# EFFECT OF BENTONITE AS PARTIAL REPLACEMENT OF CEMENT ON RESIDUAL PROPERTIES OF CONCRETE EXPOSED TO ELEVATED TEMPERATURES



## Sardar Farhan Mushtaq

Fall 2017-MS Structural Engineering 00000206028

## MASTERS IN STRUCTURAL ENGINEERING

**MS Thesis Advisor:** 

Dr. Ather Ali

NUST Institute of Civil Engineering (NICE) School of Civil and Environmental Engineering (SCEE) National University of Sciences and Technology (NUST)

Islamabad, Pakistan (2020)

This is to certify that

Thesis titled

# EFFECT OF BENTONITE AS PARTIAL REPLACEMENT OF CEMENT ON RESIDUAL PROPERTIES OF CONCRETE EXPOSED

## TO ELEVATED TEMPERATURES

Submitted by

#### Sardar Farhan Mushtaq

Fall 2017-MS Structural Engineering

00000206028

Has been accepted towards the partial fulfillment

of

the requirements for the award of degree of

Master of Science in Structural Engineering

Dr. Ather Ali Assistant Professor NUST Institute of Civil Engineering (NICE) School of Civil and Environmental Engineering (SCEE) National University of Sciences and Technology (NUST) Islamabad, Pakistan

## THESIS ACCEPTANCE CERTIFICATE

Certified that final copy of MS thesis written by **Mr. Sardar Farhan Mushtaq**, Registration No. (**00000206028**), of MS Structure 2017 Batch (NICE) has been vetted by undersigned, found completed in all respects as per NUST Statutes/Regulations, is free of plagiarism, errors, and mistakes and is accepted as partial fulfillment for award of MS/MPhil degree.

Signature
<u> </u>
Name of Supervisor Dr. Ather Ali
Date:
Signature (HoD)
Date:
Signature (Dean/Principal)

Date: \_\_\_\_\_

## Acknowledgements

First of all, thanks to ALLAH Almighty as with His blessings, His will and His eternal love, I am able to complete my thesis.

I would like to show gratitude towards my family and teachers for their continuous support and love throughout my career. I always found them alongside me while encouraging me in achieving my goals.

In particular, I would like to extend my sincere gratitude for my supervisor Dr. Ather Ali and co supervisor Dr. Rao Arsalan Khushnood for their consistent guidance during this whole time. I have been so grateful to have them as a mentor, who responded to my queries so promptly. Thanks to them for the motivation they have given me in my hard times. His positive outlook really inspired me and gave me confidence. I also feel pleased to thank Dr. Rana Faisal Taufail for their guidance.

Last but not the least, special thanks to lab staff and all my friends as well other faculty persons for putting their time and hard work with me during various stages of the project.

## Abstract

This study investigated the effect of elevated temperature on the mechanical, physical and durability properties of normal strength concrete, modified with bentonite. The bentonite concrete was cast by substituting cement with bentonite content in proportions of 5, 10 and 15% by weight. Mechanical tests were conducted (compressive and splitting tensile strength). Further, durability performance (mass loss and sorptivity) and specimen properties (elastic modulus, stress–strain behavior, ductility and energy absorption) were evaluated and discussed. The results demonstrated that samples incorporating bentonite showed better fire endurance than the control mix. The inclusion of bentonite in concrete decreased the mass loss of the specimens exposed to high temperatures. The performance of bentonite concrete was better in terms of mechanical behavior (compressive and tensile strength) than that of conventional concrete, and it had high resistance to water absorption. Higher ductility and energy absorption capacity was observed for the concrete specimen containing bentonite than its counterpart control specimens.

## **TABLE OF CONTENTS**

Acknow	wledgements	iv
Abstra	nct	v
List of ]	Figure	viii
List of	Tables	X
1. Int	troduction	1
1.1	General	1
1.2	Research objectives	3
1.3	Research tasks	4
1.4	Research significance	4
1.5	Thesis outline	4
2 Lit	terature review	6
2.1	General	6
2.2	Cement	6
2.3	Aggregates	6
2.4	Concrete	6
2.5	Bentonite	7
2.5	5.1 Uses of Bentonite	7
2.5	5.2 Challenges with the use of bentonite in concrete	7
2.6	Superplasticizer	7
2.7	High temperature testing on heating and loading regimes	8
3 Ex	xperimental Program	
3.1	General	
3.2	Materials	10
3.3	Mix proportions and preparation of specimen	14
3.4	Target temperatures for test methods	15

3	.5	Tes	t equipment and procedure1	5
	3.5.	1	Compressive strength test	7
	3.5.	2	Splitting tensile strength	3
	3.5.	3	Stress-strain curve	3
	3.5.	4	Elastic modulus	3
	3.5.	5	Mass loss	3
	3.5.	6	Sorptivity19	9
	3.5.	7	Ductility	9
	3.5.	8	Energy Absorption	9
4	Res	ults a	and discussion	)
4	.1	Cor	npressive strength	)
4	.2	Ten	sile strength	5
4	.3	Stre	ess-strain response	7
4	.4	Mo	dulus of elasticity, Ductility and Energy Absorption29	9
4	.5	Sor	ptivity	2
4	.6	Mas	ss loss	5
5	Con	clus	ions40	)
6	Rec	omn	nendations	1
7	Ref	erend	ces42	2

## List of Figures

$FIGURE 1: LOADING \ AND \ HEATING \ CONDITIONS \ FOR \ TEST \ METHODS \ A) \ STRESSED \ B) \ UNSTRESSED$
C) RESIDUAL
FIGURE 2: X-RAY DIFFRACTION OF POWDERED BENTONITE
FIGURE 3: ELECTRIC FURNACE FOR HEATING THE SAMPLES TO TARGETED TEMPERATURES16
FIGURE 4: UNIVERSAL TESTING MACHINE FOR MECHANICAL TESTING
FIGURE 5: PLOT OF RESIDUALS FOR BC15 MIX
FIGURE 6: COMPRESSIVE STRENGTH FOR CONTROL MIX AND BENTONITE MODIFIED CONCRETE
MIXTURES AT AMBIENT AND TARGETED TEMPERATURES A) COMPRESSIVE STRENGTH B)
RELATIVE COMPRESSIVE STRENGTH
Figure 7 Micrographs of 10BT exposed to elevated temperature a) $200^{\circ}$ C b) $400^{\circ}$ C c)
600°C d) 600°C
FIGURE 8: VARIATION OF SPLITTING TENSILE STRENGTH AT AMBIENT AND TARGETED
TEMPERATURES FOR CONTROL MIX AND BENTONITE MODIFIED CONCRETE A) ABSOLUTE
SPLITTING TENSILE STRENGTH B) RELATIVE SPLITTING TENSILE STRENGTH27
FIGURE 9: STRESS-STRAIN RESPONSE AT AMBIENT AND ELEVATED TARGETED TEMPERATURES
A) CM b) 5BT c) 10BT d) 15BT
FIGURE 10: DUCTILITY OF THE TESTED SPECIMENS
FIGURE 11: VARIATION IN MODULUS OF ELASTICITY OF CONTROL MIX AND BENTONITE
MODIFIED CONCRETE AT AMBIENT AND ELEVATED TEMPERATURES (A) ABSOLUTE ELASTIC
MODULUS, (B) RELATIVE ELASTIC MODULUS
FIGURE 12: SORPTIVITY AT AMBIENT AND TARGETED TEMPERATURE FOR CONTROL MIX AND
BENTONITE MODIFIED MIX
FIGURE 13: SORPTIVITY AT AMBIENT AND TARGETED TEMPERATURE FOR CONTROL MIX
FIGURE 14: SORPTIVITY AT AMBIENT AND TARGETED TEMPERATURE FOR 5% BENTONITE MIX 34
FIGURE 15: SORPTIVITY AT AMBIENT AND TARGETED TEMPERATURE FOR $10\%$ bentonite mix
FIGURE 16: SORPTIVITY AT AMBIENT AND TARGETED TEMPERATURE FOR 15% BENTONITE MIX
FIGURE 17: MASS LOSS VARIATION FOR DIFFERENT MIXES AT TARGETED TEMPERATURE
FIGURE 18: TGA TEST FOR CONTROL MIX AT TARGETED TEMPERATURE
FIGURE 19: TGA TEST FOR 5BT AT TARGETED TEMPERATURE

FIGURE 20: TGA TEST FOR 10BT AT TARGETED TEMPERATURE	
FIGURE 21: TGA TEST FOR 15BT AT TARGETED TEMPERATURE	

## List of Tables

TABLE 1: PHYSICAL AND CHEMICAL PROPERTIES OF CEMENT    11
TABLE 2: PHYSICAL PROPERTIES OF FINE AND COARSE AGGREGATES       12
TABLE 3: CHEMICAL COMPOSITION OF BENTONITE    13
TABLE 4: PROPERTIES OF SUPER PLASTICIZER
TABLE 5: DETAIL OF MIXTURE PROPORTIONS AND STRENGTH PROGRESSION
TABLE 6: EXPERIMENTAL AND PREDICTED COMPRESSIVE STRENGTH USING RESPONSE SURFACE
Methodology23
TABLE 7: RELATIVE RESIDUAL SPLITTING TENSILE AND COMPRESSIVE STRENGTH
TABLE 8; COMPRESSIVE STRENGTH, MODULUS OF ELASTICITY, ENERGY ABSORPTION, TENSILE
STRENGTH AND DUCTILITY AT TARGETED TEMPERATURES

## **CHAPTER 1**

## **1** Introduction

#### 1.1 General

Concrete is regarded as one of the most broadly used construction material in world because of the fact that it has large number of resources, high adaptability and matured process for production. With appropriate combination of cement, additional cementitious components and less water to cement ratio, different types of concrete are produced for different purposes. For instance, steel fiber reinforcement is used to enhance tensile strength and to bridge the micro cracks but it increases the cost, self-weight and is also ineffective in delaying micro cracks due to large spacing and less interlocking[1-3]. Most of the time the concrete structures become progressively worse when some challenging environments are faced. Moreover, they also require repairs before their service life is ended which are very costly.

A good concrete material should have two criteria; i.e. it should be good in its fresh and hardened state. Concrete should be consistent and cohesive. It must be consistent to such a limit that we can compact it without applying excessive effort, and also it must be not that much cohesive to produce segregation which will reduce the homogeneity of concrete. For a concrete material to be efficient in hardened state, it must have durability and compressive strength[4, 5].

In today's world concrete with higher strength and higher performance is being used and for its production, the ratio of water to binder should be reduced and binder content should be high. To achieve lower water to binder ratio, super plasticizers are used in the concrete. Also, to achieve low porosity and permeability different types of cementitious materials can also be added in addition with concrete. Silica fume is added in concrete mixtures which cause lower porosity and permeability. It is one of the most popular pozzolan because their oxides (SiO<sub>2</sub>) reaction consumes calcium hydroxides, which are products of hydration of ordinary Portland cement. Pozzolanic reactions results are the lower heat release, lime consumption, strength development and smaller pore size distribution[6].

During fire or near furnaces and reactors, concrete is being exposed to elevated temperature[7]. These exposures can result into decrement of mechanical properties like elastic modulus, strength and stability of volume which leads to structural failures. For determination of load carrying capacity and for re-establishing of structures which are damaged from fire, it is

important to study properties retained after exposure to elevated temperature[8]. Exposure of specimens to heat can considerably change the physical structure and composition of concrete. Above110°C temperature, significant amount of water which is chemically bonded release through calcium silicate hydrate (CSH) gel[9]. Internal stresses are increased and micro cracks are also produced from 300°C due to expansion of aggregates. Calcium hydroxide (CH) is an important product of hydration reaction is also dissociated at 530°C which can result into the shrinkage of concrete. Cracking and crumbling of concrete are occurred when CaO is turned into calcium hydroxide (CH). Surface cracking and spalling are visible effects of high temperature[10]. This exposure may also cause some color changes. Above 500°C, the transitions due to high temperatures are more noticeable. The changes which concrete experiences on this temperature level are mostly considered irrevocable[11]. Above 600°C, CSH gel, which gives strength to the cement paste also decomposes. With further increase of temperature at 800°C, concrete usually break down. Moreover, some other minerals which are present in the cement paste are converted into the glass phase[8]. This results in changes of the microstructure, strength degradation and durability of concrete[11].

As we know that after water concrete is second most consumed material on earth, with **4 billion tons** of the material produced every year and growing population and its needs for abidance is pushing the production of cement to far extents. The production of cement involves the emission of CO2 in massive quantities. As a result, the industry generates approximately **8%** of global CO2 emissions. Still the need for the material cannot be denied, only in October 2018 Pakistan produced **3945 tons** of cement and the demand is only going to grow when Pakistan transitions into the upcoming construction phase. There is a need for replacement of this massive cog in our lives with a material that is viable, environment friendly, cheap and readily available and most importantly has the same structural properties as cement if not better. One option is to replace the cement in concrete with a pozolonic substance that can act as a substituent and reduce the cost and environmental harms.

The extensive use of concrete as a building material requires having sound knowledge regarding its behavior at high temperatures[12]. In addition, technology advancements have resulted in increasing demand to develop materials for various purposes that cannot be met by using conventional concrete. Moreover, in the past few years, the cost of, and demand for, cement has increased significantly[13]. Therefore, it is necessary to replace cement with a material that will not affect the residual properties of concrete, but rather will enhance them after exposure to high temperatures and will also be cost-effective.

In this regard, the characteristics and suitable use of clays as an alternative to cement in concrete have been studied for decades[14]. Previously, different materials were used to improve the overall behavior of concrete exposed to elevated temperature, such as polypropylene fibres [15], steel fibres [16], carbon nanotubes [17], fly ash [18] and metakaolin [19]. However, these materials are either expensive or not available freely. Hence, this study uses bentonite as partial replacement of cement in concrete. Bentonite is freely available in Jahangira [20] and Karak [21], Khyber Pakhtunkhwa, Pakistan. Bentonite is a clay mineral and possesses pozzolanic properties [22]. In previous studies, bentonite was incorporated as partial replacement of cement, to make a cost-effective concrete mix. Using bentonite as partial replacement of cement will result in reducing greenhouse gas emissions because cement production emits a huge volume of greenhouse gases [23]. These hazardous gases can travel throughout the world in a week's time [24]. Hence, to support green living, cement production should be reduced and supplementary materials, such as bentonite, should be used. Therefore, this study investigates the behavior and residual properties of bentonite, when used as partial replacement of cement, in concrete mixtures exposed to high temperatures. Tests on residual properties, such as compressive strength, splitting tensile strength, stress-strain response, ductility, sorptivity and weight loss, were performed along with microscopic investigation and the results were compared with control samples, which were tested at room temperature. The empirical findings of this study can be used as input for the design and analysis of concrete structures.

#### **1.2 Research objectives**

- > To Study the material and mechanical properties of concrete incorporating bentonite.
- > To evaluate the performance of these modified mixes under residual test methods
- To study the microstructure of heat-treated samples by field emission scanning electron microscopy
- To study the effect of bentonite on ductility, energy absorption and water absorption of concrete.
- To develop simplified mathematical relationships for expressing material properties as a function of temperature

### 1.3 Research tasks

To accomplish research objectives following tasks were performed.

- ➢ Literature review.
- Test set up which includes furnace, splitting tensile test assembly, protective steel assembly.
- > Perform tests on concrete samples exposed to elevated temperature.
- Evaluate and analyze experimental results.
- Conclusions and recommendations.

#### 1.4 Research significance

Fire is regarded as harmful hazards from which structures are threatened with during the service life. Hence, the requirement to characterize the mechanical and material behavior of bentonite incorporated concrete at high temperatures becomes significant. The fine particles of bentonite material act as filler agent and lead to reduction of micro pores and produce a compact concrete which brings about the suspicion about spalling of such concrete at elevated temperature.

Limited research work is being implemented to observe the behavior of bentonite in concrete at ambient temperatures and there is no reliable data available on higher temperature properties of these bentonite reinforced concrete. For predicting the elevated temperature properties, an elaborative experimental data and mathematical models are required. This study provides mechanical, physical and micro structural properties of normal strength concrete modified with various proportions of bentonite up to the required targeted temperature of 800°C.

#### 1.5 Thesis outline

The research executed to achieve the mentioned objectives is presented in five chapters.

In chapter 1, effect of fire on concrete, bentonite, research objectives and research significance are being discussed.

Chapter 2 describes literature review in detail and about properties of bentonite in general. Elevated temperature response of control mix and effect of bentonite at ambient temperature have been discussed in detail, in addition to that cooling method for heat treated samples are discussed.

Chapter 3 deals with the test setup. It explains which types of equipment are used to evaluate mechanical properties. Furthermore, it presents an overview of the test procedure which describes the ways and methods to determine mechanical properties.

Chapter 4 provides evaluation, analysis and discussion for results of material property tests. Further simple relationships expressing material properties as a function of temperature were developed and explained.

Chapter 5 provides detailed conclusions

## **CHAPTER 2**

## 2 Literature review

#### 2.1 General

The construction industry in Pakistan is growing day by day, and demands for construction material such as steel, crush, sand, and cement is growing accordingly. So far so Pakistan is meeting up with material demand. But it can all change soon. As we know these materials are not renewable in theory but these practices are not up held in field because of lack of knowledge and engineering practices. Like all resources the raw material required for cement production can also run out, same as the raw material used for filler.

#### 2.2 Cement

Cement is a binder material that mixed up with filler material when wet and hardens up to a stone like material that can be molded when it's in plastic state. Cement is rarely used on its own as a building material but other fillers like sand are in corporate in it. It is mixed with aggregates and used as mortar for masonry, or with sand or gravel and used as concrete. It has density of 3.15g/cubic centimeter. Ordinary Portland cement in conformity with ASTM C150 [25] was used.

#### 2.3 Aggregates

Construction is being done all over the world. So, the use of the concrete has increased a lot. Concrete is used twice as much as any other construction materials like bamboo(wood), metals (steel, aluminum) etc. Increased rate of construction has resulted in the excessive use of the concrete which directly increases the consumption of natural aggregates. Major aggregates of the concrete are stone, gravel and sand. Stone and gravel in crushed state are categorized as coarse aggregates and sand due to its fineness falls in the category of fine aggregates. It is predicted that globally the production of crushed stone and gravel will grow by an average of 7.7% annually. And the expected growth rate of sand is predicted to be 3.9% per annum.

#### 2.4 Concrete

Concrete is a composite building material produced using a mixture of broken stone or rock, sand, bond and water which can be spread or filled in shape and forms a stone like mass on hardening. Concrete is the most well-known contrived material on earth. Most concretes utilized are lime-based concretes, for example, plain cement concrete or concretes made with other hydraulic cements, for example, bond fondue. However, asphalt concrete which is very

common now and then utilized for road surfaces, is also a type of concrete, where the bond material is bitumen.

#### 2.5 Bentonite

Bentonite is absorbent clay that is formed by the breakdown of volcanic ash. It consists mostly of silicon dioxide and aluminum oxide, also some trace amount of calcium and magnesium oxide and other minerals. The chemical properties of bentonite depend upon its constituent that make it a perfect pozzolanic material. Like most pozzolans it has the capacity to make C-S-H bonds.

#### 2.5.1 Uses of Bentonite

The main use of bentonite in concrete is that we use it in partial replacement of cement and the replacing of bentonite leaves us with many benefits, enhancing the properties and increase in strength being some of them. Also, bentonite is cheaper than cement so partially replacing bentonite with cement drastically lowers the project cost. Bentonite produces no pollutants because it occurs naturally unlike cement so while using it you are also reducing the greenhouse effect caused by the CO2 produced while the production of cement. Achieving all that does not come with the cost of structural strength loss. Initial strength of bentonite incorporated concrete will be lesser then normal concrete but the final strength would be more than conventional concrete.

#### 2.5.2 Challenges with the use of bentonite in concrete

Bentonite is naturally available in form of crush rocks and these rocks need to be converted in fine powder like cement in order to be use as a partial replacement of cement in concrete. To crush the rocks intensive use of ball machine is required 5000 rounds per 5kg at least. Also, bentonite rocks have to be zero moisture content before we crush it. This imposes that any additional moisture in material will cause problems like clogging and can lead to the failure of process all together. The crushed rock then needs to be passed from sieve no. 200 and only the material passed from the sieve can be used in concrete. The passed material is very low in yield only producing 500g of fine material out of 5kg crushed rock. All this processing consumes a lot of energy which is not efficient.

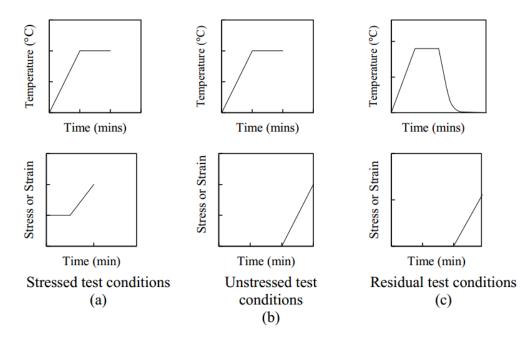
#### 2.6 Superplasticizer

When partially replacing the cement with bentonite the mixture increases so a lot of water is required to achieve desired or set workability. Since the W/C ratio is constant so amount of water can't be increased, hence the concrete produces with same amount of water as normal

concrete will have either negligible or no slump. This problem is solved by the introduction of super plasticizer in the mixture. It added according to certain percentage of weight of binder.

#### 2.7 High temperature testing on heating and loading regimes

There are three different testing methods available for heat treatment and testing samples which are named as stressed, unstressed and residual. In stressed condition, specimen is loaded to 40% of ultimate strength in pre-heating stage. The sample is then heat treated to required



*Figure 1: Loading and heating Conditions for test methods a) stressed b) unstressed c) residual* 

temperature in a recommended ramp time and then temperature is kept constant for suitable time to accomplish thermal steady state. Then further load is applied till sample failure. Schematically it is shown in Figure 1(a). This condition accomplishes actual life fire action. It is a complex system and special laboratories are established to study fire properties of members under this condition.

In unstressed situation, the sample is not subjected to pre-loading. Sample is heated up to target temperature and then held constant for some time at that temperature to attain thermal steady state. Then the sample is tested without any heat loss by covering it with thermal jacket insulation. Results obtained are different from stressed conditions but they give reasonably exact idea about how structure will perform under actual fire condition. Schematic illustration is shown in Figure 1(b).

In residual situation, specimen is heat treated under thermal steady conditions and then sample is cooled down to room temperature. Sample is then loaded till failure. The loading and heating regimes are shown in Figure 1(c). This condition is least accurate but it is easy and simple in execution and due to that reason, most of research associated to high temperatures is established on this method.

## **CHAPTER 3**

## **3** Experimental Program

#### 3.1 General

This chapter discourse the methodology and procedures adopted to achieve research goals. The detailed process from preparation of specimen to testing techniques executed to achieve the results has been explained.

Mechanical, material, and physical properties of concrete is required for studying and assessing the performance of mixes incorporating bentonite at high temperatures. Compressive strength, tensile strength, stress-strain response, elastic modulus and mass loss are important material properties. There is sufficient data available in literature on material properties of concrete but no authentic data is available on elevated temperature properties of mixtures incorporating bentonite.

In order to establish the effect of fire on material properties of concrete replaced with bentonite, mechanical and material property tests at high temperature were carried on reference concrete mix and bentonite incorporated concrete mixes which include mechanical strength testing like compressive and tensile strength tests, stress strain behavior, elastic modulus, micro structural properties, sorptivity and mass loss. All the mechanical and material properties thus evaluated were presented in form of graphs, and generated data was compared with control mixes as well. The collected data was used to formulate simplified mathematical models for various material properties of these modified mixtures as a function of temperature in range of 23-800°C. Experimental design detail, standards, test setup and procedures of testing are described in this chapter.

#### 3.2 Materials

Ordinary Portland cement (OPC) Type-I in conformity with ASTM C150 was used in the preparation of investigated concrete formulations [25]. Properties of OPC are presented in Table 1. The summarized oxide composition in Table 2 is experimentally attained via X-Ray fluorescence (XRF) technique.

Compound	Cement
Silicon dioxide (SiO2)	19.2
Aluminium oxide	9.67
(Al <sub>2</sub> O <sub>3</sub> )	
Ferric oxide (Fe 2O3)	3.28
Magnesium oxide (MgO)	1.66
Calcium oxide (CaO)	60.1
Sodium oxide (Na2O)	0.81
Potassium oxide(K 2O)	1.18
Phosphorus pentoxide (P <sub>2</sub> O <sub>5</sub> )	0.068
Sulphur trioxide (SO3)	2.65
Titanium oxide (TiO <sub>2</sub> )	
Zinc oxide (ZnO)	

## Table 1: Physical and chemical properties of cement

Natural sand as fine aggregate was used. The natural crushed aggregates of maximum size of 12.5 mm were used as coarse aggregates. Table 2 summarizes the properties of fine and coarse aggregates. Cement was partially replaced with bentonite (5%, 10% and 15%). The results of XRD of bentonite are shown in Fig 1. The average size of bentonite particles was 4  $\mu$ m to 5  $\mu$ m. Super plasticizer was used to make the mix workable [26], and the properties of super plasticizer are shown in Table 4.

Aggregate Type	Coarse Aggregate	Fine Aggregate
Maximum size	12.5mm	
Water absorption (%)	0.8%	1.46%
Specific gravity (g/cm <sup>3</sup> )	2.6417	2.474
Bulk density (Kg/m <sup>3</sup> )	1432	
Crushing value	21	
Fineness modulus		1.7

Table 2: Physical properties of fine and coarse aggregates

Compound	Bentonite
Silicon dioxide (SiO2)	54.25
Aluminium oxide	20.29
(Al 2O3)	
Ferric oxide (Fe 2O3)	8.70
Magnesium oxide (MgO)	4.1
Calcium oxide (CaO)	7.18
Sodium oxide (Na2O)	1.24
Potassium oxide(K 2O)	3.95
Phosphorus pentoxide (P <sub>2</sub> O <sub>5</sub> )	1.107
Sulphur trioxide (SO3)	
Titanium oxide (TiO <sub>2</sub> )	0.9
Zinc oxide (ZnO)	0.18

## Table 3: Chemical composition of bentonite

Table 4: Properties of super plasticizer

Properties	Values
Туре	ViscoCrete-3100
Colour	Colorless to Yellowish
Consumption	0.5-1%
Air contents	Less than 1%
Freezing point	0°C
Form	Liquid
Density (at 25°C)	1.085kg/Lit

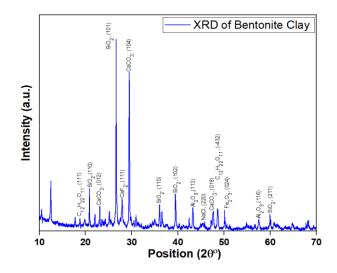


Figure 2: X-Ray Diffraction of Powdered Bentonite

#### **3.3** Mix proportions and preparation of specimen

A control mix (CM) formulation and three modified formulations of bentonite concrete containing 5%,10% and 15% bentonite were prepared using water to cement ratio of 0.55. Third generation superplasticizer (Sika ViscoCrete-3110) 0.5% and 1% by weight of cement was used to adjust the slump of concrete mixture to 50 mm. Cylindrical specimens (100 mm x 200 mm) were prepared and de-molded after 24 hours of casting. For curing, specimens were placed in controlled conditions as specified in ASTM C192 ( $23^{\circ}C \pm 2^{\circ}C$ , 95% humidity) [27]. Development of compressive strength was observed by ASTM C39 [28]. Table 5 shows the mix design detail and the strength progression of analyzed formulations.

Mix	<b>Cement</b> kg/m <sup>3</sup>	<b>Bentonite</b> kg/m <sup>3</sup>	Water *(w/c=0.58) kg/m <sup>3</sup>	<b>Fine Aggregate</b> kg/m <sup>3</sup>	Coarse Aggregate kg/m <sup>3</sup>
СМ	778.05	0	451.27	906.15	985.202
5BT	739.145	38.90	451.27	906.15	985.202
10BT	700.245	77.80	451.27	906.15	985.202
15BT	661.343	116.7	451.27	906.15	985.202

## Table 5: Detail of mixture proportions and strength progression

## **3.4** Target temperatures for test methods

The target temperatures achieved in this study are 23°C, 200°C, 400°C, 600°C and 800°C.

## 3.5 Test equipment and procedure

The concrete samples were subjected to high temperatures by using an electric furnace (shown in Figure 3) with capability to sustain temperatures up to 1100°C. Electric furnace was assembled with a temperature rate controller and hold time controller.



Figure 3: Electric Furnace for heating the samples to targeted temperatures

In order to obtain the compressive strength values by ASTM C39 [28], concrete samples were examined using a universal testing machine (UTM) having 1000 KN capacity to resist compressive loading (shown in Figure 4). Loading rate of 0.20 MPa/second was applied to record the response in compression. Modulus of elasticity was obtained as per ASTM C469 procedure [29]. For tensile strength, concrete specimens were tested diametrically by ASTM C496 method [30]. All the exposure temperatures were provided with holding time of 2 hours and 30 minutes. In order to achieve uniform thermal state a minimum holding time of 2 hours is required as concrete has low thermal conductivity [31, 32]. The targeted temperatures in this study were 23°C, 200°C, 400°C, 600°C and 800°C. RILEM test procedures were adopted for heating rate and hold times. The heating rate for specimens to reach targeted temperature was 5°C/minute. Samples were allowed to cool down slowly to ambient temperature with the door of furnace kept open to avoid thermal shock. Prior to strength tests, sorptivity and mass loss test were performed before and after the heat exposure.



Figure 4: Universal testing machine for mechanical testing

## 3.5.1 Compressive strength test

After achieving the thermal steady state temperature condition, samples were cool down to ambient temperature in air and then shifted to compression testing machine in case of residual test conditions.

One specimen was tested at each required temperature. If results were found ambiguous or outliers, additional testing was done to confirm results. To draw a comparison amongst results of different concrete types and testing conditions, relative compressive strengths were calculated using following relationship.

 $Relative \ Compressive \ Strength = \frac{Strength \ at \ targeted \ temperature}{Ambient \ temperature \ strength}.....EQ.1$ 

#### **3.5.2** Splitting tensile strength

The heat-treated samples were allowed to cool down to room temperatures in residual test conditions. The samples were loaded diametrically at the loading rate of 0.02MPa per second and the peak load was recorded. The tensile strength were calculated in accordance with ASTM C496 [30].To compare tensile strength of different types of concretes under different test conditions, relative tensile strength was calculated using following relationship.

 $Relative Tensile Strength = \frac{Strength at target temperature}{Ambient temperature strength} \qquad .....EQ.2$ 

#### 3.5.3 Stress-strain curve

Compressive strength tests were undertaken to achieve stress-strain response of concrete cylinders. Data was obtained by failing cylinders under gradually rising loads at precise loading rate of 0.2MPa/second.

#### 3.5.4 Elastic modulus

The elastic modulus of concrete samples was evaluated using the stress-strain curves. Chord modulus of elasticity was calculated using relationship described in ASTM C469 [30].

E = Chord modulus of elasticity

 $S_2 = Stress$  against 40% of ultimate load

 $S_1 = Stress$  against longitudinal strain of 0.00005

 $\epsilon_1$  = Longitudinal strain against S<sub>1</sub>=0.00005

 $\epsilon_2$ = Longitudinal strain against S<sub>2</sub>

#### 3.5.5 Mass loss

To calculate variation in mass of heat-treated concrete samples, they were weighed before and after heat treatment. For residual conditions, heat treated samples were cool down to ambient temperature before mass loss measurements. The specimens then transferred to sensitive balance (1000th of a kilogram) and weighed to record the change in weight at a particular target temperature. Relative mass loss measured at different temperatures was calculated from the following relationship.

$$Mass \ loss = \frac{Mt}{M}....EQ.4$$

M<sub>t</sub>= Mass at temperature (T)

M = Mass at ambient temperature

## 3.5.6 Sorptivity

Sorptivity is a property that symbolizes the tendency of a material to absorb water through capillarity action [33]. Sorptivity parameter and is used extensively to evaluate the resistance of concrete that is exposed to an aggressive environment [34]. The sorptivity test was performed according to ASTM C 1585 [35].

## 3.5.7 Ductility

The ductility of the tested specimens was calculated as the ratio of the ultimate strain of concrete to the corresponding yield strain. The yield strain was calculated as the strain matching to 65% unconfined compressive strength of concrete.

## 3.5.8 Energy Absorption

Energy absorption was obtained by calculating the area under the stress–strain curve, and the area up to ultimate strain was considered for all mixes [36]

### **CHAPTER 4**

## 4 Results and discussion

Results of tests for mechanical properties such as tensile strength, compressive strength, stressstrain response, modulus of elasticity, energy absorption, ductility, sorptivity, micro structural analysis, visual assessment and mass loss under different test condition are analyzed for control mix and bentonite incorporated mix.

#### 4.1 Compressive strength

The compressive strength at room temperature mainly depends on water–cement ratio, the aggregate type and aggregate size, the aggregate-paste interface transition zone, the curing condition and the admixtures [37]. At elevated temperatures, compressive strength is a function of the additives used, the room temperature strength and the heating rate [38]. Up to 200 °C, hydrothermal changes occur due to loss of absorbed water [39]. Micro-cracking occurs at 300 °C due to strength loss because of generation of internal stresses [8]. Above 400 °C, calcium hydroxide Ca(OH)<sub>2</sub> disintegrates, resulting in further strength reduction [40]. At about 600 °C, strength reduces along with the reduction of stiffness, due to disintegration of the calcium silicate hydrate (CSH) gel [41]. Between the temperature range of 600 °C to 800 °C, calcium carbonate (CaCO<sub>3</sub>) decomposes, resulting in further loss of strength [39, 42].

The experimental results for axial compression are shown in Figure 6 and Tables 6 and 7. The compressive strength of 28.8, 29.1, 30.3 and 28.6 MPa was recorded for mix CM, 5BT, 10 BT and 15 BT respectively. Fig. 6 and Table 7 show that the compressive strength of the specimen increased as the bentonite content increased, up to 10% bentonite. Further increase of bentonite, beyond 10%, reduces the compressive strength of the concrete cylinders. The best possible reason for the increase in strength with bentonite is the pozzolanic reaction, which takes place slower than the hydration of cement, coupled with the micro-filler effect caused by bentonite. Ahmad et al. [43] found that the compressive strength reduced with the increment in the percentage of bentonite used as the cement replacement. Studies on this topic have reported varying results, which is clearly due to the dissimilar heating temperatures used to prepare the bentonite sample. Notably, bentonite should be heated at 200 °C to achieve better strength of bentonite concrete.

The exposure temperature (200 to 800 °C) had a detrimental effect on the compressive strength of concrete, as shown in Fig. 6 and Table 7. Residual compressive strengths of 28.0 MPa, 23.7 MPa, 20.8 MPa, 13.3 MPa and 6.6 MPa were recorded for the control specimens exposed to heating regime of 23 °C, 200 °C, 400 °C, 600 °C and 800 °C, respectively. This result shows 16, 26, 52 and 76% reduction in the compressive strength of the samples exposed to 200, 400, 600 and 800 °C, respectively, compared with the compressive strength of the unheated specimen. The compressive strength of 5BT (5% Bentonite) at the heating regime of 23 °C to 800 °C was recorded as 29.1 MPa, 24.7 MPa, 21.6 MPa, 14.1 MPa and 7.1 MPa, respectively. This result shows 15, 25, 51 and 76% decrease in the compressive strength at the exposure temperatures of 200, 400, 600 and 800 °C, respectively, compared with that of the unheated specimen. The compressive strength of 10BT (10% Bentonite) at 23 °C, 200 °C, 400 °C, 600 °C and 800 °C was recorded as 30.3 MPa, 26.4 MPa, 23.5 MPa, 17.4 MPa and 12.1 MPa, respectively. Thus, the compressive strength at the exposure temperatures of 23 °C, 200 °C, 400 °C, 600 °C and 800 °C was lower by 13%, 22%, 42% and 60%, respectively, than that of the unheated sample. Moreover, the compressive strength of 15BT (15% Bentonite) at 23 °C, 200 °C, 400 °C, 600 °C and 800 °C was recorded as 28.6 MPa, 23.2 MPa, 19.7 MPa, 12.9 MPa and 7.9 MPa, respectively. The recorded strength loss at exposure temperatures of 23 °C, 200 °C, 400 °C, 600 °C and 800 °C was 19%, 31%, 55% and 78%, respectively, compared with the compressive strength of the unheated specimen.

It can be observed from these results that the samples containing bentonite showed better performance at room temperature than the control mix did. The main reason behind this difference may be the pozzolanic reaction that occurs more slowly than the hydration of cement and also the filler effect that bentonite provides to the mix. The test results also showed significant improvement in the residual compressive strength of concrete with bentonite. The concrete containing 10% bentonite showed superior performance in terms of post-heated residual compressive strength. This is because the heat capacity of the mix containing bentonite is better than that of the control mix, and the thermal conductivity of the mix containing bentonite is low, which results in improvement of the microstructure and reduction in the propagation of micro-cracks. It can also be observed that the loss of compressive strength below 400°C is very low.

The regression equations developed for the post-heated compressive strength of CM, BC5, BC10 and BC15 mix using the response surface methodology are shown in Eq.1, 2, 3 and 4,

respectively. No experimental study is available for post-heated bentonite concrete. These equations can provide a guideline for comparing the test results of post-heated concrete. The equations were developed using statistical modelling using the multi regression method. The normal plot of residuals is shown in Fig. 5, which reveals that the developed regression model is significant. The low difference between the predicted and the adjusted  $R^2$  shows the validity of the developed regression model.

Compressive Strength(MPa) = 
$$30.038 - 0.028 * T_e = 0.9808$$
 (1)

Compressive Strength (MPa) = 
$$30.665 - 0.0279 * T_e R^2 = 0.9760$$
 (2)

Compressive Strength (MPa) = 
$$31.77 - 0.0232 * T_e R^2 = 0.9493$$
 (3)

Compressive Strength (MPa) = 
$$29.211 - 0.0264 * T_e R^2 = 0.9929$$
 (4)

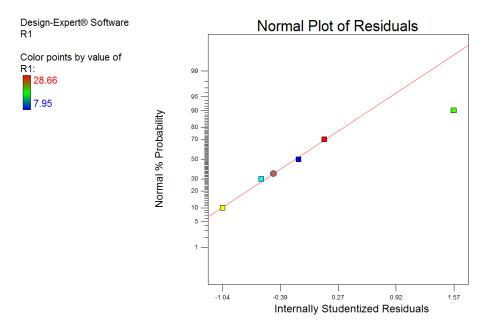


Figure 5: Plot of residuals for BC15 mix

ID	Exposure temperature (°C)	Compressive strength MPa (Experimental)	Compressive strength MPa (Predicted)	% Error	Residual (R <sup>2</sup> )
	23	28.805	29.394	2.00381	0.9808
	200	23.79	24.438	2.651608	0.9808
СМ	400	20.88	18.838	-10.8398	0.9808
	600	13.33	13.238	-0.69497	0.9808
	800	6.67	7.638	12.67347	0.9808
	23	29.1	30.021	3.067853	0.9760
	200	24.7	25.065	1.456214	0.9760
5BC	400	21.69	19.465	-11.4308	0.9760
	600	14.12	13.865	-1.83916	0.9760
	800	7.1	8.265	14.09558	0.9760
	23	30.31	30.1314	-0.59274	0.9493
	200	26.47	26.025	-1.70989	0.9493
10BC	400	23.59	21.385	-10.311	0.9493
	600	17.48	16.745	-4.38937	0.9493
	800	12.1	12.105	0.041305	0.9493
	23	28.66	28.6038	-0.19648	0.9929
	200	23.23	23.931	2.929255	0.9929
15BC	400	19.75	18.651	-5.89245	0.9929
	600	12.93	13.371	3.298183	0.9929
	800	7.95	8.091	1.742677	0.9929

Table 6: Experimental and predicted compressive strength using Response SurfaceMethodology

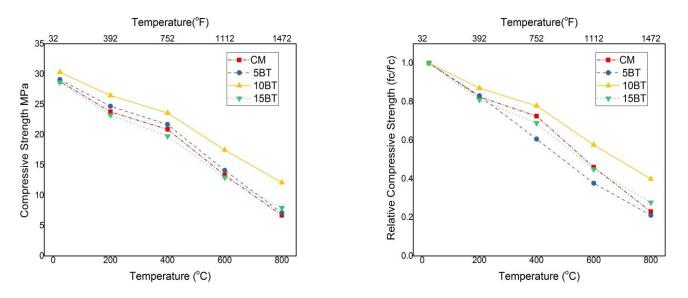
Residual Compressive Strength							Residual Splitting Tensile Strength				
Samp le Name	23° C	200° C	400° C	600° C	800° C	23° C	200° C	400° C	600° C	800 °C	
СМ	1	0.83	0.72	0.46	0.23	1	0.69	0.48	0.28	0.13	
5BC	1	0.85	0.75	0.49	0.24	1	0.70	0.55	0.32	0.18	
10BC	1	0.87	0.78	0.58	0.40	1	0.75	0.62	0.40	0.24	
15BC	1	0.81	0.69	0.45	0.28	1	0.73	0.60	0.30	0.17	

Table 7: Relative Residual Splitting Tensile and Compressive Strength

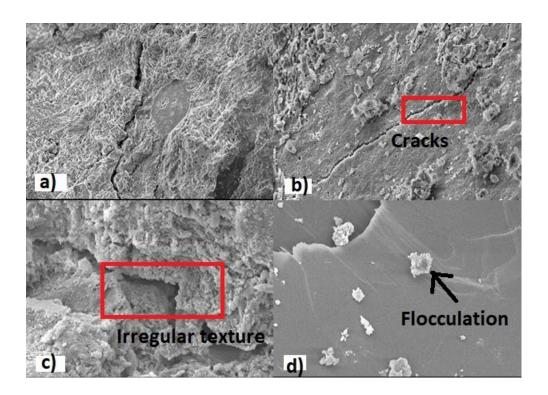
 Table 8; Compressive Strength, Modulus of Elasticity, Energy Absorption, Tensile Strength

 and Ductility at Targeted Temperatures

ID		Compressive	Modulus of	Energy	Tensile	Dustility
	Temp.	Strength	Elasticity	Absorption	Strength	Ductility
		(MPa)	(GPa)	(J/m <sup>3</sup> )	(MPa)	
СМ	23°C	28.805	26.18	436.2	3.25	0.4
	200°C	23.79	22.27	398.1	2.24	0.47
	400°C	20.88	17.613	389.3	1.56	0.68
	600°C	13.33	13.75	459.7	0.91	0.75
	800°C	6.67	8.60	523.6	0.42	0.83
5BT	23°C	29.10	27.10	436.7	3.42	0.52
	200°C	24.70	22.90	371.6	2.39	0.59
	400°C	21.69	17.86	413.8	1.88	0.83
	600°C	14.12	13.94	481.4	1.094	0.87
	800°C	7.10	8.89	489.9	0.615	0.96
10BT	23°C	30.31	27.93	450.2	3.76	0.85
	200°C	26.47	23.36	418.9	2.82	0.77
	400°C	23.59	18.48	485.8	2.33	0.84
	600°C	17.48	14.44	673.4	1.504	0.94
	800°C	12.10	9.37	867.7	0.902	1.03
15BT	23°C	28.66	25.56	423.2	3.51	0.83
	200°C	23.23	21.54	364.1	2.56	0.79
	400°C	19.75	15.00	416.6	1.82	0.87
	600°C	12.93	12.30	444.5	1.053	0.99
	800°C	7.95	8.13	607.5	0.596	1.04



*Figure 6: Compressive strength for control mix and bentonite modified concrete mixtures at ambient and targeted temperatures a) Compressive strength b) Relative compressive strength* 



*Figure 7 Micrographs of 10BT exposed to elevated temperature a)* 200°*C b)* 400°*C c)* 600°*C d)* 600°*C* 

#### 4.2 Tensile strength

The splitting tensile strength test was performed according to ASTM C 496[44]. Figure 8 illustrates that the splitting tensile strength increased with an increment of bentonite, up to 10%, and that further increments of bentonite resulted in decreasing the splitting tensile strength. The figure also shows that increasing the exposure temperature resulted in decreased splitting tensile strength.

The exposure temperature (200 °C to 800 °C) had a damaging effect on the splitting tensile strength of concrete, as shown in Fig. 8 and Table 7. A residual tensile strength of 3.2 MPa ,2.2MPa, 1.5 MPa, 0.9 MPa and 0.4 MPa was recorded for the control sample at 23 °C to 800 °C with an increment of 200°C after 200°C, respectively. This result shows 31%, 52%, 72% and 87% loss of tensile strength at 200 °C, 400 °C, 600 °C and 800 °C, respectively. Similarly, the splitting tensile strength recorded for 5BT (5% Bentonite) at 23 °C, 200 °C, 400 °C, 600 °C and 800 °C was 3.4, 2.3, 1.8, 1.09 and 0.6 MPa respectively. This result shows 30%, 45%, 68% and 82% loss of splitting tensile strength at 200 °C, 400 °C, 600 °C and 800 °C, respectively. The splitting tensile strength recorded for 10BT (10% Bentonite) at 23 °C, 200 °C, 400 °C, 600 °C and 800 °C was 3.7 MPa, 2.8 MPa, 2.3 MPa, 1.5 MPa and 0.9 MPa, respectively. This result clearly shows that the loss of tensile strength at 200 °C, 400 °C, 600 °C and 800 °C was 25%, 38%, 60% and 76%, respectively. Similarly, the splitting tensile strength recorded for 15BT (15% Bentonite) at 23 °C, 200 °C, 400 °C, 600 °C and 800 °C was 3.5 MPa, 2.5 MPa, 1.8 MPa, 1.0 MPa and 0.5 MPa, respectively. This result shows a loss of 27%, 40%, 70% and 83% splitting tensile strength at 200 °C, 400 °C, 600 °C and 800 °C, respectively. The best possible reason for the increase at elevated temperatures in the splitting tensile strength of the mixes that incorporate bentonite can be the better heat capacity of bentonite. Moreover, bentonite provides a filler effect to the concrete because the size of the bentonite particles is smaller than that of the cement particles.

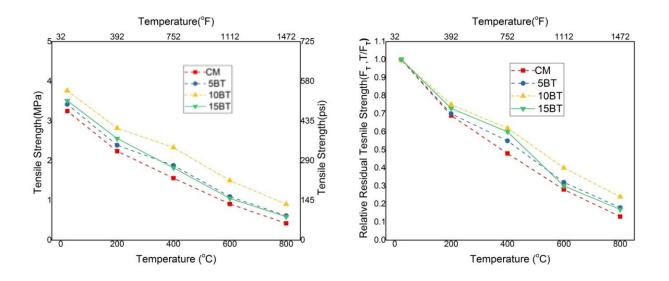


Figure 8: Variation of splitting tensile strength at ambient and targeted temperatures for control mix and bentonite modified concrete a) Absolute splitting tensile strength b) Relative splitting tensile strength

#### 4.3 Stress-strain response

To envisage the behavior of bentonite in concrete that is exposed to elevated temperatures, it is essential to consider the stress-strain behavior of the control specimens that are exposed to high temperatures for comparison purposes. When concrete is exposed to heat, physical and chemical reactions occur at high temperatures, such as the moisture loss, the dehydration of the cement paste and the disintegration of the aggregate. This phenomenon is responsible for internal micro-cracking and the loss of strength of concrete that is exposed to elevated temperatures. The experimentally obtained stress-strain curves reveal that as the temperature increased, the stress decreased and the peak strain increased. The stress-strain behavior is illustrated in Fig. 9. The recorded peak stress and the corresponding peak strain of the control mix (CM) at 23 °C was 28.805 MPa and 0.00214 mm/mm, respectively. At 200 °C, the peak stress decreased by 17% and the peak strain increased by 12%. At 400 °C, the peak stress decreased by 28% and the peak strain increased by 35%. Similarly, at 600 °C and 800 °C, the peak stress decreased by 54% and 77% and the strain increased by 114% and 357%, respectively. The test stress-strain response of the specimens after exposure to elevated temperatures are shown in Figure 8.

The stress–strain behavior of the CM and 5BT (5% Bentonite) followed a similar trend up to the elastic limit. After the elastic limit, significant increase in strain was observed in the 5BT mix compared with the CM mix. Further, this effect was more evident for a higher temperature range (400 to 800 °C). At 23 °C, the recorded peak stress and the corresponding strain were 29.1 MPa and 0.00217 mm/mm, respectively. At 200 °C, the peak stress decreased by 15% and the corresponding strain increased by 12%. The residual strength at 200 °C still retained about 90% of the original unheated value. The peak stress decreased by 25% at 400 °C, and the strain increased by 38%. Similarly, at 600 °C and 800 °C, the peak stress decreased by 51% and 76% and the peak strain increased by 125% and 360% respectively. It can be concluded from Figure 6 that the stress–strain relationship significantly changes with the increase in temperature. The strength decreases, whereas the deformation increases with temperature. These changes follow a trend similar to that mentioned in a previous study on conventional concrete that was exposed to elevated temperatures [45].

Similarly, 10BT (10% Bentonite) behaves better than CM, 5BT and 15BT. It can be observed from Fig. 8 that at 23 °C, the peak stress and strain was 30.31 MPa and 0.00219 mm/mm. Fig. 8 also shows that at 200 °C and 400°C, the stress decreased by 13% and 22% whereas the strain increased by 14% and 46%, respectively. Similarly, at 600 °C and 800 °C, the stress decreased by 48% and 60% and the strain increased by 137% and 388%, respectively. The peak strain increased with the increase in the exposure temperature for 10BT. The cement mortar structure became loose after high temperature exposure, which is attributed to the pore expansion owing to the vaporization of the absorbed water. During the cooling phase, the ionized CaO decomposes from calcium hydroxide [Ca(OH)<sub>2</sub>], absorbs water and then reverts to Ca(OH)<sub>2</sub>. Thus, it results in the increase of the concrete volume. The damage gathered during the cooling process further lessens the residual strength. This is a possible explanation for the change in the peak strain. Figure 9 shows that the recorded ultimate stress and the corresponding strain at 23 °C for 15BT (15% Bentonite) was 28.6 MPa and 0.00218 mm/mm, respectively. At 200 °C and 400°C, the stress decreased by 19% and 31% and the corresponding strain increased by 13% and 37%, respectively. Similarly, at 600 °C and 800 °C, the stress decreased by 55% and 72% and the strain increased by 120% and 354%, respectively. This result reveals the enhanced deformation capacity of concrete specimens containing 5% bentonite that are exposed to elevated temperatures (200 to 800 °C).

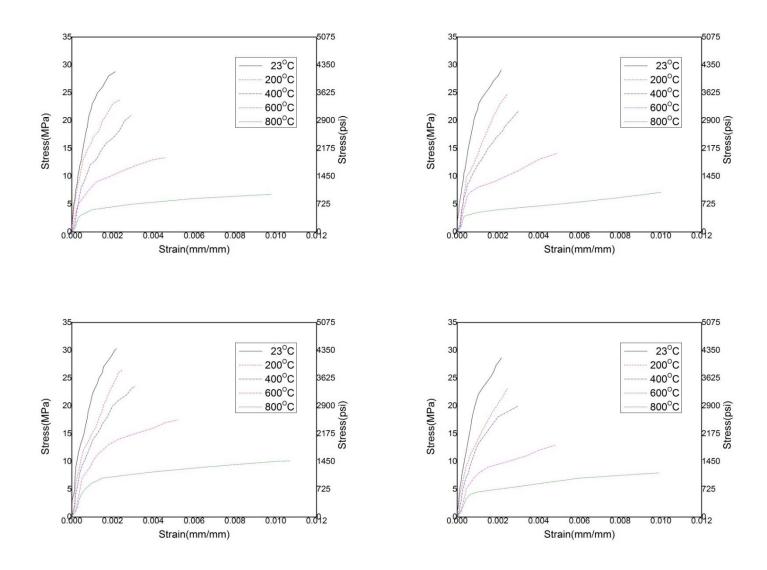


Figure 9: Stress-strain response at ambient and elevated targeted temperatures a) CM b) 5BT c) 10BT d) 15BT

### 4.4 Modulus of elasticity, Ductility and Energy Absorption

The modulus of elasticity was calculated according to ASTM C469 [46]. The modulus of elasticity was calculated on strain corresponding to 40% of the compressive stress for each targeted temperature. The results are shown in the form of absolute and relative loss of elastic modulus. Figure 8 clearly shows that the elastic modulus increased with the increase in the percentage of bentonite up to 10%. Beyond 10%, the elastic modulus started decreasing. It can also be observed that the elastic modulus started decreasing with the increase in the exposure temperature. It can be predicted from Fig. 11 that the loss of elastic modulus is almost the same for all mixes.

It can be clearly observed from Figure 11 and Table 7 that the modulus of elasticity for the control mix at 23 °C, 200 °C, 400 °C, 600 °C and 800 °C was recorded as 26.1 GPa, 22.2 GPa, 17.6 GPa, 13.7 GPa and 8.6 GPa, respectively. Similarly, the loss of absolute modulus at 200 °C to 800 °C was 15%, 33%, 47% and 67%, respectively. For 5BT (5% Bentonite), the modulus of elasticity at 23 °C to and 800 °C was recorded as 27.1 GPa, 22.9 GPa, 17.8 GPa, 13.9 GPa and 8.8 GPa, respectively. The loss of modulus for 5BT (5% Bentonite) was recorded as 15%, 34%, 49% and 67% at 200 °C to 800 °C, respectively. Figure 11 also shows that the modulus of elasticity for 10BT (10% Bentonite) at 23 °C to 800 °C was recorded as 27.9 GPa, 23.3 GPa, 1.4 GPa, 14.4 GPa and 9.3 GPa, respectively. For 10BT (10% Bentonite), the loss of modulus was recorded as 16%, 34%, 48% and 76% at 200 °C to 800 °C, respectively. The modulus of elasticity for 15BT (15% Bentonite) at 23 °C to 800 °C was recorded as 25.5 GPa, 21.5 GPa, 15 GPa, 12.3 GPa and 8.13 GPa, respectively. From Fig. 10, it can be observed that the loss of modulus for 15BT (15% Bentonite) was recorded as 16%, 30%, 52% and 78% at 200 °C to 800 °C, respectively.

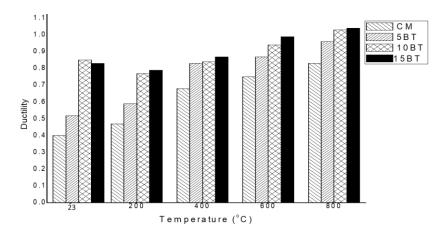


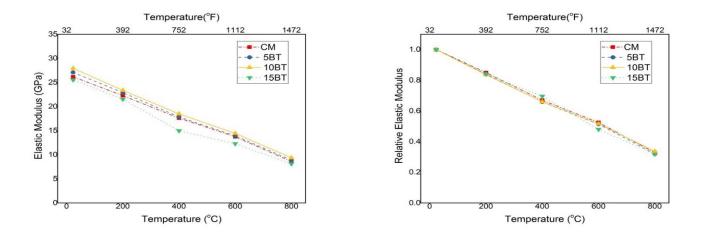
Figure 10: Ductility of the tested specimens

The ductility of the specimens was measured as the ratio of the ultimate strain of concrete to the matching yield strain. The yield strain was calculated as the strain corresponding to 65% unconfined compressive strength of concrete. Figure 10 and Table 7 show that ductility values of 0.4, 0.47, 0.68, 0.75 and 0.83 were recorded for the CM mix (0% bentonite) at 23 °C, 200 °C, 400 °C, 600 °C and 800 °C, respectively. A similar trend of ductility was observed for 5 BT (5% bentonite). Ductility values of 0.52, 0.59, 0.83, 0.87 and 0.96 were recorded for 5 BT at 23 °C to 800 °C, respectively. Higher ductility values were observed for the 10 BT mix

than for the CM and 5 BT mixes. An enhancement of 53% and 19% in ductility was observed for 5BT and 10BT compared with that for CM at 23 and 800°C, respectively. For this group (10BT), ductility values of 0.85, 0.77, 0.84, 0.94 and 1.03 were recorded at 23 °C to 800 °C, respectively. Further, ductility values of 0.83, 0.79, 0.87, 0.99 and 1.04 were recorded for 10BT at 23 °C to 800 °C, respectively. It was concluded that the highest and lowest ductility values were observed for 15 BT and CM mix irrespective of the corresponding temperature. However, the ductility values of 10 BT and 15 BT were very close to each other.

The stress–strain results showed that mixture having bentonite showed higher peak stress as well as peak strain. Thus, the energy absorption of all formulations was computed and compared. Energy absorption was quantified by calculating the area under the stress–strain curve, and the area up to ultimate strain was considered for all mixes [36]. The area under the

stress–strain curve for all mixes was calculated and compared with that for the control mix. Table 7 shows the energy absorption capacity of all mixes. It can be clearly observed that energy absorption increased by incorporating bentonite up to 10%, and that beyond that level, energy absorption started decreasing. The performance of the mix containing 10% bentonite was superior to that of all the other mixes. At 23 °C to 800 °C, energy absorption was recorded as 450.2 J/m<sup>3</sup>, 418.9 J/m<sup>3</sup>, 485.8 J/m<sup>3</sup>, 673.4 J/m<sup>3</sup> and 867.7 J/m<sup>3</sup>, respectively. This is because the addition of bentonite, up to an optimum amount, improved the microstructure of the mix, which resulted in an improvement of the inelastic deformation without compromising the load carrying capacity. This increased energy absorption is attributed to the presence of bentonite, which efficiently resists the propagation of cracks.



*Figure 11: Variation in modulus of elasticity of control mix and bentonite modified concrete at ambient and elevated temperatures (a) absolute elastic modulus, (b) relative elastic modulus* 

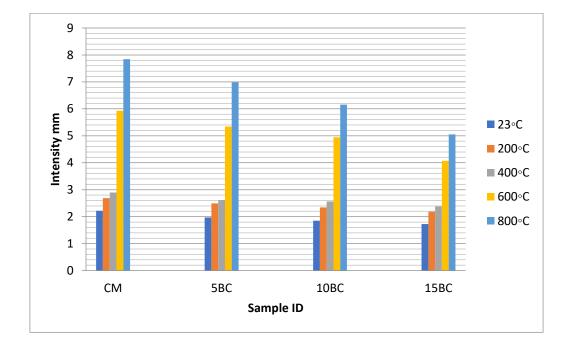
#### 4.5 Sorptivity

Sorptivity is a property that characterizes the ability of a material to absorb water through capillarity [33]. Sorptivity parameter and is used extensively to assess the resistance of concrete that is exposed to an aggressive environment [34].

The sorptivity test was performed according to ASTM C 1585 [35]. The test results displayed that the water absorption reduced as the cement replacement by bentonite increased. The reduction in water absorption is attributed to the chemical reaction between natural pozzolans and calcium hydroxide of hydrated cement paste that consumes more lime. Further, since the particle size of bentonite is lesser than that of conventional cement, it results in the packing of the binder and hence reduction of porosity. This reduction in porosity is responsible for improved resistance to water absorption. The reported test results of sorptivity are in agreement with those of the previous literature [43].

Figure 12 shows that water absorption by capillary action decreases by increasing the percentage of bentonite at 23 °C. The figure clearly shows that water absorption also increases with the increase in exposure temperature because free and absorbed water vanishes with a rise in temperature beyond 200 °C. Water absorption increases drastically as the temperature rises above 400 °C. For all mixes, water absorption at 600 °C and 800 °C increases to more than 150% of the water absorption at 23 °C. From this discussion, it is evident that water absorption

decreases with increase in bentonite content, regardless of the target temperature. The size of bentonite particles is smaller than cement, it provides a filler effect and makes concrete less porous, resulting in less water absorption under capillary action. Moreover, at high temperatures, bentonite behaves better than the control sample, due to the greater thermal capacity of bentonite, resulting in decreased crack propagation. Figure 13,14,15 and 16 shows sorptivity of individual samples at ambient and targeted temperatures.



*Figure 12: Sorptivity at ambient and targeted temperature for control mix and bentonite modified mix* 

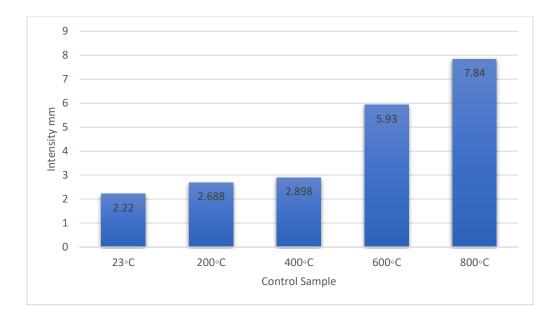


Figure 13: Sorptivity at ambient and targeted temperature for control mix

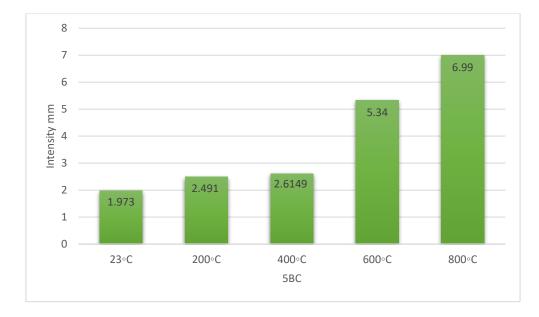
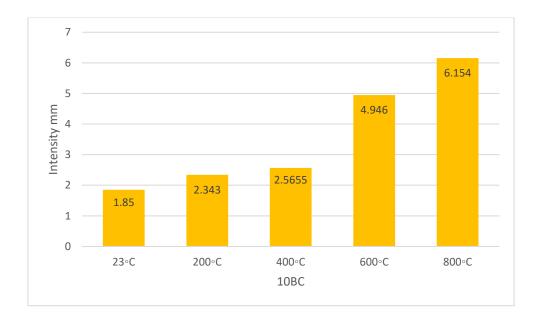


Figure 14: Sorptivity at ambient and targeted temperature for 5% bentonite mix



*Figure 15: Sorptivity at ambient and targeted temperature for 10% bentonite mix* 

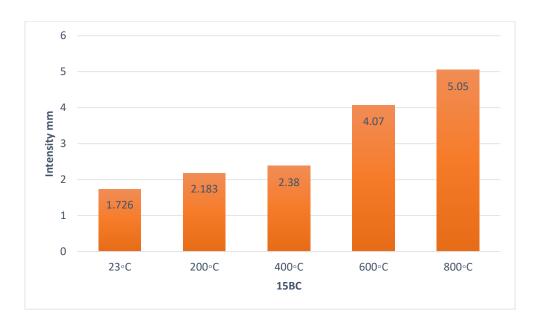


Figure 16: Sorptivity at ambient and targeted temperature for 15% bentonite mix

#### 4.6 Mass loss

Mass loss is a very important characteristic in the evaluation of the durability of concrete. From previous studies, it can be noted that the mass loss in normal strength concrete is lower than that in high strength concrete [47]. This is because in normal strength concrete, there is enough room for the free water to easily vaporize without generating pore pressure, unlike in high strength concrete.

It is evident from Figure 17 that the mass loss increases with temperature, and it is also shown that the mass loss decreases when the quantity of bentonite is increased up to 10%. Post this, mass loss again decreases. The mass recorded for the CM at 200 °C, 400 °C, 600 °C and 800 °C was 97.1%, 95.3%, 92.2% and 86.1% and the mass loss was 2.9%, 4.7%, 7.8% and 13.9%, respectively. Further, 97.5%, 96.4%, 93.2% and 87.4% mass was recorded for 5BT (5% Bentonite) at 200 °C to 800 °C and mass loss was recorded as 2.5%, 3.6%, 6.8% and 12.6%, respectively. The figure also shows that the mass of 10BT (10% Bentonite) at 200 °C, 400 °C, 600 °C and 800 °C was 98%, 97%,95.1% and 89.5% and mass loss recorded was 2%, 3%, 4.9% and 10.5%, respectively. For 15BT (15% Bentonite), mass of 97.8%, 96.7%, 94% and 88% was recorded at 200 °C to 800 °C and mass loss was recorded as 2.2%, 3.3%, 6% and 12%, respectively. From this discussion, it can be concluded that the mixes containing bentonite behaved better than the control mix. At high temperatures, bentonite behaves better than cement because of its greater heat capacity and lower thermal conductivity. High heat capacity does not support the propagation of cracks and results in better microstructure of the bentonite mix, compared with the control mix. The mass loss determined from thermogravimetric analysis was in good agreement with the measured mass loss. TGA test of control and 5 BT mix exposed to elevated temperature is shown in Fig. 18 and 19 respectively. Mass loss of 3.64, 5.60, 8.19, and 12.23% was recorded for control sample exposed to 200, 400, 600 and 800°C respectively. Lowest mass loss (only 12.23%) was recorded for control specimen at high exposure temperature (800°C). Mass loss of 3.65, 5.61, 8.19 and 12.24% was recorded for 5 BT sample exposed to 200, 400, 600 and 800°C respectively. It is worth noting that the mass loss values for control specimen and 5 BT were very close to each other with similar trend. TGA test of 10 BT and 15 BT mix exposed to elevated temperature is shown in Fig. 20 and 21 respectively. Based on TGA test, mass loss of 4, 6, 8 and 13% was recorded for 5 BT sample exposed to 200, 400, 600 and 800°C respectively. Mass loss of 4.14, 6.14, 8.65 and 12.20% was recorded for 10 BT sample exposed to 200, 400, 600 and 800°C respectively. Similarly, for 15 BT mix, mass loss of 3.49, 5.60, 7.91 and 13.28% was recorded for sample exposed to 200, 400, 600 and 800°C respectively. Based on these recorded test results, it is concluded that 10 BT incurred lowest mass loss (12.20%) which is the lowest mass loss at 800°C as compared to its counterparts. Figure 18,19,20 and 21 shows the results of TGA at targeted temperature.

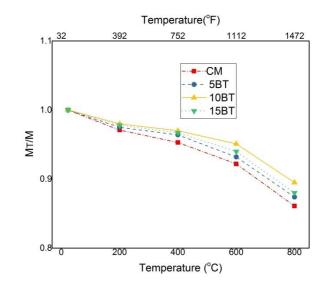


Figure 17: Mass loss variation for different mixes at targeted temperature

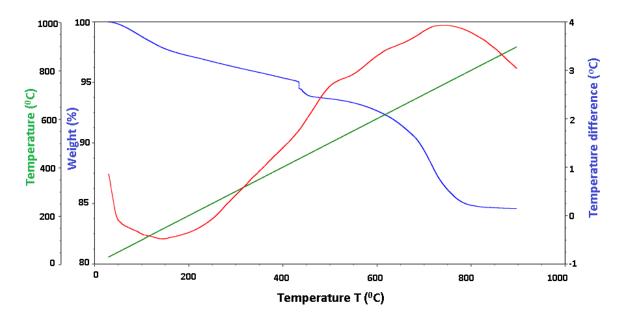


Figure 18: TGA test for control mix at targeted temperature

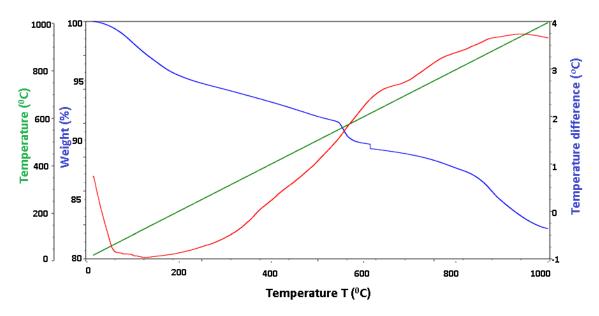


Figure 19: TGA test for 5BT at targeted temperature

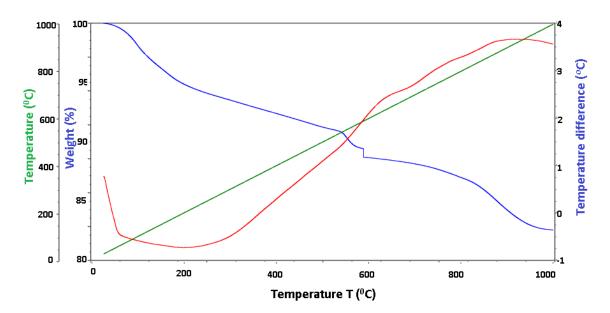


Figure 20: TGA test for 10BT at targeted temperature

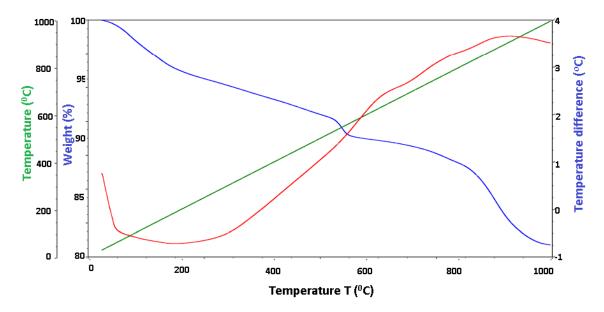


Figure 21: TGA test for 15BT at targeted temperature

## **Chapter 5**

## **5** Conclusions

Results were generated from this study on the behavior of concrete when incorporating different proportions of bentonite (5%, 10% and 15%) and were compared with the results for conventional concrete, when heated to temperatures of 200 °C, 400 °C, 600 °C and 800 °C, to report new data on the fire endurance of concrete. Based on this study, the following conclusions can be drawn:

- 1. Samples containing 10% bentonite showed better performance in terms of residual compressive strength at all targeted temperatures. Residual compressive strength increases due to the greater fire endurance of bentonite compared with that of cement.
- 2. The residual tensile strength followed a trend similar to that of the residual compressive strength. The performance of the sample containing 10% bentonite was superior to that of all other samples.
- 3. The water absorption of bentonite concrete increased with temperature irrespective of the percentage replacement of cement by bentonite. The bentonite content in concrete resulted in enhanced resistance to water absorption. The increase was attributed to the improved microstructure and the filler effect of bentonite.
- 4. The bentonite content in concrete slightly improved its mass loss. However, the mass loss increased at a higher percentage of bentonite (15%) in concrete.
- 5. The bentonite concrete with 10% bentonite content showed significant improvement in the energy absorption capacity. This finding shows that the bentonite concrete can be utilised in practical engineering applications that require high energy absorption.
- 6. The ductility of the concrete specimens improved significantly with increased bentonite content. A clear trend of ductility was observed, which shows that the ductility increases with the percentage of bentonite.
- 7. The performance of the concrete containing 10% bentonite content was superior, in terms of ultimate strength and the corresponding peak strain response, to that of the concrete mixes containing other percentages of bentonite content.

# **6** Recommendations

- Stressed and unstressed properties should be checked
- A complete investigation of structural behavior should be studied for members made up of bentonite concrete
- Use of bentonite along with polypropylene fibres and study their effect on fire performance

# 7 References

- 1. Park, S.H., et al., *Tensile behavior of ultra high performance hybrid fiber reinforced concrete*. Cement and Concrete Composites, 2012. **34**(2): p. 172-184.
- 2. Meng, W. and K.H. Khayat, *Experimental and numerical studies on flexural behavior* of ultrahigh-performance concrete panels reinforced with embedded glass fiberreinforced polymer grids. Transportation Research Record, 2016. **2592**(1): p. 38-44.
- 3. Wille, K., D.J. Kim, and A.E. Naaman, *Strain-hardening UHP-FRC with low fiber contents*. Materials and structures, 2011. **44**(3): p. 583-598.
- 4. Neville, A.M. and J.J. Brooks, *Concrete technology*. 1987: Longman Scientific & Technical England.
- 5. Chouhan, P., S. Jamle, and M. Verma, *Effect of Silica Fume on Strength Parameters of Concrete as a Partial Substitution of Cement.* IJSART3, 2017. **5**: p. 3-7.
- Mazloom, M., A. Ramezanianpour, and J. Brooks, *Effect of silica fume on mechanical properties of high-strength concrete*. Cement and Concrete Composites, 2004. 26(4): p. 347-357.
- 7. Sakr, K. and E. El-Hakim, *Effect of high temperature or fire on heavy weight concrete properties.* Cement and concrete research, 2005. **35**(3): p. 590-596.
- Hertz, K.D., *Concrete strength for fire safety design*. Magazine of concrete research, 2005. 57(8): p. 445-453.
- Khoury, G., et al., *Modelling of heated concrete*. Magazine of concrete research, 2002.
   54(2): p. 77-101.
- 10. Hertz, K.D. and L.S. Sørensen, *Test method for spalling of fire exposed concrete*. Fire safety journal, 2005. **40**(5): p. 466-476.
- 11. Luccioni, B.M., M.I. Figueroa, and R.F. Danesi, *Thermo-mechanic model for concrete exposed to elevated temperatures*. Engineering Structures, 2003. **25**(6): p. 729-742.
- 12. Arioz, O., *Effects of elevated temperatures on properties of concrete*. Fire safety journal, 2007. **42**(8): p. 516-522.
- Phan, L.T., J.R. Lawson, and F.L. Davis, *Effects of elevated temperature exposure on heating characteristics, spalling, and residual properties of high performance concrete.* Materials and structures, 2001. **34**(2): p. 83-91.
- 14. Kalifa, P., F.-D. Menneteau, and D. Quenard, *Spalling and pore pressure in HPC at high temperatures*. Cement and concrete research, 2000. **30**(12): p. 1915-1927.

- 15. Shihada, S., *Effect of polypropylene fibers on concrete fire resistance*. Journal of civil engineering and management, 2011. **17**(2): p. 259-264.
- 16. Lau, A. and M. Anson, *Effect of high temperatures on high performance steel fibre reinforced concrete*. Cement and concrete research, 2006. **36**(9): p. 1698-1707.
- Baloch, W.L., et al., *Effect of elevated temperatures on mechanical performance of normal and lightweight concretes reinforced with carbon nanotubes*. Fire technology, 2018. 54(5): p. 1331-1367.
- Xu, Y., et al., *Impact of high temperature on PFA concrete*. Cement and concrete research, 2001. **31**(7): p. 1065-1073.
- Andrejkovičová, S., et al., *Bentonite as a natural additive for lime and lime-metakaolin mortars used for restoration of adobe buildings*. Cement and Concrete Composites, 2015. 60: p. 99-110.
- 20. Memon, S.A., et al., *Utilization of Pakistani bentonite as partial replacement of cement in concrete*. Construction and building materials, 2012. **30**: p. 237-242.
- 21. Mirza, J., et al., *Pakistani bentonite in mortars and concrete as low cost construction material*. Applied Clay Science, 2009. **45**(4): p. 220-226.
- Saba, S., et al., *Microstructure and anisotropic swelling behaviour of compacted bentonite/sand mixture*. Journal of Rock Mechanics and Geotechnical Engineering, 2014. 6(2): p. 126-132.
- 23. Hendriks, C.A., et al. *Emission reduction of greenhouse gases from the cement industry*. in *Proceedings of the fourth international conference on greenhouse gas control technologies*. 1998. Interlaken, Austria, IEA GHG R&D Programme.
- 24. Ramanathan, V. and Y. Feng, *Air pollution, greenhouse gases and climate change: Global and regional perspectives.* Atmospheric environment, 2009. **43**(1): p. 37-50.
- 25. Astm, C., 150. Standard specification for portland cement, 1994.
- 26. Faroug, F., J. Szwabowski, and S. Wild, *Influence of superplasticizers on workability of concrete*. Journal of materials in civil engineering, 1999. **11**(2): p. 151-157.
- Safiuddin, M. and N. Hearn, *Comparison of ASTM saturation techniques for measuring* the permeable porosity of concrete. Cement and Concrete Research, 2005. 35(5): p. 1008-1013.
- Assaad, J.J., J. Harb, and E. Chakar, *Relationships between key ASTM test methods* determined on concrete and concrete-equivalent-mortar mixtures. Journal of ASTM International, 2009. 6(3): p. 1-13.

- 29. Khaliq, W. and H.A. Khan, *High temperature material properties of calcium aluminate cement concrete*. Construction and Building Materials, 2015. **94**: p. 475-487.
- 30. Song, P. and S. Hwang, *Mechanical properties of high-strength steel fiber-reinforced concrete*. Construction and Building Materials, 2004. **18**(9): p. 669-673.
- 31. Li, B., Zhong 2011 J. of. Mater. Sci. 46(17): p. 5595-5614.
- Baloch, W.L., R.A. Khushnood, and W. Khaliq, *Influence of multi-walled carbon nanotubes on the residual performance of concrete exposed to high temperatures*. Construction and Building Materials, 2018. 185: p. 44-56.
- 33. Hall, C., *Water sorptivity of mortars and concretes: a review*. Magazine of concrete research, 1989. **41**(147): p. 51-61.
- 34. Lockington, D., J.-Y. Parlange, and P. Dux, *Sorptivity and the estimation of water penetration into unsaturated concrete*. Materials and Structures, 1999. **32**(5): p. 342.
- ASTM, C., 1585. Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic-", ASTM Int, 2013: p. 4-9.
- 36. Marara, K., Ö. Erenb, and İ. Yitmena, *Compression specific toughness of normal* strength steel fiber reinforced concrete (NSSFRC) and high strength steel fiber reinforced concrete (HSSFRC). Materials Research, 2011. **14**(2): p. 239-247.
- 37. Mehta, P.K. and P.J. Monteiro, *Concrete microstructure, properties and materials*. 2017.
- Kodur, V., Properties of concrete at elevated temperatures. ISRN Civil engineering, 2014. 2014.
- 39. Khoury, G., Compressive strength of concrete at high temperatures: a reassessment.Magazine of concrete Research, 1992. 44(161): p. 291-309.
- 40. Li, M., C. Qian, and W. Sun, *Mechanical properties of high-strength concrete after fire*. Cement and concrete research, 2004. **34**(6): p. 1001-1005.
- Phan, L.T. and N.J. Carino, *Effects of test conditions and mixture proportions on behavior of high-strength concrete exposed to high temperatures*. ACI Materials Journal, 2002. 99(1): p. 54-66.
- 42. Khaliq, W., *Mechanical and physical response of recycled aggregates high-strength concrete at elevated temperatures.* Fire safety journal, 2018. **96**: p. 203-214.
- 43. Ahmad, S., et al., *Effect of Pakistani bentonite on properties of mortar and concrete*. Clay Minerals, 2011. 46(1): p. 85-92.
- 44. ASTM, C., Standard test method for splitting tensile strength of cylindrical concrete specimens. 2011.

- 45. Felicetti, R. and P.G. Gambarova, *Effects of high temperature on the residual compressive strength of high-strength siliceous concretes*. ACI materials Journal, 1998.
  95: p. 395-406.
- Astm, C., 469. Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression," American Society for Testing and Materials, Pennsylvania, 2002.
- 47. Ali, F., *Is high strength concrete more susceptible to explosive spalling than normal strength concrete in fire?* Fire and materials, 2002. **26**(3): p. 127-130.