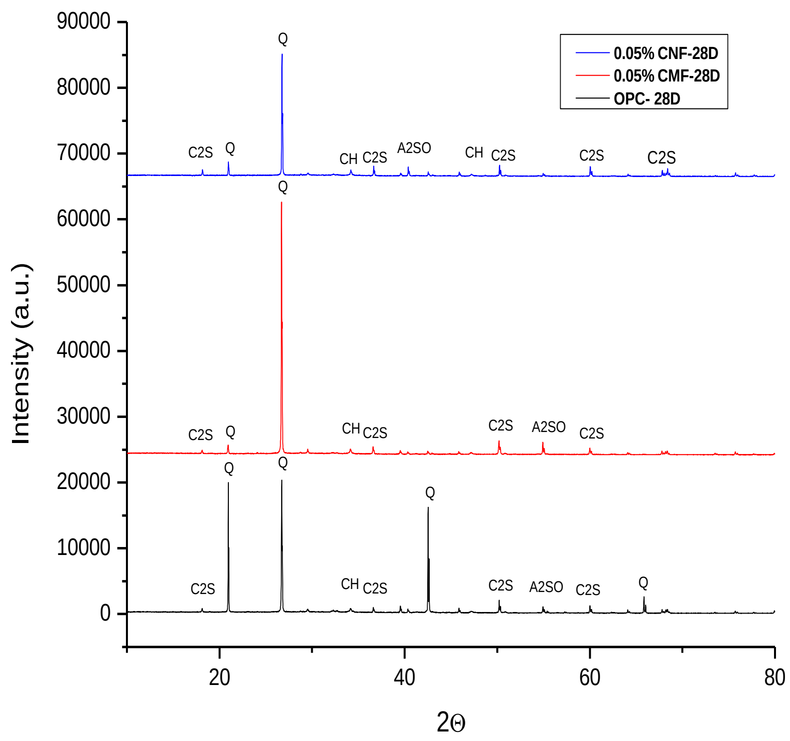


Figure – 4.8(b)



4.6 X-RAY DIFFRACTION (XRD)

Only samples which caused increase in compressive, flexural and tensile strength properties were selected to XRD. Hence only samples got tested at 0.0% and 0.05% CMF and CNF. It is observed that addition of 0.05% by weight of CMF some bonding between gel C-S-H and needle grown in the modified CM. While the main compound identified was calcium silicate hydrate and calcium hydroxide (CH). It is clear from Figure 4.9 that XRD pattern of CMF/CNF control cement mortar containg optimize 0.05% CMF/CNF. It has a crystalline phase at 2θ angle which indicate the presence of quartz between platelet in the peak of intensity between 67,000 to 9000 (A.U.). This is due to the pozzolonic reaction of CMF/CNF with the free line liberated during process of hydration.



Notations:

Q – Quartz
C_2S – Dicalcium silicate
CH – Calcium hydroxide
A_2SO – Dialuminium silicate oxide

Figure 4.9: XRD pattern for plain and CNF/CMF modified cement paste hydrated for 28 days (optimize addition 0.05%)

4.7 SCANNING ELECTRON MICROSCOPY

SEM was carried out at the ages of 3 days, 7 days and 28 days for optimize concentration 0.05% CNF/CMF by weight of cement. The microstructure shows the formation of C-S-H gel, needle hydrates and voids. The same micrograph shows on the hydrated products such as CSH, CH etc. The test samples were obtained from central part of specimen. Figure shows the microstructure of the cement paste without CNF/CMF. From Figure 4.10(a), (d) and (e) it is concluded that C-S-H gel existed in the form of gel and SEM image also shows growing needles like crystal embedded in the gel and shows a good bond between gel and needle. This shows the evidence of improvement in strength of CMF as well as CNF at optimized 0.05% replacement by cement. Hence the mechanical properties like compressive strength etc. are much improved in the modified CM at all ages (3 days, 7 days and 28 days). This clearly depicts the better performance of CNF than CMF as interpreted from the images of micrograph Figures 4.10(b), (c), (f), (g), (h) and (i).

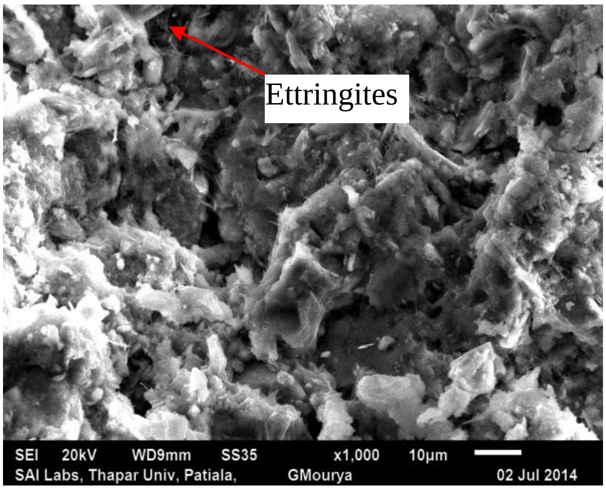
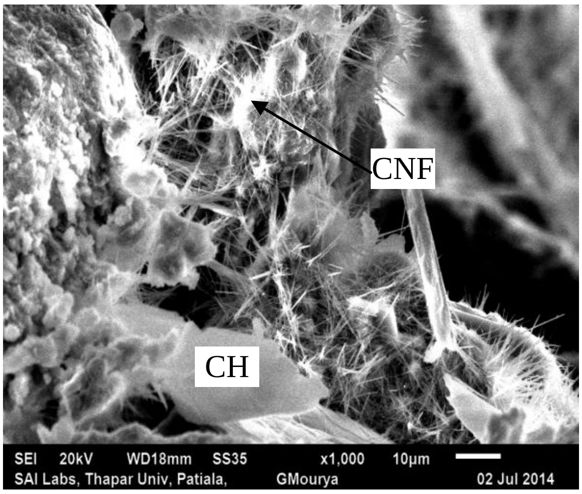
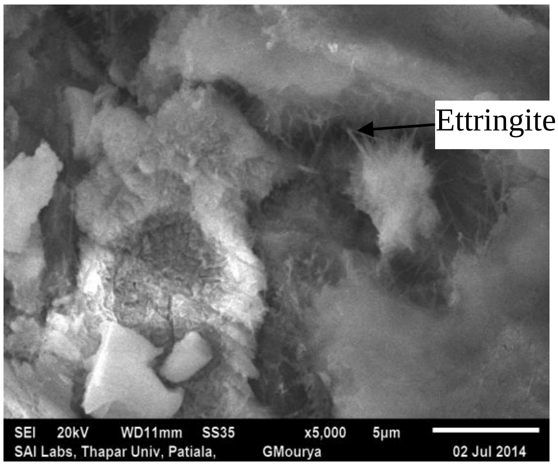


Figure 4.10(a): SEM micrograph of control OPC (3 days hydrated) showing Ettringites



(b): SEM micrograph of CNF (3 days hydrated) showing CNF & CH

Figure 4.10(c): SEM micrograph of CNF 0.05% (7 days hydrated) showing Calcium Hydroxide



(d): SEM micrograph of control OPC (7 days hydrated)

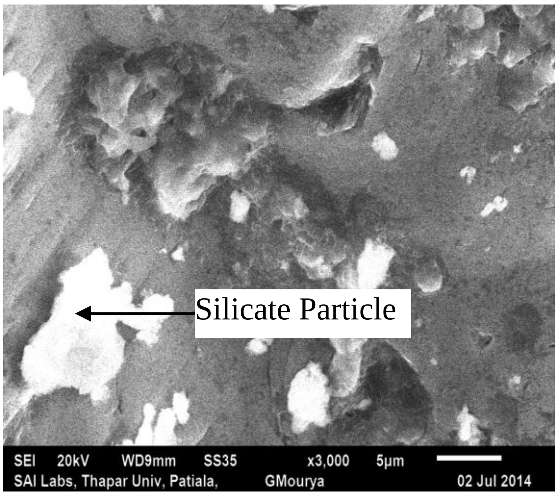
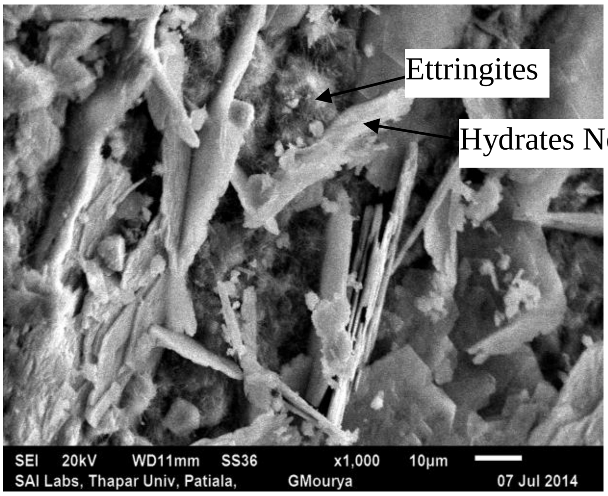


Figure 4.10(e): SEM micrograph of OPC control sample (28 days hydrated) showing silicate particles



(f): SEM micrograph of CMF 0.05% (28 days hydrated) showing Ettrigites and Hydrated Needles

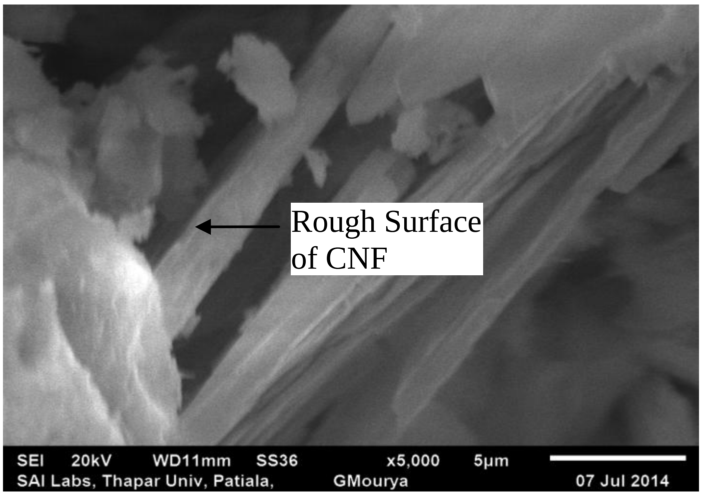


Figure 4.10(g): SEM micrograph of CNF 0.05% (28 days hydrated) showing Rough Surface of CNF

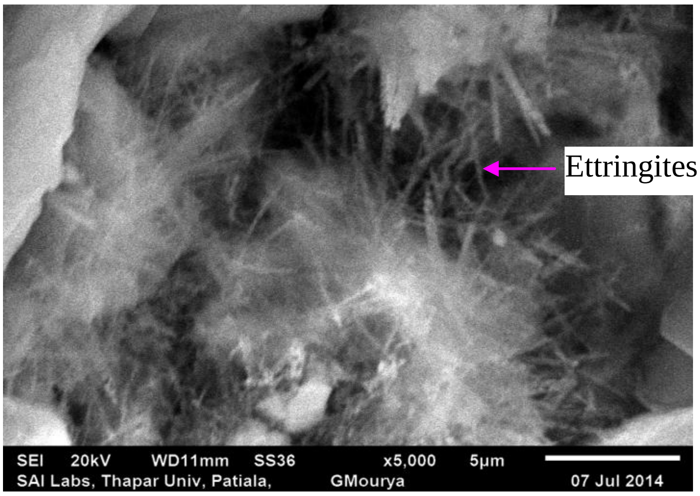


Figure 4.10(h): SEM micrograph of CMF 0.05% (28 days hydrated) showing Ettringites

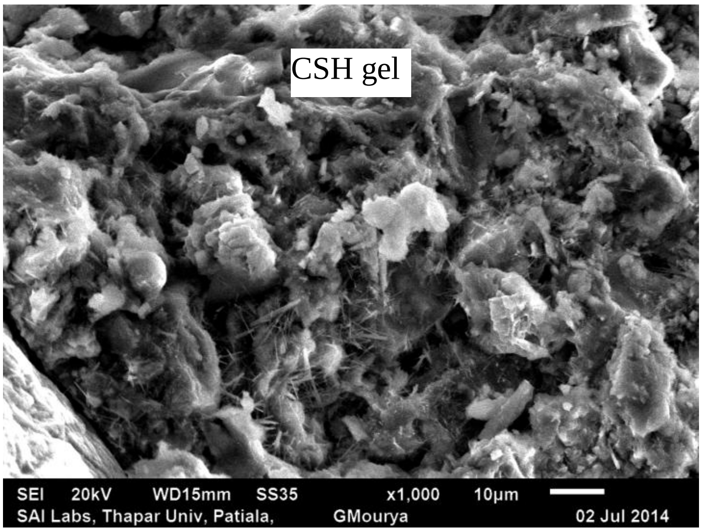


Figure 4.10(i): SEM micrograph of CMF 0.05% (28 days hydrated) showing CSH gel

4.8 CLOSING REMARKS

In this chapter, the test results were discussed. It is clear from the result that nano carbon fiber act as filler/reinforcement in mortar mix to increase the strength. Conclusion were discussed which are drawn out from the results of the previous chapter. Well dispersed CNF improve the strength & stiffness of cement mortar. Excess concentration leads to poorly dispersed CNF lumps inside the mortar and have a negative impact of strength (M_0 2011).

CHAPTER – 5
CONCLUSIONS AND FUTURE SCOPE

5.1 GENERAL

High potential has been predicted for nano technology application in construction & even minor improvement in material could bring large accumulated benefits. The role an application of the carbon nano fiber and carbon micro fiber with cement mortar have been reviewed and discussed in details. The present study aims to improve the scare data base of fresh and hardened test on the cement mortar with and without using CNF/CMF to study the effect of above carbon fiber on the properties of mortar and give some inside to improve the properties of mortar. The present experimental study was conceived following the general purpose of testing new sustainable building processes and modern production system aimed at saving natural raw materials and reducing energy consumption.

5.2 CONCLUSIONS AND MAJOR FINDINGS

Based on the scope, materials, techniques, procedure and other parameter associated with this work. The following conclusion and recommendation can be stated as under:

5.2.1 Effect of Dispersion by Surfactants

Nanoshel carbon nano fibers and carbon micro fibers are hydrophobic and will consequently not dispersed well in water. Surfactant such as Triton – X100 and polyisobutylene succinimide (PIBSI) have proven to be helpful in dispersing and maintaining CNFs/CMFs in a stable suspension. There is a progressive increase in strength test results by addition of CNF/CMF in coagulation form as compared with control mix samples or mortar.

5.2.2 Effect of CNF/CMF on Compressive Strength

- Incorporating optimized CNFs/CMFs to cement mortar mixtures generally enhance their mechanical properties.
- Compressive strength of modified cement mortar by CMF for aqueous mixing at optimized level 0.05% shows replacement of cement by weight increase in

strength for 3, 7 and 28 days by 9.05%, 10.25% and 10.49% (CMF) compared to control mixes.

- Similarly for CNF addition, progressive increase in strength for 3, 7 and 28 days by 11.90%, 15.64% and 17.23% respectively, as compare to control mixes sample.
- The addition of mix 0.075%, 0.1%, the strength decreases by 10.4% for 28 days (CMF) and 8.79% for 28 days (CNF) as compared to control mix samples.

5.2.3 Effect of CNF/CMF on Split Tensile Strength

- Split tensile strength shows progressive increase in strength with addition of optimised carbon nano fiber and carbon micro fiber in collided form to cement mortar for 7 days, 14 days and 28 days. The strength in case of 7 days increases by 12.87% (incase of M_2 mix), in case of 14 days, increases by 24.42% for M_2, in case of 28 days for M_2 increases by 26.13% by addition of CMF. However, in addition of CNF optimised concentration 0.05%, the strength in case of 7 days increases by 20.45%, 14 days increases by 31.35%, 28 days increases by 31.83% as compared to control samples.
- The addition of more CNF i.e. M_4 mix the strength at 28 days decreases by 17.66% as compare to control mortar sample. While CMF mix M_4 decreases by 16.21% as compare to control mortar samples.

5.2.4 Effect of Carbon Fiber on Flexural Strength

- Flexural strength shows progressive increase in strength with the addition of CNF and CMF in mix M_2 for 7, 14, & 28 days. The strength incase of CMF modified mortar, 7 days increases by 14.22%, in case of 14 days, 17.74% and 28 days as 20.26% as compare to control mix samples.
- Similarly in case of CNF modified cement mortar, strength of 7 days increases by 17.49%, in case of 14 days, increases by 18.93% and in case of 28 days, increased by 25.58%.
- The addition of more percentage CNF & CMF in cement mortar at 0.1% decrease the strength by 14.20% for 28 days (CMF) and for 28 days decreases the strength by 13.10% (CNF).

5.2.5 The Effect of CNF on Water Absorption and Bulk Density

The increase in the percentage/amount of carbon nano fiber decrease the water absorption of mortar specimen which can be seen in the result in previous chapter 4. It can be result of the enhancement in permeability mechanism of mortars due to super-pozzolonic performance of carbon nano particle. The fine particles of pozzolonic block the channels connecting the capillary pores in the cement paste and generate a more homogonous distribution of C-S-H gel resulting in less pore structure and permeable voids. Similar effect on the bulk density have also been observed which is evident from the test result in previous chapter.

5.2.6 Effect of Carbon Nano Fiber and Carbon Micro Fiber on Microstructural Properties

- The results from XRD represent the higher formation of CSH and more consumption of CH.
- SEM micrographic show that the microstructure is appeared quite dense, compact which can be explained by great surface area.
- Though the SEM images, correlation with the mechanical properties was observed from 28 days. SEM micrograph, C-S-H gel existed in the from of less amounted, ‘stand-along’ clusters, lapped & connected to together by little amount of needle hydrates and hence high compressive strength at 28 days as compare to 3 or 7 days.

5.3 WORK LIMITATIONS

There are limitations in present study as the study of effect of CNF/CMF in proportion of mortar is a vast subject of interest for research work.

- The limited quantity of CNF/CMF due to its uncommon availability & high coast allowed only for limited scope of experiment to be conducted.
- The scope of present study is limited to use of nano silica in cement mortars. This can be further used in concrete as partial substitute to the cement.
- The replacement of cement with CNF/CMF was 0.025%, 0.050%, 0.075% & 0.1% the intervals can further be decreased in future studies.
- More powerful & through mixing was needed than the hand mixing.

- This study is limited to the mixes without using superplasticizer. Therefore, further studies can be cement out preparing the mix using superplasticizer.
- Some of test employed in this study such as SEM are not common in the construction industry. Which made it difficult to compared against reference data.

5.4 SUGGESTION FOR FUTURE RESEARCH WORK

- Considering all aspects, it is suggested that optimal use of carbon fiber from micro to nano scale to obtain more strengthened cement mortar has to be established, which will result in long lasting concrete structure in the future.
- Codes of practice should be introduced for testing connection material with nano composite (nano fiber & nano tube) to determine how to choose the suitable percentages for each particle size of CNF to be added to concrete & appropriate water to cement ratio and superplasticizer amount to be added in order to obtain the required properties for each application.
- Feasibility studies must for conducted in order to evaluate if it is economic to add those carbon fiber (Nano/micro) to cement mortar and concrete or note to discover the possible application in construction material in general.
- The durability test including chemical test rapid chloride permeability test etc. needs to be conducted, examined and validated in further studies.

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