Effect of multiwalled carbon nanotubes on compressive behavior of high strength concrete at elevated temperature for mass concreting

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A thesis submitted for the partial fulfilment

of the requirement for the degree of

Master of Science in

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Submitted by

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DECLARATION

I certify that the research work titled "**Effect of multiwalled carbon nanotubes on compressive behavior of high strength concrete at elevated temperature for mass concreting**" is my own work . This work has not been presented elsewhere for assessment. The material that has been used from other sources it has been properly acknowledged/referred

Signature of Student

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<u>DEDICATION</u>

This thesis is dedicated to **Muhammad wa Aal e Muhammad (Peace be Upon Him)**; minaret of knowledge and wisdom, my beloved **Mother** and my beloved **Father** who always supported me through thick and thin of my life, my beloved **brothers** who cheered me when I was sad and always motivated me. I would also like to extend my heart most gratification to respected **Dr. Syed Hassan Farooq** and my **advisor Dr. Muhammad Shahid Siddique** whose guidance made me able to finish my research work.

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I am thankful to ALLAH (S.W.T) by grace of which I am able to complete my research.

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(*Engr. Kashan Nisar)*

Abstract

The need for high-rise buildings has increased in recent years as the population has grown. Hence, high-strength concrete has grown more popular than standard-strength concrete. However, the fundamental problem with high-strength concrete (HSC) is that it undergoes numerous undesired chemical changes and thermal fracture on exposure to high temperatures. This quickly jeopardizes the use of high-strength concrete in structural applications. As a result of these challenges, it is necessary to build more thermally stable structures. So, in order to compensate this issue carbon nanotubes (CNT) are induced in this research. The mechanical properties such as compressive strength, flexural strength stress-strain response, elastic modulus, Compressive toughness, mass loss, as well as detoriation caused by the exposure of elevated temperatures were studied. The analyzed formulation was heated up to 200, 400 ,600 and 800 ^{o}C at a heating rate of 5 ^{o}C /mint and then tested for residual conditions. Carbon nanotubes (CNTs) modified concrete showed a higher compressive strength, elastic modulus, flexural strength, and compressive toughness as compared to the control specimens. According to the visual inspection modified specimens showed less cracking at higher temperatures. Moreover, the effect of plastering high strength concrete with CNTs showed better strength retention on exposure to fire. Conclusively, utilization of CNTs in high strength concrete is efficient as fire resistance concrete.

TABLE CONTENTS

Contents

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

AASHTO	-	American Association of State Highway and Transportation Officials
CNT	-	Carbon Nano Tubes
MWCNTs	-	Multi-walled Carbon Nano Tubes
ASTM	-	American Society for Testing and Materials
CS	-	Compression Strength
FS	-	Flexural Strength
C-H	-	Calcium Hydroxide
C-S-H	-	Calcium silicate hydrate
Fc’	-	28 days Compressive strength
E	-	Elastic Modulus
Tc	-	Compressive toughness
TI	-	Toughness index
SF	-	Silica Fumes
GGBSF	-	Ground granulated blast-furnace slag
SP	-	Superplasticizers
HRWRs	-	High range water reducers

Chapter 1

INTRODUCTION

1.1 General

Concrete is a building material that is composed of cement, fine aggregates (sand), and coarse aggregates, all mixed together with water. Due to its great strength, durability, and raw material availability, it is one of the most often used construction materials. Portland cement is one of the most common synthetic substance that is commonly used in concrete production. It is most likely utilized more than any other man-made commodity on the earth. Normal strength concrete (NSC) has been pushed to the sidelines in recent years, owing to its inability to meet performance demands. But on the other hand high strength concrete is becoming popular due to its excellent mechanical qualities, including compressive and tensile strength. It is also more resistant to chemical assaults when compared to conventional strength concrete . Furthermore, because of its dense microstructure, high strength concrete has limited permeability(Daivari et al., 2016).

High Strength Concrete (HSC) is good in various ways but it undergoes many undesirable chemical changes when it is exposed to elevated temperatures. When HSC is exposed to fire, it experiences a lot of thermal cracking and negative chemical changes, which puts its structural usage in jeopardy.(Phan & Carino, 2003). At elevated temperature pore pressure increases due to the evaporation of water in the pores which causes the abrupt embrittlement and subject massive tensile stresses on concrete inner core (Kodur & Dwaikat, 2010).Concrete core has a smaller heat dispersion than the surrounding layer. The outside layers of concrete are subjected to excessive heat due to their higher specific heat and lower thermal conductivity, and heat transfer to the core layers is slow. This mechanism slows total heat transport to the interior layers of concrete, resulting in thermal inertia (Bažant et al., 1996). Tensile thermal stresses are usually caused by an increase in heat gradient due to thermal inertia which results in fissures causing spalling and cracks (Shah et al., 2015).

Recently, nanotechnology research and development has made its way into construction materials. Nanotechnology manipulates matter at the molecular level with tolerances of less than 100nm. A lot of nanoparticles allotropes are available as shown in figure 01, which can be used in cementious matrices to improve its mechanical performance in terms of self-sensing ability, enhanced compressive , tensile and flexural strength(Power et al., 2018a).

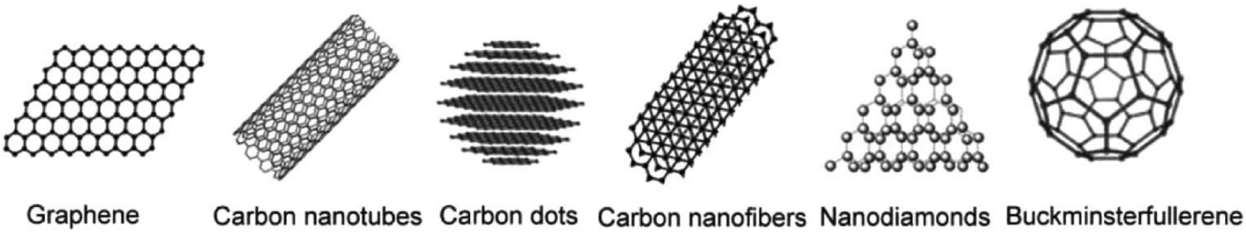


Figure: 01 - Different allotropes of Carbon nanoparticles (Power et al., 2018b).

There are two types of Carbon Nanotubes (CNTs) on the basis of structural level. Single walled carbon nanotubes and multi-walled carbon nanotubes as shown in figure 02.

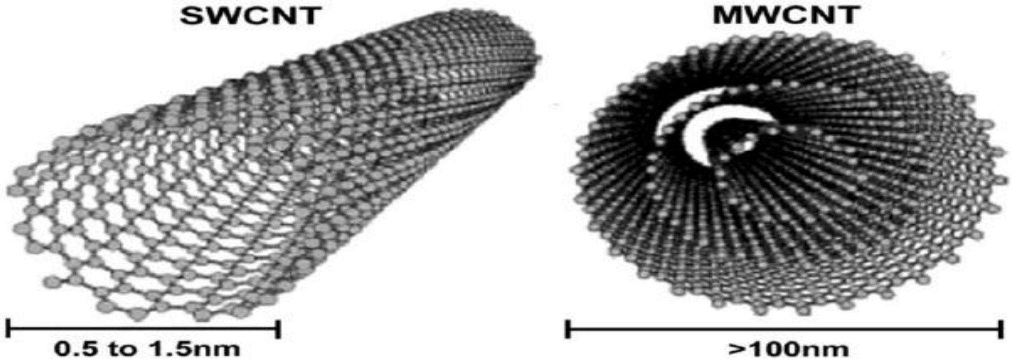


Figure: 02 – Diametric range of Carbon nano tubes(Malhotra et al., 2015).

When exposed to high temperatures, carbon nanotubes (CNTs) exhibit attributes comparable to carbon nanotubes (CNFs) in terms of compressive, tensile, and flexural strength, as well as fracture resistance(Sadiq et al., 2021). Carbon nanotubes (CNTs) have a high tensile strength, elasticity modulus, and yield strain. They may be widely distributed in concrete paste, and their bond with the paste is stronger than that of larger fibers since they are nano-materials. They provide excellent opportunities to develop CNTs based reinforced concrete structures (Sobolev, 2016)

1.2 Research Objective

a. To study the mechanical behavior of multiwalled carbon nano tube high strength concrete at elevated temperature for mass concreting.

b. To study the effectiveness of multiwalled carbon nano tube plaster in improving high strength concrete's thermal resistance at elevated temperature for mass concreting

1.3 Research Overview

The following tasks are carried out in order to achieve research objectives.

- Literature review
- Test set up such as Flexural test assembly, protective steel assembly for furnace, compression test assembly
- Performance of high temperature tests
- Evaluation of experimental results
- Conclusion and further recommendations

1.4 Problem Statement

As the world's population has expanded in recent years, so has the need for high-rise structures. At high temperatures, high-strength concrete, on the other hand, endures a cascade of undesirable chemical changes and thermal cracking, jeopardizing its structural value. This is mostly due to huge tensile strains caused by pore water vaporization at the micro level of concrete. As a result, it becomes necessary to construct more thermally stable structures. Carbon nano tubes could be effective for this because of its crack bridging, strengthening, and heat stability. So far, a lot of study has been done on carbon nano tubes (CNTs), but it's all been done at ambience level. There hasn't been much research done on their behavior at elevated temperature. Carbon nanotubes (CNTs) have gotten a lot of interest from scientists. However, all of this was largely focused on concrete’s compressive strength. Although some research was done to study its behavior at high temperatures, these few studies focused on one parameter, such as replacing a certain percentage of cement with carbon nano tubes, but no one applied them to the outer surface of concrete cylinders and studied their behavior at high temperatures because carbon nano tubes have a melting point of 3500 ^{o}C. Moreover, all of it has been executed at the ambience level. Until now, research on their behavior at elevated temperatures has been lacking. Hence, objective of this research is to determine the mechanical properties of high-strength concrete(HSC) after the induction of MWCNTs .

1.5 Organization of Thesis

Thesis can be categorized in the following chapters as:

Chapter 1 Comprises of introduction about concrete and carbon nanotubes along with their purpose and usage .Furthermore problem description, research objectives and the scope of the investigation has also been discussed.

Chapter 2 This chapter gives a detail overview about the literature review, materials such as CNTs, silica fumes etc., along with their usage in concrete. Furthermore, HSC performance under high elevated temperature , required test methods and research gap are also discussed in this chapter.

Chapter 3 This chapter provides an outline of the testing techniques, along with methods to determine mechanical properties such as strengths (compressive and flexural) etc. Further it also gave a brief description about several sorts of equipments and testing standards that are being used to assess the mechanical properties of specimens.

Chapter 4 This chapter gives a detail interpretation of the obtained experimental results. Such as variation in strength (Compressive and flexural) with comparison to Control HSC specimens along with other mechanical properties such as stress-strain response, compressive toughness, Crack energies, modulus of elasticity, visual evaluation and mass loss.

Chapter 5 This chapter gives a brief description about the results obtained from the analysis of induced and plaster specimens .Such as about their strength (Compressive and flexural) increase at ambient level along with the strength retainment at elevated temperatures ranging from 23 ^{o}C to 800 ^{o}C. In addition to all such conclusions further recommendations are also given.

CHAPTER 02

LITERATURE REVIEW

2.1 Introduction

This chapter gives a basic overview of Carbon Nano Tubes (CNTs), their production and usage in cementitious matrices along with their mechanism with high strength concrete. As we know that concrete is most widely used material in construction field due to its high compressive strength, higher modulus of elasticity, simplicity of preparation, water tightness, resistance to corrosive agents etc., concrete has become a major constituent in our building's infrastructure. The compressive strength of concrete gives a good idea about its properties such as concrete with a higher compressive strength has high modulus of elasticity, tensile strength, lower permeability, and good durability. The usage of HSC has become quite prevalent since the development of superplasticizers (SP) or high range water reducers (HRWRs). In terms of greater durability and economics, HSC has a lot of benefits. Because of the smaller cross sections, the use of HSC in high-rise building columns has become a supplementary technique in contemporary construction practices. Several studies on the behavior of concrete at high temperatures have been conducted. When compared to NSC, the data demonstrate that HSC performs poorly at higher temperatures, despite its superior performance in all other situations so for that purpose it is necessary to develop a high strength concrete that is resistant to fire up to some extent as compared to conventional high strength concrete so for that purpose nano material are being used in this research.

2.2 Nano Materials

Materials with at least one external dimension ranging from 1 to 100 nanometers are known as nanomaterials. At least half of the particles in a numerical size distribution must be 100nm or less, according to the European Commission's definition. Nanomaterials can be found in nature as byproducts of combustion processes, or they can be created artificially (Zhang & Li, 2009).

2.3 Carbon-based cementitious nanotubes composites

Since the 1990s, carbon-based cementitious nano composites have been explored. There are two types of carbon nanotubes such as single walled carbon nanotubes (SWCNT) with a diameter of 0.75-3nm and a length of 1-50um and multi walled carbon nanotubes (MWCNT) with a diameter of 2–30nm and a length of 0.1–50um(Jalali et al., 2005).

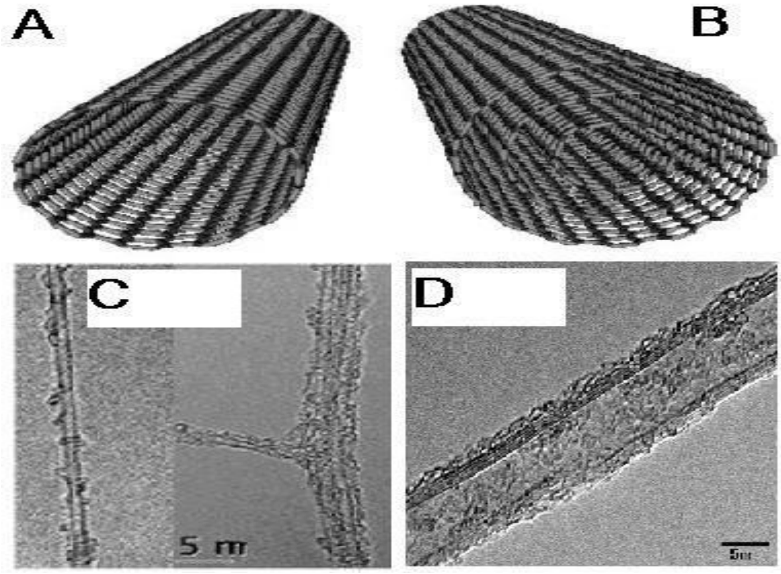


Figure 03:- SWCNT and MWCNT schematic structure and TEM pictures. (A) Schematic structure of SWCNT and **(B)** MWCNT. The transmission electron microscope (TEM) images of a **(C)** SWCNT and **(D)** MWCNT(Etemadi et al., 2014).

2.4 Carbon nanotubes (CNTs)

Carbon nanotubes can define as tubes whose diameter is less than 100 nm. Radushkevich and Lukyanov found these tubes in 1952 (Berber et al., 2000).Carbon nanotubes are carbon-based nanomaterial that are obtained from graphite. Single-walled carbon nanotubes (SWCNTs) and multiwalled carbon nanotubes (MWCNTs) are the two varieties of carbon nanotubes. SWCNTs, on the other hand, have a diameter of 0.75-3nm, whilst MWCNTs have a diameter ranging from 2-30 nm. Carbon nanotubes (CNTs) are cylindrical shells formed by rolling graphene sheets into a continuous cylinder(Zhang & Li, 2009).

2.5 Application of Carbon nanotubes (CNTs)

Many efforts are being made to broaden the unique applications of this versatile material in a variety of industries. Nanotechnology has been a major concentration in various fields of material sciences, electronics, energy, and medicine etc., in addition to that nanomaterial have lately penetrated the construction field as well. CNTs can play a key role in the building sector by altering cementitious composites. CNTs have exceptional material qualities, such as high elasticity and strength, which can make cementitious composites superior to traditional composites. Hence, now CNTs are used in a variety of industries, including construction, building materials, medicine, batteries, capacitors, and membranes as shown in figure 04 (Wilson, 2002a).

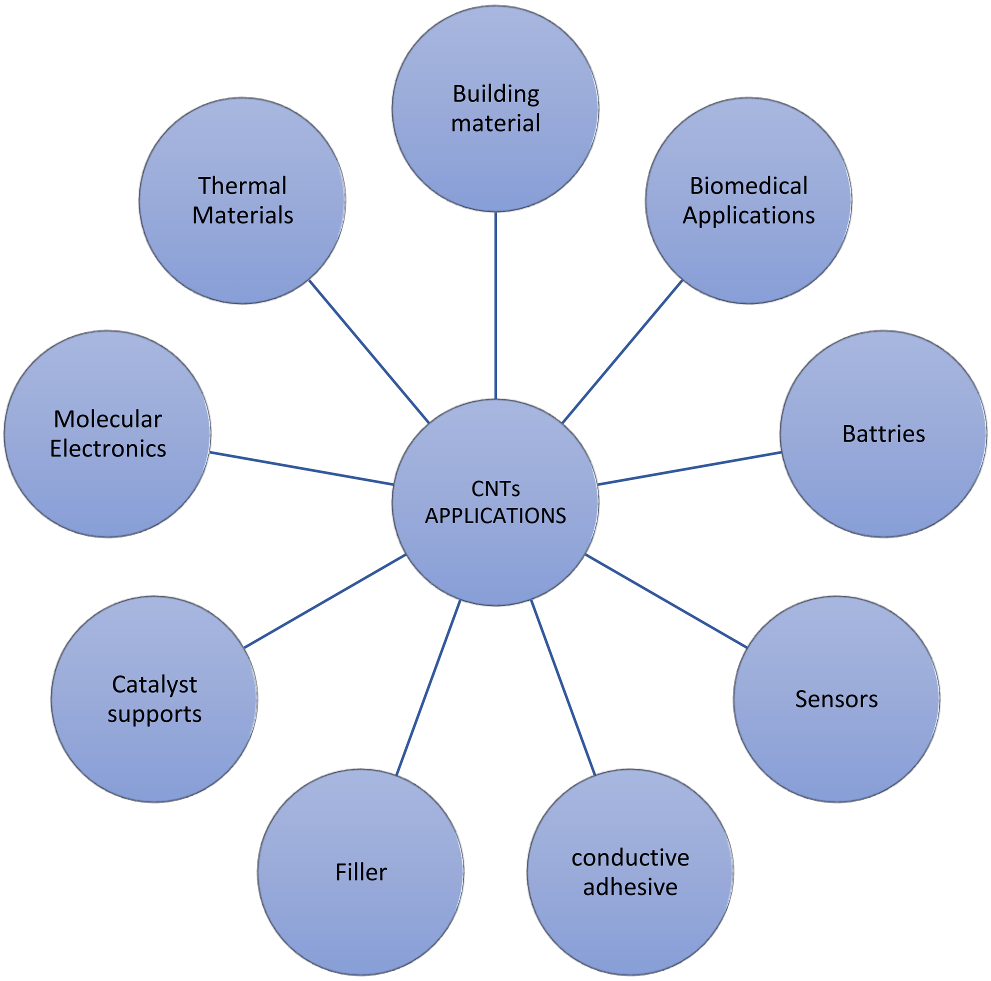


Figure 04: - Various Applications of CNTs

2.6 Properties of Carbon nanotubes

Carbon nanotubes have the following characteristics:

- The electrical conductivity of carbon nanotubes (CNTs) is extremely high.
- Carbon nanotubes have a high elongation-to-failure ratio of 18%.
- Tensile strength of carbon nanotubes is quite great.
- CNTs are very flexible and may be twisted to almost any shape without causing harm.
- CNTs have low coefficient of thermal expansion
- CNTs are strong emitters of electron fields.

(Wilson, 2002b)

Hence , from all of the characteristics as mentioned above we can conclude that carbon nanotubes have good thermal stability and crack bridging properties .Some of the properties of Multi-walled Carbon Nanotubes (CNTs) used in this project are shown below in table 01.

Table 1: Properties of the carbon nanotubes that were utilized

Parameters	**Properties**
Inner diameter (nm)	3-6 nm
Purity%	>95%
Density (g/cm^3)	2.0
Surface area per unit weight (m^2/g)	210-290
Apparent density (g/cm3)	0.10
Outer Diameter (nm)	5-15nm
Electrical conductivity (S/cm)	8-10

2.7 Dispersion of Carbon nanotubes (CNTs)

Carbon nanotubes are attracted to each other due to the presence of van der waal's forces. So in order to overcome them ,the traditional procedure involves the use of a surfactant such as gum acacia (GA) and ultrasonic pulse velocitor as they give opposite charges to the nanoparticle for the expansion throughout the concrete mix, but due to lack of equipment we induced a new method in which we dry mix the Carbon nano tubes with the cement with help of Hobert Mixer (HM) at revolution of 360 rev/mint as shown in figure 05.

(a) (b)

Figure 05: - A pictorial representation of CNT dispersion (a) before mixing (b) after mixing in Hobert Mixer.

2.8 High Strength Concrete (HSC)

Concrete can be categorized as high strength concrete if its compressive strength is equal to or greater the 40 Mpa. As a result, in comparison to normal concrete, the preparation of high strength concrete (HSC) demands more quality control and analysis(Babu, 2013). In present era in order to use high strength concrete the proportion of water to cement in its production is reduced which ultimately demands the use of admixtures such as super plasticizers. To ensure a low pore size distribution and workability, normally we use silica fume for such purposes as silica oxides (SiO2) react with calcium hydroxides, which are hydration products of normal Portland cement, it is one of the most preferred pozzolans. which leads to lower heat release, increased strength, lime consumption, and a narrower pore size dispersion(Sahoo et al., 2019).

2.9 Silica Fume

After silicon alloys are treated in electric arc furnaces, silica fume is created as a by-product. So, the first step in making high-quality quartz is to heat it to 2000 degrees Celsius. After that, silica fumes are created by oxidizing silicon dioxide(Claisse, 2014).

The characteristics of silica fume are as follows:

- We can obtain greater early compressive strength by using SF
- We can get higher modulus, tensile, and flexural strength by using SF
- Toughness and bond strength can be raised by using SF
- Durability and abrasion resistance can be increased by using SF

2.10 Silica fume reaction mechanism

Silica fume is a significant supplemental cementitious binder for the production of high strength concrete. Silica fume densifies the concrete microstructure through its pozzolanic reaction particularly in the paste-aggregate interface zone .When blended with concrete, silica fume is initially inert. A pozzolanic reaction between silica fume and Calcium Hydro-oxide(C-H) occurs in many of the spaces around hydrated cement particles, yielding additional Calcium Silicate Hydrate (C-S-H) gel. This added C-S-H not only improves the compressive, flexural, and bond-strength properties of the concrete, but also provides a significantly denser matrix, especially in locations that would otherwise remain as microscopic voids prone to hazardous material penetration (Jensen, 2012).

2.11 Loading and heating regime-based testing technique

Most test programs that are being used to examine the deterioration of concrete's material characteristics at increasing temperatures employ one of three test methods: stressed, unstressed, or unstressed residual property tests. In the pre-heating step of the stressed test, a load of magnitude equal to 40% of the member capacity is applied. The member is heated at a set pace until it reaches the specified temperature, at which point it is maintained at that degree and no additional heating is allowed. A complicated assembly of furnace and loading equipment is required to perform tests under this loading and heating regime, in which the sample is placed in the furnace while the load is applied. Because unique preparations are necessary, it is a complex method that is not usually found in structural engineering laboratories. This feature is usually found in laboratories dedicated to studying the fire characteristics of buildings. This loading and heating regime accurately depicts the real-life situation of structures in the case of a fire. Unstressed test circumstances are the second testing scenario. The specimen or part is not loaded in preheating circumstances in this case. In contrast to the stressed approach, the member or specimen is not loaded, and heating is administered at a constant pace until the appropriate temperature is reached. The temperature is then maintained at a consistent level, and load is added at a predetermined pace until the structure fails. This heating and loading regime do not accurately represent the structural circumstances during a fire. Due to the lack of stressed test condition equipment, this testing condition becomes increasingly important. As compared to the findings obtained under unstressed test setting to those of stressed test conditions, they follow the same pattern, and the building under fire behaves similarly in each of these scenarios.

2.12 Previous research on the mechanical properties of high-temperature concrete

This section contains research papers that summarize flexural strength, elastic modulus, compressive strength , energies, compressive stress-strain behaviour, and loss of mass at high temperatures. Heating rate, testing procedures (stressed, unstressed, or unstressed residual), size of the specimens, moisture content during heating, type of course and fine aggregates, strength (NSC or HSC), cement content etc.

2.12.1 Compressive Strength

The essential input parameter in the structural design of RCC components is compressive strength, which is the most fundamental and crucial mechanical characteristic of concrete. Concrete is structurally active in compression as compared to most of the grey constructions. The change in concrete compressive strength as a function of temperature must be understood as an input parameter for the construction of structural prediction models for RCC components subjected to increased temperatures(Lawson et al). From an evaluation of several research papers it can be determined that the variability of concrete compressive strength as a result of fire is dependent on a number of variables, including strength at room temperature (NSC or HSC), nature of loading heating regimen (stressed, unstressed, or unstressed residual), heating rate, coarse aggregate type (normal weight siliceous and calcareous, or lightweight), and a variety of other variables.

As microstructure of high strength concrete (HSC) is dense and impermeable, vapor pressures generated by high temperatures rarely dissipate, resulting in unavoidable degradation in HSC compressive strength. HSC loses up to 40% of its room temperature strength between 100°C and 400°C, while NSC loses 10 to 20% of its room temperature strength in the same temperature range(Lawson et al.).

Recent research done by (Ergün et al., 2013) looked at the impact of two different cement concentrations, 250 and 350 kg/m3, on concrete behaviour at high temperatures. He employed limestone aggregates, ordinary Portland cement (OPC), a 0.50 w/c ratio, and a 2°C/min heating rate. Compressive strength testing was carried out using 150X150X150 mm cubes. Mixtures with cement concentrations of 250 and 350 kg/m3 had room temperature compressive strengths of 28.16 and 48.99 MPa, respectively. After test specimens were subjected to five different target temperatures: 100, 200, 400, 600, and 800 degrees Celsius, he looked at how compressive strength altered under unstressed residual conditions. To guarantee that each specimen attained thermal equilibrium, each temperature was held constant for 45 minutes. He arrived to the conclusion that the amount of cement in concrete had no influence on its behaviour at high temperatures.

According to (Georgali et al.)The rate of heating and the temperature at which it is heated have an impact on the qualities of concrete that has been exposed to high temperatures. Due to the quick heating rate, a higher vapor pressure develops, resulting in concrete fractures. When concrete is heated to 200°C or above, its compressive strength begins to deteriorate. Furthermore, according to (Suhaendi & Horiguchi, 2005), high-temperature residual compressive strength of high-strength concrete drops to 20% and 36% at 200 °C and 400 °C, respectively, as compared to room temperature. Furthermore, another author in their work found that compressive strength of high strength concrete samples were 70%, 67%, 53% and 37% at 100, 200, 400 and 600^{O}C respectively as compared to that of room temperature(Khaliq & Waheed, 2017). Furthermore in another research done by (Sedaghatdoost & Behfarnia, 2018) it was found that 0.1 % replacement of CNTs by weight of cement showed an optimum results as 0.1 wt% MWCNT showed an increase in the compressive, tensile, and flexure strength by 35, 8 and 11.2%, respectively. Similarly, in another research done by (Viana et al., 2020) found that 0.1% replacement by weight of cement showed an optimum results .It was also found that rate of heating has a great impact on strength detoriation as at fast rate of heating (9°C/mint) strength detoriates faster then slow rate of heating(2°C/mint).In other research done by (Lu et al., 2016) it was found that 0.05% replacement of CNTs showed an optimum results as introduced a new method of dispersion as compared to the standard practice of sonification so from all this literature review it can be concluded that method of dispersion , rate of heating and amount of CNTs effects the strength variations for different concrete mixes.

2.12.2 Flexural strength Test

The capacity of a material to withstand deformation generated by applied load is known as flexural strength. Most materials fail by tension rather than compression because they have low tensile stress. The maximum resistance to tensile stress before failure is defined as flexural strength(Gillani et al., 2017).The Modulus of rupture, also known as flexural strength of concrete, is an indirect measure of unreinforced concrete's tensile strength. It can be alternatively defined as the extreme fiber stresses when a part is bent. In addition to external force, warping, steel corrosion, drying shrinkage, and temperature gradients can all cause tensile stresses.

A number of tests and investigations about impact of CNTs in concrete has been conducted, one of which was conducted by (Mohsen et al., 2019) who discovered that the impact of CNT usage on concrete flexural strength, strain capacity, permeability, and microstructure was investigated in this study. It was found that Concrete batches weighing 0, 0.03, 0.08, 0.15, and 0.25 % by wt of cement. When compared to 0 percent CNT concrete, concrete with high CNT concentrations of 0.15 and 0.25 % by wt of cement enhanced flexural strength by more than 100 percent.

Similarly, (Naji et al., 2021) discovered that when carbon nano tubes were substituted with 0, 0.25, 0.5, 0.75, and 1 % by wt of cement the most significant improvement was observed when MWCNTs equivalent to 1 % by weight of cement were replaced. Compressive strength, splitting strength, and flexural strength were all increased by 38, 48, and 56 percent, respectively. MWCNTs increase the flexural behaviour of RC beams by up to 29 percent beyond the reference beam's ultimate load bearing capability

2.12.3 Stress-Strain Compressive response

Concrete's stress-strain compressive response is a critical analysis since it is a required input parameter in mathematical models that anticipate the reaction of a concrete structural element. To use a numerical approach such as finite element analysis to simulate the reaction of a concrete structural part exposed to fire, a constitutive model of concrete must be provided that can capture the strains at various stress levels and temperatures. As the temperature rises, concrete becomes more porous, and this increase in porosity leads concrete to become more ductile under compression. (Kodur et al., 2014).

Under unstressed circumstances, a study was conducted in which the stress-strain response of HSC mixtures containing fly ash was measured. The temperatures exposed were 250°C, 450°C, 550°C, 650°C, 750°C, and 850°C, with a heating rate of 2°C/min. For capturing the stress-strain response, they used a strain-controlled approach. The cylinders were 80 x 300 mm in size and had a compressive strength of 91.8 MPa at room temperature(Pachideh & Gholhaki, 2019).Similarly, many other researches has been conducted to capture stress-strain response such as (Cheng et al.) investigated the stress-strain behaviour of HSC under residual circumstances at various high temperatures. The rate of heating was 2°C/min. The compressive strength at room temperature was 71.4 MPa. The test specimens were cylinders measuring 100 x 200 mm. The stress-strain response was measured using a strain-controlled loading technique. They discovered that when exposure temperatures rise, the slope of stress-strain graphs decreases. They also discovered that peak strains at higher temperatures can be up to four times higher than peak strains at lower temperatures.

2.12.4 Elastic Modulus

Concrete's modulus of elasticity is an essential material property since it is used to determine the deflections of a concrete structural element. It is also an essential input parameter for RCC and elastic frame analysis. The w/c ratio, the age of the concrete, the processes used to condition it after casting and the type of aggregates used all influence the elastic modulus of concrete(Kodur et al., 2004). The elastic modulus of concrete, like other material characteristics, changes with temperature.

One of the research in this regard was conducted by (Xiao et al., 2016) as a heating rate of 2.5°C/min and a hold time of 2.5 hours was implemented and its impact on elastic modulus was studied along with other mechanical properties. The water-to-cement ratio used was 0.24, with a powder content of 580 kg/m3. Ground Granulated Blast-Furnace Slag(GGBFS) was used to replace 20% of the cement mass, while silica fume was used as 10% replacement by weight of cement. At room temperature, the compressive strength and elastic modulus of HSC were 68 MPa and 38GPa respectively. Prisms with dimensions of 100 x 100 x 300 mm were utilised. At temperatures ranging from 23 to 200°C, they discovered a significant drop in elastic modulus. The decrease grew steadier from 200 to 400°C, but from 400 to 600°C, the most intense rate of degradation was seen. At temperatures above 600°C, the rate of elastic modulus loss slowed. The slower rate of loss in elastic modulus after 600°C was attributed to calcination of limestone aggregates

Similarly in another research conducted by (Khaliq & Waheed, 2017) it was found that the elastic modulus of high-strength concrete samples falls as temperature rises; at 600°C, it was lowered by up to 40%.In another research conducted by (Suhaendi & Horiguchi, 2005), they found that elastic modulus of high strength concrete subject to high temperature reduces to 25% and 57% at 200ºC and 400ºC, respectively, as compared to room temperature.

In Another study conducted by (Khaliq & Taimur, 2018) on unstressed and residual test circumstances. They concluded that the loss in elastic modulus was high in the unstressed test conditions as compared to the residual test condition. They also found that at 400, 600, and 800 degrees Celsius, the decrease in elastic modulus was 26 %, 67%, and 87 %, accordingly, In contrast to room temperature in an unstressed test condition.

2.13 Research Gap

As we know that Carbon nanotubes (CNTs) are widely used commodity .But when it is used in the field of construction it experiences the problem in the dispersion part. So for that purpose the conventional method of dispersion up till now is ultra-sonification. Now the problem with sonification is that it is a very time-consuming process and secondly, it can only be done in the laboratories . So due to these problems we couldn't implement CNTs in field construction hence, by considering this issue we are checking the behaviour of Hobert mix carbon nanotubes for mass concreting in field. In addition to that, effectiveness of carbon nanotubes as plaster membrane against fire has been studied in this research. As no literature on its behaviour is available up till now.

Chapter 3

EXPERIMENTAL PROGRAM

3.1 General

A complete test program with varied concrete mix regimes is required to study the behavior of HSC reinforced with carbon nanotubes when subjected to increased temperature. The test thesis will include compressive and flexural strength tests, post crack energies, stress-strain graphs, elastic modulus, pre-crack energies, toughness index, and mass loss. Various authors have explored high strength concrete in depth in the literature, but there is no one set of studies that covers the mechanical characteristics of CNTs intruded in high strength concrete when it is exposed to fire as CNTs as cement substitute and as cement plaster. Basic mechanical tests, such as compressive and flexural strength tests, stress strain response, elastic modulus, and mass loss, will be performed at temperatures of 23°C, 200°C, 400°C, 600°C, and 800°C under residual test conditions to better understand the fire and spalling behavior of high strength concrete. This chapter goes through the specifics of the test program and technique, as well as the materials and mix regime. All of the mechanical and material parameters that were investigated were graphed, and the produced data was compared to control mixtures. Type of materials, techniques, and procedures employed in this investigation are described in depth in this chapter.

3.2 Experiment Design

Cylindrical specimens of dimension (300mmx150mm) along with small beam specimens of dimension (100mmx100mmx400mm) were fabricated from each batch of concrete mixes. After 28-days of curing these specimens were then exposed to different temperatures ranging from 23℃-800℃ . For compressive strength tests ASTM C-39 was used while for Flexural strength testing ASTM C239M was used. Residual test method was used to study the elevated temperature mechanical and material properties of the mixes. The material properties of all types of specimens were evaluated using this test method.

3.3 Materials

This section discusses the materials used in the experimental inquiry. Ordinary Portland Cement (OPC) Type-I in accordance with ASTM C150 (Ferraro & Ishee, 2010). OPC-Type 1 was utilised as a main binder in the formulations under investigation. Fine aggregate is from Lawrencepur with a fineness modulus of 2.34, whereas coarse aggregate is limestone coarse aggregate with a maximum size of 12.5 mm from Khairabad. Furthermore, varying percentages of multiwalled carbon nano tubes (MWCNTs) were employed as cement replacement and cement plaster along with 3rd generation Super-plasticizer and silica fumes. Table 02 gives a general idea about the physical properties of fine and coarse aggregates.

Table 02: Physical properties of aggregates

Aggregate type	**Limestone aggregate**	**Fine aggregate**
Size (mm)	12.5	-
Specific gravity (g/cm^3)	2.68	2.65
Impact value	9.5%	-
Maximum size of aggregate (mm)	12.5	-

3.4 Mix proportion

In this step we developed six formulation as such (0.05-CNTs, 0.1-CNTs, 0.15-CNTs, 0.1P-CNTs, 0.2P-CNTs, 0.3P-CNTs) along with the control Specimen. Carbon nanotubes(CNTs) dose of 0.05%,0.1% and 0.2 % by wt of cement was added to modify the concrete specimens. In case of plaster specimens 0.1%,0.2% and 0.3 % of CNTs were used as cement plaster by wt % of cement. All mixes are made with a low water/cement ratio of 0.35, with 3rd generation plasticizer concentration of 1% by weight of cement as shown in table 03. After 24 hours, the specimen were demolded and cured in water for 28 days . To analyze the strength evolution of formulations, compressive strength tests were done according to ASTM C39.While flexural tests were conducted according to ASTM C239M

Table 03: Mix Design proportions in Kg/m3

Mix ID	Cement		Silica Fume	Sand		Coarse Aggreg-ate	Carbon nanotubes		Water	Superplasticizer
	In concrete	In mortar	In concrete	In concrete	In mortar	In concrete	In Concrete	In mortar	In concrete	In concrete
HSC	612	--	61.2	694	--	1457	0	--	236	6.12
0.05-CNT	612	--	61.2	694	--	1457	0.306	--	236	6.12
0.1-CNT	612	--	61.2	694	--	1457	0.612	--	236	6.12
0.15-CNT	612	--	61.2	694	--	1457	0.918	--	236	6.12
0.1P-CNT	612	630	61.2	694	2120	1457	--	0.630	236	6.12
0.2P-CNT	612	630	61.2	694	2120	1457	--	1.260	236	6.12
0.3P-CNT	612	630	61.2	694	2120	1457	--	1.890	236	6.12

3.5 Dispersion and characterization of carbon nanotubes (CNTs)

Carbon nanotubes are carbon-based nanomaterial that are obtained from graphite. In theory, carbon nanotubes (CNTs) are cylindrical shells formed by rolling graphene sheets into a continuous cylinder(Zhang & Li, 2009). CNTs can play a key role in the building sector by altering cementitious composites. CNTs have exceptional material qualities, such as high elasticity and strength, which can make cementitious composites superior to traditional composites(Wilson, 2002b). But when they are mixed with concrete proportions the main issue that arises in its dispersion is that Carbon nanotubes tends to gets attracted to each other , due to the presence of Van der Waal's forces. Usually in order to properly disperse them the conventional method is that we use ultra-sonification with Gum-Acacia (GA) as surfactant with the help of Ultrasonic Pulse velocitor but due to lack of equipment we induced a new method in which we dry mix the Carbon nano tubes with the cement with help of Hobert Mixer (HM) as shown in figure 05.

3.6 Sample preparation

For each kind of formulation, concrete samples with dimensions of 150 x300 mm were casted for cylinder specimens and 100x100x400 mm were casted for beam specimens. For each temperature variation 02 samples were casted as shown in table 05 and 06. A trowel was used to finish the top surface of newly produced concrete. Concrete stayed in molds for 24 hours after pouring, then samples were de-molded and placed in curing tanks for 28 days in a controlled environment of 23 °C as shown in figure 06 and 07(A&B). After which Compressive strength tests and flexural strength tests were performed in accordance with ASTM C39 and ASTMC239M respectively, to verify the strength development.

Figure 06: Depicting the preparation phase

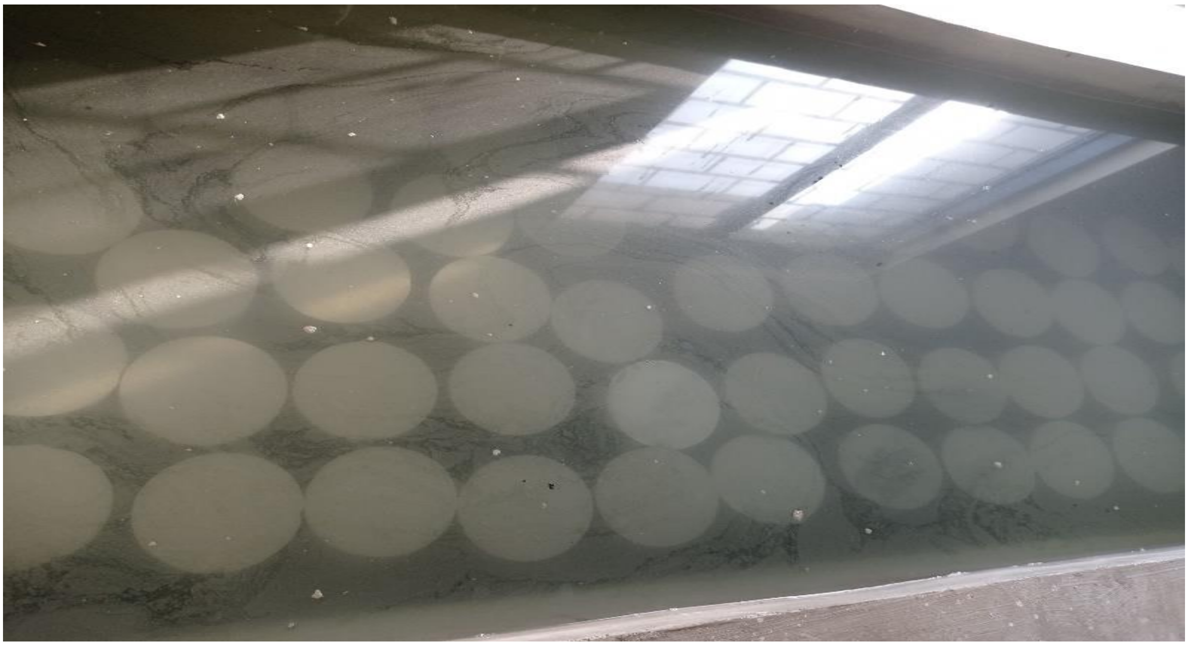


Figure 7A: Curing Phase of Cylinders

Figure 7B: Curing Phase of Beams

Table 04: Detail on number of test specimens, temperature levels and test conditions for Cylinders

Mix type	Exposure temperature (°C)	No of samples	Remarks
Cylinder specimen size 300×150 mm and heating rate of 5°C/minute were used.			
HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.05C-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.1C-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.15C-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.1CP-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.2CP-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.3CP-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	

Table 05: Detail on number of test specimens, temperature levels and test conditions for Beams**.**

Mix type	Exposure temperature (°C)	No of samples	Remarks
Prism specimen size 400×100x100mm and heating rate of 5°C/minute were used.			
HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.05C-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.1C-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.15C-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.1CP-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.2CP-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	
0.3CP-HSC	23	2	For residual test conditions
	200	2	
	400	2	
	600	2	
	800	2	

3.7 Fire loading characteristics

The test findings of samples that have been subjected to high temperatures are influenced by two main fire loading factors. Heating rate and targeted temperatures. Due to a lack of suitable higher temperature testing standards, these two properties were chosen based on previous research on concrete specimens at high temperatures.

3.8 Target temperature, heating rate and hold time for test method

From the previous studies the targeted temperatures of 23°C, 200°C, 400°C, 600°C and 800°C were selected for the tests as they are the most often used target temperatures for evaluating the quality of concrete samples. Aggregate types also have a crucial role in defining the fire behavior of concrete because calcareous aggregates tend to desiccate at 800°C so for that purpose strong aggregates with the impact value of 9% were used. The heating rate used in various researches to assess high temperature mechanical characteristics of concrete had a range of (2-5)/minute. In this study, a heating rate of 5°C per minute was used. As proper hold time must be supplied to the sample under heat treatment in order to achieve thermal steady state so for that purpose thermocouple were used to determine exact hold time for the specimens to achieve the targeted temperature. A typical thermocouple is simply a wire combination that is used to detect temperature via data logger. The thermocouples employed in this investigation were K-type thermocouples. A suitable hold time according thermocouple values was given for all the mixes, such that they can achieve thermal steady state.

3.9 Compressive strength test ($f_{c'\,T}$)

To minimize thermal spalling, all of the cylinders were allowed to come down to ambient temperature before being examined under residual conditions. The ASTM C39 standard was used to measure compressive strength. As there are no other testing standards available in the literature that include high-temperature compression testing. In compression tests, the loading rate is 0.2MPa per second was implemented as mentioned in ASTM. At each target temperature, cylinders were tested in compression testing machine as shown in figure 17.

Figure 8: Compressive Strength testing machine.

3.10 Flexural Strength (*R*)

The modulus of rupture also known as flexural strength of unreinforced concrete is referred to as an indirect measure of tensile strength. When bending a member, the modulus of rupture may alternatively be defined as calculation of the extreme fiber stresses caused due to the bending moment. Other factors, such as warping, corrosion of steel, drying shrinkage, and temperature gradient, may contribute to tensile stresses in addition to external force. In order to maintain a continuous and shock-free load on the specimen the load was applied at constant rate. The load will be applied to the tension face at a rate of 0.9 to 1.2 MPa per minute [125 and 175 psi per minute] in accordance with (ASTM C239M) .Flexural test assembly according to ASTM C239M is shown in figure 18 along with Universal testing machine used for this purpose in figure 19 .

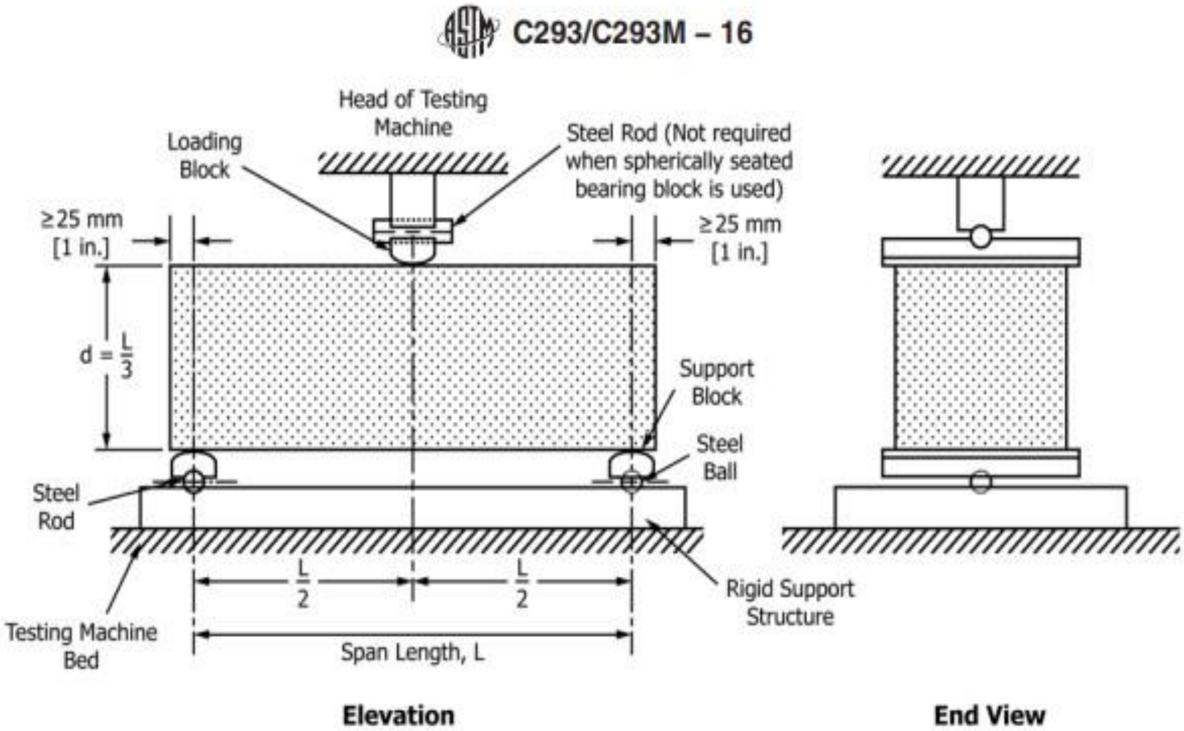


Figure 9: Flexural Test Assembly (ASTM C-239M)

In order to determine the Modulus of rupture following mathematical formulation is used

modulus of rupture

$$R = \frac{3\,PL}{2bd^2}$$

Where:

R = Modulus of rupture (flexural strength), MPa [psi],

P = Applied maximum load N [lb.],

L = Effective span length, mm [in.],

b = Specimen Width, mm [in.],

d = Specimen Depth, mm [in.].

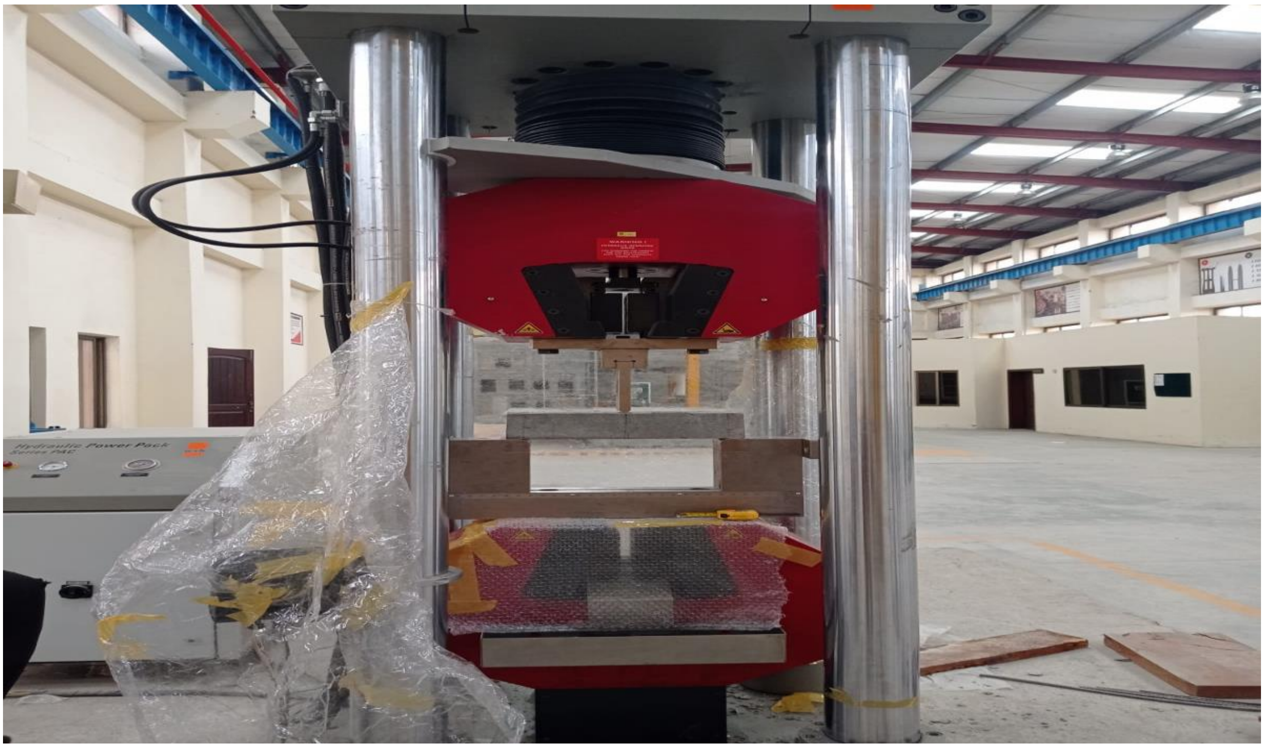


Figure 10: Universal Strength testing machine for flexural strength test

3.11 Stress-strain curve

Load-deformation data was captured using a data collection system coupled to a compression testing machine in order to produce stress-strain curves, which were then plotted. Cylinder and beam specimens data was collected and plotted for stress-strain curves for the loading rate of 0.2mpa/sec and 1.2mpa/mint respectively.

3.12 Elastic modulus

The stress-strain curves defined in ASTM C469 were than used to assess secant elastic modulus (E) concrete specimens.

$$E = \frac{S2 - S1}{€ - 0.000005}$$

E = Elasticity of Chord modulus

S2 = Value of stress against 40 % ultimate stress

S1= Value of stress against the longitudinal strain

ε =Value of Longitudinal strain against S1

3.13 Mass loss

Weighing concrete samples before and after heat treatment was done to see whether there was a difference in mass. The following relationship was used to compute relative mass loss at various temperatures.

$$\text{Mass Loss} = \frac{\text{Mt}}{\text{M}}$$

Mt = Mass at Specific temperature (T)

M = Mass at ambient temperature

CHAPTER 4

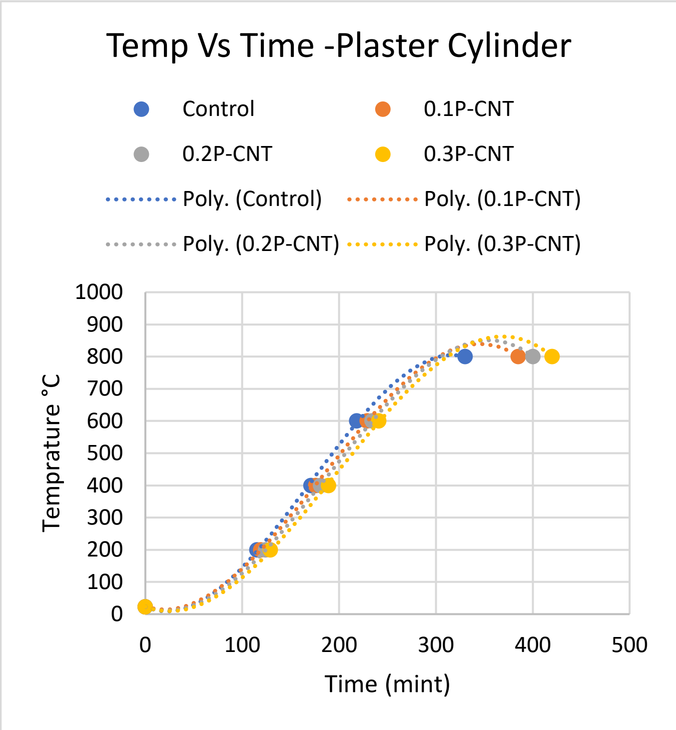
RESULTS AND DISCUSSIONS

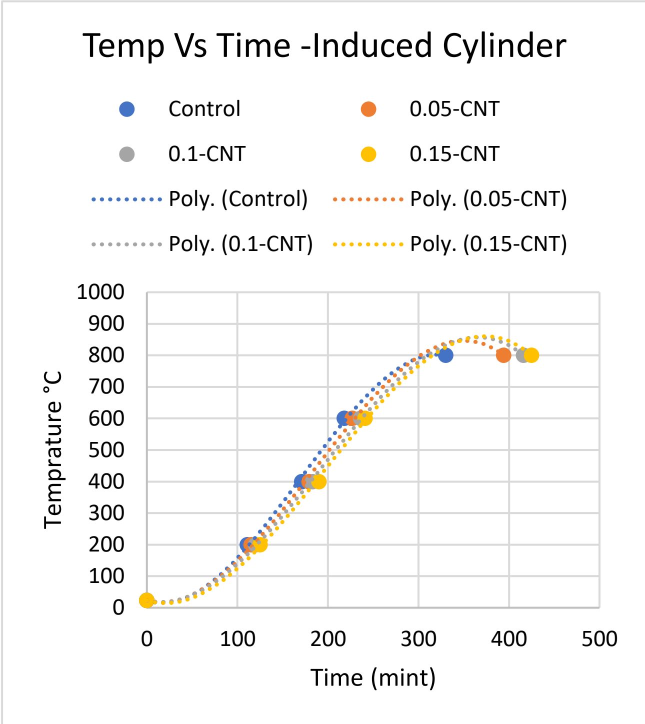
4.1 General

This chapter gives a detail interpretation of the obtained experimental results. Such as variation in strength (Compressive and flexural) with comparison to Control HSC specimens along with other mechanical properties such as stress-strain response, compressive toughness, Crack energies, modulus of elasticity, visual evaluation and mass loss.

4.2 Time-Temperature Variations

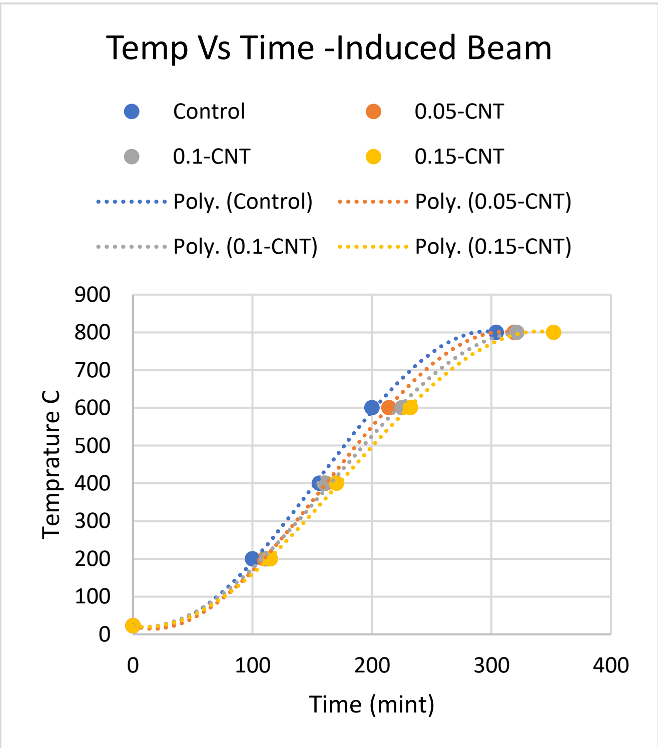
To obtain compressive and flexural strength, all specimens were evaluated according to ASTM-C39 & ASTM C239/C239M-16 respectively. The stress-strain curves were produced using a regulated loading rate of 0.2 Mpa/Sec in case of cylinder specimens .While for the beam specimens a loading rate of 1.2 Mpa/mint was used according to the standard ASTM. Based on the parameters that were given, the stress-strain findings of the response curves were also utilised to determine modulus of elasticity. For temperature control a furnace of 1200 °C capacity was used in which a computerized temperature-time controller was installed to control time and to maintain it according to the desired conditions. To record the change in temperature and ensure that equal heat distribution is being provided to the concrete specimens, K-type thermocouples were implanted in the core of a typical samples. Temperature readings for representative samples were taken during the testing, which aided in determining the needed hold time and heating rate according to RILEM test protocols.("Recommendation of RILEM TC 129-MHT 'Test Methods for Mechanical Properties of Concrete at High Temperatures' Modulus of Elasticity for Service and Accident Conditions," 2004). To obtain the desired temperatures, the samples were heated at a controlled rate of 5°C/min. A hold period of 2.5 hours (150 minutes) was given to preserve thermal equilibrium (stationary state) conditions based on temperature-time graphs as shown in figure 11 and figure 12**.** The mentioned figures show the temperature-time variation of control HSC samples as well as multi-walled carbon nanotubes specimens. To minimise heat shock, all of the specimens were allowed to cool down up to ambient temperature. The targeted temperature was 23°C (ambient), 200°C, 400°C, 600°C, and 800°C, respectively. The weights of all the specimens were recorded before and after the fire exposure to calculate mass loss.

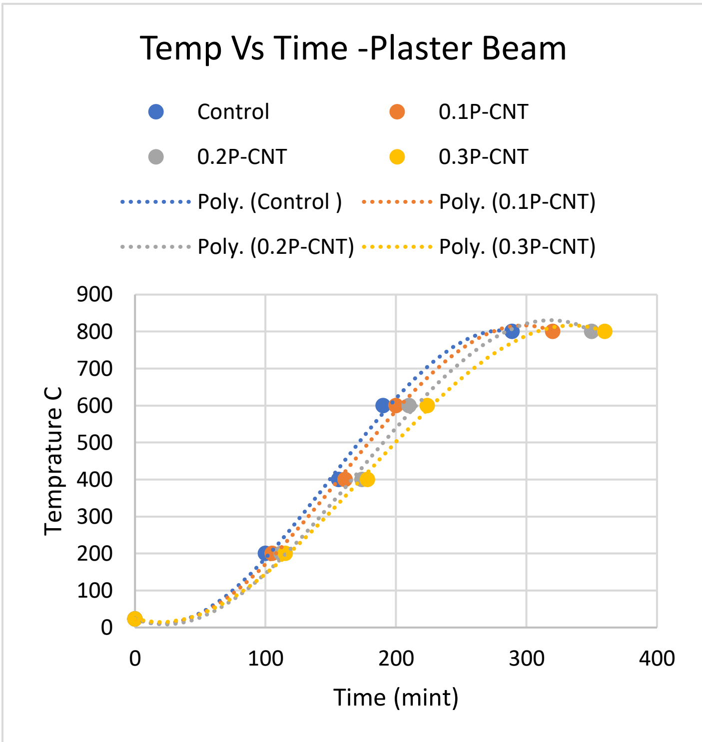




(a) (b)

Figure 11 : Temperature vs time variation for (a) plaster cylinders (b) induced cylinders





(a) (b)

Figure 12: : Temperature vs time variation for (a) induced beams (b) plaster beams

4.3 Compressive strength

4.3.1 For induced CNTs

All concrete specimen formulations (such as 0.05-CNT, 0.1-CNT, and 0.15-CNT) cylinders showed an increase in the compressive strength due to the inducement of carbon nanotubes as compared to the control specimens. The inclusion of nanotubes improved the microstructure of modified materials (0.05-CNT, 0.1-CNT, and 0.15-CNT), and the compressive strength of the samples rose by 10%, 21%, and 33% respectively, when evaluated at ambient temperatures as shown in figure 13. Using nano media as a reinforcement in the host matrix, the microstructure was improved own to filling of gaps, which led a boost in strength. Under residual conditions, both high-strength concrete and modified high-strength concrete exhibit a similar pattern of relative residual compressive strength loss up to 100 ℃. However, the strength loss becomes more obvious between 200 and 800 degrees Celsius in control samples that eventually spalled above 600 degrees Celsius. Every single changed formulation demonstrated superior strength retention throughout a range of temperatures (from 23 to 800 °C) as compared to the control HSC formulation. The behaviour of (0.05-CNT, 0.1-CNT, and 0.15-CNT) was almost identical . Heat is dispersed uniformly in the presence of thermally conductive nanotubes in the cementitious matrix, reducing the effects of thermal stress on the structure. The fundamental cause of the formation of heat pockets, which eventually results in thermal cracking, is the concentration of thermal stresses.(Latif Baloch et al). At 600 °C, 0.05-CNT, 0.1-CNT, and 0.15-CNT kept around 33 percent, 40 percent, and 49 percent of their strengths, respectively, whereas control samples retained about 29 percent of its original compressive strength. At 800 °C, 0.05-CNT, 0.1-CNT and 0.15-CNT preserved 12%, 16%, and 23% of their strength respectively, while control HSC spalled above 600 °C. Thermally conductive medium not only assisted to efficiently distribute thermal stresses, but they also aided to minimise thermal inertia.

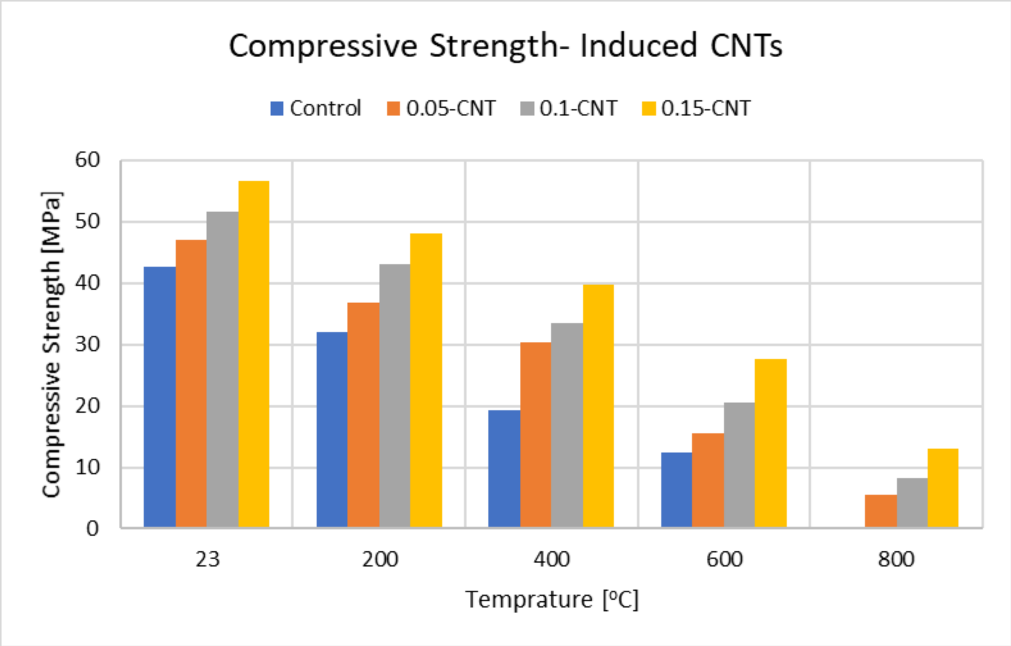


Figure 13: Showing Compressive Strength Variations For Induced CNTs-HSC

4.3.2 For Plaster CNTs

Carbon nano tubes were placed externally in case of plaster specimens to act as membrane against fire .The specimens with 0.1, 0.2, and 0.3% CNT plaster retain their properties much better as compared to the control specimens as shown in figure 14. At 200 °C first substantial change in hydrothermal state was observed due to the loss of both free and adsorbed moisture. At 400°C 0.3P-CNT plaster specimens out-performed all of the other formulations. At 600 degrees Celsius, the 0.1P-CNT, 0.2P-CNT, and 0.3P-CNT maintained up to 35%, 40%, and 43% respectively, whereas the control specimen retained up to 29 percent. Additionally, at 800 °C, 0.1P-CNT, 0.2P-CNT, and 0.3P-CNT preserved up to 16%, 17%, and 24% of their original properties, respectively.

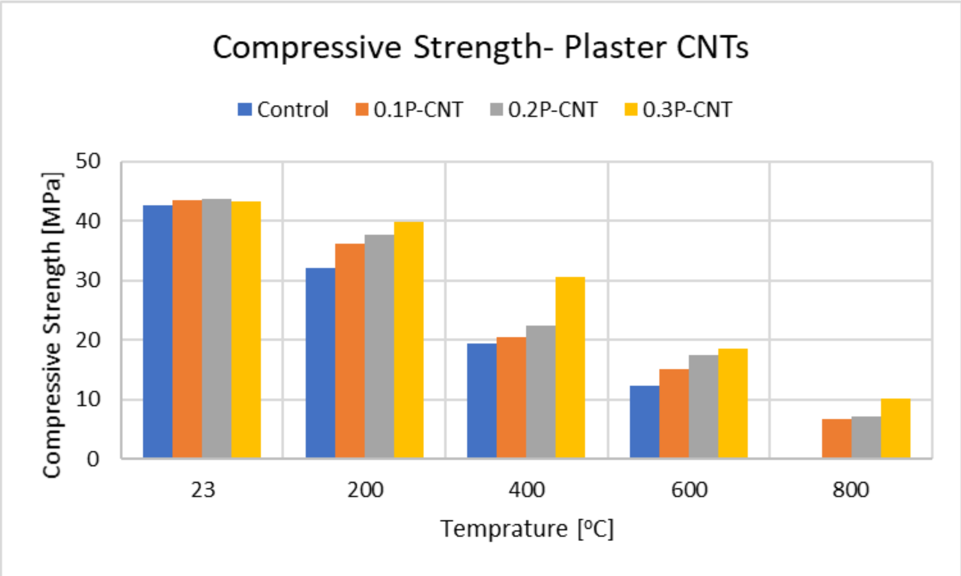


Figure 14: Showing Variation for Carbon nano tube Plaster HSC specimens

4.4 Stress Strain Evaluation

4.4.1 For induced Specimens

Temperature-dependent Stress Vs Strain values are shown for the Carbon nanotubes Induced specimens. All the Cylinder specimens were [150x300 mm] according to ASTM C-39. In all of the Figures it can be seen that there was no specific difference in strength retainment between 0.15-CNT and 0.1-CNT formulations up to 200^{o}C.But after that 0.15 % modified samples outperformed all of the other modified formulations as at 200 , 400 ,600 and 800 ^{o}C they retained up to 84 %,71%,49% and 23% of their original values. While 0.1 % modified samples at 200, 400 ,600 and 800 ^{o}C retained up to 83 %, 64 %,40% and 16% of their original values. And 0.05 % of the modified samples at 200, 400 ,600 and 800 ^{o}C retained up to 78%, 65% ,34% and12% of their original values.

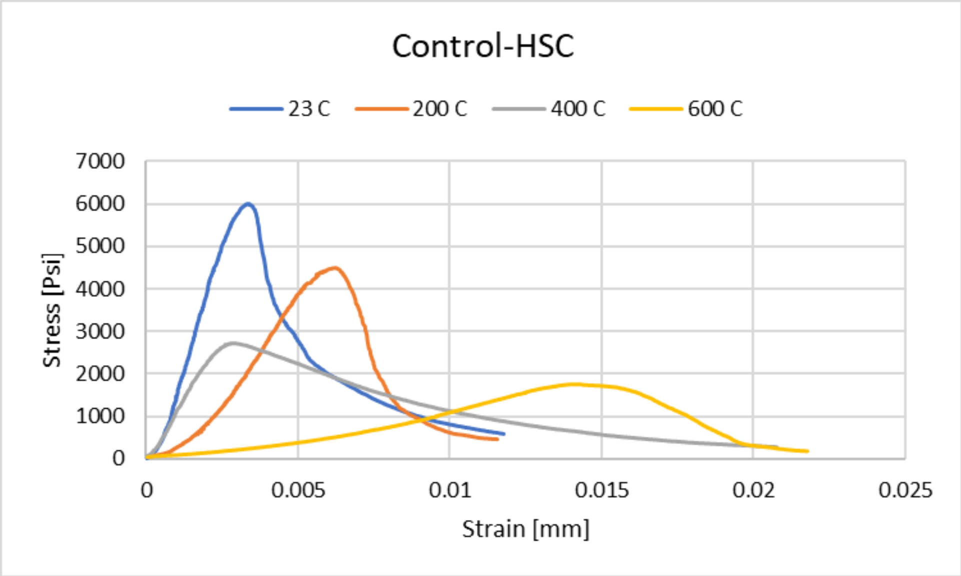


Figure 15: Variation of Stress-Strain evaluations for Control Cylinders

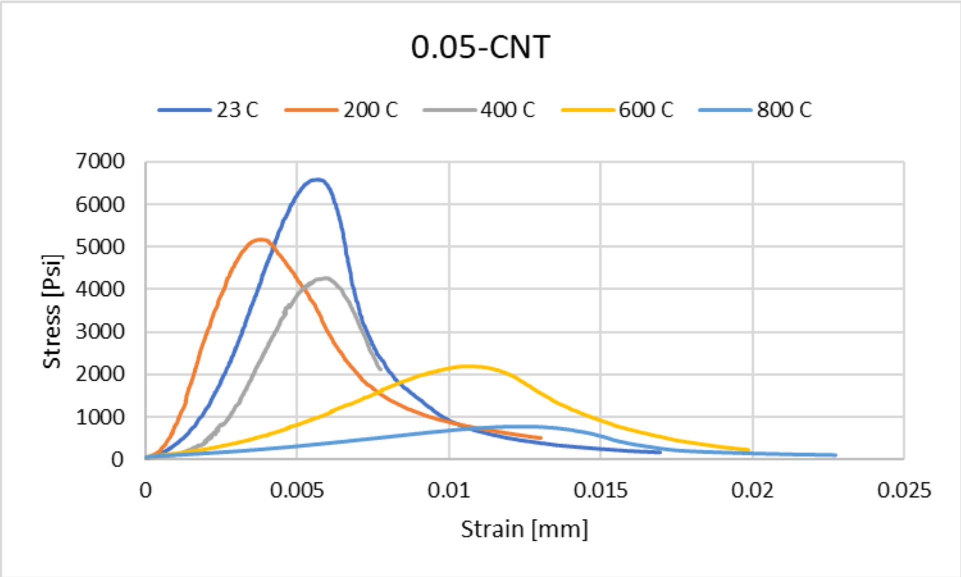


Figure 16: Variation of Stress-Strain evaluations for 0.05-CNT induced Cylinders

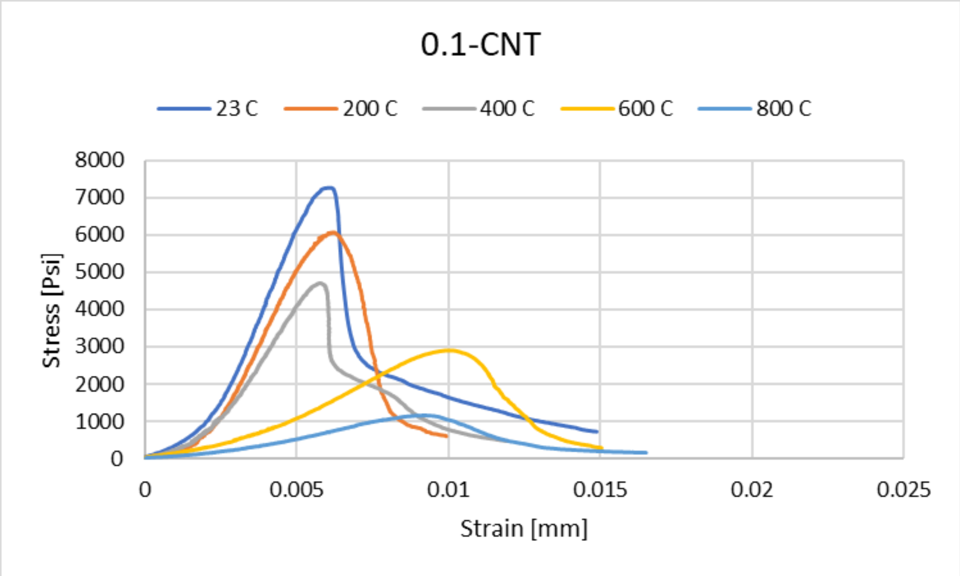


Figure 17: Variation of Stress-Strain evaluations for 0.1-CNT induced Cylinders

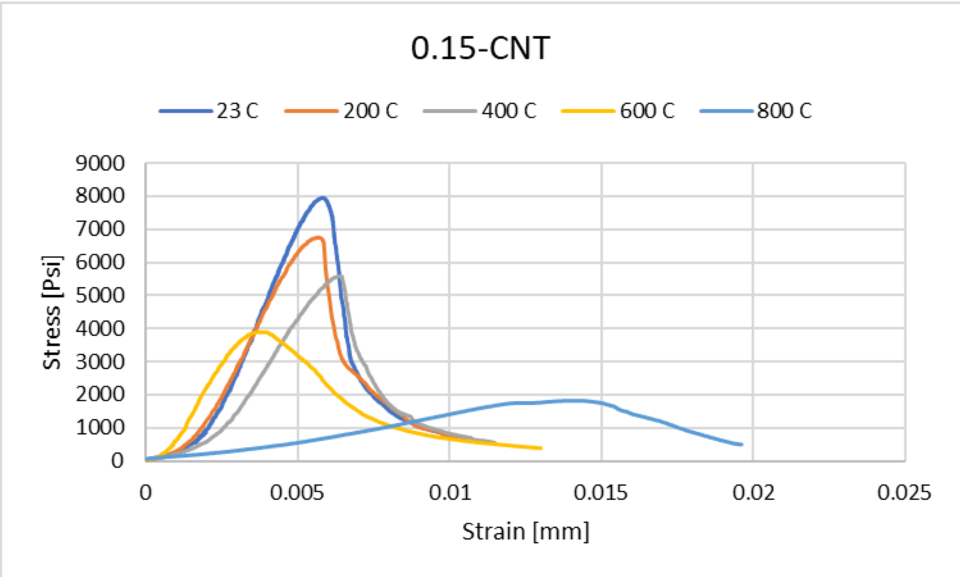


Figure 18: Variation of Stress-Strain evaluations for0.15- CNT induced Cylinders

4.4.2 For plaster Specimens

Temperature-dependent Stress Vs Strain values are shown for the Carbon nanotubes plaster specimens. All the Cylinder specimens were [150x300 mm] according to ASTM C-39. In all the Figures it can be seen that 0.3P plaster modified samples outperformed all the other modified formulations as at 200 , 400 ,600 and 800 ^oC they retained up to 91%, 71%,43% and 29% of their original values. While 0.2P plaster modified samples at 200, 400 ,600 and 800 ^oC retained up to 85%,51%, 40% and 17% of their original values. And 0.1P plaster of the modified samples at 200, 400 ,600 and 800 ^oC retained up to 83%, 47%,34% and 15% of their original values.

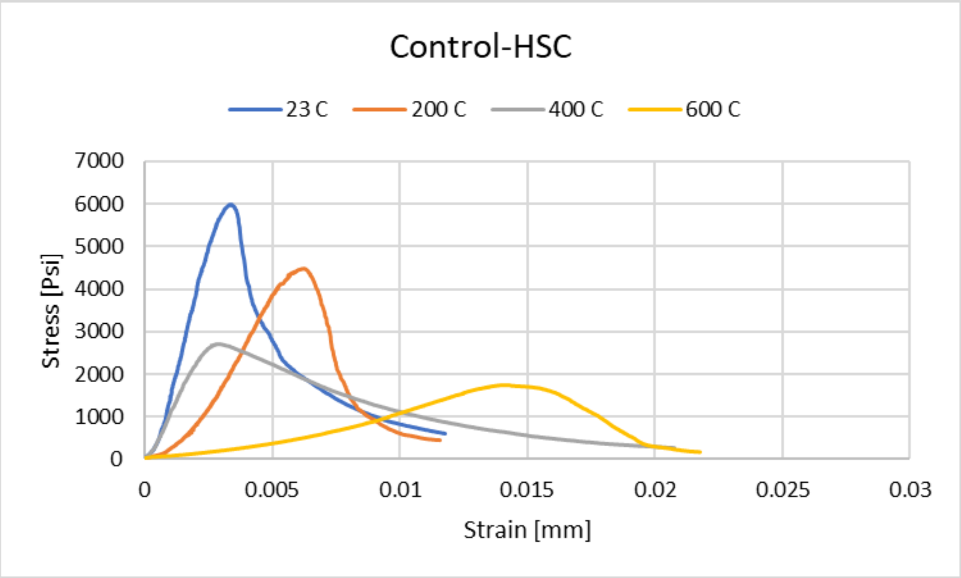


Figure 19: Stress-Strain variation for CNT Control Cylinders

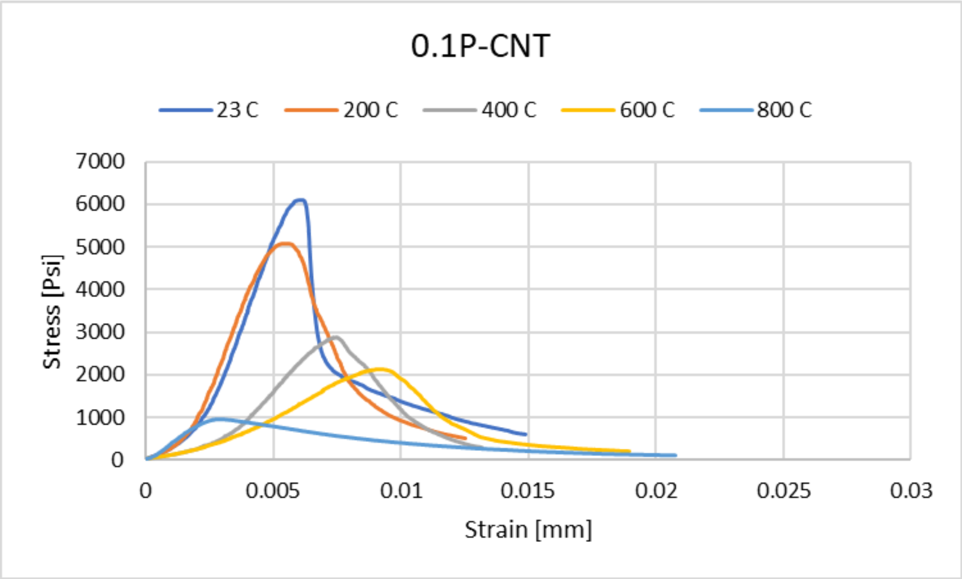


Figure 20 : Stress-Strain variation of for 0.1P-CNT Plaster Cylinders

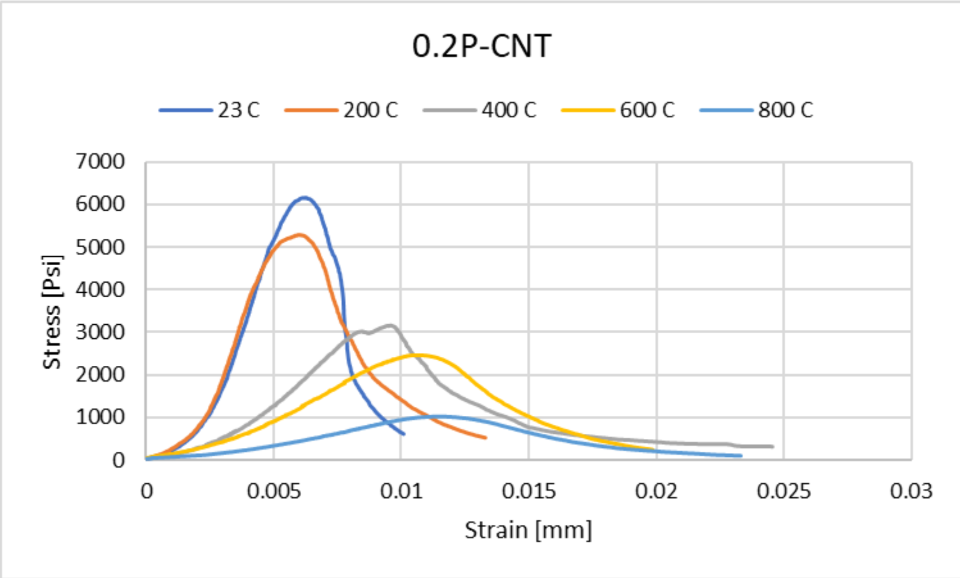


Figure 21 : Stress-Strain variation for 0.2P-CNT plaster cylinders

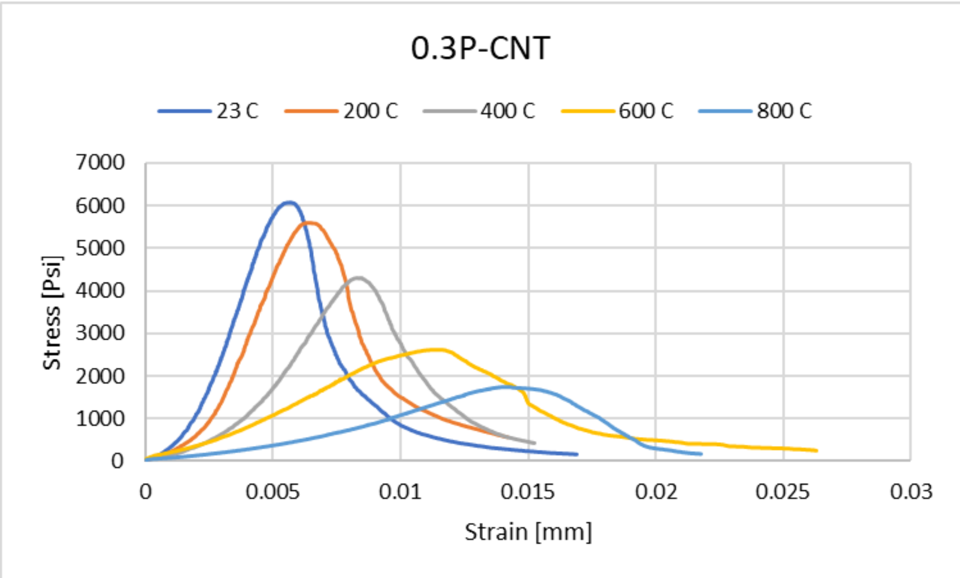


Figure 22 : Stress-Strain variations for 0.3P-CNT Plaster Cylinders

4.5 Flexural Strength

4.5.1 for Induced CNTs

The flexural strength of unreinforced concrete, commonly known as the Modulus of rupture, is an indirect indicator of the material's tensile strength. The modulus of rupture, while bending a member, may also be described as the measure of the severe fiber stresses that occur as a result of bending. In addition to external force, other variables such as warping, steel corrosion, drying shrinkage, and temperature gradient may contribute to tensile stresses. According to the patterns, when temperatures rise, both types of concrete, namely control (HSC) and modified (0.05-CNT, 0.1-CNT, and 0.15-CNT) Beams, lose flexural strength. The results show that all of the induced specimens exhibit excellent increases in strength at ambient levels as modified 0.05-CNT, 0.1 CNT, and 0.15 CNT exhibit increases in strength by 14%, 19%, and 50% respectively as shown in figure 23, when compared to Control specimens, and that they retain their properties significantly better than Control specimens. Furthermore, at 600 °C, the 0.05-CNT, 0.1-CNT, and 0.15-CNT specimens preserved about 46%, 53%, and 48% of their original strength respectively, while the control specimen retained nearly 40% of its original strength. At 800 °C, the control specimen spalled because to the significant temperature fluctuation, while the 0.05-CNT, 0.1-CNT, and 0.15- CNT specimens, in contrast to the control specimen, kept about 37%, 41%, and 39% of their strength at 800 °C, respectively from this we can conclude that although at ambient level 0.15% formulation outperformed all the other formulations but at elevated temperatures 0.1% formulation retained their strength better as compared to the other formulations.

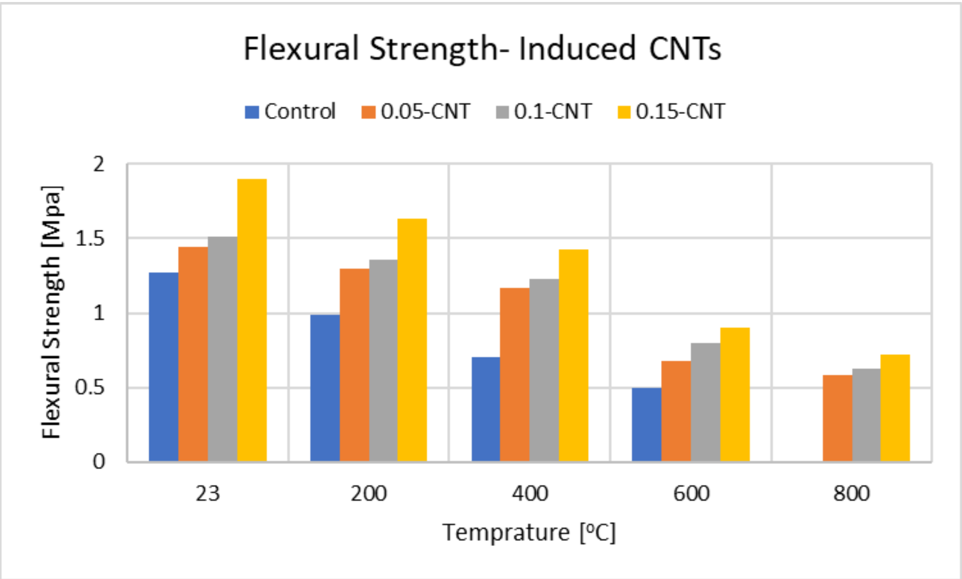


Figure 23: Depicting the flexural strength variation for induced CNTs

4.5.2 For Plaster CNTs

The specimens in which a membrane of carbon nanotubes was provide as plaster preserved their properties much better than those of the control specimens. However, the total Flexural strength of concrete did not improve at ambient level as Carbon Nanotubes were employed as plaster because the CNTs were put externally, The specimens treated with 0.1, 0.2, and 0.3 CNT kept their mechanical qualities much better than the control specimens. At 200 °C first substantial change in hydrothermal state occurs, which results in the loss of both free and adsorbed moisture. In this way, up to 400 °C, it showed a similar trend. At 600 degrees Celsius, the 0.1P-HSC, 0.2P-HSC, and 0.3P-HSC maintained up to 43%, 44%, and 46%, respectively, whereas the control specimen retained up to 34%. Additionally, at 800 °C, 0.1P-CNT, 0.2P-CNT, and 0.3P-CNT preserved up to 36%, 37%, and 38% of their original properties, respectively as shown in figure 24.

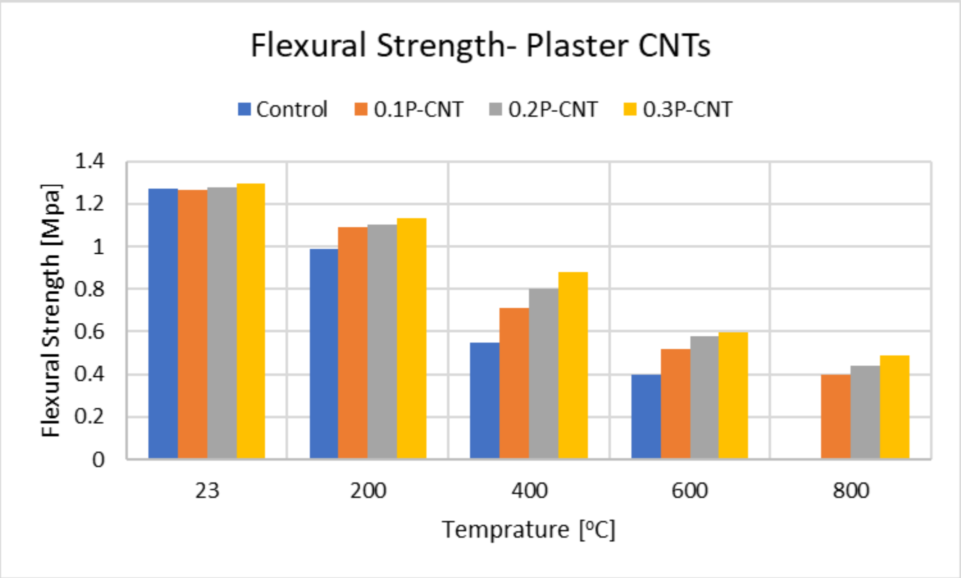


Figure 24: Showing the Flexural Strength Variation for Plaster CNT-HSC

4.6 Load Vs Displacement Variations

4.6.1 For induced specimens

The ultimate load at which specimens failed at different temperatures was measured using a load system for flexural tests. Temperature-dependent Load Vs Displacement values are shown for the Carbon nanotubes Induced specimens. All the beam specimens were [100x100x400mm] according to ASTM C293/C239M-16.In all the Figures as shown below it can be seen that 0.1 % modified samples outperformed all of the other modified formulations in terms of strength retainment at elevate temperatures as at 200 , 400 ,600 and 800 ^{o}C they retained up to 87%, 79%, 57% And 41% of their original values. While 0.15 % modified samples at 200, 400 ,600 and 800 ^{o}C retained up to 85%, 77%, 57 % and 37% of their original values. And 0.05 % of the modified samples at 200, 400 ,600 and 800 ^{o}C retained up to 84%, 67% ,49% and35% of their original values

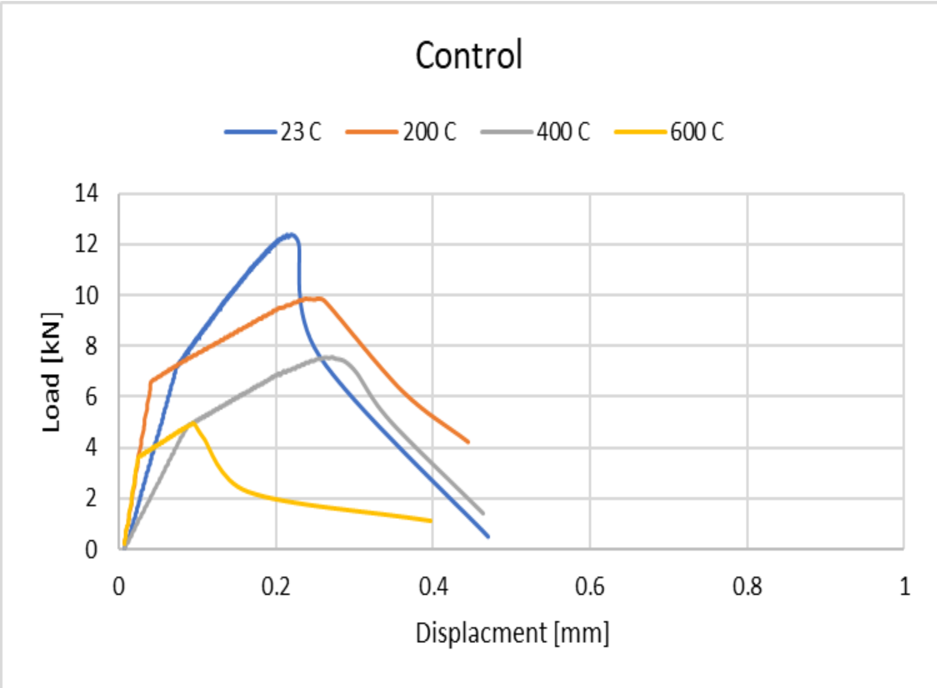


Figure 25: Load vs Deflection variations of Control beam Specimens at different elevated temperatures.

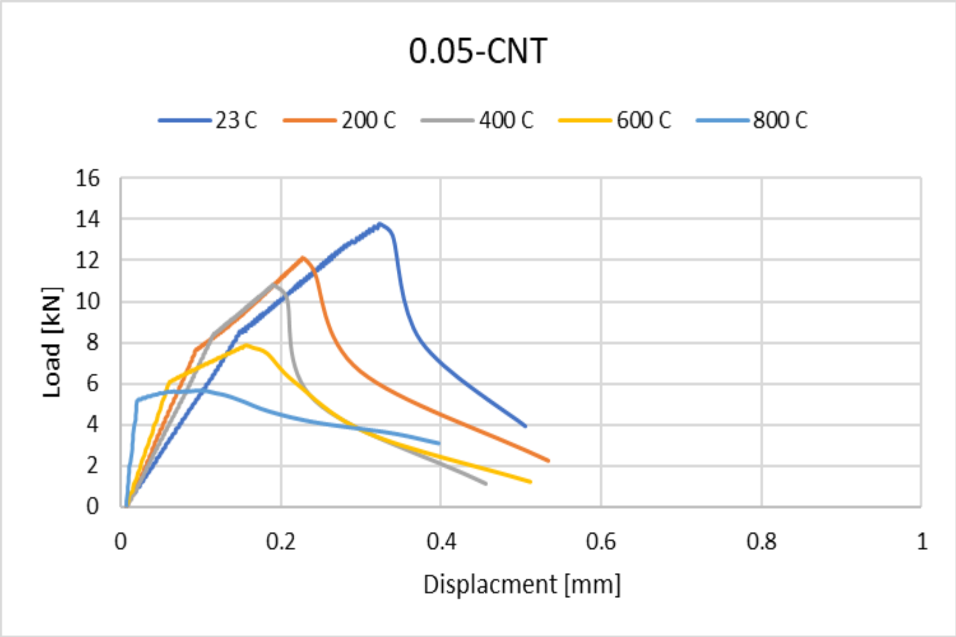


Figure 26: Load vs Deflection variations of induced 0.05-CNTs beam Specimens at different elevated temperatures.

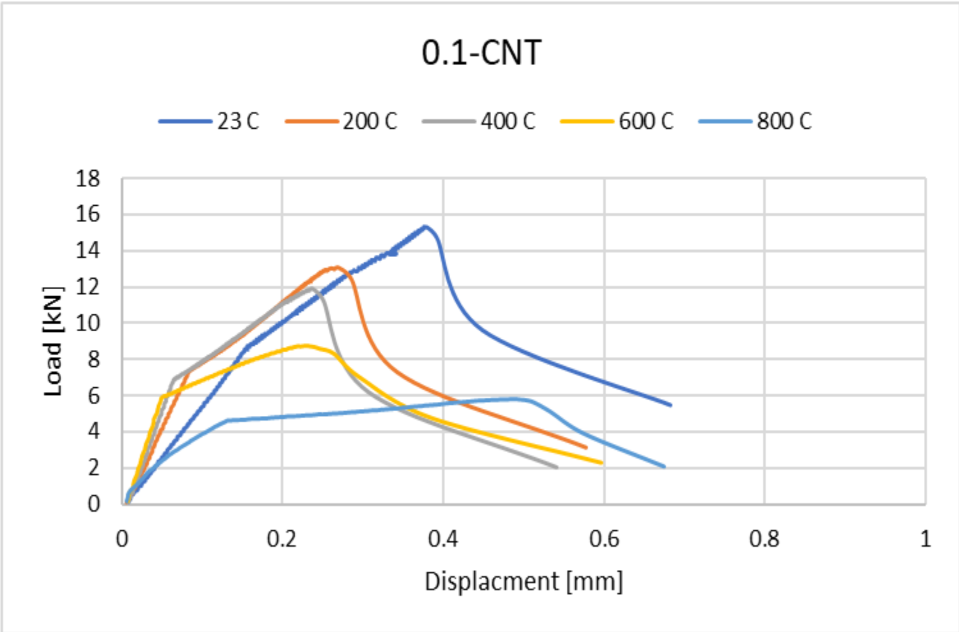


Figure 27 : Load vs Deflection variations of induced 0.1-CNTs beam Specimens at different elevated temperatures.

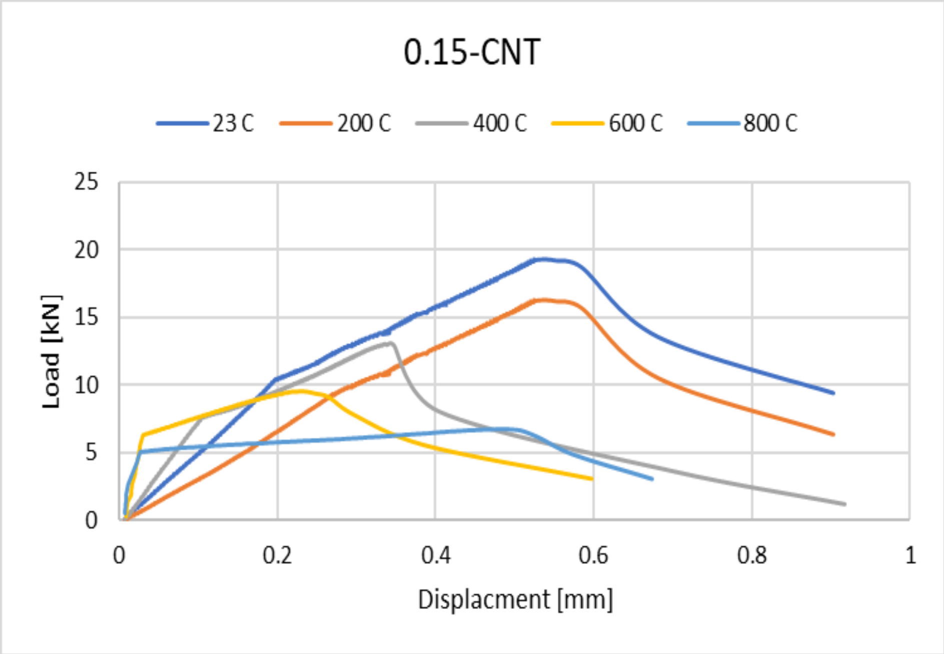


Figure 28 : Load vs Deflection variations of induced 0.15-CNTs beam Specimens at different elevated temperatures.

4.6.2 For Plaster beam

The ultimate load at which specimens failed at different temperatures was detected during flexural test assembly. Temperature-dependent Load Vs Displacement values are shown for the Carbon nano-tubes Plaster specimens. All of the beam specimens were [100x100x400 mm] according to ASTM C293/C239M-16. . In all the Figures as shown below it can be seen that there was no specific difference in strength retentions for all the formulations .But 0.3P plaster modified samples outperformed all the other modified formulations as at 200 , 400 ,600 and 800 ^{o}C they retained up to 80%, 67%,47% and 36% of their original values. While 0.2P plaster modified samples at 200, 400 ,600 and 800 ^{o}C retained up 79%, 62%, 47% and 35% of their original values. And 0.1P plaster of the modified samples at 200, 400 ,600 and 800 ^{o}C retained up to 78%, 60%, 44% and 32 % of their original values.

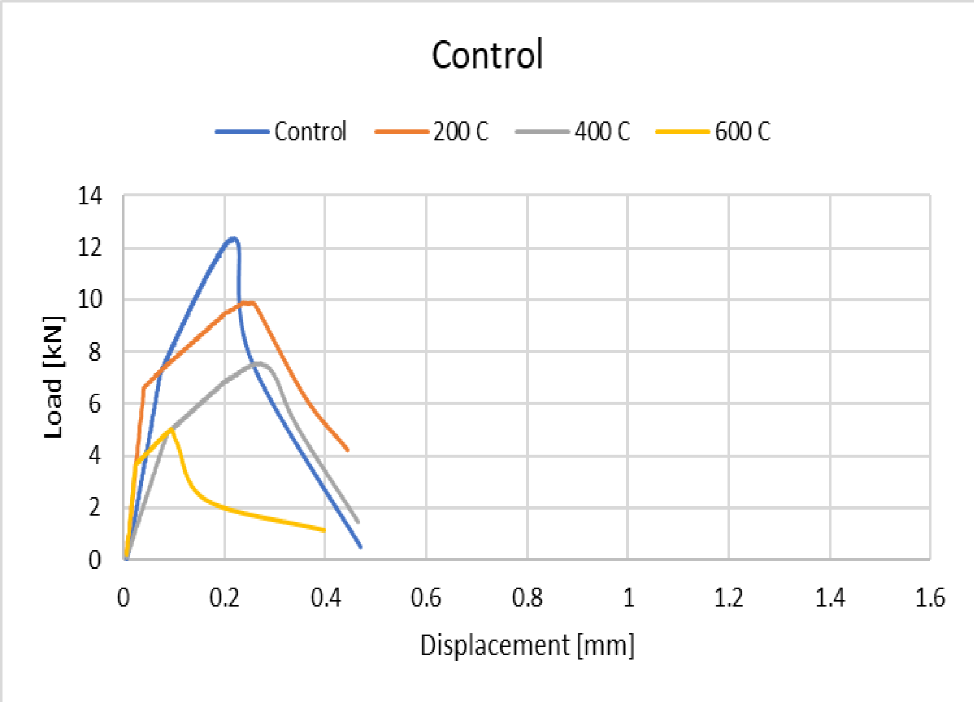


Figure 29: Load vs Deflection variations of Control beam Specimens at different elevated temperatures.

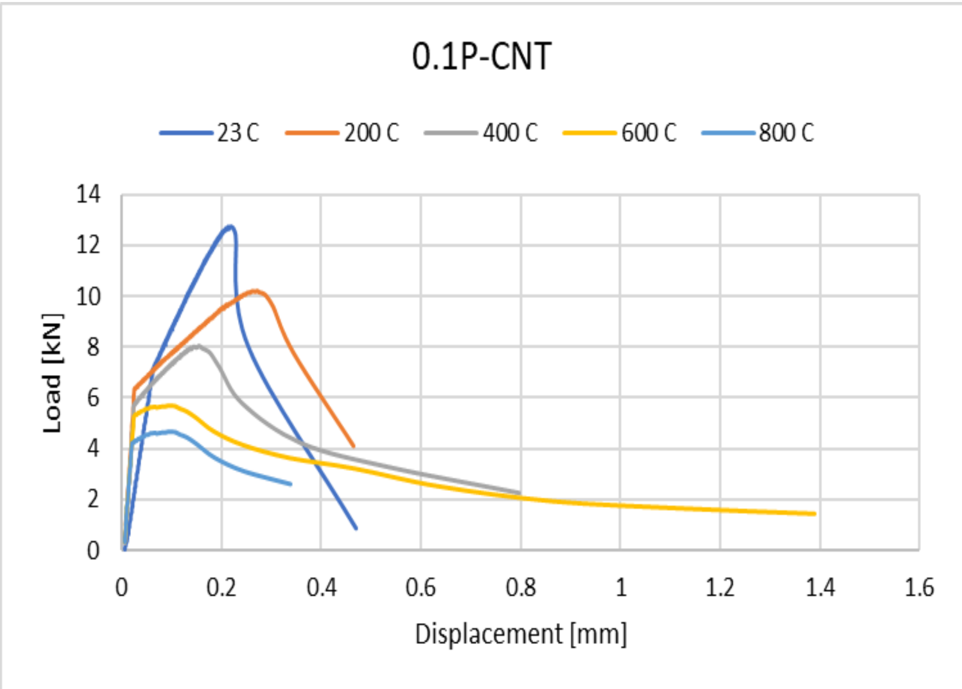


Figure 30: Load vs Deflection variations of 0.1P-CNT plaster beam Specimens at different elevated temperatures.

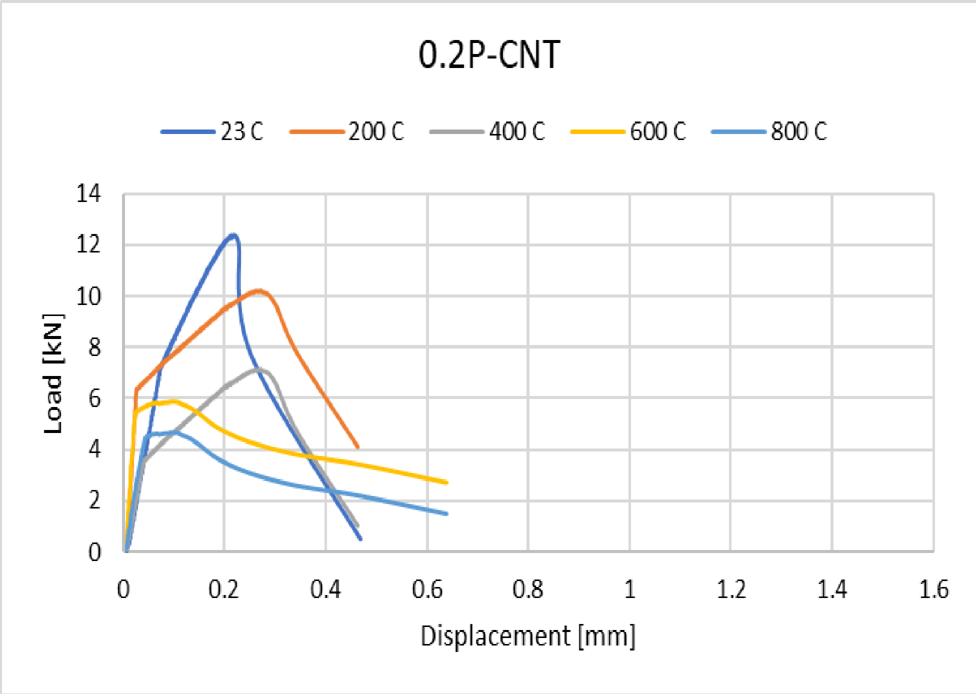


Figure 31: Load vs Deflection variations of 0.2P-CNT plaster beam Specimens at different elevated temperatures.

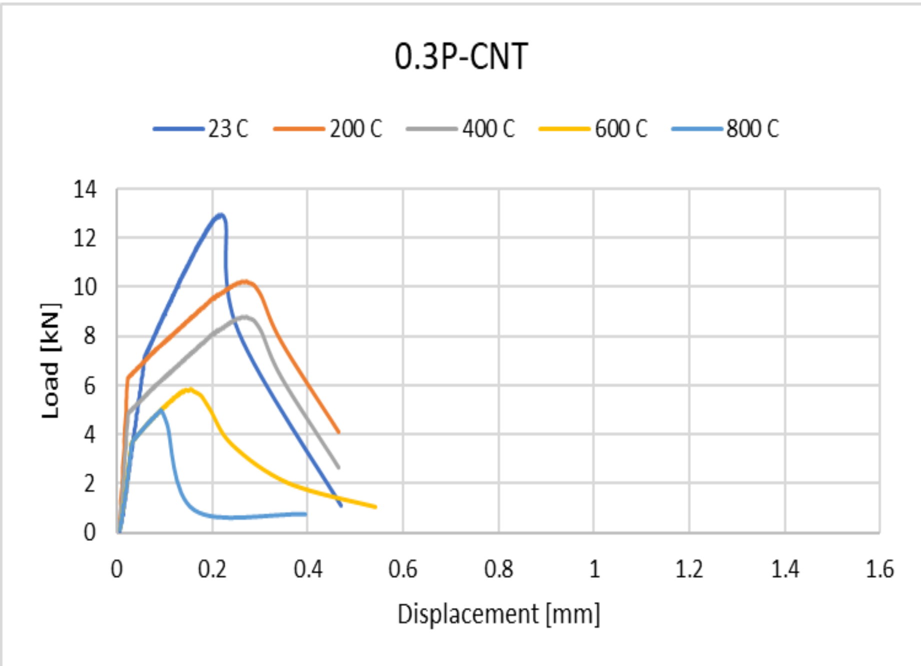


Figure 32: Load vs Deflection variations of 0.3P-CNT plaster beam Specimens at different elevated temperatures.

4.7 ELASTIC MODULOUS

4.7.1 For Induced Specimens

To assess the strength of HSC and modified HSC concrete (0.05-CNT, 0.1-CNT, and 0.3CNT), compressive stress-strain data must be used to compute elastic modulus (E), which is described by ASTM C469(Taira de Vasconcellos et al., n.d.). In order to investigate the elastic moduli, strains equal to 40% of the stress value are taken into account. The average increase in 0.05-CNT, 0.1-CNT, and 0.3-CNT is around 7%, 10%, and 16% in case of Cylinder specimen respectively. However, Beam specimens shows nearly 4%, 9%, and 20% increase in elastic modulus as compared to control specimen as a result of nano modification in the ambient environment. Based on elastic modulus decomposition it is clear that modification increases the retention of modulus and decreases the relative loss in changed samples, particularly in the range up to 400 to 800 ^{o}C, due to the existence of nano-reinforcement and their reinforcing activity. The results were predicted since the compressive strength of these mixes was less affected than that of the control specimen, and they exhibited ductile behaviour in contrast to the control specimen. In comparison to HSC which spalled after 600 ^{o}C the modified samples preserved 35 %, 40 %, and 47 % of their elastic modulus at 800 ^{o}C for the 0.05-CNT, the 0.1-CNT, and the 0.15-CNT respectively in case of cylinder specimens. Similarly, the modified samples preserved 58%, 61 % and 64 %, of their elastic modulus at 800 ^{o}C for the 0.05-CNT, the 0.1-CNT, and the 0.15-CNT respectively in case of beam specimens as shown in figure 33 and 34.

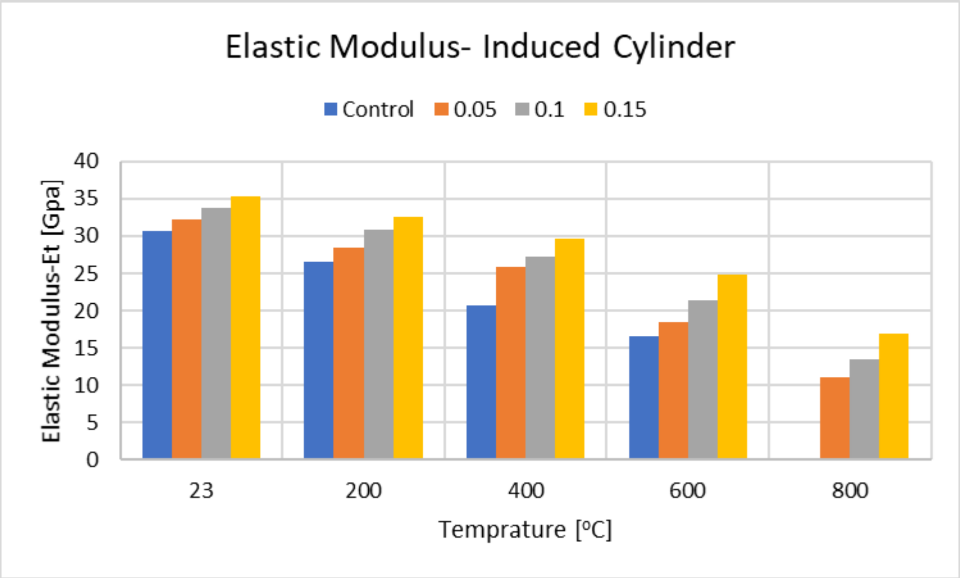


Figure 33: Elastic Modulus variation for the CNT-induced Cylinder specimens

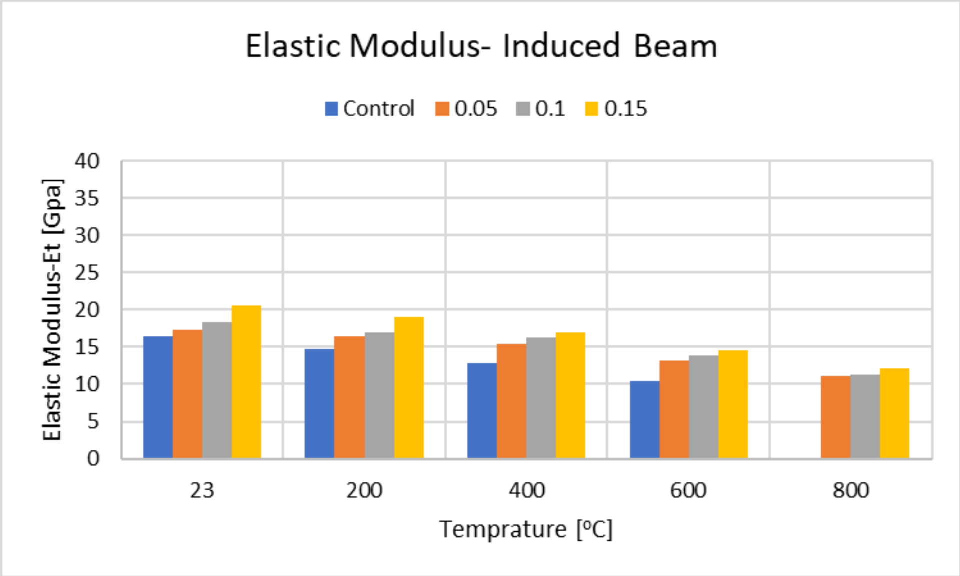


Figure 34: Elastic Modulus variation for the CNT-induced Beam specimens

4.7.2 For Plaster Specimens

It is necessary to determine elastic modulus (E) for HSC and modified concrete (0.1P-CNT, 0.2P-CNT, and 0.3P-CNT) at high temperatures by utilizing the ASTM C469 equation for compressive stress-strain curves at increased temperatures.(Taira de Vasconcellos et al., n.d.). When carbon nanotubes were utilised as plaster, no increase in elastic modulus was detected at the ambient level; nevertheless, the plaster specimens kept their characteristics better than the control specimens. To be specific, at 600 ºC, 0.1P-CNT , 0.2P-CNT , 0.3P-CNT kept about 66.68%, 66.69 % and 68.9 percent of their original values, respectively, whereas the control specimen retained 45 percent of its original values for beam as shown in figure 35.

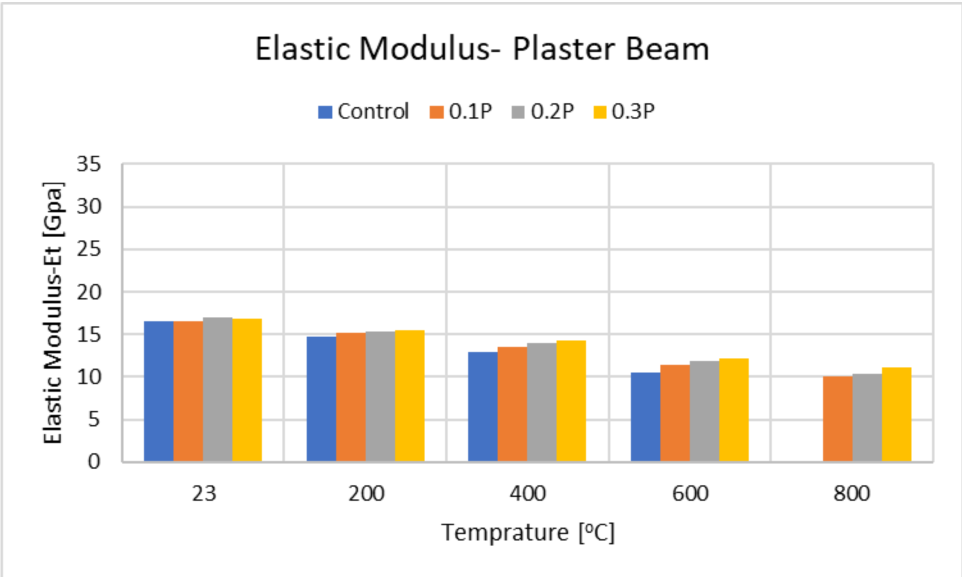


Figure 35: Depicting the Elastic Modulus variation for the Plaster Beam specimens

Additionally, 0.1P-CNT, 0.2P-CNT, and 0.3P-CNT formulations for Cylinder preserved 59%, 63 %, and 65 %, respectively, at 600 degrees Celsius, whereas the control specimen retained 49.86 percent at the same temperature. More crucially, although control specimens were unable to keep their strength after 600 ^{o}C, cylinder specimens at 800 C kept around 39%, 41%, and 48% of their original qualities, depending on the temperature. And Beam specimens kept around 51%, 60%, and 62% of their original qualities, according to the obtained results as shown in figure 36.

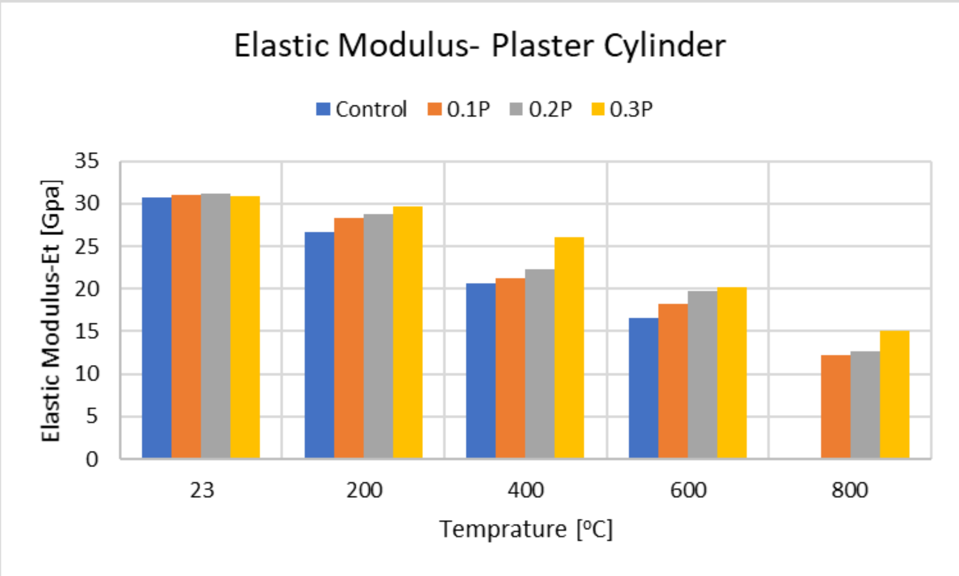


Figure 36: Elastic Modulus variation for the Plaster Cylinder specimens

4.8 Pre-Crack Energies

The energy absorbed by concrete specimens from the start point to the yield point may be calculated using stress-strain graphs. For all specimens, the area beneath this region was computed. To eliminate mistakes, the average of the regions was taken for each class, which comprised of three typical samples. Below are the values and comparisons for several types of composites with various carbon nanotubes (CNTs) percentages.

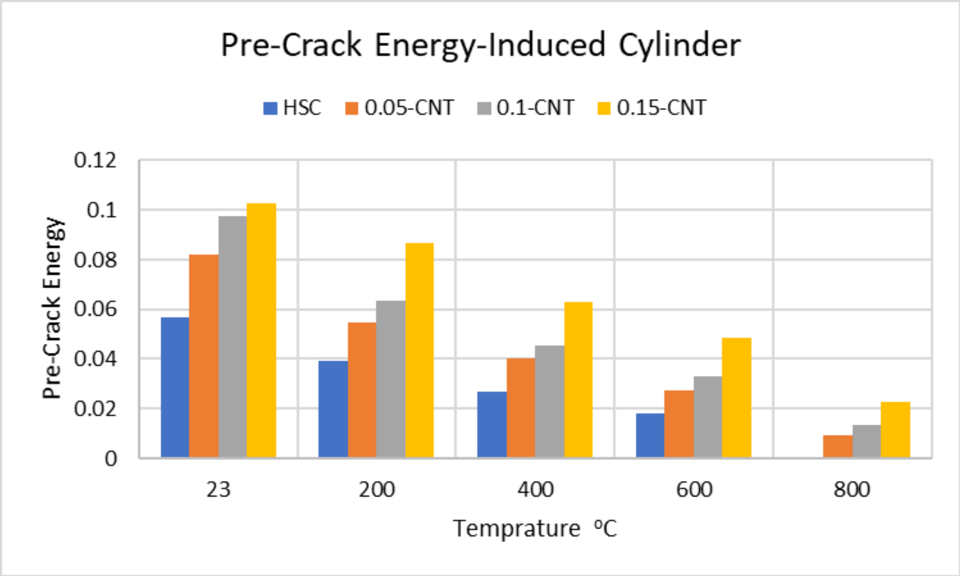


Figure 37: Pre-crack Energies of Carbon Nanotubes induced Cylinders

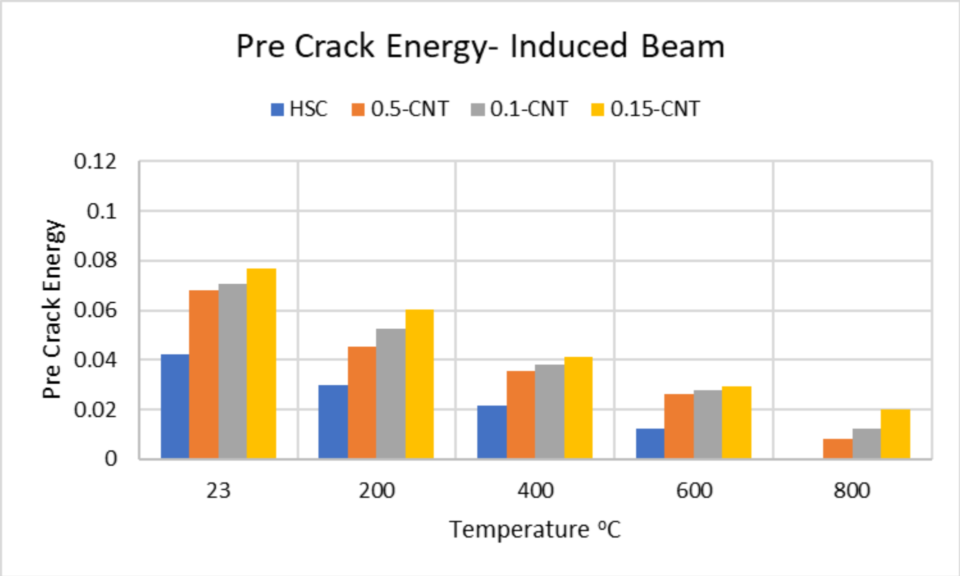


Figure 38: Pre-crack Energies of Carbon Nanotubes induced Beams

Figures 37 and 38 shows a systematic variation in Induced Carbon nanotubes (CNTs), concrete specimens where the specimens replaced 0.15 % CNT shows highest pre-crack energy as compared to Control specimens.

4.9 Total Crack Energies

The total energy absorbed by the concrete before eventual collapse is referred to as the TEC value. It is calculated using the total area under the stress-strain curve of specimens. For TEC values, the area under the curves was determined. Below are the values and comparisons for several types of composites with various carbon nanotubes (CNTs) percentages

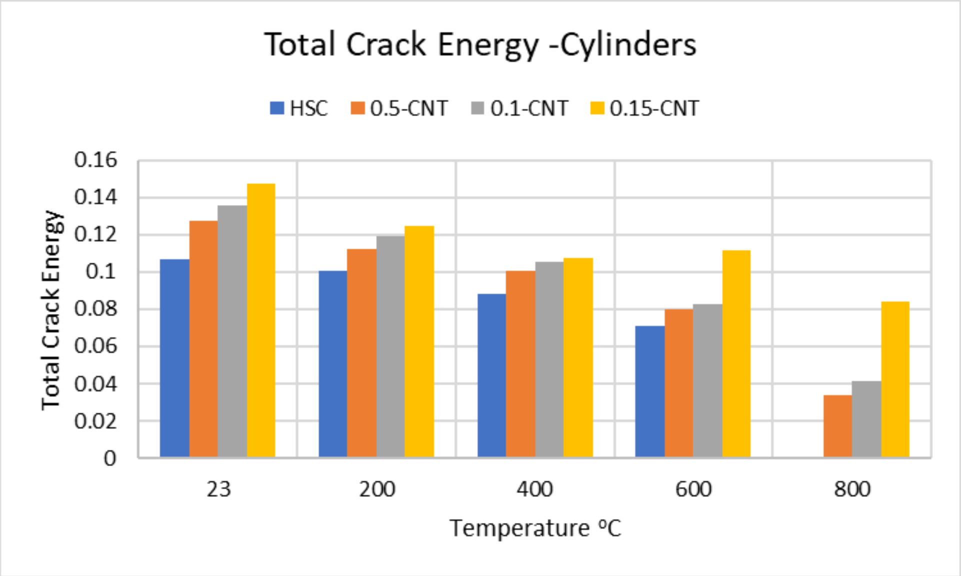


Figure 39: Total-crack Energies of Carbon Nanotubes induced Cylinders

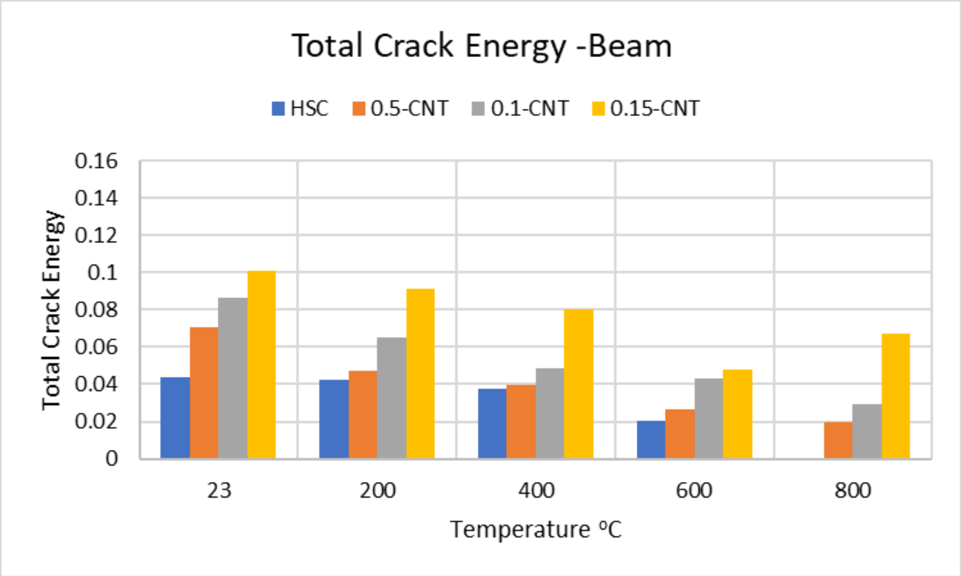


Figure 40: Total-crack Energies of Carbon Nanotubes induced Beams

Figure 39 and 40 shows a systematic variation in Induced Carbon nanotubes (CNTs), concrete specimens where the specimens replaced 0.15 % CNT shows highest pre-crack energy as compared to Control specimens.

4.10 Toughness Index (TI)

Toughness index (TI) is calculated using the area under the stress-strain curve, which reveals how much energy the specimen absorbs before failure and forecasts how well it can resist deformation under compression. The results of the stress-strain response indicate that CNT-containing samples are more likely to rupture at higher strains and shatter at higher stresses. Toughness Index (TI) was tested versus exposure temperature to quantify the increase in fracture energy of all examined formulations, as indicated in the table below. The toughness index (TI) is utilised to effectively evaluate increases in energy absorption by modified samples including CNTs as shown in figure 41 and figure 42. Toughness index is given as follows:

$$\frac{\text{Tc of modified specimen at a particular temperature}}{\text{Tc of control specimen at that particular temperature}}$$

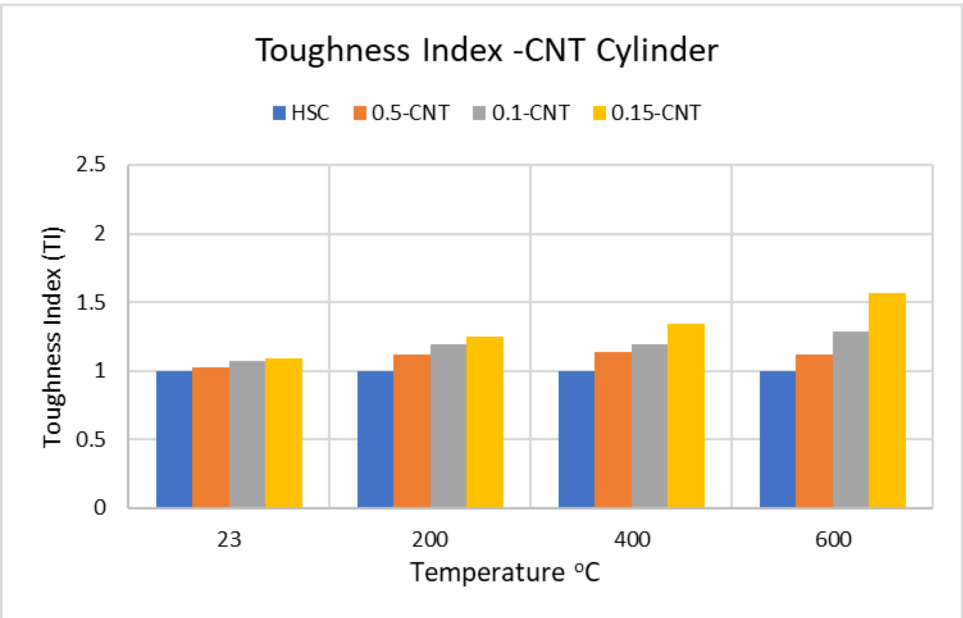


Figure 41: Toughness index (TI) of Carbon Nanotubes induced Cylinders

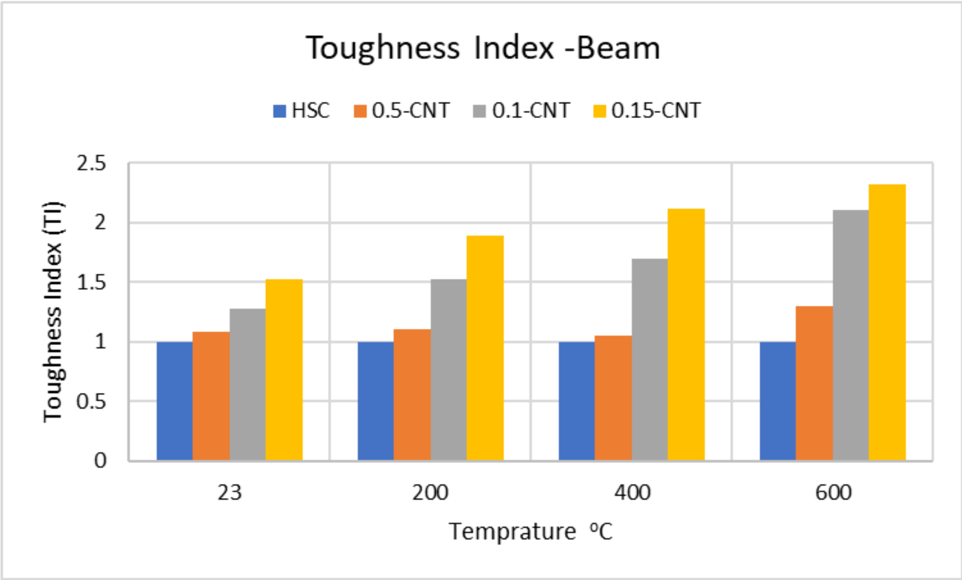
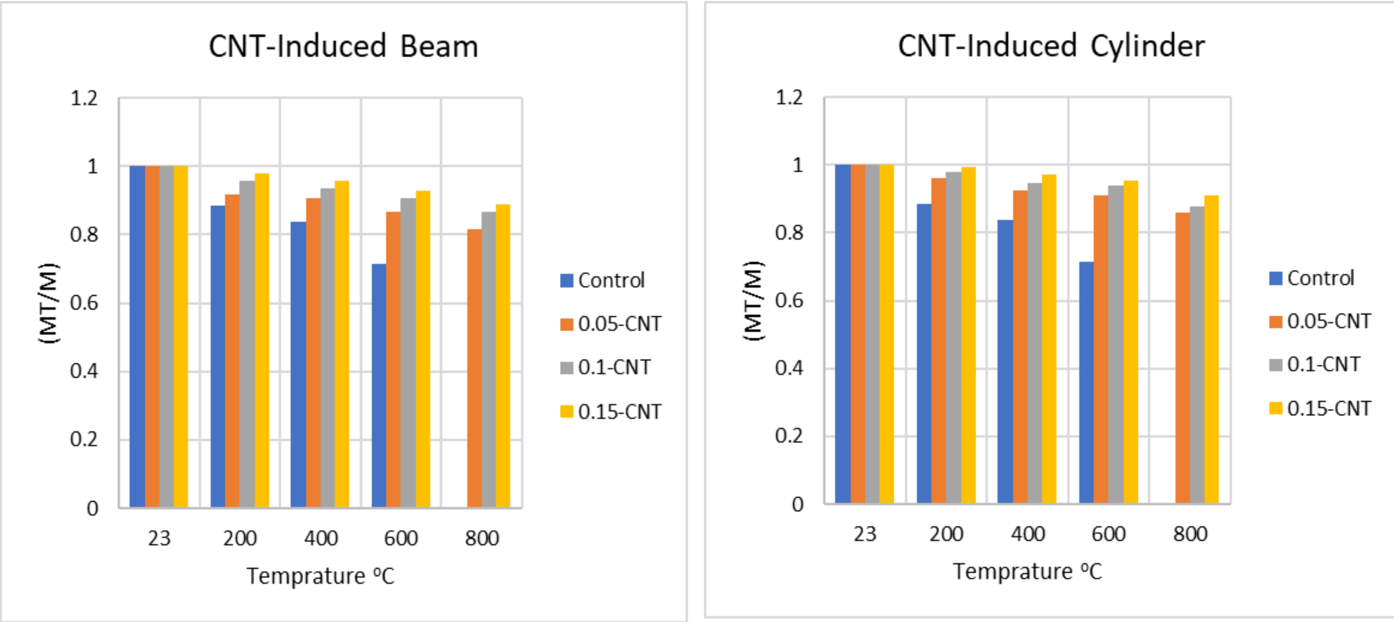


Figure 42: Toughness index (TI) of Carbon Nanotubes induced Beams

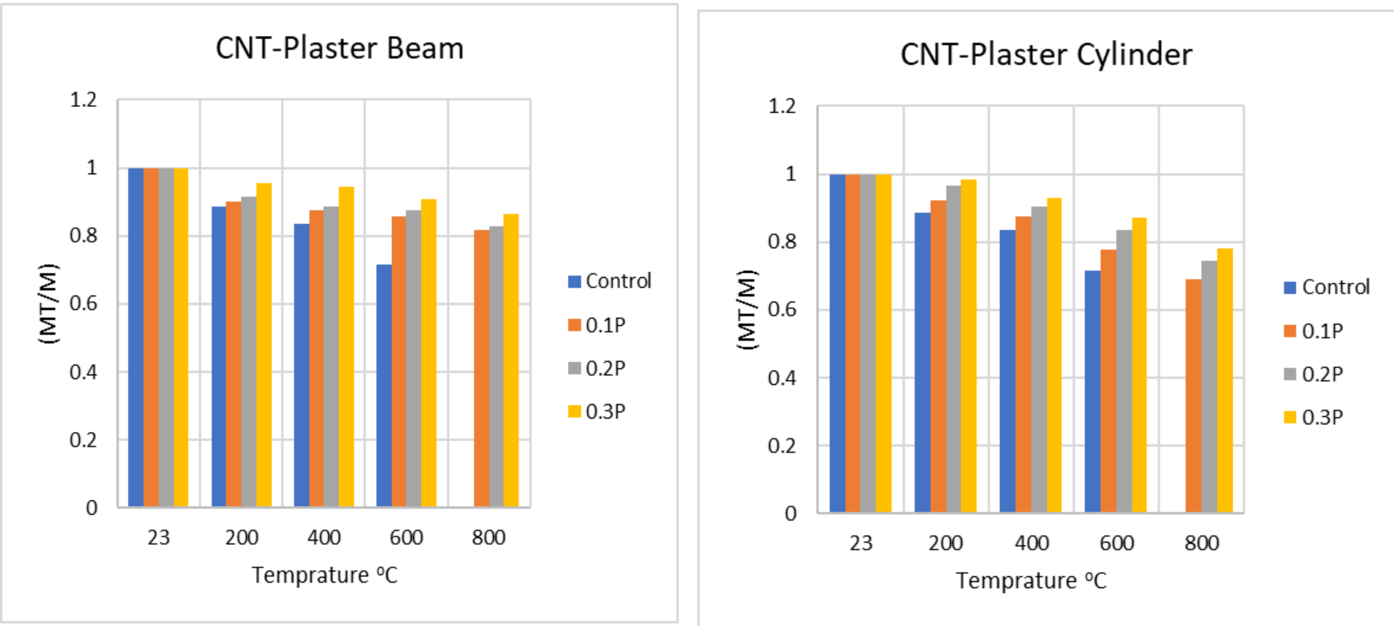
4.11 Mass loss

The presence of moisture in different forms (free, absorbed, capillary), as well as adsorbed and chemically coalesced water, may be determined by measuring mass loss (Malik et al., 2020). The ratio of mass loss increases as the temperature rises, and it may be used to determine the extent of progressive degradation and microstructure damage. hydrate decomposition and removal of water from concrete matrix are two of the most significant contributions to mass loss(Khaliq & Khan, 2015)There is a significant association between residual strength and mass loss caused by moisture disturbances in concrete due to the binding matrix in concrete. As mix water is critical to the development of concrete's strength, the removal of moisture during dehydration is critical to the reduction of concrete's strength and the degradation of the concrete matrix. The amount of moisture lost rises in direct proportion to the temperature. The differential rates are influenced by the microstructure's density, hydro-thermal processes, and the bonding state of the water in the concrete mix. The mass loss of various formulations is depicted in the diagram below.



(a) **(b)**

Figure 43: Showing Mass Loss (a) for induced CNT Beam Specimens (b) for induced CNT Cylinder Specimens



(a) **(b)**

Figure 44: Showing Mass Loss (a) for plaster CNT Beam Specimens (b) for plaster CNT Cylinder Specimens

4.12 Visual Assessment of Carbon Nanotubes Specimens:

A preliminary visual investigation is carried out to ascertain the extent of the fire's destruction. It is crucial to analyze the damaged structure by performing a thorough investigation and making precise judgements about the severity of the fire, such as visual degradation, fire size, and spread pattern, and so on. A concrete specimen's rough surface texture is the macrostructure visible with the naked eye. The formation of discrete fractures is visible on visual inspection of control and modified HSC exposed to 23-800 °C, as shown in respective tables. Changed samples had more evenly distributed cracks and less cracking when compared to the control sample, which spalled between 600 and 800 °C. Between 23 to 200 °C, no significant cracking was observed, which can be attributed to less disintegration of the aggregate-paste bond and a milder thermal gradient between the specimen's core and surface. Cracking was detected in control HSC at temperatures over 200 °C, which was attributed to the thick microstructure, which prevents simple pore pressure release and is prone to cracking. The changed samples, on the other hand, showed no such breaking. After 600 °C, the control specimens showed severe spalling, whereas the changed specimens showed moderate fractures. In comparison to 0.2 and 0.1-CNT, the amount of cracking in 0.3-CNT was reduced. Due to the induction of nanoparticles in the 0.3-CNT concrete matrix has benefited the concrete matrix in evenly distributing thermal stresses.

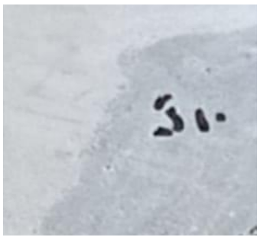
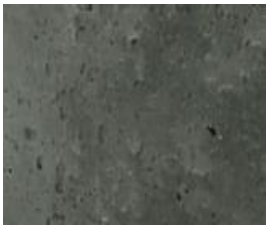
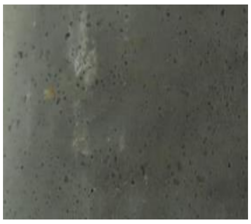
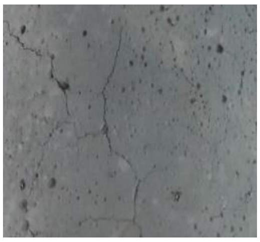
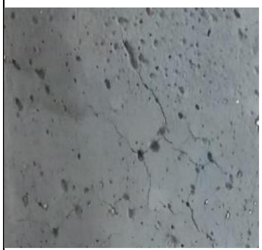
Temp (ºC)	**0.15CNT**	**0.1CNT**	**0.05CNT**	**HSC**
200				
400				
600				
800				Specimen Spalled after 800 ^{o}C

Figure 45: Represents Carbon Nanotubes induced Cylinder Specimens Visual Representation

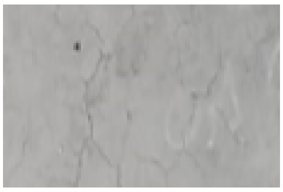
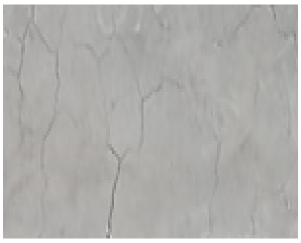
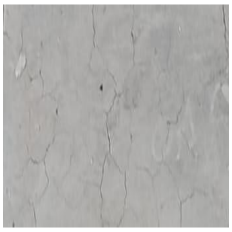
Temp (ºC)	0.15CNT	0.1CNT	0.05CNT	HSC
200				
400				
600				
800				Specimen Spalled after 800 ºC

Figure 45: Represents Carbon Nanotubes induced Beam Specimens Visual Representation

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions and Recommendation

The following are my findings that can be deduced from the observations

- In comparison to control HSC samples modified with CNTs displayed physical damage at elevated temperatures ranging from 23 to 800 ^{o}C.

- The compressive strength of the concrete matrix was enhanced both before and after fire exposure because of the induction of CNTs.

- Nearly 10% ,21% and 33 % of compressive strength was increased at ambient temperature for formulation of 0.05-CNT,0.1-CNT and 0.15-CNT respectively. This was owing to nanotubes' reinforcing activity.

- The results of specimen induced with 0.15 % CNT showed a optimum behavior as they retained up to 49 % of their initial compressive strength while the specimens induced with 0.1% and 0.05 % retained up to 40% and 33 % of their original values at 600 ^{o}C. while control specimens only retained up to 29% of their original compressive strength.

- While at 800 ^{o}C the induced cylinder specimens retained nearly 12%, 16% and 23% of their original compressive strength respectively.

- The addition of well distributed carbon nanotubes (0.05CNTs-HSC, 0.1 CNTs-HSC, and 0.15 CNTs-HSC) in concrete formulations improves their pre and post fire tension resilience. Above the critical temperature of 400^{o}C

- For the formulations of 0.1P-CNT,0.2P-CNT and 0.3P-CNT at 600 ^{o}C the plaster cylinder specimens retained 34%, 40% and 43% of their original compressive strength respectively. While at 800 ^{o}C they retained nearly 16%, 17% and 24% of their original compressive strength respectively.

- The addition of carbon nanotubes increased the flexural strength of the concrete beams up to 14% ,19% and 50 % for formulations of 0.05-CNT,0.1-CNT and 0.15-CNT respectively. Furthermore, induced specimens and plaster specimens both kept their characteristics far better than HSC specimens.

- In case of beam specimens at 600 ^{o}C they retained nearly up to 44% 45% and 47% of their original flexural strength .While at 800 ^{o}C they retained 36% ,37% and 39 % of their original flexural strength.

- Due to the presence of high-density C-S-H gel, lower thermal inertia, and fracture restraining properties of carbon nanotubes. CNT reinforced concretes sustain less internal structural damage than control specimens.

- Higher mass loss is observed in HSC compared to modified concrete formulations which showed better mass retention at elevated temperatures

5.2 Recommendations

- Use of sea shells alongside Carbon nanotubes and their effect on fire endurance of such matrices.
- Use of CNTs with other fibers i.e. SF, PP and glass fiber in HSC under elevated temperature conditions.

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