DURABILITY STUDY ON PARTIAL REPLACEMENT OF DHOLPUR STONE WASTES AS COARSE AGGREGATE IN CONCRETE

Ph.D. Thesis

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by

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This thesis is dedicated to

***My Parents** (Dr. Selvaraju and Ms. Vijayalakshmi),*

Beloved Brother (Dr. Abu)

&

Civil Engineers in the family

Mr. Rajamohan, Mr. Sudheesh & Mr. Ramsundar

CERTIFICATE

This is to certify that the thesis entitled **"Durability Study on Partial Replacement of Dholpur Stone Wastes as Coarse Aggregate in Concrete"** being submitted by **Sanjeev Kumar K.S. (ID. NO. 2014RCE9052)** is a bonafide research work carried out our supervision and guidance in fulfilment of the requirement for the award of the degree of **Doctor of Philosophy** in the Department of Civil Engineering, Malaviya National Institute of Technology Jaipur, India. The matter embodied in this thesis is original and has not been submitted to any other University or Institute for the award of any other degree.

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DECLARATION

I, Sanjeev Kumar, declare that this thesis entitled, "**Durability Study on Partial Replacement of Dholpur Stone Wastes as Coarse Aggregate in Concrete"** and the work presented in it, are my own, I confirm that:

- This work was done wholly or mainly while in candidature for a research degree at this university.
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- Where I have consulted the published work of others, this is always clearly attributed.
- Where I have quoted from the work of others, the source is always given. With the exception of such quotations, this thesis is entirely my own work.
- I have acknowledged all main sources of help.
- Where the thesis is based on work done by myself, jointly with others, I have made clear exactly what was done by others and what I have contributed myself.

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(Sanjeev Kumar K.S.)

ABSTRACT

Sandstones are generally composed of grains of quartz and other minerals of fairly uniform size which are often smooth and rounded. These grains are held together by a cementing material which may be siliceous or ferruginous. The toughness of sandstone depends mostly on the nature of this cementing material. While utilising sandstone into concrete, different properties of sandstones has to be studied in detail to know the potential use of sandstone as a part or whole replacement of coarse aggregates. This replacement depends on many factors, like; mineralogical composition, micro structure, moisture absorption capacity, presence of carbonates and many other parameters.

In this study, tests were analysed for sandstone procured from Dholpur, eastern most part of Rajasthan state in India. Sandstone waste generation is very much higher in this particular region and it is estimated that Rajasthan alone produces 900 million tons of sandstone waste thus leading to a large dumping of these materials without any particular utilisation. In order to overcome this massive dumping of sandstone wastes and to lessen the use of natural aggregates, a study was carried out to find out the effective use of these sandstone wastes in concrete. At initial stage, preliminary tests were done to study the morphology, compressive strength based on combined gradation, clay content percentage and Rietveld curve fitting was done to identify the major constituents in the sandstone aggregate. Partial replacement of such aggregates would prevent the usage of natural aggregates which are in the zone of depletion, thus protecting the natural resources and reducing landfilling of mine wastes.

In this study, M30 grade of concrete was designed as per IS 10262: 2010, with water cement ratio of 0.40. However, to find the scattering of strength plots, two more water-cement ratios of 0.35 and 0.45 was adopted. Control mix consists of 0% quartz sandstone and replacement of coarse aggregates was done for 0-100%, in the multiples

of 20%. The properties of concrete, like; compressive strength, flexural tensile strength, abrasion resistance, pull-off strength, water permeability, water absorption, resistance to acid attack and sulphate attack, carbonation, depth of chloride penetration and corrosion of steel reinforcements were tested and their micro structures were analysed using Scanning Electron Microscopy (SEM). Thermogravimetric analysis (TGA) and action of fire were also studied.

The microscopic study revealed the presence of increased void fractions in the concrete samples containing quartz sandstone aggregates. These void fractions were found to enhance the thermal resistance of concrete based on the reduced weight loss upon heating them and also assumed to improve insulation properties by hindering the heat transfer in the material. It was discovered that the compressive strength, flexural strength and the sulphate attack resistance were inferior to that of control concrete; but upto a certain level of replacement, they showed a better resistance to pull off and carbonation. Also, the decrease in strength was found to be comparable to that of control specimens. It was observed that the quartz sandstones might be utilised as a partial replacement of coarse aggregates up to 40% without considerable decrease in its preferred strength.

The efficient utilisation of these quartz sandstone wastes in concrete can reduce the substantial amount of landfill that is used for dumping them and also provide a valuable source of supplementary aggregate used in the making of cement concrete contributing to the overall sustainability.

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CHAPTER-1

INTRODUCTION

1.1 Background

Concrete is the third most widely used substance in the world after air and water. The concrete has become one of the major discovery that has eased the construction activities around the globe. Concrete being a versatile material can be formed into any shape and size. The ingredients of concrete are cement, aggregates, water and sometimes with admixtures. The most important ingredient in concrete is the ordinary looking grey powder known as cement. The Ordinary Portland Cement (OPC) production has raised concern among the practitioners regarding the sustainability in the concrete industry (Ban et al., 2017). The concrete also has 60 to 75 percentage of the total volume as aggregates, which is also a significant ingredient.

The world is experiencing an acute shortage of natural aggregates which are used in concrete manufacturing. The strict environmental protection laws and the dying rivers have raised a query against the availability of natural aggregates for construction activities. The construction industries have also started to use large number of by-products from other industries (Kovler 2012).

Stone waste disposal has become one of the increasing drawbacks on the environmental sustainability. Uses of stones like marbles, granites and sandstones as a building material has increased and the disposal of unused stones possess a severe threat to the society. Every year tons of stone wastes have been generated, and it was estimated that almost 25% of stone goes as wastes while excavation and processing them. Over the years, the quarrying area has expanded, the use of modern machinery had led to the cutting of hardest of stones and exposing the soft underbelly of sandstones. With increased urbanisation, industrialisation and technological innovations, large amount of stone junks have been generated by mining, cutting and polishing activities. International natural stone production is plenty and many European and Asian countries have indulged in stone quarrying like India. These lands generate an enormous amount of quarry wastes which is being dumped without any particular utilisation. Some of these waste materials include stone wastes (marble, granite, sandstone) incineration ash, copper tailings, flyash, carbon steel slag, coal waste, mine waste, ceramic waste, construction and demolition waste, foundry slag, wood ash, furnace slag, welding slag, ISG slag, wollastonite, waste tire rubber etc. Reduction of raw material generation and increase in waste utilisation being the two key parameters in reducing material throughput. At the same time, waste prevention, recycle and reuse reduces the need for virgin materials. It also reduces the associated environmental impacts (Gazi et al., 2012; Van Ewijk et al., 2016). The best approach in overcoming these wastes management problems is to promote large volume recycling of these waste materials (Ken et al., 2015).

Quarries are the ones which generate most of the stone wastes and the lack of disposal plans create an enormous environmental concern. Since quarrying is usually done in open cast method which involves exploration, blasting, transportation and disposal of waste rocks, it greatly affects the environmental surroundings. Tremendous pressure on soil and water resources are generated by quarrying activities, and the dumping of waste rocks in open areas disrupts drainage, diverts the streams into agricultural lands resulting in flooding of crop fields (Stehouwer et al., 2006).

Use of concrete has been increasing all over the world and producing green concrete is the best solution to tackle environmental problems. The surplus use of construction materials such as aggregates and cement is the primary reason behind the environmental degradation. Upon excessive extraction and usage of these materials could lead to a lot of environmental issues. There has also been an urge of concern in the utilisation of by-products and waste materials in cement concrete. On utilisation of these wastes in concrete has not only economic benefits but also reduces the landfilling problems, saves energy and also numerous ecological advantages (Xin Yu et al., 2016; Blankendaal et al., 2014; Kim Hung Mo et al., 2016). In the recent years, the infrastructure companies are facing many challenges to improve the sustainability in the initial stages of concrete production and also by finding an appropriate eco-friendly material to replace the growing demand for natural aggregates.

Quite a number of researches has been carried out on the utilisation of waste materials in cement concrete as an alternative for conventional overused aggregates and clinker. Flyash has been used by many researchers as a fractional substitute of cement for the production of Portland Pozzolana Cement (PPC). Ashes like rice husk ash, baggase ash and bottom ash has been utilised as a filler and partial replacement of cement in concrete production. Stone wastes, copper slag, ceramic wastes, ISF slag, construction and demolition waste etc. have been utilised as partial and/or complete replacement for coarse and fine aggregates in concrete. The idea of replacing overused natural aggregates (fine and coarse aggregates) with waste products has been encouraged by the construction industry to promote sustainability (Thomas et al., 2013, 2015; Abdul and Hossein, 2016). The accumulation of stone wastes has also been increasing throughout the year and the land requirements to dump these wastes becomes a significant burden for civil and environmental engineers. With the increase in output and wastage of natural stones like sandstones, the best solution lies in utilising them in cement concrete as a part replacement for aggregates.

1.2 Problem Formulation

Although there are some literature focussing on the types of sandstone and their significant effect on concrete's strength, there is very limited research focussing on durability properties, thermal effects of concretes containing quartz sandstone aggregates. Many researchers have studied the mechanical properties and density alone. A proper study is needed on the durability characteristics, like; resistance to acid attack, thermal effects, corrosion of steel reinforcements, etc. The author prefers to take the Dholpur sandstone (a quartz sandstone) for a thorough study on its utilisation. This study using quartz sandstone (Dholpur sandstone) in concrete was further required on the aspects of strength and durability properties.

In this study, M30 grade of concrete was designed and examined in the first series with a water-cement ratio of 0.40. Quartz sandstone (Dholpur stone) was replaced as a substitute for coarse aggregates varying from 0% to 100% (at 20% interval). The properties of concrete, like; compressive strength, flexural tensile strength, abrasion resistance, pull-off strength, water permeability, water absorption, resistance to acid attack, resistance to sulphate attack, carbonation, depth of chloride penetration, corrosion of steel reinforcements, etc. were tested. Also, Thermogravimetric analysis (TGA), action of fire and their micro structures were analysed using Scanning Electron Microscopy (SEM). The properties of concrete with water-cement ratios of 0.35 and 0.45 were also studied as the second and third series to evaluate the disparity in different properties.

1.3 Objectives of the Work

The objectives of the work carried out are as follows:

- If the quartz sandstones (Dholpur stone) may be used a fractional/whole substitute for aggregates in concrete, the environmental pollution caused by discarding waste sandstones can be prevented to a greater extent.
- A quantum of natural coarse aggregates may be saved due to the substitution of quartz sandstones in concrete.
- This could be an effective system of reusing/disposal of the waste quartz sandstones (Dholpur stone)
- The expenditure of construction projects can be minimised by reducing the utilisation of costlier natural coarse aggregates.
- The waste accumulation that destroys the beauty of nature can be avoided to a greater extent.
- In overall, the utilisation of this waste material is beneficial to the environment and also economical in producing a sustainable concrete.

1.4 Organisation of thesis

This thesis consists of five chapters.

Chapter-1, Introduction: This chapter introduces the work carried out in the thesis. The research significance and the objectives have been described

Chapter-2, Literature Review: This chapter reviews the literature on the effective utilisation of solid waste materials including the use of sandstones in cement concrete and identifies the research gap.

Chapter-3, Materials and Methods: This chapter illustrates the description of the materials used in the making of quartz sandstone concrete. The chapter also elaborates the method of testing and the testing procedures of concrete in green and hardened state.

Chapter-4, Results and Discussions: This chapter explains the analysis of results obtained in the testing procedure and its discussions.

Chapter-5, Conclusions and Recommendations: The conclusions derived are compiled and the recommendation for future work are discussed in this chapter.

This thesis also presents abstract, acknowledgements at the beginning and references, bibliography, a brief biodata of the author at the end.

CHAPTER-2

LITERATURE REVIEW

2.1 Solid Waste Materials

The Department of Environmental Conservation, New York states that a solid waste material can be categorised to any type of garbage, refuse, sludge from any wastewater plant, and all other discarded materials of any type including solid, semisolid, liquid or gaseous materials. These waste materials can be generated from industrial, commercial, mining and agricultural operations, and also from communal activities.

Solid waste materials include- fly ash, rice husk-bark ash, bottom ash, bagasse ash, ceramic wastes, carbon steel slag, copper slag, coal waste, incineration waste, limestone waste, mine waste, marble sludge waste, construction and demolition waste, plastic waste, furnace and welding slag, sandstone waste.

Fly ash otherwise known was pulverised fuel ash is a finely divided residue from the combustion of powdered coal collected by an electrostatic precipitator. Rice husk-bark ash is obtained as a residue from burning rice husk-bark as a fuel in power plants. Similarly bagasse ash is obtained from sugarcane mills. Bottom ash is the agglomerated particles of ash formed as a by-product of coal combustion in pulverized coal furnaces. Incineration ash is produced as a result of burning municipal solid waste in an incinerator.

Fabrication industries generate wastes like welding and furnace slag. Carbon steel slag is the by-product generated during the refining of carbon steel in an arc furnace. One ton of carbon steel produces almost 10 kg of carbon steel slag. Copper is an industrial by product obtained during the matte smelting and refining of copper.

Similarly coal waste is a by-product of energy industry. Foundry slag is a by-product of metal casting process at metal foundries. These are high quality silica sands used to make moulds for casting. Ceramic waste are the non-biodegradable waste of sanitary ware like wash bowels, floor and roof tile, medical and laboratory vessels. Mine waste is generally the stone waste generated from mining activities.

2.2 Utilisation of Solid Waste Materials

Different types of solid waste materials are being utilised in the manufacture of cement concrete as a substitute for aggregates, partial replacement for cement, a filler, fibre etc. Depending on their cementitious properties, they can be either inert or reactive.

2.2.1 Reactive Materials (Pozzolanic Materials)

Pozzolanic materials are either siliceous materials or siliceous and aluminous materials which do not have any cementitious value when they exist as such. But when finely grounded, they can chemically react with calcium hydroxide in the presence of moisture to form cementitious properties (Shetty, 2005).

A simple pozzolanic reaction can be shown by:

$$\text{Pozzolan} + \text{Calcium hydroxide} + \text{Water} \longrightarrow \text{C-S-H (Gel)}$$

2.2.1.1 Fly Ash

Fly ash is one of the widely used pozzolanic material around the world. In India, almost 75 million tons of fly ash is being generated every year from the thermal power plants (Shetty, 2005). As the disposal of fly ash becomes a huge environmental concern, its efficient use in concrete is highly recommendable. It has become the most used ingredient to improve the strength and performance of cement concrete. Usage of right amounts of flyash in concrete helps in reducing the water demand for attaining the desired workability. As the quantity of water in concrete is reduced, a substantial amount of bleeding and drying shrinkage is reduced. It also helps in reducing the heat of hydration while replacing cement with flyash (Shi et al., 2008; Tan et al., 2012).

2.2.1.2 Silica Fume

Silica fume, generally known as microsilica, is a product from the result of reducing high purity quartz with coal in an electric arc furnace during the manufacture of silicon or ferrosilicon alloy (Neville, 1995). Silica fumes rises as an oxidised vapour which cools, condenses and collected. The particles are generally spherical in shape and are extremely fine being less than 1 micron with an average diameter of about

0.1 micron, which is almost 100 times smaller than that of cement particles (Shetty, 2005; Azavedo et al., 2012; Bagheri et al., 2012; Neville, 1995). Silica fumes can increase the denseness by filling up the pores of cement paste, thereby generating a stronger concrete. When compared to fly ash at room temperature, silica fumes are much more reactive in the presence of moisture. When added to concrete, it makes the mix cohesive and attains lesser slump values. Segregation and bleeding of concrete can be minimised if added in proper dosage.

2.2.1.3 Rice Husk Ash

Rice husk ash is produced by incinerating rice husk in a restrained manner. When properly burnt, it has a high amount of silicon dioxide which can be used as a concrete admixture. Rice husk ash contributes to inpermeability of concrete and thereby increases the concrete strength (Shetty, 2005). It also helps in the refinement of pore structure and also reduced the expansion, plastic shrinkage and thermal cracking. Due to the pozzolanic reaction taking place, calcium hydroxide in hydrated Portland cement paste gets consumed. This enables the concrete in improving the resistance to acid attack and chloride penetration.

2.2.1.4 Metakaolin

A refined kaolin clay that is calcined under controlled conditions to create amorphous aluminosilicate that in reactive in concrete is termed as metakaolin. Like other pozzolans, metakaolin reacts with lime, by-products of cement hydration and helps in densification of cement paste and also increases the strength and decreases the permeability of concrete (Nabil, 2006). Metakaolin reacts with calcium hydroxide and produces additional cementing compounds which holds the concrete together. Erhan et al. (2008) found the addition of metakaolin increased the strength of concrete, reduced the drying shrinkage strain, enhanced the pore structure and made the concrete more impervious.

2.2.1.5 Ground Granulated Blast Furnace Slag

Ground Granulated Blast Furnace Slag (GGBS or GGBFS) is obtained by rapidly quenching molten iron slag (a by-product of iron and steel-making) from a blast furnace in water or a stream, to form a glassy sand like granular material that is then dried and finely ground to powder. Around 7.8 million tonnes of GGBS is produced in India every year which is mainly used for the manufacture of slag cement (Elke et al., 2012; Qingtao et al., 2012; Moruf et al., 2014).

Qingtao et al. (2012) reported a mass loss of concrete with GGBFS was less than 8% than that of Ordinary Portland Cement (OPC) concrete. Carbonation depth was higher in test specimens and the effect of temperature on its compressive strength was more pronounced foe the concrete with GGBFS. Bagheri et al (2012) replaced GGBFS for cement at 15%, 30% and 50% and silica fumes replaces cement at 2.5%, 5%, 7.5% and 10%. It was reported that the silica fumes had only lesser effect in improving the strength of concrete with slag, while there was a considerable improvement noticed in the durability of concrete.

Moruf et al. (2014) noticed the pore filling ability of GGBFS in concrete which helped to enhance the compressive strength of concrete by 20%. The strength achievement was obtained in less than 3 days when compared to gradual strength achievement in normal concrete.

2.2.2 Inert Materials

There are quite a number of inert materials that are used in concrete. The commonly used inert materials in India are the stone wastes, different type of flags and recycled aggregates. However on the thesis point of view, stone waste materials are considered here.

2.2.2.1 Stone Waste Materials

With an increase in the innovation of technologies and the usage of materials, the amount of waste has been generated to a greater extent.

- The increase in constructional activities has surged the use of new materials and also has led to the disposal of existing ones.
- The large amount of resources being consumed in the present day scenario imposes unpredictable loss in biodiversity and amenities.
- Hence, the utilisation of these stone wastes in cement concrete might reduce the landfilling problems and also curtails the use of conventional natural aggregates.

There exists a variety of building stones which has been utilised as a construction material from the ancient times. Marble, granite and sandstone are some of the commonly used building stones. These building stones are generally cut into smaller blocks or pieces to get the desired shape and size. Some of the stones are cut and shaped to use as tiles, slabs and cladding material. During the process of cutting, almost 20-25% of the stone is lost as waste which is termed as stone wastes. The one which is comes as a mixture of water and stone dust is generally termed as slurry waste.

Concrete is a composite construction material composed of cement paste, aggregates, water and admixture. Aggregates are generally gravel or crushed rocks of granite, marble or sandstones. The overall objective is to make a concrete that is easy to mix, place, compact, finish and sometimes transport. The proportioning of overall quantities in a concrete mix is very important since it affects the properties of hardened concrete.

The marble, granite and sandstones industries generate both solid wastes and slurry wastes. Solid wastes from these industries include discarded stone waste from the mining activities. The solid wastes from stone industries can also be the scraps from mining and processing units. Slurry wastes include the mix of water and stone dust as a result of cutting/sawing and polishing stones.

2.2.2.2 Utilisation of Solid Stone Waste Materials

Stone is one of the most commonly occurring building material around the world. Some of the types of solid stone materials include marble, granite, sandstone, limestone and slate etc. There is an enormous increase in the demand of such stones in the construction industry.

2.2.2.3 Marble Stones Wastes in Concrete

Marble stones is an ancient construction material that is being widely used in the production of concrete. The word marble denotes a shining stone or a crystalline rock. These rocks are generally the result of metamorphism taking place in the several sedimentary carbonate rocks. The rocks thence formed are a result of various carbonate crystals locked together. There are various types of marbles that exist based on the colour and the location where it is found. The overall marble production is dominated by four countries that include Italy, China, India and Spain. Italy and China being the major producers of marble followed by India and Spain. Most of the marble stones are used as a constructional aggregate in the manufacture of cement concrete. In accordance with the United States Geological Survey, crushed marble aggregates amounts to 11.8 million tons in 2006 (https://www.usgs.gov).

At present scenario, the increased demand for natural aggregates has given rise to find an alternative source of building material. On the other hand, the marble waste production has also been greatly increasing. The wastes which are retained after processing and cutting gets clogged in the aquifers causing much more problems. So inorder to utilise the wastes effectively and also to reduce the use of conventional aggregates, marble aggregates was used in concrete by many researchers. Hebhoub et al., (2011) reported that 75% substitution of marble aggregates could be utilised confirming to concrete production standards. Another study done by Binici et al., (2008) reported that a concrete mix with 100% marble aggregate mixed with GGBS showed better compressive, flexural strengths. The utilisation of marble wastes in concrete not only contributes to an eco-friendly concrete but also improves the national economy. It was also found that the use of marble dust at 40%, saves 4 US$ while used for paving block production (Rai et al., 2011; Gencel et al., 2012).

Sardinha et al., (2016) conducted studies on durability properties of concrete containing very fine marble sludge. He noticed that the durability characteristics worsened as the water-cement ratio and percentage of marble sludge increased. The optimum dosage of marble sludge was witnessed to be 5% and 10%. The fresh concrete properties greatly depended on super plasticisers. Ulubeyli et al., (2016) noticed an improvement in the durability properties of normal concrete and self-compacting concrete. The water absorption characteristics, resistance to sulphate attack and chloride ingression was found to be improved when marble aggregates were used in cement concrete. Sadek et al., (2016) conducted a study in utilisation of marble slurry in self-compacting concrete. The results indicated that high volumes of fly ash of about 50% by weight of cement content (400 kg/m^3) can be utilised successfully in cement concrete. The authors also suggested to use silica fume to enhance hardened concrete property. Similar research was done by Tennich et al., (2015) utilising marble and tile waste as mineral additive in self-compacting concrete. The fresh concrete was tested against tests such as V-Funnel test, flow test, L-Box tests. The hardened concrete was tested for compression and tensile strength test along with Ultra sonic pulse velocity testing. The output of research clearly showed a sufficient result in the utilisation of marble slurry as mineral additives.

Aliabdo et al., (2014) studied the reuse of marble dust for the production of cement and concrete. The test results indicated that the marble dust blended with cements upto 15% replacement by weight remains as an acceptable limit. The results also showed that the marble dust replaced as fine aggregate in concrete production, enhances the physical and mechanical properties. Due to its filler effect and also inactive role in the hydration process, the marble dust is found to be a sustainable material to be used in cement and concrete production. Similar research was carried out by Talah et al., (2015) on utilising marble powder on high performance concrete. The studies indicated a positive mechanical characteristics and also beneficial resistance to chloride migration.

2.2.2.4 Granite Stone Waste in Concrete

India is a country which is endowed with almost 20% of overall granite deposits around the world. There has been a continuous lack of space for landfill and also degradation to environment due to the granite waste generation (Lokeshwari and Jagadish 2016). Granite fines which are produced during the process of cutting are carried by water and stored in tanks. After evaporation of water, the sludge is disposed in an inefficient manner (Arshad et al., 2014). The important motive of a waste management system is to utilise the waste in most efficient way by protecting the environment and also by doing it in a more economical way. The industries of granite are very much similar to that of other stone industries that generates large amount of stone wastes inform of powder while cutting and sawing (Dhanapandian et al., 2009). Thus a sustainable use way of utilising these wastes are by utilising them in the manufacture of cement concrete.

Li et al., (2016) studied on the utilisation of granite dust in manufactured sand concrete. The results indicated that the workability of fresh concrete containing manufactured sand was improved by adding granite dust. Hamza et al., (2011) utilised granite slurry waste as a fine aggregate replacement for the production of bricks. The test results revealed that the recycled products such as granite slurry had physical and mechanical properties that qualifies for the manufacture of bricks. Soltan et al., (2016) studied the possibility on using granite waste in the production of light weight aggregate. The results indicated that the viscosity of liquid phase in the aggregates was governed by the presence of silica and alumina when firing. It was found that the manufactured light weight aggregates with granite waste can be used as thermal insulators after firing at 1200°C.

Bacarji et al., (2013) studied on the sustainability perspective on granite waste as concrete fillers. Granite waste being an inactive material in the pozzolanic activity can be used as a filler material in the production of cement concrete. The results indicated that the 5% replacement level showed no considerable change in the properties of fresh concrete as well as in mechanical properties. Singh et al (2016) worked on the use of granite powder as a fine aggregate replacement in concrete. The adopted replacement levels were 10%, 20%, 30% and 50%. The slump values of granite added concrete produced a lesser slump with a lesser workability due to the angular shaped rough aggregates. Similar fines replacement was done by Raghavendra et al., (2015). The granite fines was noticed to have a satisfactory workability when compared to standard concrete mix upto 25% replacement.

Vijayalakshmi et al., (2013) studied the changes in slump values as the granite fines were added in concrete. A maximum replacement of 25% was adopted and the concrete was casted with fixed water-cement ratios. However a decreased workability was observed by the researcher, some of the authors have established a contrary data. One such contrary result was published by Adigun (2013) while using crushed granite dust as a replacement for natural fine aggregate. For a fixed water-cement ratios of 0.42 and 0.45, an increase in slump values was observed as the granite fines were increased in the mixes. Similar increase in workability was observed by Felixkala and Sethuraman (2013) as the granite waste was used as a sand replacement in cement concrete.

On reviewing the strength of products having granite wastes, Olaniyan et al. (2012) used granite fines as fine aggregate replacement in the manufacture of sandcrete blocks. The sandcrete blocks were manufactured with replacements of granite at 0% to 100%. A maximum compressive strength by sandcrete block with 15%

granite fines was observed by the authors. Chiranjeevi Reddy et al. (2015) utilised granite fines as natural fine aggregate replacement in the production of concrete. The replacement levels were adopted to be 2.5%, 5%, 7.5% and 10%. They observed a maximum compressive strength at 7.5% replacement level of fine aggregates with granite fines. The maximum replacement of 10% showed a comparable result of mechanical properties with that of control mix.

Divakar et al., (2012) investigated the effect of granite dust as fine aggregate replacement in concrete with replacements varying from 0% to 50%. They observed a considerable increase of flexural strength upto 5.41% at 5% replacement level. However at other replacement levels, a significant decrease in the flexural strength was observed. Ramos et al., (2013) used granite dust and superfine granite dust in cement mortars. The replacements were done for cement at 5%, 10% and the split tensile strength of the mortar was observed. A continuous decrease in split tensile strength was observed as the superfine granite dust was added in mortar.

2.2.2.5 Sandstone Waste in Concrete

Concrete is widely used as a construction material in modern society. With the growth in urbanisation and industrialisation, the demand for concrete is increasing. Therefore, raw materials and natural resources are required in large quantities for concrete production worldwide. At the same time, a considerable quantity of industrial waste, agricultural waste and other types of solid material disposal are creating environmental issues (Aprianti et al., 2016). So it becomes necessary to find an alternative source for raw materials and to lessen the wastes being dumped (Brito & Amorim, 2009) (Aggarwal et al., 2014).

While utilising aggregate of different sizes, proper grading and its effect on strength should be studied to find out the exact ratio in which they can be mixed together. Van Vliet and Van Mier (2000) studied the size effect of strength and fracture energy of concrete and sandstone. It has been recognised for ages that the size effect can have a significant effect on the nominal strength. Thus sandstone of different sizes used in concrete would have a varying effect on its corresponding strength and further it is important to grade these aggregates when used in concrete (S. Kumar et al., 2016).

The term sandstone denotes a rock formed by grains which are sand-sized. Quartz sandstones have a framework of about 90% quartz and with the limited amount of some other grains like feldspars and lithic fragments. Sandstones are sedimentary type of rock which are composed of rock grains and silt sized particles. They are of different types based on the geological property, elemental framework and most of the sandstones has quartz and feldspar due to abundance of them in earth's crust. Being a widespread aggregate resource, sandstones are widely used in concrete construction around the world (Mackechnie, 2006). The geological properties of sedimentary rocks are fairly diverse such as quartzite, arkose, subarkose and greywacke aggregate that may produce a range of hardened concrete properties. Therefore, it is important that the aggregate can be easily characterized to obtain predictable concrete properties (Yilmaz and Tugrul, 2012).

The factors generally influencing the strength of concrete are the amount and type of cement, w/c ratio, aggregate type and grading, workability of fresh concrete, mineral admixtures used, chemical additives, curing conditions and time, etc. (Kilic et al., 2008). Previous studies have showed the importance of aggregate packing and grading to enhance the performance of concrete (Sobolev, 2004; Fuller and Thompson, 1906; David, 2007; Sobolev and Amirjanov, 2010). Changes in aggregate proportions and its influence on concrete properties such as strength and modulus of elasticity was studied by several researchers (Abrams, 1918; Gilkey, 1961; Neville, 2000; Thierry and Larrard, 1994; Vorobiev 1977; Larrard, 1990; Larrard and Belloc, 1997; Hansen, 1965; Shilstone, 1990; Newton, 2005; Sobolev and Amirjanov, 2008; Fennis, 2011). Similarly when sandstone used in concrete, compressive strength of resulting concrete might vary and it is being accepted as the most important mechanical property of structural concrete.

Since sandstones are porous materials, it can absorb more moisture when compared to conventional coarse aggregates. Influence of moisture is well known to decrease the mechanical property of construction material like sandstones (Marques et al., 2012; Adriaens et al., 2014). There are different types of sandstones and they contribute differently towards the strength of concrete. Clay content in sandstone approximately reduces the compressive strength of concrete to about 40-50% and the presence of carbonate in sandstones have a better bonding between cement and aggregate than those containing clay particles (Yilmaz and Tugrul, 2012). Quartz sandstones are those which have a lesser quantity of clay and it approximates to about 2.7g/kg as measured by methylene blue test. Specification BS EN 12620:2002+A1:2008, limits the fines content in recycled aggregate at 3% and in recycled concrete aggregate at 5%, since the fines content influences the water absorption property and which in turn affects the drying shrinkage of concrete (Kumar et al., 2016).

The rapid advancement of construction sector, in combination with economic growth, consequently indirectly requires considerably high amount of production and consumption of construction minerals, such as rock materials (aggregate) and sand (Ismail et al., 2013; Madurwar et al., 2013). On the other hand, the mining and quarry industry, should guarantee the adequate and continuous supply of raw materials as a producer of construction minerals to the construction sector to sustain the economic development of the country. If there occurs a shortage in these resources, leads to increased construction cost and which in turn affects the overall national development on infrastructure (Hamid et al., 2006). In countries like India, sandstones are mined to a greater extent leading to large amount of landfilling. Thus the use of sedimentary rock like sandstones can reduce the cost of construction materials, reduce the amount of sandstone wastes that need to be land filled and also reduce the transportation and energy cost which is needed to import virgin natural aggregates thereby reducing the use of virgin aggregates and conserve natural resources (Han and Thakur, 2014).

2.3 Research Gaps Identified

From the literature studies the following observations were made

1. Most of the studies focussed on mechanical properties of sandstone. Proper studies on the durability characteristics were not identified.
2. Gradation study of using natural aggregate and sedimentary aggregate was not observed in the literature studies.
3. The studies on sandstones were mostly outside India. There would be differences in quality of sandstone and all other constituent materials.
4. Studies on abrasion, thermal effects and acid attack resistance of quartz sandstones were not observed in the literature study.
5. Considerable use of sedimentary aggregate in concrete were found to be very less in the literature study.

CHAPTER- 3

MATERIALS AND METHODS

3.1 Introduction

In this chapter, the properties of materials used and the experimental programme along with the methodologies are elaborated. The methodologies of mechanical and durability properties of cement concrete containing Dholpur Aggregates (quartz sandstones) as a part replacement for natural coarse aggregates have been reported. Trial mixes and preliminary study was done to ensure the workability, compressive strength and the dosages of plasticiser with different quantities of Dholpur aggregates to be added in the main casting. Based on the test results, the proportions of constituent materials used in the main casting were decided and modified accordingly. Three series of concrete mixes were designed as per IS 10262: 2009. In the first series, M30 concrete grade with a water-cement ratio of 0.40 was fixed for a maximum exposure condition according to IS 10262:2009 by varying the quantity of Dholpur Aggregate and labelled as series 1.

There is always a greater interest in research towards the effect of water to cement ratios on certain property changes like strength, shrinkage and cracking potential of internally cured concrete (Zhutovsky & Kovler, 2017). Hence, in the next two series, water-cement ratios of 0.35 and 0.45 were adopted. The mechanical and chemical properties of these materials are presented here and the methodology of tests on concrete, like; resistance to compression and flexure, pull off strength, resistance to abrasion, water permeability, carbonation, acid and sulphate resistance, corrosion etc. are presented in this Chapter.

For attaining the goal of concrete containing quartz sandstones, the physical properties of materials viz. cement, fine aggregates, coarse aggregates and Dholpur aggregates (quartz sandstones) were determined as per the procedures of Indian Standard Codes. Control mix containing solely natural aggregates and the various other concrete mixes with different dosages of Dholpur aggregates were prepared and tested for fresh and hardened concrete properties.

3.2 Material Investigations

The physical and mechanical properties of cement, fine aggregates, coarse aggregates and the Dholpur sandstones (quartz sandstone) were determined. The physical properties include specific gravity, fineness of aggregates, water absorption and the mechanical properties like compressive strength of cement was carried out according to the Indian Standards. The constituent materials used in the production of cement concrete are:

(a) Cement: Ordinary Portland cement of 43 grade was obtained from the local market (Commercial name: Binani). The procured cement confirms to IS 8112: 2013.
(b) Fine Aggregate: Natural river sand confirming to IS 383: 1970 was used. (Obtained from river Banas; Zone II)
(c) Coarse Aggregate: Aggregates of sizes 20 mm and 10mm were procured locally. The natural coarse aggregates confirms to IS 383: 1970.
(d) Dholpur Aggregates (Quartz sandstones): Dholpur aggregates of sizes not greater than 25mm and 10mm were used. The aggregates were obtained from a mining region in the Dholpur district of Rajasthan state in Northern India.
(e) Water: Potable water available in the Institute was used.
(f) Super Plasticiser: Gilenium Sky 8777 (BASF manufactured)

3.2.1 Properties of Cement

The properties of cement such as normal consistency, soundness, initial and final setting time, fineness and compressive strength (3, 7, 28 days) were tested as per Indian Standards IS 8112: 1989 and IS 4031: 1996.

The Vicat apparatus was used to measure the normal consistency of cement. Normal consistency denotes the consistency in which the plunger of Vicat apparatus is made to penetrate a depth of 5-7 mm from the bottom the mould. At first, cement paste is prepared using with the weighed quantities of cement and water. Gauging was done between 3-5 minutes and the cement paste was placed in the mould. The plunger of the Vicat apparatus was released and allowed to sink into the cement paste. With varying the percentages of water, trail mixes were done to attain the normal consistency of the cement paste. The depth of penetration was noted down.

To determine the soundness of cement Le-Chateliar's apparatus was used. The measured quantities of cement and water were mixed properly (0.78 times the water required for normal consistency) to prepare the cement paste. The prepared paste was placed in the mould and the glass pieces were used to cover the both sides of the mould. The mould was then placed in water maintained at 27 ° ± 2 °C after 30 minutes. The sample was kept in water for the next 24 hours. The distance between the indicators were measured accurately and the whole assembly was kept in boiled water for 30 minutes. The distance between the two indicators was again measured accurately. The difference between the initial and final reading gives the soundness of cement.

To measure the initial and final setting time of cement, the cement paste was prepared according to Indian standards by mixing 0.85 times the water required for the normal consistency. The cement paste was placed in the Vicat mould and the plunger was released to measure the depth of penetration. The period elapsed between the time water was added to the cement and the time at which needle fails to pierce the cement paste at 5.0 ± 0.5 mm from the bottom of mould was reported as the initial setting time of cement. The annular attachment needle was used to determine the final setting time. The period elapsed between the time water was added to the cement and the time at which needle makes an impression on the surface of mould was reported as the final setting time.

To determine the fineness of cement, 100 grams of cement was sieved over 90 micron sieve. The residue left over the sieve was weighed. The ratio of the residue left over to the initial measured cement is expressed as percentage. The ratio expressed should not be more than 10%.

In order to determine the compressive strength of cement, a total of 9 mortar cubes with an area of 50 cm^2 was prepared by mixing cement and the standard sand in the ratio of 1:3. The quantity of water taken accounts to (P/4+3) % of the combined weight of sand and cement. P denotes the percentage of water required for normal consistency. The mould was then filled in 2 layers with each layer tamped 20 times. The mould was then vibrated for 2 minutes and kept in room temperature for 24 hours. After demoulding the specimens, they were tested for resistance to compression at 3, 7 and 28 days.

The properties of cement are shown in Table 3.1 and Table 3.2

Table 3.1: Physical properties of cement

S. No.	Requirements	Test results (Lab)	As per Test Certificate Binani*	Requirements as per IS-8112-1989	
1	Consistency	32%	30-35%	-	
2	Fineness	1%	292 m^2/kg	10%	225 (minimum)
3	Setting Time				
	Initial	80 min.	60 -100	30 (Minimum)	
	Final	164 min.	160-200	600 (Maximum)	
4	Compressive Strength				
	3 days	25		23 (Minimum)	
	7 days	36		33 (Minimum)	
	28 days	45		43 (Minimum)	
5	Specific Gravity	3.15		-	

* Test certificate (No. R/15-21, dated 11-06-2013) issued by Binani Cements Limited, India.

Table 3.2: Chemical composition of cement

S. No.	Requirements	Test results	Requirements as per IS-8112-1989
1	Lime saturation factor	0.96	0.66-1.02
2	Ratio of alumina to that of iron oxide	1.70	0.66 (Minimum)
3	Insoluble residue (% by mass)	2.69	3.0 (Maximum)
4	Magnesia (% by mass)	1.77	6.0 (Maximum)
5	Sulphuric anhydride (% by mass)	2.25	3 (Maximum)
6	Total loss on ignition (%)	2.66	5 (Maximum)
7	Total chlorides (%)	0.020	0.10 (Maximum)

[a] Test certificate (No. R/15-21, dated 11-06-2013) issued by Binani Cements Limited, India.

3.2.2 Properties of Fine Aggregate

The river sand procured from river Banas was used as the fine aggregates for the production of cement concrete. The fine aggregates were tested for fineness, water absorption and specific gravity as per the parts of Indian Standard code IS 2386: 1963.

As the fine aggregate particles consists of particles of various sizes, their size distribution should be analysed to understand their suitability in making concrete. For this purpose, sieve analysis was carried out. The Indian standard set of sieve sizes were adopted i.e. 80 mm, 40 mm, 20 mm, 10 mm, 4.75 mm, 2.36 mm, 1.18 mm, 600 micron, 300 micron and 150 micron. A pan was kept at the bottom to collect the finer particles that pass through all the standard sieve sizes. A kilogram of oven dried sand was taken randomly and was placed in top of sieve after cooling at room temperature for 24 hours. The set of sieves was then closed and placed in a sieve shaker. After shaking, the particles in sieve was collected and the percentage passing through each sieves were calculated.

A pycnometer was used to find out the specific gravity of fine aggregates. The empty weight of pycnometer was taken as W_1. The fine aggregates were oven dried at 60°± 5 °C for 3 days (72 hours) and then cooled at room temperature for 24 hours. The pycnometer was then filled to about one-third with fine aggregate and the weight of pycnometer along with aggregate was taken as W_2. The pycnometer was the half filled with water and stirred vigorously to remove the entrapped air. The pycnometer was then completely filled with water and the weight of pycnometer and sand along with the water was taken as W_3. The water and sand was removed from the pycnometer and refilled with water alone. This weight was taken as W_4. The specific gravity was calculated as:

$$\text{Specific Gravity (G)} = \frac{W_2 - W_1}{(W_2 - W_1) - (W_3 - W_4)}$$

Inorder to determine the water absorption of fine aggregates, the sample was washed properly to remove the dirt. Further it was then dipped in distilled water, where the temperature was maintained to be at 27 ° ± 2 °C for 24 ± ½ hours. After the end of soaking period, the aggregates were taken out and surface dried for 10 minutes. The initial weight W_1 was noted down. Then the aggregates were placed in a tray and oven dried at a temperature of 100°± 5°C for 3 days. After removing it from the oven, the aggregate was cooled at room temperature and the weight W_2 was taken. The water absorption of the aggregate was then calculated as:

$$\text{Water Absorption} = 100 \times \frac{W_1 - W_2}{W_2}$$

The results of various tests conducted n fine aggregates are given below in Table 3.3

Table 3.3: Physical properties of Fine Aggregate

S. No.	Name of the test		Fine Aggregate
1	Sieve Analysis	Sieve size	% Finer
		80 mm	100.00
		40 mm	100.00
		20 mm	100.00
		10 mm	100.00
		4.75 mm	094.45
		2.36 mm	088.40
		1.18 mm	073.10
		600 micron	042.54
		300 micron	011.84
		150 micron	001.36
2	Specific Gravity		2.63
3	Water Absorption		1.5 %
4	Fineness Modulus		2.88

3.2.3 Properties of Coarse Aggregate

The natural coarse aggregates having two different sizes (20 mm and 10 mm) were used. The physical properties like specific gravity, sieve analysis and water absorption were determined separately for 20 mm and 10 mm as per different parts of IS 2386: 1963.

The procedure of sieve analysis was done similar to that of fine aggregates. A total of 5 kg of oven dried and cooled sample at room temperature was sieved in the

Indian standard sieve sizes. The weight retained on each sieve was measured individually to calculate the percentage of material passing through each sieve. The physical properties such as water absorption and specific gravity was done similar to that fine aggregate testing. The physical properties of coarse aggregate are given in Table 3.4.

Table 3.4: Physical properties of Coarse Aggregate

S. No.	Name of the test		Coarse Aggregate	
			20 mm	10 mm
1	Sieve Analysis	Sieve size	% Finer	
		80 mm	100.00	100.00
		40 mm	100.00	100.00
		20 mm	67.02	100.00
		10 mm	0.70	95.5
		4.75 mm	0.12	28.8
		2.36 mm	0.00	0.00
		1.18 mm	0.00	0.00
		600 micron	0.00	0.00
		300 micron	0.00	0.00
		150 micron	0.00	0.00
2	Specific Gravity		2.67	2.56
3	Water Absorption (%)		0.24	0.26
4	Fineness Modulus		7.24	5.58

3.2.4 Properties of Dholpur stone (Quartz sandstone)

The test methods adopted to determine the physical properties of Dholpur stone is similar to that methods used for natural coarse aggregates. Two different sizes of Dholpur aggregates were adopted for the study (25 mm and 10 mm). Images of Dholpur stone are given in Figure 3.1.The sieve analysis of Dholpur aggregates are given in Table 3.5.

Fig 3.1: Dholpur aggregate (Quartz sandstone) aggregates of sizes 25 mm and 10mm.

Table 3.5: Sieve Analysis of Dholpur Coarse Aggregate

Name of the test		Coarse Aggregate	
		25 mm	10 mm
Sieve Analysis	Sieve size	% Finer	
	80 mm	100.00	100.00
	40 mm	100.00	100.00
	20 mm	72.70	100.00
	10 mm	1.90	98.45
	4.75 mm	0.10	7.88
	2.36 mm	0.00	0.00
	1.18 mm	0.00	0.00
	600 micron	0.00	0.00
	300 micron	0.00	0.00
	150 micron	0.00	0.00

The Dholpur aggregates were procured from a mining site at Dholpur, Rajasthan, India. The aggregates were tested for physical properties like specific gravity, water absorption, modulus of rupture, compressive strength and abrasion. The properties of Dholpur stones tested are given in Table 3.6.

Table 3.6: Properties of Dholpur aggregates

S.No.	Technical Information	Sample condition		Value	Standards used
1	Water Absorption, % by weight	-		1.36	IS 2386-(Part III)-1963
2	Specific Gravity	-		2.43	IS 2386-(Part III)-1963
3	Modulus of rupture (MPa)	dry	parallel to rift	16	ASTM C-99
			perpendicular to rift	9	
		wet	parallel to rift	13	
			perpendicular to rift	8	
4	Compressive Strength (Mpa)	dry	parallel to rift	115	ASTM C-170
			perpendicular to rift	108	
		wet	parallel to rift	106	
			perpendicular to rift	91	
5	Abrasion	Average wear, mm	-	1.89	IS 1237

Dholpur aggregates (quartz sandstone) crystal structure was further studied using X-ray diffraction in which X-Rays are produced by a cathode ray tube and are refined to produce monochromatic radiation which is directed towards the sample. When there is an interaction between the sample and incident rays it leads to a constructive interference while satisfying Bragg's law. X-Ray diffraction was done to characterise the crystalline minerals in the quartz sandstone and further to crystal structure was studied using Rietveld refinement. Rietveld curve fitting refers to an iteration method whereby the X-ray diffraction pattern is attempted to be recreated by compiling it from the peaks of the constituent phases at varied proportions (higher content – higher peaks) and finding the most likely composition by best fit.

X-Ray analysis confirmed the presence of small quantity of clay in form of muscovite and feldspar (microcline) and the major part being quartz. Table 3.7 shows the concentration of elements and the figure 3.2 shows the Rietveld curve fitting.

Table 3.7: Element concentration in percentage from X-Ray diffraction

S. No.	Chemical formula	Chemical name	Concentration %
1	SiO_2	Silica	81.045
2	Al_2O_3	Aluminium Oxide	8.225
3	K_2O	Potassium Oxide	5.592
4	MgO	Magnesium Oxide	0.523
5	TiO_2	Titanium Oxide	0.442
6	Fe_2O_3	Iron (III) Oxide	0.388
7	CaO	Calcium Oxide	0.273
8	Na_2O	Sodium Oxide	0.220
9	SO_3	Sulphur trioxide	0.072
10	Ba	Barium	0.055
11	Zr	Zirconium	0.055
12	P_2O_5	Phosphorus pentoxide	0.048
13	Ce	Cerium	0.030
14	Cr	Chromium	0.029
15	Rb	Rubidium	0.013
16	Cl	Chloride	0.012
17	Sr	Strontium	0.009
18	MnO	Manganese (II) oxide	0.006
19	Cu	Copper	0.003
20	Pb	Lead	0.003
21	Ni	Nickel	0.002
22	Zn	Zinc	0.002
23	Y	Yttrium	0.002
24	Th	Thorium	0.002
25	Nb	Niobium	0.001
26	Ga	Gallium	0.001

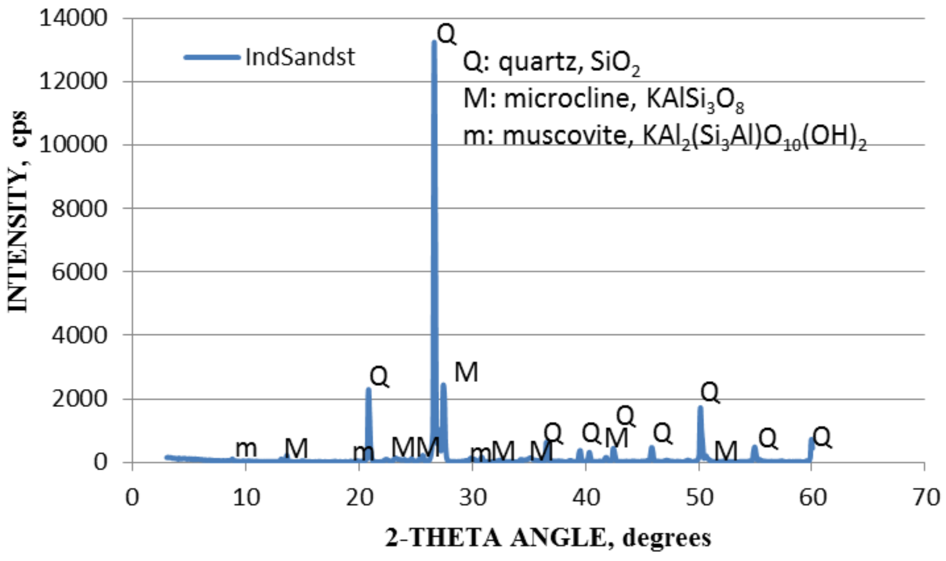

Figure 3.2: Rietveld Curve fitting showing quartz and small quantities of clay

Particles less than 2 µm in diameter may have a determinable effect on concrete strength and the presence of clay in aggregate could reduce the compressive strength of concrete up to 40–50%. In order to determine the amount of clay in sandstone wastes methylene blue test was done in accordance to BS EN 933-9:1999. Methylene blue is a heterocyclic aromatic chemical compound and at room temperature it appears solid, odourless, dark green powder form. During the test, methylene blue dye is added in small increments to the continuously stirred sample (water slurry) and a drop of slurry is placed on filter paper at a time. Adding more dye and sampling the slurry is repeated until no more dye can be absorbed by the sample and hence there is a blue halo is seen around the dot (Fig. 3.3). MB value for the sandstone wastes was found to be 2.7 g/kg. Specification BS EN 12620:2002+A1:2008, limits the fines content in recycled aggregate at 3% and in recycled concrete aggregate at 5%, since the fines content influences the water absorption property and which in turn influences the drying shrinkage of concrete. This latter is limited at 0.075% in all of the above standards and indirectly puts a control on the fines content.

Figure 3.3: Figure showing methylene blue test

3.2.4.1 Sampling and Testing for SEM

A preliminary study was also done on the powdered samples using scanning electron microscope to know the compatibility of quartz sandstone for the partial replacement of natural aggregates. The samples were thoroughly degreased and dried to eliminate any outgassing from organic contamination and water. The accelerated electrons having large amount of kinetic energy is made to dissipate into variety of signals by electron-sample interactions. Secondary electron produces SEM images that are used to study the crystal structure and mineral orientations. These electrons are important as it shows morphology and topography of samples. Non-destructive tests like SEM serves as an important study for analysing the use of sandstones in concrete since there is no volume loss in material and the same sample can be used repetitively. S-4700 Field Emission Scanning Electron Microscope (FE-SEM) was used to image the samples.

3.2.4.2 SEM Analysis

It was evident from the images that the sandstone particles have a dense structure and presumably low porosity/low water absorption, which is beneficial from technical point of view (i.e. the water demand would be low). On the other hand, there are fairly sharp cleavages and the smaller particles show some elongation, which adversely affect the rheology/flow characteristics of fresh concrete and can be mitigated by increasing the w/c ratio or applying super plasticizers. The two effects (low water absorption and angular shape) compensate each other, so all in all, based on its morphology, this material appears to be suitable for concreting. The SEM images of natural aggregate and quartz sandstone in shown in Fig. 3.4.

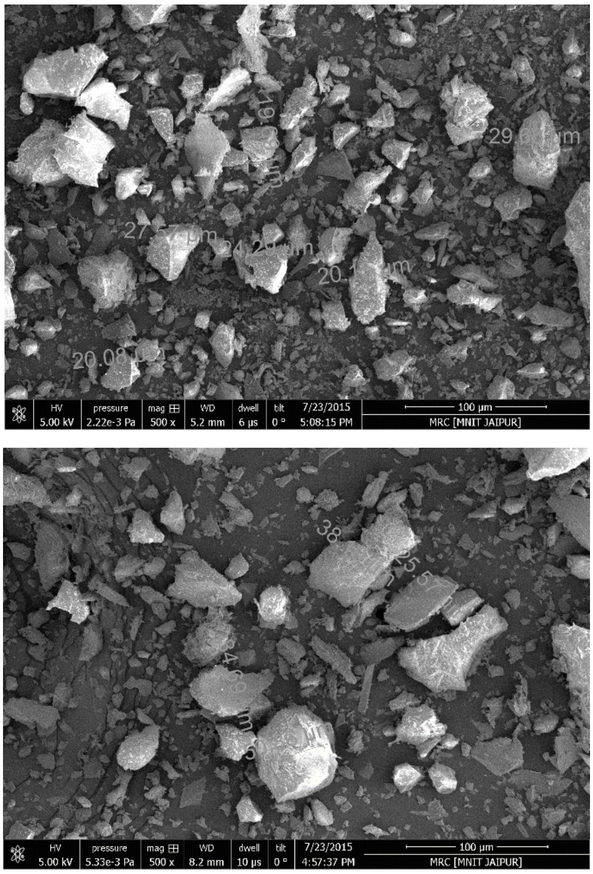

Figure 3.4: SEM images of Natural (top) and Dholpur coarse aggregates (bottom).

3.2.5 Super Plasticiser

A second generation poly carboxylic ether based superplasticiser was used. Master Glenium Sky 8777 free from chloride, low alkali and compatible with all types of cement. This super plasticiser has been primarily developed for applications in high performance ready-mix concrete to facilitate total performance control**.**

3.3 Testing of Concrete in Fresh Stage

Fresh concrete stage is the stage where the concrete can be moulded to any shape possible. This stage is generally stated as plastic stage. One important factor that determines the strength of concrete is compaction of fresh concrete. Sufficient workability will improve compaction of concrete mixes. Workability of concrete determines the ease at which concrete can be mixed, placed and compacted. There are many such methods like slump test, compaction factor etc., to determine the workability of fresh concrete. Compaction factor test is mostly used in this research work since it is a more rational method than the slump test and is suitable for dry mixes with low slump. Compaction factor is the ratio of partially compacted concrete to that fully compacted concrete.

3.3.1 Workability of Concrete by Compaction Factor Test

The compaction factor test was used to determine the workability of fresh concrete. The compaction factor test was preferred over slump test due to its accuracy and its applications for low water-cement ratios.

Figure 3.5: Concrete mixer and compaction factor apparatus

The compaction factor apparatus shown in Figure 3.5 consists of two hopper vessels A and B with a hinged bottom and a bottom cylinder, C. After the removal of dirt, cleaning all the vessels and oiling, the vessel A was filled with fresh concrete sample. The hinged door at the bottom of vessel A is opened so that the concrete falls into vessel B. The hinge door of vessel B is opened to make the fresh concrete fall into bottom cylinder C. The surplus concrete in the cylinder was struck off with a steel float. The weight of concrete W_1 was calculated after subtracting the cylinder weight. This cylinder was then vibrated to attain full compaction and the extra concrete was added to fill. Now the weight W_2 of fully compacted concrete is taken by subtracting the weight of cylinder. Compacting factor is calculated by W_1/W_2.

The reason for using Compaction factor test is for more sensitive and precise measurements of workability. It also helps to indicate small variations in workability over a wide range (http://iitk.ac.in/ce/materials/4.html.

3.4 Testing of Hardened Concrete

Inorder to study the effects of Dholpur stones (quartz sandstones) as a part replacement for coarse aggregates in cement concrete, properties like resistance to compression and flexure, pull-off strength, oven dry density, water absorption and abrasion resistance were measured as per the standards given in various codes.

The tests to determine the compressive strength and flexural tensile strength were performed after 7, 28, 90 days of water curing. The tests for water absorption, dry density, pull-off strength and abrasion resistance tests were performed after 28 days of water curing. A minimum of three samples were taken to determine the average readings of the various tests performed. In accordance with IS 516-1959, specimens stored in water should be tested immediately on removal from water. In our research, the specimens were allowed to dry for 15-20 minutes before testing.

3.4.1 Bulk Density Test

The bulk densities of concrete samples were determined as per IS 6441:1972 (Part-I) from 100 mm concrete cubes specimens. The samples cured for 28 days and oven dried at 60° ± 5 °C for 3 days were used for this test. The specimens were cooled at room temperature and the weight was taken immediately. The weight divided by the volume of cubes gives the bulk density of the concrete.

3.4.2 Compressive Strength Test

To test the compressive strength, concrete cube specimens of size 100 mm were cast while percentages of Dholpur stone (Quartz sandstone) (0% to 100% in the multiples of 20%) and varying water-cement ratios (0.35, 0.40 and 0.45). The concrete specimens were then demoulded after 24 hours of casting and stored in a curing tank with fresh water maintained at a temperature of 27°± 2°C. The specimens were then tested for compressive strength at 7, 28 and 90 days of curing. The average of three specimens were taken as the compressive strength of concrete sample.

Figure 3.6: Compression Testing Machine

The cube specimens were then tested using the compression testing machine and the specimens were subjected to load on both the sides. The load without shock was then applied gradually at the rate of 140 kg/sq cm/min until the specimen breaks gradually. The maximum load was recorded and the compressive strength was calculated by dividing the maximum load at failure divided by the cross sectional area of the specimen. The compressive strength testing machine is shown in Fig. 3.6.

3.4.3 Flexural Tensile Strength Test

Concrete beam specimens of size 100mm × 100mm × 500mm were casted with varying percentages of Dholpur sandstones and with varying water-cement ratios.

The specimens after casting were demoulded after 24 hour and kept for curing in clean fresh water. The temperature of the water was maintained at 27°°± 2°C. The beam specimens were tested on a flexural testing apparatus for 7, 28 and 90 days as per IS 516:1959. Inorder to obtain average value, three specimens were tested from each sample.

Figure 3.7: Flexural Tensile Strength Testing Machine

The concrete beam specimens were placed in the flexural tensile strength testing machine (as shown in Figure 3.7). The axis of specimen was carefully aligned with the axis of loading device. The machine arrangement had a four point loading and the rate of loading was kept at 180 kg/minute. The load was gradually increased and the maximum load at which the specimen fails are recorded.

3.4.4 Abrasion Resistance Test

Inorder to measure the resistance to wear, abrasion test was carried out. The test was performed according to IS 1237:1980 on a 28 days cured 100mm sample (samples oven dried at 60°± 5°C for 3 days). The weight of the specimens taken were measured closest to 0.5 grams. The grinding path of the abrasion testing apparatus was filled evenly with abrasion powder (20 grams). The abrasive powder used for the test was rounded in shape and have aluminium oxide content not less than 95% by mass; in the sieve analysis 0-15% by mass can be retained on 212 micron IS sieve, minimum 15% should be retained on 212 micron IS sieve, minimum 70% shall be retained on 180 microns IS sieve and not more than 3% shall be passed through 150 micron IS sieve.

The concrete cube specimen was fixed to the holding device of the abrasion testing machine (Figure 3.8) such that the surface to be abraded faces the disc. A specimen with 100 cm^2 surface area was subjected to a load of about 600 N. (According to code, the load shall be 300 N for the surface of 50 cm^2. As the surface was doubled, the load was also doubled). The grinding disc with abrasion powder is made to move with 30 revolutions per minute. With the help of brush, the abrasive powder is made to feed back into the grinding path. After 22 revolutions, the grinding path and the face of the concrete specimen is brushed to remove the abrasive powder. Now, the specimen is kept in the holding device after turning an angle of 90° about vertical axis in the clockwise direction. After the complete brushing of old abrasive powder, fresh ones of 20g are fed into the grinding path. This procedure is repeated 9 times and the total revolutions accounts to 220. When this procedure is over, the concrete specimen is reweighed and the average loss in thickness,‘t’ is calculated. As per the standard code, in general purpose tiles, the average maximum wear shall not exceed 3.5mm and wear on any individual specimen shall not exceed 4 mm. For heavy duty floors, a maximum of 2 mm and 2.5 mm is allowed.

The average loss in thickness, ‘t’ is calculated by the following equation

$$t = \frac{(W_1 - W_2)\ \times V_1}{(W_1 \times A)}$$

Where,

t = Average loss in thickness (mm)

W_1 = Initial mass of the specimen (grams)

W_2 = Final mass of the specimen (grams)

V_1 = Initial volume of the specimen (mm^2)

A = Surface area of the specimen (mm^2)

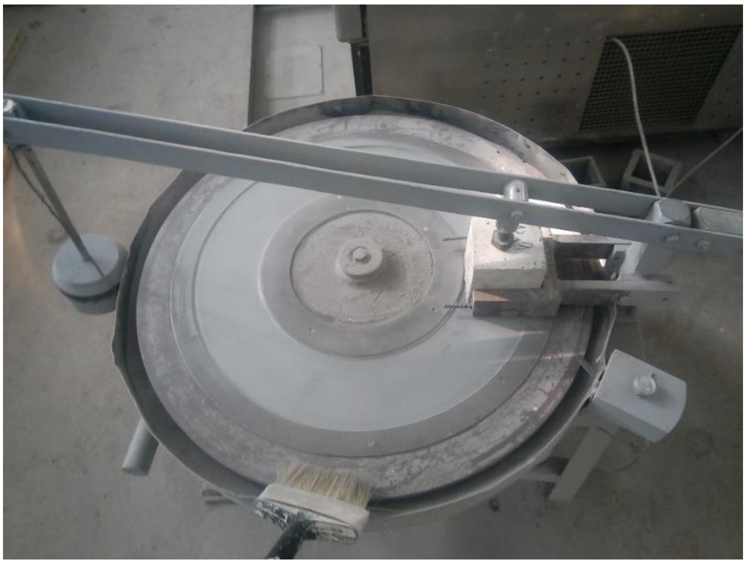

Figure 3.8: Abrasion Testing Machine

3.4.5 Pull-Off Strength Test

The tensile strength of the cover zone of the concrete specimens is termed as pull-off strength (test procedure as per BS 1881 Part 207:1992). This test was performed on concrete samples after 28 days of curing. After cleaning the surface of the specimen, discs of 50 mm diameter were perfectly bonded with the help of epoxy adhesive. The concrete samples were tested after 24 hours after pasting. The standard loading rate of about 5-10 kN/ minute was applied. Pull off strength is the force required to pull-off the disc, along with the surface area of concrete (Figures 3.9, 3.10)

Figure 3.9: Pull-off set up on the concrete samples

Figure 3.10: Procedure showing Pull-off strength testing

3.5 Durability Properties of Concrete containing Dholpur Stone (Quartz sandstone) Aggregates in Hardened State

The durability properties of concrete containing Dholpur stone was analysed by performing the water permeability test, water absorption test, acid attack test, chloride ion permeability test, carbonation test and corrosion test. All the durability tests were carried on 28 day cured concrete sample.

3.5.1 Water Absorption Test

Normal water absorption test and water absorption by capillary action were performed for the concrete samples with various replacements of Dholpur aggregates (quartz sandstones).

3.5.1.1 Normal Water Absorption Test

The test for water absorption was carried out as per ASTM C 642 (2006). The IS code (IS 15658:2006) was not used due to the fact that the specimens should be dried at a ventilated oven at a temperature of 107° ± 7°C for 24 hours. Also the procedure given by American Standards were found to be easily carried out at laboratory facilities.

The samples cured for 28 days and oven dried at 60° ± 5 °C for 3 days were used for this test. The initial weight W_1 was taken after the cubes were kept in room temperature for 24 hours. Then the cubes were immersed in water such a way that 50 mm water is maintained on the top surface of the specimen. After 48 hours, the cubes were taken out of the water and allowed to drain for a minute by placing on a dry cloth and the final weight W_2 was taken immediately. The water absorption values of concrete with Dholpur stone (Quartz sandstone) are compared with the water absorption values of the control specimens.

The measured water absorption was expressed as a percentage of the dry weight of the specimen.

$$Water\ Absorption\ = \frac{W_2\ - W_1}{W_1} \times 100$$

3.5.1.2 Water Absorption by Sorptivity

Water absorption testing was carried out according to ASTM C 1585-04. The permeability of the pore system is one of the most important factors for determining the performance of concrete in aggressive environments. The rate of ingress of water is mostly controlled by absorption due to capillary rise for an unsaturated concrete. This test method is used to determine the rate of absorption (sorptivity) of water by hydraulic cement concrete by measuring the increase in the mass of a specimen resulting from absorption of water as a function of time when only one surface of the specimen is exposed to water (Figure 3.11). The exposed surface of the specimen is immersed in water and water ingresses of unsaturated concrete predominantly by capillary suction during the initial contact with water. The only difference of sorption from permeability is the driving force for water ingress in concrete being the capillary suction rather than a pressure head. Specimens of size 100 mm × 100 mm × 100 mm, cured for 28 days and oven dried at a temperature of 65 ± 5°C for seven days, were used for testing.

Figure 3.11: Test setup for sorptivity showing wax coated sides.

3.5.2 Water Permeability as per DIN 1048

Water acts the transport medium for aggressive chemicals like chloride or sulphate ions which leads to the deterioration of concrete. One of the characteristics that influence the durability of concrete is its resistance to ingress of water and other potentially harmful substances.

The test for water permeability was carried out according to DIN 1048 (Part 5) on 28 day cured concrete sample. The specimen size of 150 mm were used for this test. The use of German code was preferred over IS code (IS 3085: 1965) for performing this test because the rate of water penetration as per IS code is very low. According to DIN 1048, the concrete specimen should be subjected to a constant water pressure of 0.5 N/mm^2 from above for 3 days acting normal to the mould-filling direction (Figure 3.11). Inorder to provide a same pressure of 0.5 N/mm^2 according to Indian Standards, a water column of 50 meters would be practically impossible.

Figure 3.12: Water Penetration Apparatus as per DIN 1048

After the 3 days of exposure to a constant water pressure, the specimen were removed from the apparatus and split open at the centre into two equal halves. After 5-10 minutes drying, the highest depth of penetration was noted down and the average of the measurements were taken as the depth of penetration (Average of 3 samples). The depth of penetration is considered to be low if it is less than 3cm. At 3-6cms it is medium penetration and high penetration above 6cm (Eshmail Ganjian et al., 2009).

3.5.3 Test for Carbonation in Concrete

Carbonation in concrete is a process by which dissolution of CO_2 in the concrete pore fluid reacts with calcium from CaOH and CSH to form $CaCO_3$. Within some duration, carbonation brings down the overall alkalinity of concrete which is required to guard the steel from being corroded. The protective layer once sacrificed, the corrosion of reinforcement takes place and the concrete cover fails (Lo and Lee, 2002; Jack et al., 2002).

The stipulations set by CPC-18, RILEM were adhered to for measuring the carbonation depth. Six concrete cubes of size 100 mm were water cured for 28 days following which they were oven dried for 48 hours. Each of these cubes were cut into four equal pieces of 50 mm width and 100 mm depth with the help of a concrete cutter given as shown in Figure 3.13. These pieces were again oven dried at 60-

70°C for 2 weeks. After that two coating of epoxy paint was applied on the 100 mm surface of the prisms and left to dry as given in 3.14. After marking, the samples were introduced to the CO_2 chamber given in Figure 3.14. Inside the chamber, the relative humidity is controlled at 50-55%, Carbon dioxide concentration kept at 5 ± 0.2%, Temperature was 27 ± 20°C. After 2, 4, 6, 8, 10, and 12 weeks of CO_2 exposure, 3 pieces from each sample were taken and tested.

Figure 3.13: Concrete Cutter

The longitudinal sides of the samples were broken into two equal halves and were sprayed with phenolphthalein as a pH indicator. Colourless areas are visualised with a pH value less than 9.2 and the non-carbonated areas with a pH value greater, turns purple red. The average depth of carbonation and carbonation coefficient was reported for each specimen.

$$Carbonation\ coefficient = \frac{Carbonation\ depth}{\sqrt{\text{Time}}\ of\ Exposure\ in\ days}$$

Fig 3.14: Carbonation chamber and Epoxy coated concrete samples

3.5.4 Chloride Ion Penetration

The silver nitrate solution spray method is a simpler and quicker method to determine the chloride ion migration depth of concrete. Specimens of 100mm size water cured for 28 days were immersed for 90 days in 4% NaCl solution. To maintain a constant concentration, the NaCl solution was replaced after 24 hours of immersion, then at 3rd day and then at every 7 days. Test specimens were taken out at every 28, 56 and 90 days from the soaking tank and were split into two halves in the middle. To determine the extent of chloride ion penetration, 0.1N Silver nitrate ($AgNO_3$) solution was sprayed on these split pieces. When the presence of free chloride ions on the concrete surface is greater than 0.15% by weight of cement, $AgNO_3$ reacts with these ions and forms a white precipitate of silver chloride (AgCl). In places where there is an absence of free chlorides, hydroxide reacts with $AgNO_3$ to form a brown precipitate of silver oxide (AgO). The depth of chloride penetration is hence measured at the boundary of colour change. The silver chloride formation (white colour) occurs only (Erhan et al., 2007 & 2009).

3.5.5 Acid Attack Test

Deterioration of concrete can take place when exposed to aggressive sulphuric acid environments. There are many durability requirements to be considered for the design of concrete in aggressive environments. One such important consideration is acid attack in concrete. Since sulphuric acid is responsible for sewage systems, piping systems and waste water treatment systems. Since sulphuric acids are strong, they can abate the pH of the concrete environment and alter the hydration components which further intensifies the deterioration. This process of deterioration of concrete when exposed sulphuric acid can be divided into reactions that occur with calcium hydroxide (CH) and calcium silicate hydrates gel (CSH) (Nnadi and Marriaga, 2013). Some of the researchers found that the sulphuric acid reacts with CH and forms gypsum which increases the volume of concrete causing cracks. The reactions between tricalcium aluminate and gypsum and formation of ettringite leads to more volume expansion causing to deterioration of concrete (Monteny et al., 2000). Sulphuric acid corrosion of concrete can be characterised by the following reactions (Bassuoni and Nehdi, 2007)

$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4 . 2H_2O$$

$$CaSiO_2 + H_2SO_4 \rightarrow CaSO_4 + Si(OH)_4 + H_2O$$

$$3CaO.Al_2O_3.12H_2O + 3(CaSO_4.2H_2O) + 14H_2O \rightarrow 3CaO.Al_2.3\,CaSO_4.3H_2O$$

Gypsum ($CaSO_4 . 2H_2O$) undergoes volume expansion in concrete which induces stresses of tensile nature that causes cracking and spalling. Gypsum so formed now reacts with calcium aluminate and forms ettringite which leads to further increase in volume eventually resulting in more micro and macro cracking in concrete. Additionally, sulphuric acid tends to decalcify the calcium-silicate-hydrate (CSH) and thus destroys the cement matrix and leads to loss of strength in concrete (Bassuoni and Nehdi, 2007).

The resistance to sulphuric acid attack was evaluated out as per ASTM C 267-97 for a duration of 90 days. Sulphuric acid of 3% by weight of water was opted as a medium for this test. To perform the test, 28 day cured concrete cube specimens of 100mm size were immersed in a container filled with dilute sulphuric acid solution. To maintain a constant concentration, the H_2SO_4 solution was replaced after 24 hours of immersion, then at 3rd day and then at every 7 days. Surface treatments were not performed on the specimens attacked by acid before testing them (Emmanuel and Sami, 1988; Bassuoni and Nehdi, 2007)

The following tests were done on Acid Attacked Concrete:

3.5.5.1 Water Absorption of Acid Attacked Concrete

The water absorption test of acid attacked specimens was done to determine the changes in porosity. The concrete samples were tested for water absorption after 28, 56 and 90 days of immersion in dilute sulphuric acid solution. The test was carried out as per ASTM C 642 (2006). The concrete cube specimens of size 100mm dried in a ventilated oven at 60° ± 5 °C for 3 days and kept at room temperature for 24 hours were used and the initial weight W_1 was taken. Then the cube specimen was dipped in water by maintaining 50mm at the top surface of the specimen. After 48 hours, the specimens were taken out of the water and allowed to drain for a minute by placing over a dry cloth and the final weight W_2 is taken. The values obtained are related with the water absorption values of non-acid attacked specimens.

$$Water\ Absorption\ = \frac{W_2\ - W_1}{W_1} \times 100$$

3.5.5.2 Variation in weight (Weight loss) of Acid Attacked Concrete

The variation in weight of acid attacked specimens were done after 28, 56 and 90 days of exposure to dilute sulphuric acid solution. The concrete cube specimens of size 100mm dried in a ventilated oven at 60° ± 5 °C for 3 days and kept at room temperature for 24 hours were used and the initial weight was taken. The variation in weight was related with the initial oven dried weight before immersion in acid solution.

3.5.5.3 Compressive strength (reduction) of Acid Attacked Concrete

The compressive strength was evaluated after 28, 56 and 90 days of immersion in dilute sulphuric acid solution as per ASTM C 579-01. The compressive strength variation was compared with that of non-acid attacked specimens which were water cured for 28 days after casting. The results were expressed in terms of percentage. It is to be noted that the change in dimension of the specimen after the acid exposure was not taken into account while calculating the compressive strength.

3.5.5.4 X-ray diffraction observation of acid attacked specimen

Development of compact, lightweight, rugged, portable, and economically viable equipment for implementation of some analytical chemistry methods such as XRD has facilitated their growing semi destructive or non-destructive field applications (Peyvandi et al., 2015). Inorder to establish the formation of ettringite by XRD, 3-4 grams of powdered representative samples were taken from acid attacked specimens. The selection of specimens was based on shortest and longest exposed to magnesium sulphate solution.

3.5.6 Resistance to Sulphate Attack

Ordinary Portland cement is prone to attack of sulphates, especially to magnesium sulphates. Sulphates are mostly found in sea water, ground water, high clay content soils, mining pits, sewer pipes, organic materials in marshes etc. The common sulphates include magnesium, calcium, potassium, calcium, sodium and ammonium. Sulphates, when reacted with hydrate of calcium aluminate forms calcium sulphoaluminates. These have a volume of 227% of the volume of original aluminates. With free calcium hydroxides, sulphates form calcium sulphate initially and eventually to the formation of gypsum, ettringite and/or thaumasite. This causes several forms of concrete damage like spalling, cracking, softening, expansion, loss of strength. The expansion results in further propagation of cracks and resulting in disruption of concrete. This phenomena is known as sulphate attack, which gets accelerated if subjected to alternate wetting and drying. This mainly takes place in marine structures which are in the zone of tidal variations.

Nader et al. (2008) explained the process of sulphate attack in three steps. Due to the reaction between tricalcium aluminate in the cement and the sulphate ions from internal/external sources, ettringite forms in the cement matrix as the first step. It is shown below.

$$\underset{\substack{\uparrow \\ C_3A}}{3CaO.Al_2O_3} + \underset{\substack{\uparrow \\ \text{Gypsum}}}{3(CaSO_4.2H_2O)} + 26H_2O \rightarrow \underset{\substack{\uparrow \\ \text{Ettringite } (C_6AS_3H_{32})}}{3CaO.Al_2O_3.3CaSO_4.32\ H_2O}$$

The sulphate ions from ettringite reacts with remaining tricalcium aluminate to form tetra calcium aluminate monosulphate-12-hydrate, which is known as monosulphoaluminate. The chemical reaction is given below.

$$2(CaO.Al_2O_3) + 3CaO.Al_2O_3.\ 3CaSO_4.\ 32H_2O + 4H_2O \longrightarrow 3(CaO.Al_2O_3.SO_3.12H_2O)$$

Monosulphoaluminate

When the monosulphoaluminate is in contact with any new source of sulphate ions, it forms ettringite again.

$$4CaO.Al_2O_3.SO_3.12\ H_2O + 2(CaSO_4.\ H_2O) + 16\ H_2O \longrightarrow 3CaO.Al_2O_3.\ 3CaSO_4.\ 32H_2O$$

Ettringite

When ettringite is continuously formed within the solid concrete, it creates a lot of internal pressure that leads to expansion and cracking. Continuous and progressive loss in mass and strength of concrete occurs when it is exposed to sodium sulphate or magnesium sulphate. The sulphate ions react with calcium hydroxide from the cementitious material to form gypsum.

$$Na_2SO_4 + Ca(OH)_2 + 2H_2O \longrightarrow \text{Gypsum} + 2NaOH$$

$$Mg_2SO_4 + Ca(OH)_2 + 2H_2O \longrightarrow \text{Gypsum} + Mg(OH)_2$$

Sulphate attack test was performed according to ASTM C 1012-89. The concrete specimens of 100 mm size, cured for 28 days were used for the testing. The saturated weight of the specimens were taken and they were subjected to a continuous soaking for a period of 180 days in 3% $MgSO_4$ solution. To maintain the constant concentration, the $MgSO_4$ solution was replaced after first 24 hours of immersion, then at 3rd day, and then at every 7 days (Paulo and Kimberly, 2003; Nader et al., 2008). It is to be noted that, surface treatments were not performed for the sulphate attacked specimens prior to the test carried out. The test specimens were periodically taken out of the soaking tank and the following tests were performed.

3.5.6.1 Variation in weight

Weight measurement of sulphate attacked specimens were done after 28, 90 and 180 days of immersion. The concrete samples were taken out of the tank and allowed to surface dry at room temperature for 10 to 15 minutes and the weight was noted. The change in weight was compared to that of the initial saturated surface dried weight of the specimen.

3.5.6.2 Compressive strength

The compressive strength was evaluated after 28, 90 and 180 days of immersion in $MgSO_4$ solution. It was related with the compressive strength of non-sulphate attacked (normal concrete sample) concrete which was cured for 28 days. The results are represented in percentage. The variation in the dimension of the specimen after the exposure was not taken into account while calculating the compressive strength.

3.5.6.3 Water absorption

The water absorption capacity was evaluated (as per ASTM C 642-2006) to study the changes in porosity due to attack of sulphate salts. The concrete samples were tested for water absorption after 90 and 180 days of immersion in $MgSO_4$ solution. The concrete cube specimens of size 100mm dried in a ventilated oven at 60° ± 5 °C for 3 days and kept at room temperature for 24 hours were used and the initial weight W_1 was taken. Then the cube specimen was dipped in water by maintaining 50mm at the top surface of the specimen. After 48 hours, the specimens were taken out of the water and allowed to drain for a minute by placing over a dry cloth and the final weight W_2 is taken. The values obtained are related with the water absorption values of non-sulphate attacked specimens.

3.5.7 Test for Corrosion of Steel Reinforcements

To measure the corrosion of steel reinforcements, chloride induced corrosion technique was adopted. Macro-cell method was used to measure (monitor) the corrosion activities of embedded steel bars in concrete. The potential difference between anode and cathode across a standard resister of 100 Ω was measured.

To prepare the specimens for corrosion test, three TMT steel bars as given in Figure 3.16. (12 mm diameter and 350 mm length) were taken and properly cleaned with a wire brush for removing the dirt and rust on the surface. Rubber shrink tube was firmly affixed on the two ends of the steel bars at a length of 75 mm. This was done to prevent that area (which is exposed outside the specimen) from getting corroded.

Concrete specimens used here were of size 250 mm X 200 mm X 120 mm (as per ASTM G 109-2005) as given in Figure 3.17. The two steel bars were kept inside with a cover of 30 mm from bottom (cathode) and one steel bar with a cover of 15 mm was placed centrally at the top (anode). A reservoir of 15 mm was made on the upper surface of the specimen for ponding with 3% sodium chloride solution.

After casting, the corrosion specimens were cured in water for 28 days by maintaining the temperature at 27° ± 2°C. The specimens after removal from the curing tank were dried at room temperature for a period of 30 days. The vertical sides of specimens were then epoxy coated with two layers. With the help of electric wires, a standard resistor of 100 Ώ was connected between the common terminal of the bottom steel bars and the terminal of the top steel bar. A solution of 3% sodium chloride (by weight) was poured on the upper surface reservoir of the specimen. Then the specimens were subjected to alternate wetting and drying cycles as given in Figure 3.17 (2 weeks wetting with sodium chloride solution, followed by 2 weeks drying).

The potential measurements were taken for both the wetting and drying cycles. The initial reading for the macro-cell corrosion was taken at the beginning of the second week of the ponding and the consecutive readings were taken after every 2 weeks. A high impedance voltmeter was used to measure the potential difference between the anode and cathode as given in Figure 3.15. The macro-cell current was calculated by the following equation:

$$I_j = \frac{V_j}{100}$$

Where, Vj = Voltage across 100 Ώ resistor.

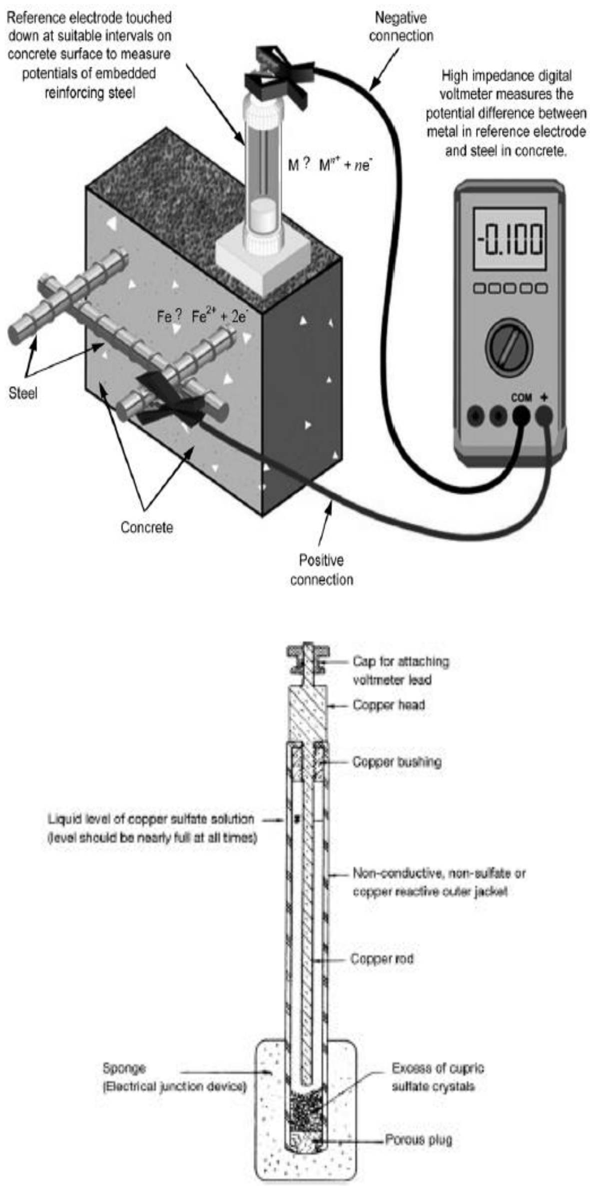

Figure 3.15: From ASTM C 876 (a) Reference Electrode Circuitry (b) Sectional view of Copper-Copper Suiphate electrode

Figure 3.16: Steel Bars and Concrete Specimens

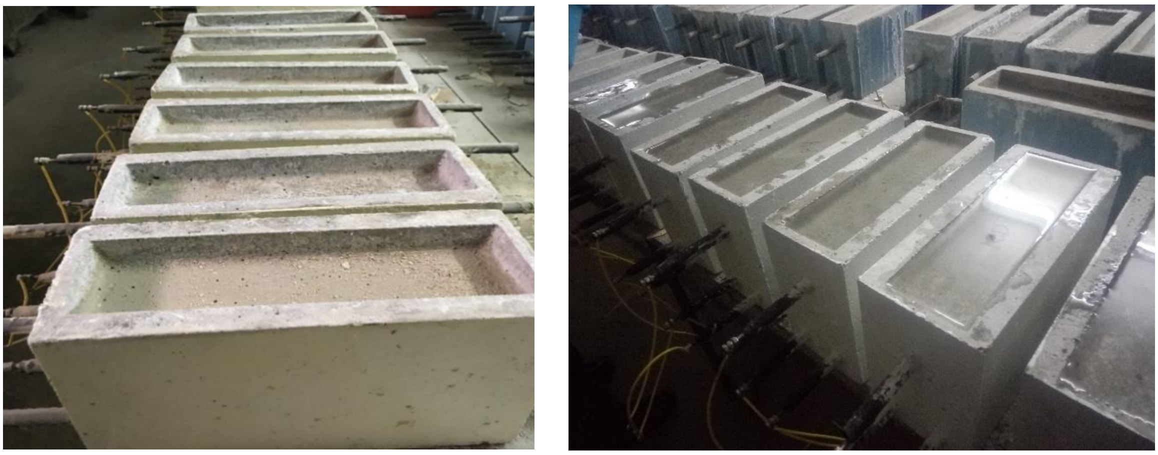

Figure 3.17: Epoxy coated specimens with 3% NaCl solution (drying and wetting cycle)

The total integrated current is obtained from the following equation:

$$TC_j = TC_{j-1} + [(t_j - t_{j-1}) \times \frac{(i_j + i_{j-1})}{2}]$$

Where,

TC = Total corrosion (coulombs)

tj = Time in seconds at which measurement of the macro cell current is carried out

ij = Macro cell current (amps) at time tj.

3.5.8 Microstructure by SEM Analysis

The microstructure was analysed with the help of a Nova Nano Field Emission Scanning Electron Microscope (FE-SEM) given in Figure 3.18. It is used to provide ultra-high resolution characterisation and analysis giving precise, true nanometer scale information. The machine gives a resolution of 1.4 nm at 1 kv (TLD-SE) and 1 nm at 15 kv (TLD-SE) The FE-SEM is coupled with an EDX detector for measuring the elemental chemical composition of the nano materials.

The instrument has a very fine cold field emission electron source, consisting of a tungsten tip cathode of only 100 nm in radius of curvature, and two anodes, the first causing electrons to be emitted from the cathode via an electric field (hence "field emission"), with the second anode then causing the emitted electrons to be accelerated at high velocity. The resulting high emission intensity allows improved resolution combined with a high signal to noise ratio.

Concrete pieces of 1cm size were cut from cube specimens and were gold coated in the coating apparatus. Then they are examined under the lens of an FE-SEM apparatus and taken the images at different focus of the lens. Cement, sand, Dholpur stones (Quartz sandstone) and silica fumes were also examined in the similar manner.

Figure 3.18: Coating device and FE-SEM machine with EDX attachment.

3.5.9 X-ray fluorescence (XRF) observation

Inorder to determine the elemental composition of concrete samples, XRF is a valid and useful method (EPA 2007). XRF is also one of the most befitting methods which permit the analyser to a lot of determinations in a very period of time when compared to rest of the methods (Proverbio and Carassiti 1997). Samples which emit X-ray intensities based on element characteristics not only depends on elemental concentration but also the chemical bindings of the element in the matrix, the degree of compaction and grain size. The concrete samples having various percentages of quartz sandstone substitution was analysed.

3.5.10 Thermogravimetric Analysis

Thermo gravimetric analysis is a type of testing that is done to determine the changes in mass or weight of a specimen with respect to change in temperature. Simultaneous Thermal Analyser (STA 6000) with Pyris Instrument Viewer software was used for analysing the samples (Figure 3.19). The sample was checked to be compatible with ceramic crucible at temperature, atmosphere and time and the nitrogen gas was connected to the analyser in the balance port with sample purge gas flow rate at 30 ml/min. The temperature was applied at the rate of 20 °C and Pyris manager was used to get the math and smoothen the curves obtained after the run was complete with furnace returning to room temperature.

Figure 3.19: Simultaneous Thermal Analyser setup

3.5.10 Action of Fire

Concrete members generally exhibit good fire resistant properties due to its low thermal conductivity, high thermal capacity and slow degradation of mechanical properties of concrete with temperature. No standard test procedures exist for high temperature testing in concrete. Most of the researchers have employed testing concrete at higher temperatures and finding its residual strength after its exposure. The concrete containing 0%, 20%, 60% and 100% of quartz sandstones were tested against fire with an increasing temperature upto 800° C (Figure 3.20).

Figure 3.20: Equipment used to regulate fire in concrete samples.

3.6. Mix Design and Trial Testing

The trial mixes series were divided into two categories. The first trail mix series consists of concrete made solely from natural/quartz sandstone (Dholpur stone) aggregates with different gradations to obtain the optimum ratio of aggregate sizes. The second trail mix series consists of replacing natural aggregates by quartz sandstones from 0% to 100% at every 10% interval (with the gradation obtained from trial mix series 1) to find the optimum replacement levels.

3.6.1 Proportioning of Trail Mix Series I

For proportioning of concrete mix with Dholpur stone (Quartz sandstone), a definite method for mix design is not available. Not many researchers have utilised this quality of sandstones in cement concrete. Some of them have worked on utilising sandstones at very high water-cement ratios. However, not many research has been carried out to replace natural coarse aggregates partially or completely

From the literature study, it was decided to use Dholpur stone (quartz sandstone) as a partial or whole replacement for coarse aggregates in cement concrete. However, a preliminary study was needed to find the optimum ratios of aggregate size to obtain a maximum dense concrete and secondly, to attain an optimum replacement percentage of Dholpur sandstone by natural coarse aggregates.

3.6.2 Mix Design Procedure for Preliminary Test

In order to achieve the best proportion of the constituent materials, trial mixes were cast and tested at every step. The trial mix was divided into two steps. One step was to find out the exact gradation at which coarse aggregates can be mixed together and the other was to find the optimum replacement level.

a) The grade of concrete (for severe exposure condition) was fixed as M30. OPC 43 grade cement conforming to IS 8112 was selected. Maximum size of aggregates was 25 mm for Dholpur coarse aggregates and 20 mm for natural coarse aggregates. Minimum cement content was 320 kg/m^3 and maximum was 450 kg/m^3.
b) For the trial mixes, the water cement ratios were fixed to be 0.38 and 0.40. The target strength for M30 grade concrete is obtained to be 38.25 N/mm^2. The fixation of water-cement ratio was fixed based on the trail mixes using superplasticiser. The total amount of cement used is determined using the water-cement ratio which is greater than the minimum amount given in code.
c) The volume of aggregate was divided into fine aggregates and coarse aggregates. As per the gradation study, the ratio of combining different size of aggregates were obtained.
d) Thus the mass of constituent materials (cement, water, aggregates, water and chemical admixture) for one cubic meter concrete was obtained.

3.6.3 Combined Gradation of Coarse Aggregates

Gradation is one of the important parameters for finding out the exact ratio of mixing different size of aggregates together. IS 383:1970 gives the grading limit of coarse aggregates for maximum sizes of aggregates 40, 20 and 16. However the maximum size of aggregate obtained was 25mm so the values were interpolated to get the exact size of grading for quartz sandstone. The expected value of percentage passing of general aggregate corresponding to size 25mm are given in table 3.8. Percentage passing for combined ratios of natural aggregates in given in table 3.9 and for quartz sandstone in table 3.10.

Table 3.8: Percentage passing for general aggregates of normal size and interpolated value

I.S. Sieve size	Percentage passing		
	40mm size	20mm size	Interpolated 25mm size
40mm	95-100	100	96-100
20mm	30-70	95-100	70-90
10mm	10-35	20-55	20-48
4.75mm	0-5	0-10	0-8

Table 3.9: Percentage passing values for combined grading of different proportions of 20 mm and 10 mm natural aggregates

Sieve size	Percentage passing of natural aggregates (20mm:10mm)		
	60:40	50:50	40:60
40	100	100	100
20	86.64	86.35	92.95
10	31.28	42.35	53.5
4.75	1.08	1.2	2

Table 3.10: Percentage passing values for combined grading of different proportions of 25 and 10mm Dholpur stone (quartz sandstone) aggregates.

Sieve size	Percentage passing of quartz sandstone aggregates (25mm:10mm)		
	60:40	50:50	40:60
40	100	100	100
20	92.56	92.26	90.35
10	41.65	51.15	61.25
4.75	3.26	3.57	3.1

3.6.4 Workability of Concrete for different proportions of Coarse Aggregates

Workability of fresh concrete was tested using a compaction factor apparatus as per IS 1199:1959. Effectiveness of concreting depends on the ease at which concrete can be placed, compacted and finished. Aggregate characteristics affect the water required for a given consistency. Sandstone aggregates tend to have higher water absorption than the natural aggregates and water requirement to maintain the same workability is a tedious process. Super plasticisers were used to maintain the compaction factor for both natural and sandstone aggregates. (C.F. - 0.85-0.9). Before partially replacing sandstone coarse aggregate for a ratio of 50:50, different proportions of individual mixes for natural and sandstone aggregates were done to obtain the exact ratio in which aggregates can be used together without affecting the gradation. Mix proportions and properties of fresh concrete are given in table 3.11 and 3.12.

Table 3.11: Mix proportions of cement concrete with varied percentages of coarse aggregates for w/c 0.38

Ingredients per cu.m	Conventional coarse (20mm:10mm)					Dholpur stone coarse (25mm:10mm)				
	40:60	45:55	50:50	55:45	60:40	50:50	55:45	60:40	65:35	70:30
Cement, kg	426	426	426	426	426	426	426	426	426	426
Water, kg	162	162	162	162	162	162	162	162	162	162
FA, kg	645	645	645	645	645	645	645	645	645	645
10 mm CA, kg	756.6	693.6	630.5	567.4	504.4	-	-	-	-	-
20 mm CA, kg	504.5	567.5	630.5	693.6	756.6					
Sandstone Aggregate 10 mm, kg	-	-	-	-	-	580.4	522.3	464.3	406.2	348.2
Sandstone Aggregate 25 mm, kg	-	-	-	-	-	580.4	638.5	696.5	754.5	812.6
Admixture, %	0.8	0.8	0.8	0.8	0.8	0.85	0.9	0.95	1.0	1.05
C.F.	0.85	0.87	0.88	0.85	0.85	0.85	0.87	0.85	0.85	0.86

Table 3.12: Mix proportions of cement concrete with varied percentages of coarse aggregates for w/c 0.40.

Ingredients per cu.m	Conventional coarse (20mm:10mm)					Dholpur stone coarse (25mm:10mm)				
	40:60	45:55	50:50	55:45	60:40	50:50	55:45	60:40	65:35	70:30
Cement, kg	388	388	388	388	388	388	388	388	388	388
Water, kg	155.2	155.2	155.2	155.2	155.2	155.2	155.2	155.2	155.2	155.2
FA, kg	698.4	698.4	698.4	698.4	698.4	698.4	698.4	698.4	698.4	698.4
10 mm CA, kg	721.7	661.5	601.4	541.3	481.1	-	-	-	-	-
20 mm CA, kg	481.1	541.3	601.4	661.5	721.7	-	-	-	-	-
Sandstone Aggregate 10 mm, kg	-	-	-	-	-	553.7	498.3	442.9	387.6	332.2
Sandstone Aggregate 25 mm, kg	-	-	-	-	-	553.7	609.0	664.4	719.8	775.1
Admixture, %	0.8	0.8	0.8	0.8	0.8	0.85	0.9	0.95	1.0	1.05
C.F.	0.86	0.89	0.88	0.85	0.85	0.88	0.89	0.8	0.85	0.87

3.6.5 Compressive Strength Test based on Combined Gradation of Aggregates

Compressive strength tests were carried out with cube size of 100 mm × 100 mm × 100 mm with varying ratios of 25 mm and 10 mm aggregates. Compressive strength was done after 7 and 28 days of curing in accordance with IS 516:1959. Surface water was removed with a dry cloth and the cubes were tested in wet condition.

The variation in compressive strength for water cement ratio 0.38 and 0.4 for natural aggregate is shown in figures 3.21 & 3.22 and for quartz sandstone in figures 3.23 & 3.24. Compressive strength results for varied ratios of combined gradation showed increasing trend till 55:45 (20mm: 10mm) natural aggregate and the same trend was noticed in quartz sandstone (Dholpur stone) aggregate till 60:40 (25mm: 10mm). The decrease in compressive strength results after attaining a specific gradation was due to increase in void spaces due to the usage of bigger sized aggregates. The exact ratio for mixing conventional coarse aggregates was found to be 55:45 (20mm: 10mm) and for quartz sandstone aggregates was 60:40 (25mm: 10mm) in order to achieve maximum compressive strength.

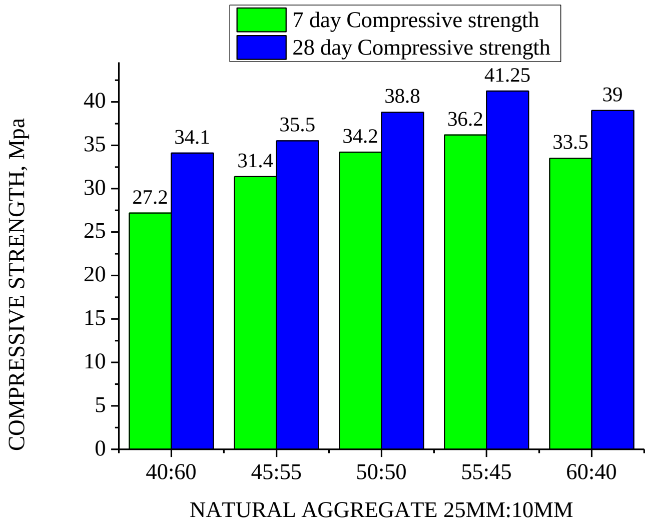

Fig 3.21: Compressive Strength of concrete containing natural aggregate with varied gradations for w/c of 0.38

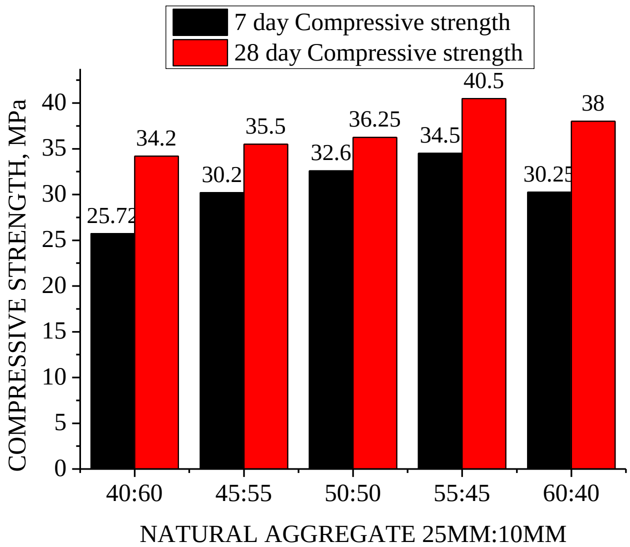

Fig 3.22: Compressive Strength of concrete containing natural aggregate with varied gradations for w/c of 0.40

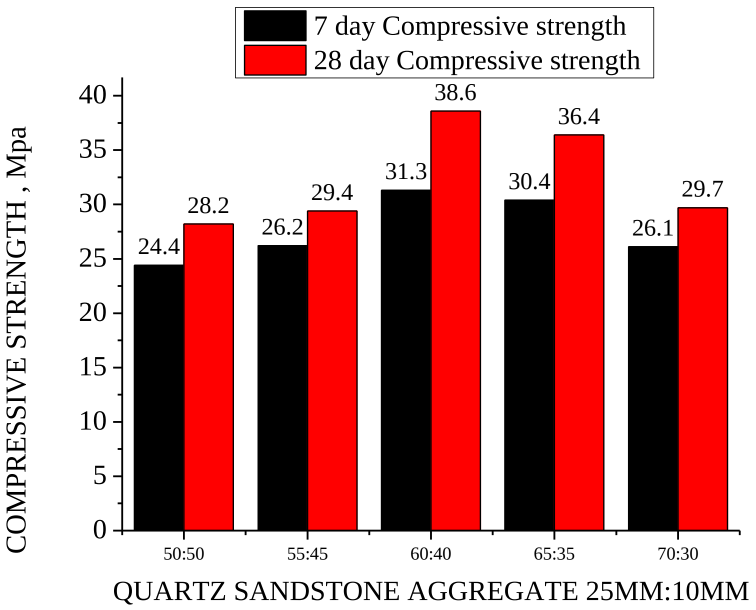

Fig 3.23: Compressive Strength of concrete containing Dholpur stone (quartz sandstone aggregate) with varied gradations for w/c of 0.38

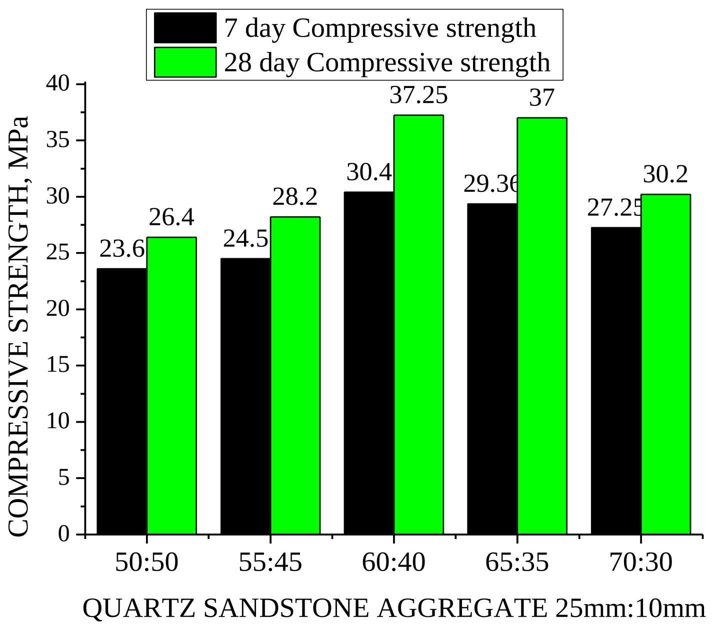

Fig 3.24: Compressive Strength of concrete containing Dholpur stone (quartz sandstone aggregate) with varied gradations for w/c of 0.40

3.6.6 Proportioning of Trial Mix Series II

M30 concrete grade was designed as per IS 10262:2010 and IS 456:2000 with water cement ratio of 0.38. The replacement of natural aggregates with quartz sandstones

(Dholpur aggregates) were done at every 10% interval upto 100% replacement (Table 3.13). Concrete cubes of sizes 100 mm x 100 mm x 100 mm for compressive strength and flexural beams of size 100 mm x 100 mm x 500 mm were casted. Concrete mixtures were casted at indoor temperatures of 25-30 °C. Compaction factor test was used to determine the workability of concrete mixes and C.F. was maintained between 0.85-0.9 by the use of super plasticisers and curing temperature of 25-27 °C was maintained in the water tank.

3.6.7 Fresh and Hardened Concrete Properties of Trial Testing Series II

Workability of fresh concrete was measured using compaction factor apparatus as per IS 1199:1959 and the same code was used for measuring density of fresh concrete. Workability and density of fresh concrete containing increased dosages of quartz sandstone aggregates were compared with the control concrete containing only the conventional aggregates. From the Table 3.13 it can be seen that density of fresh concrete decreased as the quartz sandstone aggregate was replaced for conventional coarse aggregates. This is due to the lower specific gravity of quartz sandstone aggregates.

Concrete cubes of 100 mm × 100 mm × 100 mm were cast with increasing replacement percentages of quartz sandstone as a substitute material for conventional coarse aggregate. Specimens were cast for 7 and 28 days compressive strength tests in compliance with IS 516: 1959, demoulded after 24 hours and cured immersed in a curing tank free from vibration. At test ages, any grit and surface water was removed from the concrete cubes and the test carried out without delay. The compressive strength decreased gradually due to the use of quartz sandstone aggregates (Figure 3.25).

Table 3.13: Mixture proportions and properties of fresh concrete having different replacements of quartz sandstones for trial mix series II (w/c-0.38)

Ingredients per cu.m	Percentages of quartz sandstone/ Dholpur stone (%)										
	0	10	20	30	40	50	60	70	80	90	100
Cement, kg	426	426	426	426	426	426	426	426	426	426	426
Water, kg	162	162	162	162	162	162	162	162	162	162	162
FA, kg	645	645	645	645	645	645	645	645	645	645	645
10 mm CA, kg	756.6	510.7	454.2	397.4	340.6	283.9	227.1	170.3	113.5	56.8	
Quartz Sandstone Aggregate 10 mm, kg	-	50.5	100.9	151.4	201.86	252.3	302.8	353.2	403.7	454.2	504.6
Total 10mm, kg	756.6	561.2	555.1	548.8	542.46	536.2	529.9	523.5	517.2	511	504.6
20 mm CA, kg	504.5	624.2	555.1	485.7	416.3	346.9	277.6	208.2	138.8	69.4	
Quartz Sandstone Aggregate 25 mm*, kg	-	75.7	151.4	227.1	302.8	378.4	454.2	529.9	605.6	681.3	757
Total (25mm,20mm)	504.5	699.9	706.5	712.8	719.1	725.3	731.8	738.1	744.4	750.7	757
Admixture, %	1	1	1.05	1.1	1.1	1.15	1.2	1.2	1.2	1.2	1.2
C.F.	0.9	0.9	0.89	0.88	0.88	0.87	0.87	0.88	0.87	0.88	0.86
Density,kg	2445	2405	2370	2365	2355	2345	2340	2335	2330	2295	2270

* Replacing 20mm NA by 25mm Dholpur aggregate (in available size)

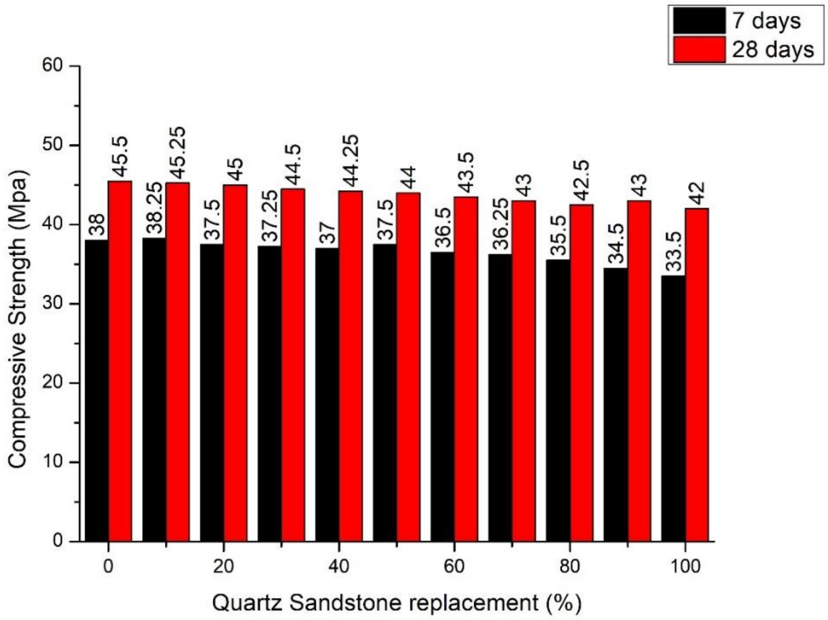

Fig 3.25: Compressive Strength of cubes for water-cement ratio of 0.38 (Trial).

Concrete beams of 100 mm × 100 mm × 500 mm size were cast with various percentages of quartz sandstone aggregates and various water/cement ratios. Flexural strength tests were carried out in accordance to IS 516: 1959 after 7 and 28 days. The test samples were stored in water at a temperature of approximately 27-29°C for 24 hours before testing. The samples were tested whilst they were still in wet condition. Loose and foreign materials were wiped off the bearing surfaces and the axis of the specimen was aligned with that of the loading device. Then load was applied at a rate of 400 kg/minute until the specimen failed to attain the peak load. The test results showed a decrease in flexural strength as the quartz sandstone was added in concrete (Figure 3.26).

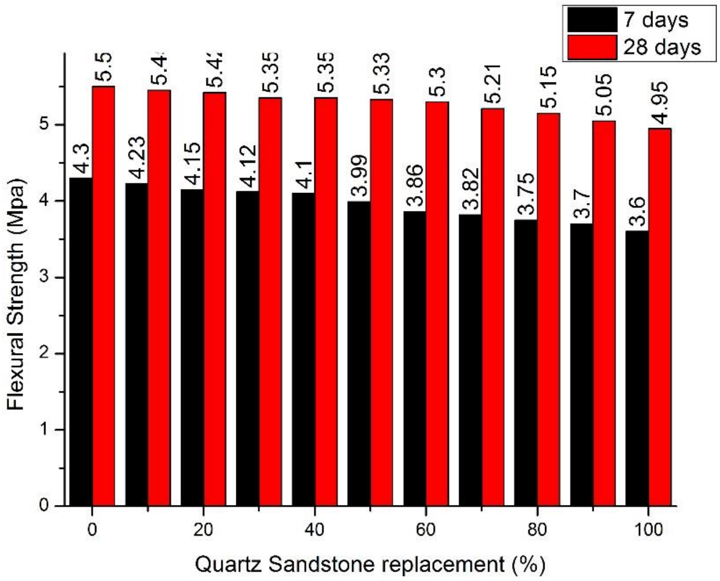

Fig 3.26: Flexural Strength of concrete beams for water-cement ratio of 0.38 (Trial)

3.7 Final Composition

Based on the results of trial mixes series (I & II), the exact ratio for mixing conventional coarse aggregates was found to be 55:45 (20mm: 10mm) and for quartz sandstone aggregates was 60:40 (25mm: 10mm) in order to achieve maximum compressive strength. Also the replacement level of Dholpur stone (Quartz sandstone) for conventional coarse aggregate was fixed to be 0% to 100% in the multiples of 20% during the main casting. Since no considerable variation in the decrease in strength plots were observed at every 10% interval, the replacement level was increased to 20%. It was further decided to study the mechanical and durability characteristics of concrete having different replacements of Dholpur stone (Quartz sandstone). In the Trial mix I with the water-cement ratio of 0.38, the workability of concrete was found to be on the lower side (CF: 0.86-0.90). Inorder to increase the workability, the amount of water was increased in the standard series. For a standard mix, M30 concrete grade was designed as with a water-cement ratio of 0.40. Also, to study the variation in different properties of concrete, the mixes with water-cement ratio of 0.35 and 0.45 were used. The mix proportions of final compositions are given in Table 3.13. The mix proportions of final composition of all the three series are similar to that of given in Table 3.14.

Table 3.14: Mixture proportions of Fresh Concrete with w/c 0.35, 0.40 and 0.45.

Water-Cement ratio	Cement (kg/m^3)	Water (kg/m^3)	Coarse aggregates 10 mm (kg/m^3)	Coarse aggregates 20 mm (kg/m^3)	Fine aggregates (kg/m^3)	Minimum-Maximum Admixture (%)
0.35	440	154	559.35	683.65	635.47	0.87-0.90
0.40	405	162	567.45	693.55	645	0.86-0.89
0.45	342.2	153.9	589.60	713.29	662.05	0.86-0.89

Dholpur stone (quartz sandstone) was partially substituted for coarse aggregate by weight from 0% to 100% in multiples of 20%. The properties of concrete with Dholpur stone were studied corresponding to three different water-cement ratios of 0.35, 0.40 and 0.45. The dosage of superplasticisers were altered to maintain a constant workability in all the concrete mixes.

On the basis of final mix design compositions, the results and discussions are described in the following chapter.

CHAPTER-4

RESULTS AND DISCUSSIONS

4.1 Introduction

The properties of individual materials and the results of trial mixes are presented in the earlier Chapter. The results obtained for concrete samples in this experimental programme and its discussion are presented in this Chapter. A total of 3 series of concrete were cast with 18 different mixes. A series with M30 grade of concrete with a water-cement ratio of 0.40 has been casted. Dholpur stone (quartz sandstone) was replaced for conventional coarse aggregate from 0% to 100% in multiples of 20%. To study the effect of change in water-cement ratio on different properties, two more series with water-cement ratios of 0.35 and 0.45 were also studied. Therefore, three series of water-cement ratios of 0.35, 0.40 and 0.45 named as Series I, II and III were studied respectively. Tests for workability, resistance to compression and flexure, pull-off strength, bulk density of hardened concrete, resistance to abrasion, water permeability, water absorption, resistance to carbonation, chloride ion penetration, acid attack, sulphate attack, corrosion of specimens were conducted to determine the fresh and hardened properties of concrete and the observations were recorded. SEM, TGA, XRD and XRF studies were also carried out. The recorded results have been tabulated and discussed here.

4.2 Properties of Fresh Concrete

The workability of fresh concrete has been calculated using compaction factor test and is presented in the Table 4.1.

Table 4.1: Workability of fresh concrete by Compacting Factor Test

Replacement percentage of Dholpur stone/Quartz Sandstone	0	20	40	60	80	100
w/c = 0.35	0.9	0.9	0.89	0.88	0.88	0.87
w/c = 0.40	0.89	0.9	0.88	0.88	0.86	0.86
w/c = 0.45	0.89	0.9	0.88	0.88	0.86	0.86

By the addition of Dholpur stone (quartz sandstone) the workability of the concrete mixes has been affected and the dosage of superplasticisers has been altered to maintain the compaction factor from 0.85-0.90 in the all the concrete mixes. The dose of superplasticisers (named as Gilenium Sky 777) has been finalised after several trial mixes and it may be upto 1.2% by weight of cement.

4.3 Properties at Hardened State

Tests to evaluate resistance to compression and flexure, pull-off strength, bulk density, abrasion resistance, water penetration, water absorption, carbonation resistance, chloride ion penetration, acid attack, sulphate attack and macro cell corrosion were conducted on the specimens for determining the strength and durability in the hardened stage.

4.3.1 Bulk Density

The bulk densities of hardened concrete mixes has been estimated by measuring the weights (in grams) and dividing by volume (cm^2). The graph showing the variations in the bulk density with respect to the percentage of Dholpur stone (quartz sandstone) is given in Figure 4.1.

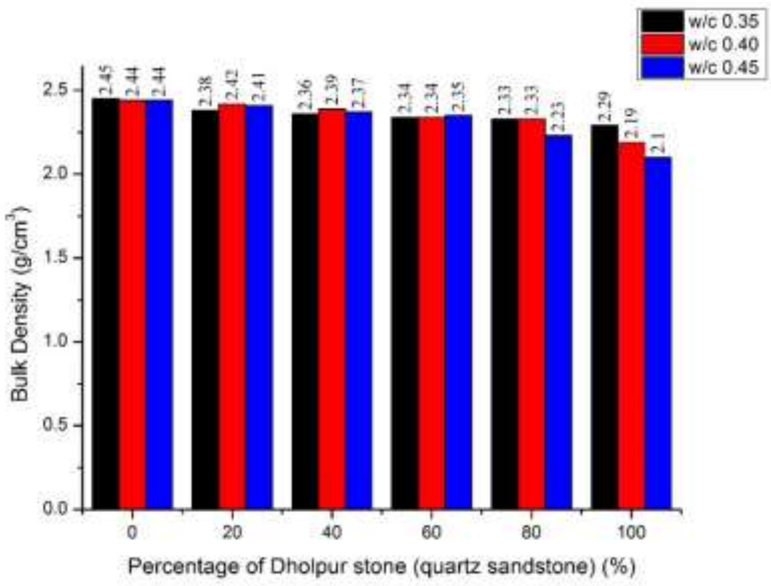

Figure 4.1: Bulk Density of specimens for all the three series

From the test results, it was seen that the bulk density of the concrete reduced with the increase in amount of Dholpur stone (quartz sandstone). For the entire series, the maximum density was recorded for the control mixes with concrete made solely from conventional coarse aggregates. The least density was observed for the concrete mixes with 100% Dholpur stone (quartz sandstone). This decrease in densities is because of utilization of quartz sandstones which are having a much less specific gravity when compared to that of conventional coarse aggregates.

4.3.2 Compressive Strength

The compressive strength of the specimens were taken after 7, 28, 90, 180 and 360 days of curing. The variations in the compressive strength with respect to their water-cement ratios and with respect to the percentage of Dholpur stone (quartz sandstone) are given in Figures 4.2-4.4.

The target mean strength (at 28 days) for M30 grade of concrete was 38.25 MPa and the actual crushing strength for control mix was obtained as 46 Mpa, 45.7 MPa and 42.3 MPa for the water-cement ratios of 0.35, 0.40 and 0.45 respectively. It was observed that the decrease in trend pattern was noticed at every percentage of replacement of quartz sandstones. A decrease of 8% in compressive strength with respect to target mean strength was observed upto the 40% replacement level. In the case of 0.35 and 0.40 water-cement ratios, compressive strength more than the target mean strength was obtained for the 100% replacement level. In the case of series III, the target mean strength was achieved upto a replacement of 40%. The maximum decrease in compressive strength was recorded to be 21% in the case of 100% replacement for the w/c 0.45.

As the quartz sandstone replacement for natural coarse aggregates increased**,** a substantial decrease in compressive strength was observed**.** This could be attributed to delay in cement hydration at the initial stages of curing due to the higher replacement level of quartz sandstones and also due to the use of sedimentary rock itself. This was also experienced by researchers (Newman and Choo, 2003; Islam et al., 2016) while utilising oil palm shells.

After 90 days, significant strength gain with respect to curing period has been observed due to the continuous pozzolanic reaction. This strength gain can also be associated with the filling up of pore spaces in the concrete with products of hydration (Thomas et al., 2015). When the water-cement ratio was 0.35, highest resistance to compression (47 MPa) was noted for the control mix and minimum compressive strength (42 MPa) was obtained for the mix having 100% quartz sandstone. The lowest compressive strength (36.2) was recorded for the mix at 0.45 water-cement ratio with 100% quartz sandstone. A compressive strength above target (38 MPa) was obtained in all the replacements at 0.35 and 0.40 water-cement ratios and till 40% replacement at 0.45 water-cement ratio. The reduction in mechanical properties of concrete containing quartz sandstones has also been reported by researchers like Yilmaz and Tugrul (2012); Mackechnie (2006). Fig 4.5 shows the broken samples of concrete specimens after compression testing.

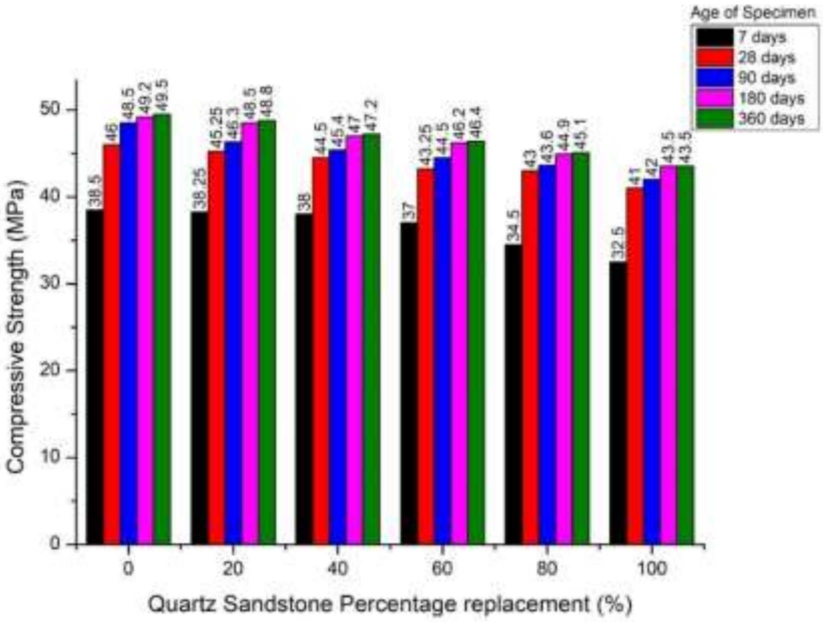

Fig 4.2: Compressive Strength of Specimens with w/c 0.35.

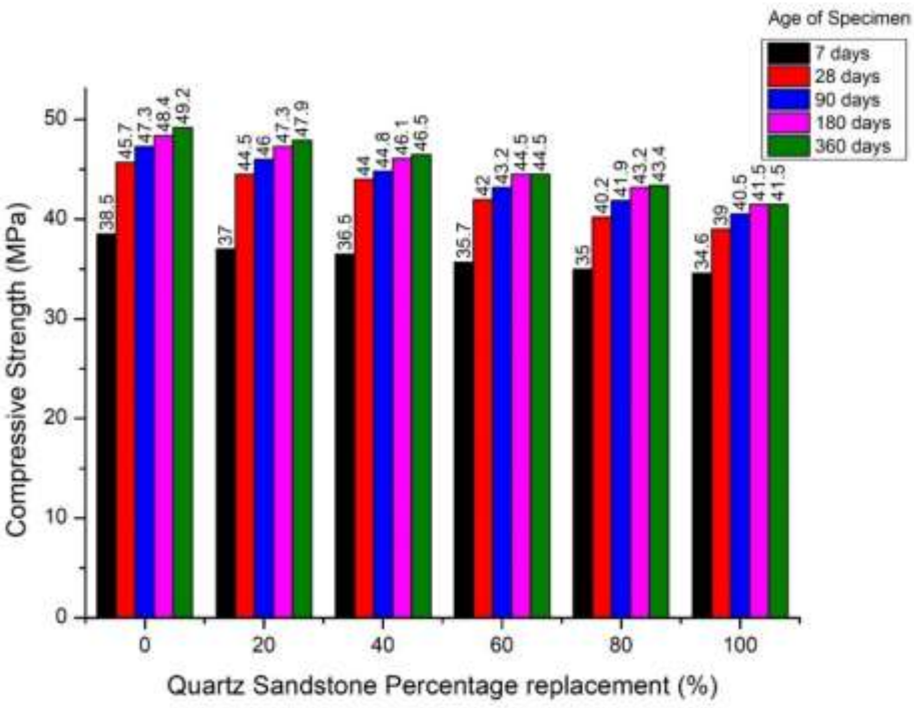

Fig 4.3: Compressive Strength of Specimens with w/c 0.40

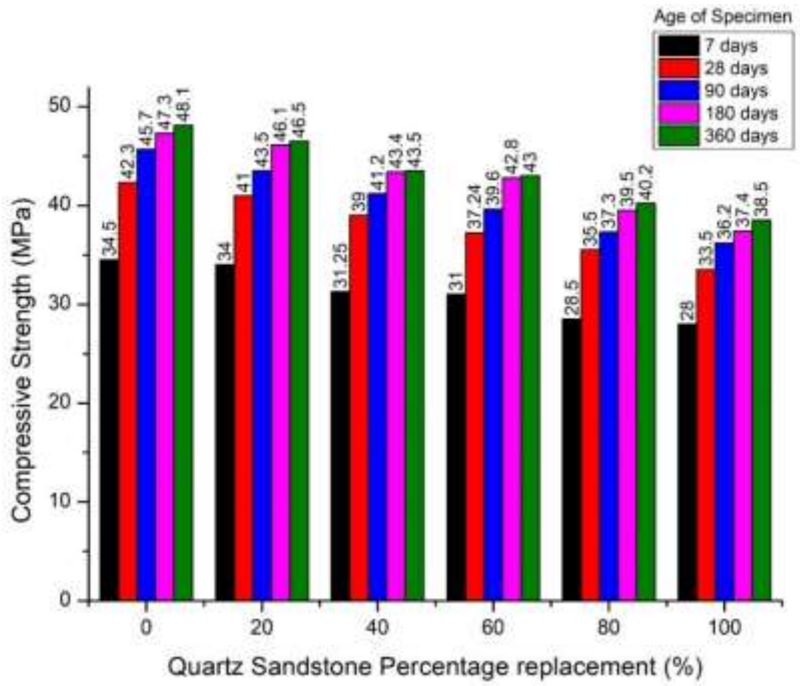

Fig 4.4: Compressive Strength of Specimens with w/c 0.45

A significant strength gain was attained at 180 days and 360days of water curing. A maximum strength of 49.5 Mpa was observed for the control concrete at w/c 0.35 and a minimum of 36.5 Mpa was observed for concrete with 100% Dholpur stone at w/c 0.45. The rate of increase in the strength gain reduced as the Dholpur stone (quartz sandstone) substitution increased in the mixes due to the porous nature of the sandstone as compared to that of natural aggregates.

Fig 4.5. Broken samples after compressive strength testing

4.3.3 Flexural Tensile Strength

Figures 4.6-4.8 show the variation in resistance to flexure observed at 7, 28, 90, 180 and 360 days with variation to the replacement percentage of Dholpur stone (Quartz sandstone). The test results showed a decrease in flexural strength as the quartz sandstone was added in concrete. All the samples showed a continuous decrease in trend pattern as the Dholpur stone (quartz sandstone) replacement increased. It was also observed that the decrease in trend was comparable until 40% replacement level in all the water/cement ratios. At 60%, 80% and 100% replacements, the trend pattern showed a major decrease in flexural strength.

A continuous decrease in strength plots was recorded when the percentage of Dholpur stone (quartz sandstone) was increased. At 7days, a maximum flexural tensile strength of 4.47 Mpa was recorded for the control concrete at 0.35 w/c and a minimum of 3.35 Mpa was observed at replacement level at 0.45 w/c.

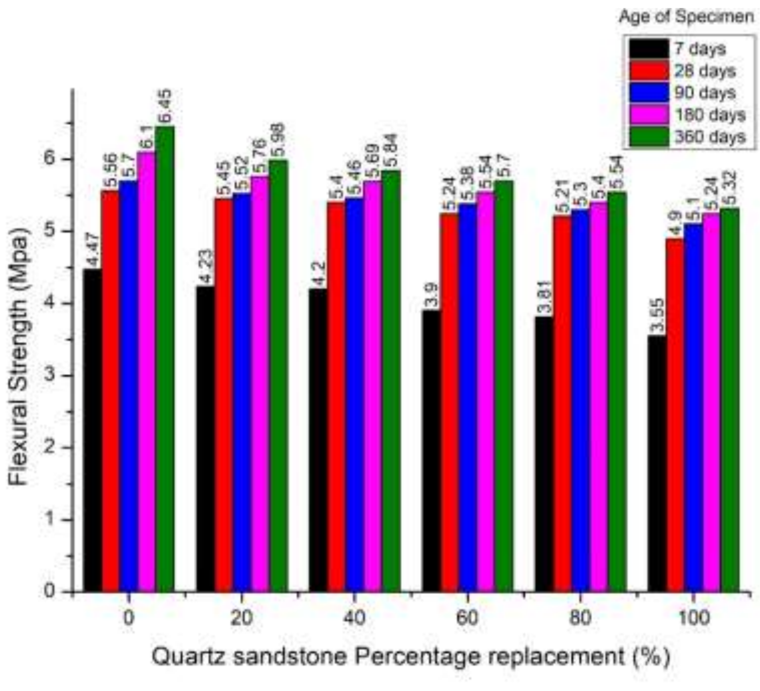

Fig 4.6: Flexural Tensile Strength of Specimens with w/c 0.35

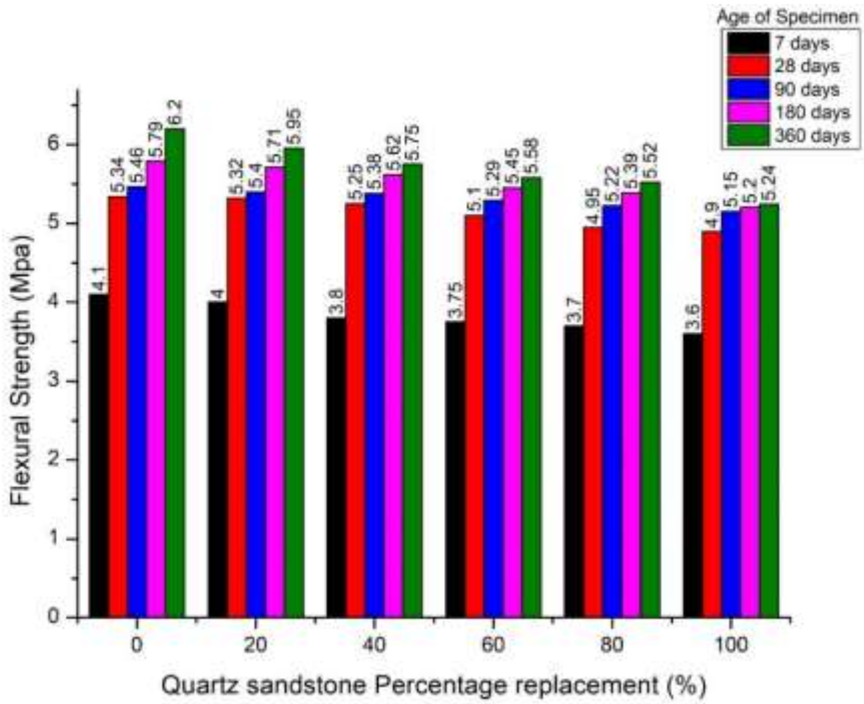

Fig 4.7: Flexural Tensile Strength of Specimens with w/c 0.40

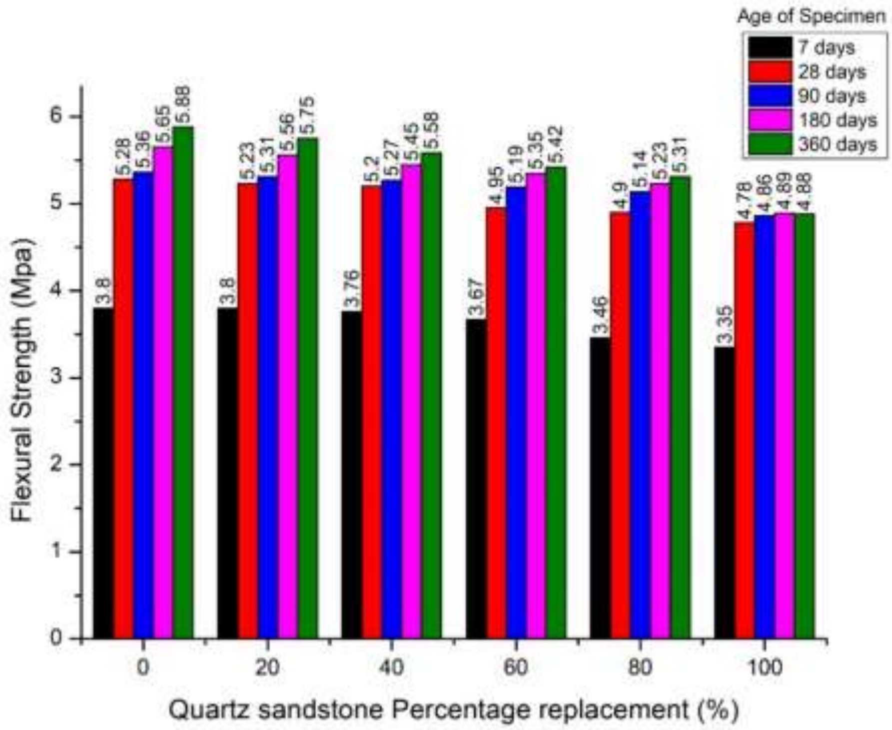

Fig 4.8: Flexural Tensile Strength of Specimens with w/c 0.45

At 28 days, flexural tensile strength reached its peak at 5.56 Mpa for the control mix at 0.35 w/c and a minimum of 4.78 Mpa was observed at replacement level at 0.45 w/c. At 90 days, a maximum flexural tensile strength of 5.7 Mpa was recorded for the control concrete at 0.35 w/c and a minimum of 4.86 Mpa was observed at replacement level at 0.45 w/c. At 180 days, a maximum resistance to flexure of 6.1 MPa was recorded for the control concrete at 0.35 w/c and a minimum of 4.89 Mpa was observed at replacement level at 0.45 w/c. At 360 days, the control concrete showed a maximum flexural tensile strength of 6.45 MPa at 0.35 w/c and a minimum of 4.88 MPa at 100% replacement level at 0.45 w/c.

The decreasing trend noticed in flexural strength results is due to the use of weaker tensile strength quartz sandstone for conventional aggregate thus resulting in the lower flexural strength of concrete. This was also observed by researchers like Yilmaz and Tugrul (2012); Mackechnie (2006). Figure 4.9 shows the arrangement and failure of test specimen under flexural loading. It was witnessed that the specimens with Dholpur stones exhibited brittle failure with a tad greater than the control specimens. As the replacement increased, the specimens failed by showing more cracks.

Fig 4.9: Arrangement and failure of test specimens under flexural loading

4.3.4 Abrasion Resistance

Deterioration of concrete can take place due to abrasion caused by various exposures such as rubbing, skidding or sliding of the object on the concrete surface. Figure 4.10 shows the variation in the depth of surface wear (abrasion) with respect to percentage of Dholpur stone (quartz sandstone). Resistance to abrasion decreased with the increase in the percentage of quartz sandstone aggregate irrespective of the water-cement ratios. This increase in abrasion depth is due to the higher proportion of sedimentary type coarse aggregate used i.e. Dholpur stone (quartz sandstone). As the replacement level increased, the depth of wear increased revealing the aggregate surfaces (Figure 4.11). The decrease in abrasion resistance may also be related to the decrease in compressive strength of samples containing quartz sandstone aggregates. The maximum abraded depth of 1.89 mm was obtained for w/c 0.45 at 100% replacement level.

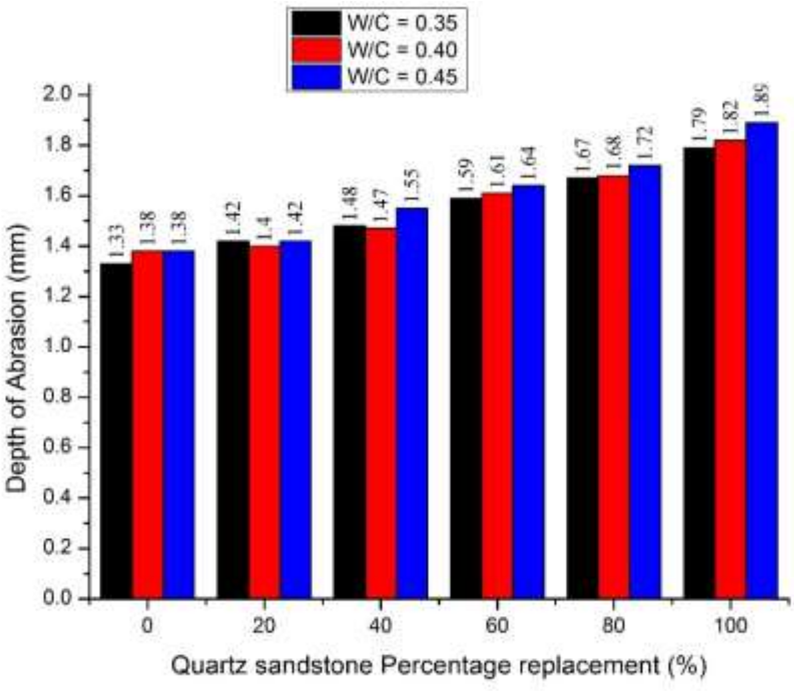

Figure 4.10: Abrasion resistance of specimens for all the three series

Figure 4.11: Abraded surface of concrete having quartz sandstone for various replacement levels

Table 4.2: Permissible Depth of Wear as per IS: 1237-1980

No	Type	Depth of wear
1	General Purpose Tiles- tiles used in floorings subjected to light loads such as office buildings, schools, colleges, hospitals and residential buildings. (i) Average wear (ii) Wear on Individual sections	 3.5 mm 4.0 mm
2	Heavy duty floor tile- tiles used in heavy traffic conditions, such as footpaths, entrances and staircases of public buildings, passages of auditoriums and storage godowns. (i) Average wear (ii) Wear on Individual sections	 2.0 mm 2.5 mm

The permissible values of depth of wear for heavy duty tiles and general purpose tiles as mentioned by IS: 1237-1980 are shown in Table 4.2. As all the observed values were less than 2 mm, quartz sandstone can be used to produce concrete tiles for heavy duty floors as well. To develop concrete for high abrasion resistance, it is desirable to use a hard surface material, aggregate, and paste with low porosity and high strength. As the quartz sandstones are not as hard as natural aggregate and the additional dosages of them may have resulted from an increase in porosity of the concrete samples. Thence an increase in wear was observed as the dosage of quartz sandstone aggregate increased for all the water-cement ratios.

4.3.5 Pull-Off Strength

The pull off strength test for the entire series of concrete specimens were done after 28 days of curing. The results are shown in Figure 4.12.

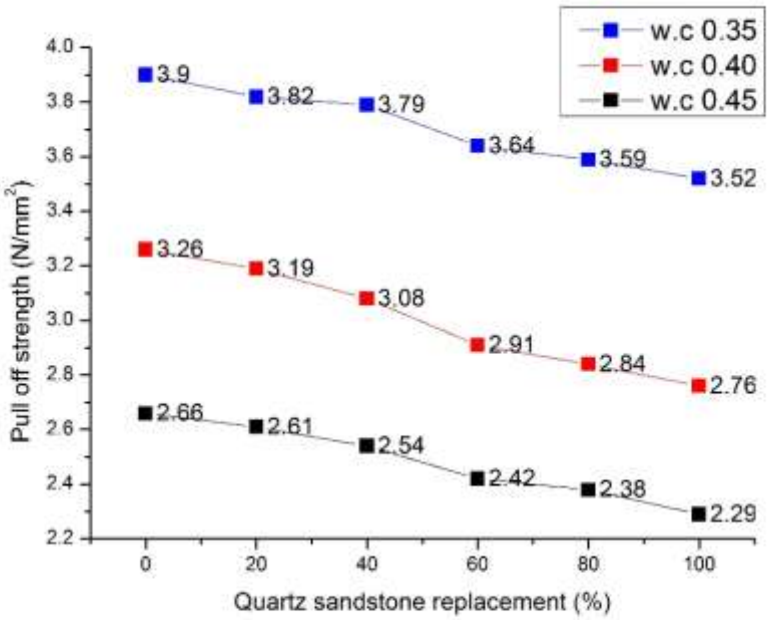

Fig.4.12: The pull-off strength of specimens for all the three series

A peak strength of 3.9 MPa was observed for the series I control mix 0.35. A steady reduction in pull-off strength results was recorded as the Dholpur stone (quartz sandstone) percentage of substitution increased. Almost a same trend of strength variation was recorded in the other mix-designs with of 0.40 and 0.45 water-cement ratios. A lowest pull-off strength of 2.29 MPa was recorded for the concrete mix with 100% Dholpur stone (quartz sandstone) replacement at 0.45 water-cement ratio.

It is quite evident that the pull-strength plots followed the trend of compressive strength. This phenomenon was also observed by researchers like Thomas et al., (2015) and Pereira and Medeiros (2012). Both the researchers have noticed a similar trend pattern of compressive and pull-off strength.

4.4 Durability Properties

The results of durability properties of concrete incorporating different replacements of Dholpur aggregates (quartz sandstones) are discussed in this article.

4.4.1 Water Absorption

The results of normal and Sorptivity (i.e. capillary water absorption) are shown below:

4.4.1.1 Normal Water Absorption

The water absorption of oven dried samples (after cooling to room temperature) were observed after 48 hours of submergence in water. The results showing the variation in the values are reported in Figure 4.13.

In series I, absorption of water by control mix was 1.12% while the absorption of specimens with 100% Dholpur stone (quartz sandstones) was 1.72%. The water absorption of concrete samples had an increasing trend as the substitution level increased. For series II, the water absorption of control mix was about 1.15% and for the mix with 100% quartz sandstones it was 1.82%. While for the specimens with water-cement ratio of 0.45, control mix recorded a water absorption value of 1.17% and for 100% substitution level it was 1.86%.

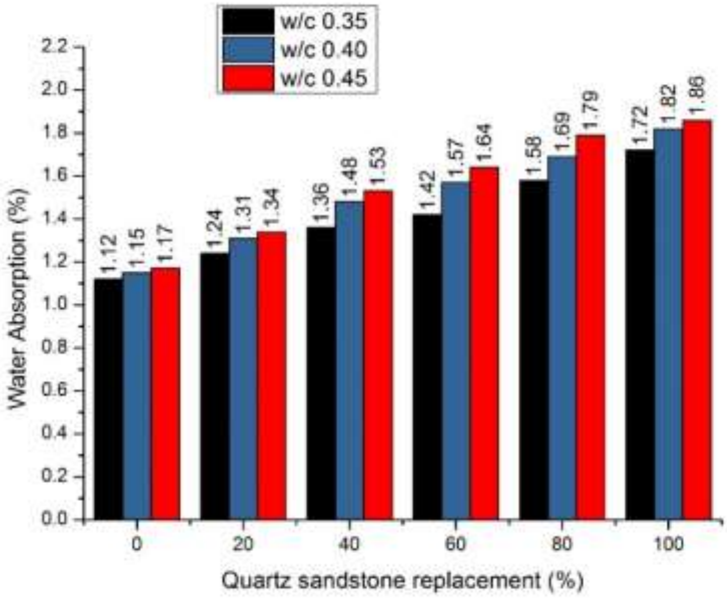

Figure 4.13: Water absorption of specimens with w/c 0.35, 0.40 and 0.45

The quantity of water absorbed by the specimens was greater in all the concrete mixes in comparison to control concrete made solely from conventional coarse aggregates irrespective of water cement ratios. This increase in water absorption in all the mixes may be due to the excessive micro voids as the sandstones were added. With the increase in water-cement ratio, the internal voids might have increased resulting in a gradual increase in the absorption values. The other reason can be inherited from the formation process of sandstone itself (i.e. sedimentation) which results in voids within the material.

4.4.1.2 Water Absorption by Sorptivity

The test results showed an increase in absorption depth as the conventional aggregates were replaced with quartz sandstone aggregates and a maximum of 0.46 mm was observed at 100% replacement level for 0.45 water/cement ratio (Figure 4.14). This increase in absorption may be related to the use of a sedimentary type of stone as a replacement of natural aggregate. Also, the increase in water/cement ratio with an increase in quartz sandstone replacement could have resulted in interconnectivity of small pores making the concrete more permeable.

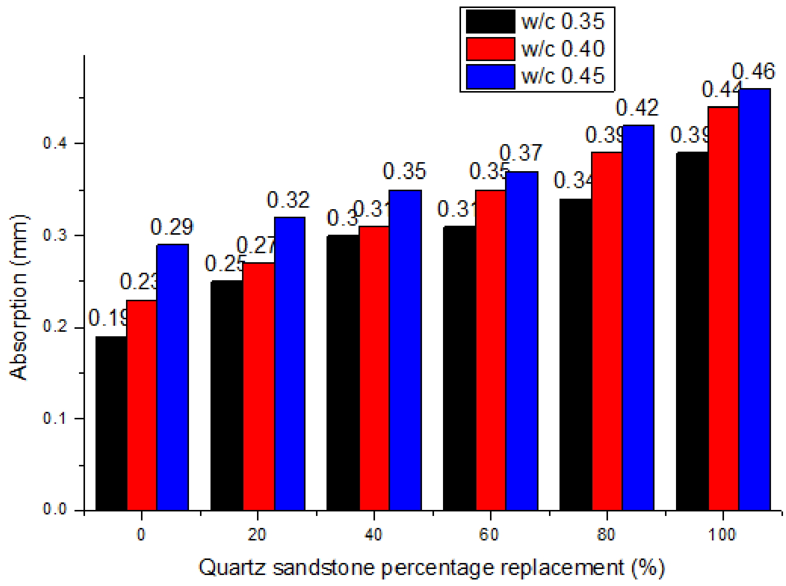

Figure 4.14: Water absorption (Sorptivity) of concrete containing quartz sandstone aggregate.

4.4.2 Water Permeability as per DIN 1048

From the test results given in Figure 4.15, depth of water penetration increased with increase in Dholpur stone (quartz sandstone) content irrespective of the water/cement ratio. For all the concrete mixes, the maximum depth of penetration was observed at 100% substitution of Dholpur stone (quartz sandstone). The maximum depth of water penetration observed was about 64 mm for water-cement ratio of 0.45 and a minimum of 9 mm was observed for control mix at 0.35 water-cement ratio.

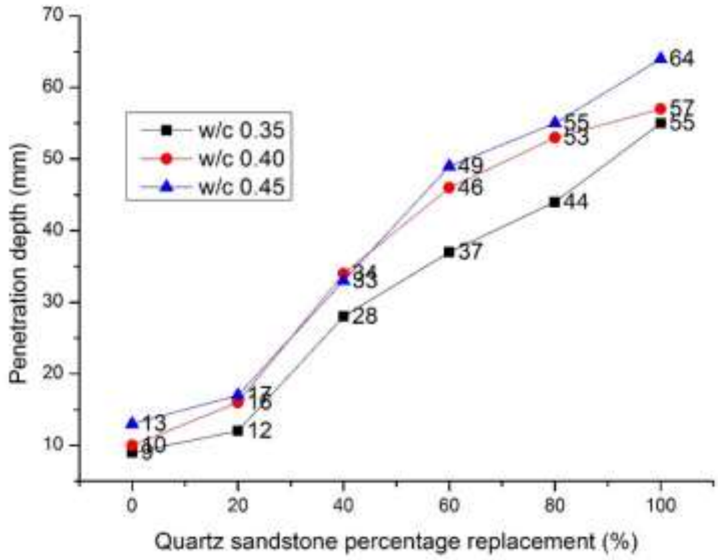

Figure 4.15a: Water Penetration of specimen for the entire three series

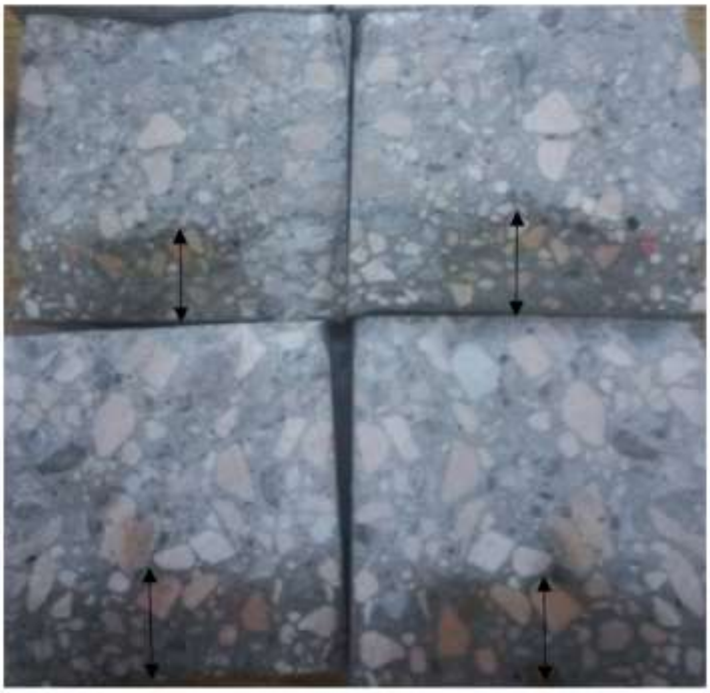

Figure 4.15b: Measurement of Depth of Water Penetration

This increase in penetration depth is due to the higher proportion of quartz sandstone aggregate used which absorbs more water when compared to conventional coarse aggregates. The increase in the intensity of micro cracks at the aggregate-cement paste interface may also have resulted in the increase in permeation as the quartz sandstone replacement was increased.

4.4.3 Carbonation Resistance

Figures 4.16-4.18 shows the results of carbonation resistance of concrete with water-cement ratios 0.35, 0.40 and 0.45. The concrete specimens showing the depth of carbonation is reported in Figure 4.19.

For series I, an improved resistance to carbonation penetration was recorded for concrete mixes with up to a 40% substitution level of sandstone aggregates. Carbonation had occurred till a maximum depth of 34 mm (90 days) was recorded for samples with complete replacement of conventional aggregates for series III whereas maximum resistance to carbonation was measured (a penetration value of 19 mm 90 days) for samples with 40% quartz sandstone replacement for series I. The depth of carbonation at 40% replacement of quartz sandstone was found to be least till 56 days for series I. However, a clear trend was not observed for series II and III. Resistance to carbonation was found to be decreasing marginally at 40% substitution of quartz sandstone as the water-cement ratio was increased. At 90 days, concrete mixes with 20% and 40% substitution by quartz sandstone showed a similar variation in carbonation depth. However, at a greater water-cement ratio (0.45), resistance to carbonation was found to decreasing at 40% substitution. The improved gradation of sandstones could be responsible for the initial decrease of carbonation depth. But the higher replacement of coarse aggregate (above 40%) and increased water-cement ratios (0.40 and 0.45) in the mixes, could have led to the widening of pore sizes in concrete thereby increasing the carbonation depth.

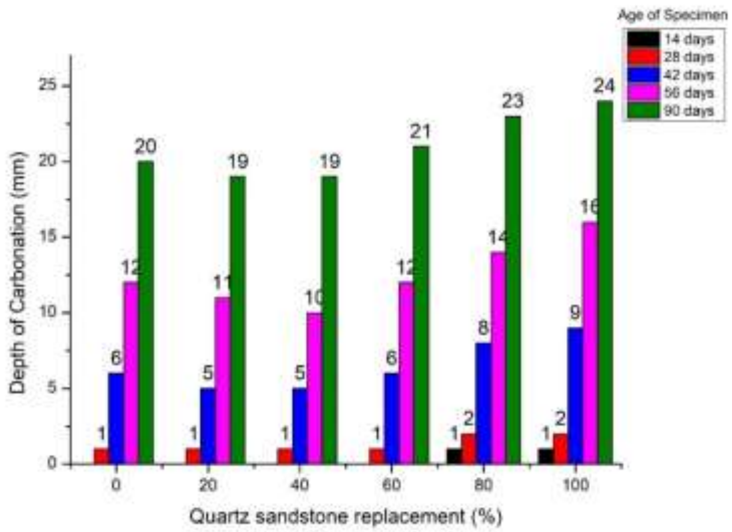

Figure 4.16: The depth of carbonation of specimens with water-cement ratio 0.35

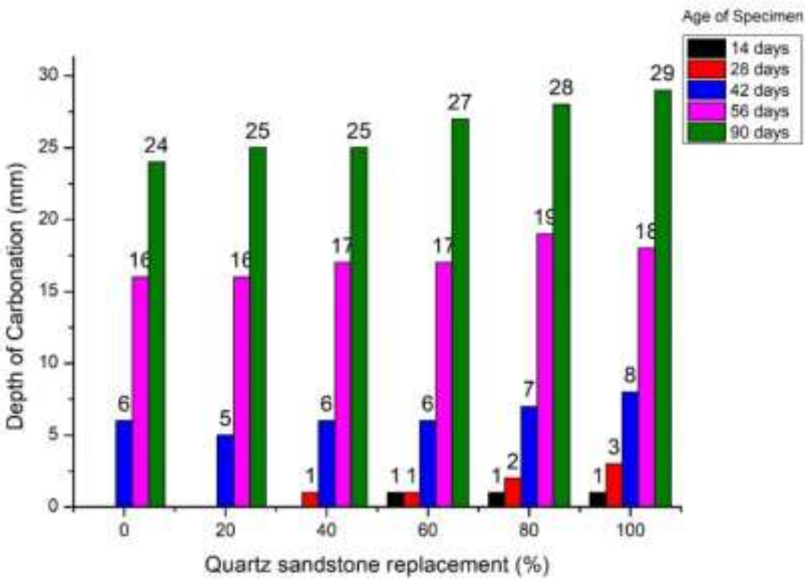

Figure 4.17: The depth of carbonation of specimens with water-cement ratio 0.40

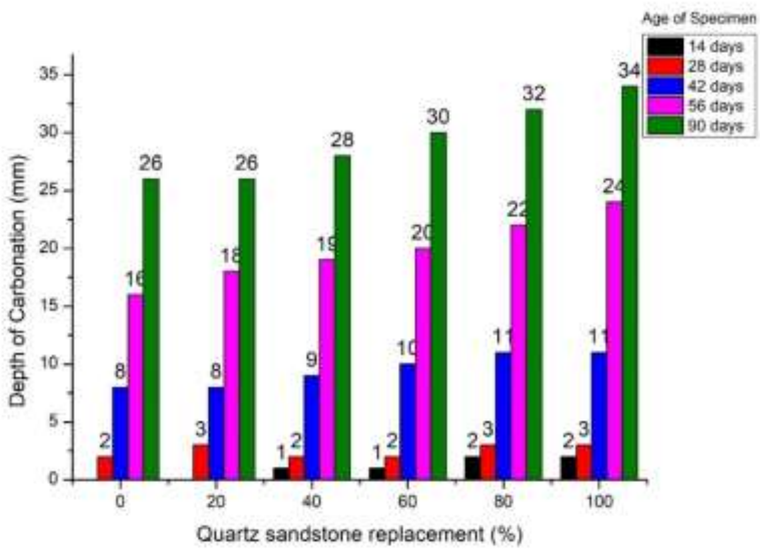

Figure 4.18: The depth of carbonation of specimens with water-cement ratio 0.45

There is quite a lack of literature which compares the carbonation depth of sandstones incorporated into concrete. However, researchers like Miguel and Jorge (2012) have studied carbonation resistance of concrete containing waste rubber tires. He found an increase in carbonation depth as the substitution of rubber was increased in concrete. Thomas (Thomas et al., 2015) found a decreasing depth till 12.5% substitution of crumb rubber following an improved gradation technique.

He found that the closely packed rubber aggregates could have prevented the entry of CO_2 gas into the concrete. A similar trend was also noticed for the concrete containing quartz sandstones. The proper gradation of quartz aggregates as described in the previous studies (Kumar et al., 2016) might have decreased the carbonation depth upto 40% substitution of quartz sandstones at lower water-cement ratio. Also, some of the research works in SCC mixes containing recycled aggregates along with low volume fly ash and high volume fly ash yielded a remarkably good carbonation resistance than normal concrete mixes (Singh & Singh, 2016a, 2016b).

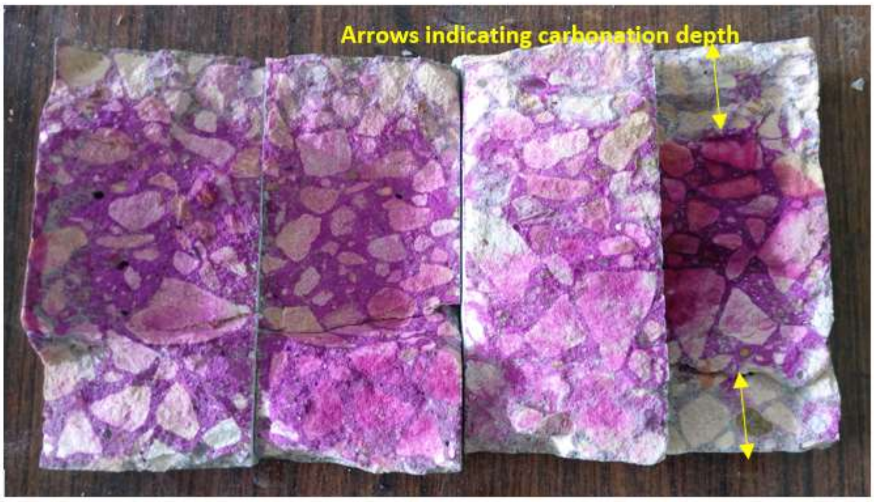

Figure 4.19: Carbonation depth of specimens with Dholpur stone (quartz sandstone)

Some of the mixes did not show any carbonation at 14 days of exposure (control concrete and 20% quartz sandstone concrete at 0.35 w/c). The carbonation coefficient calculated for the series with water-cement ratios 0.35, 0.40 and 0.45 are shown in Figures 4.20-4.22.

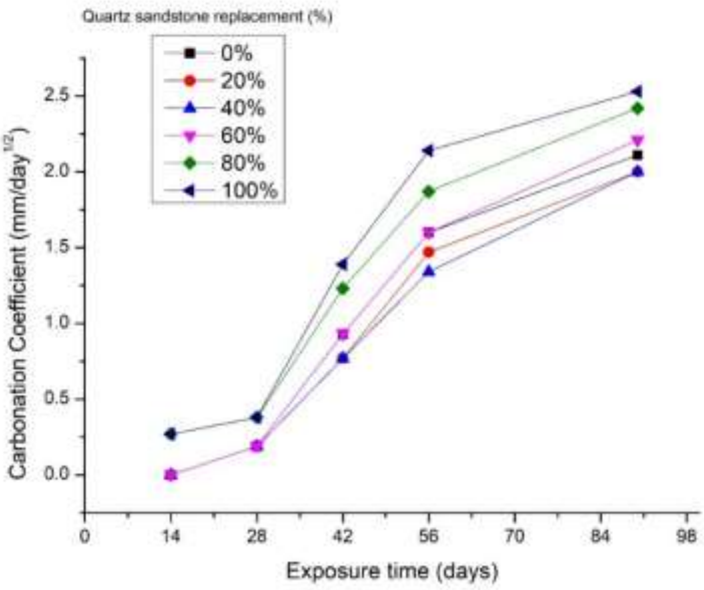

Figure 4.20: Carbonation Coefficient for the series with w/c 0.35

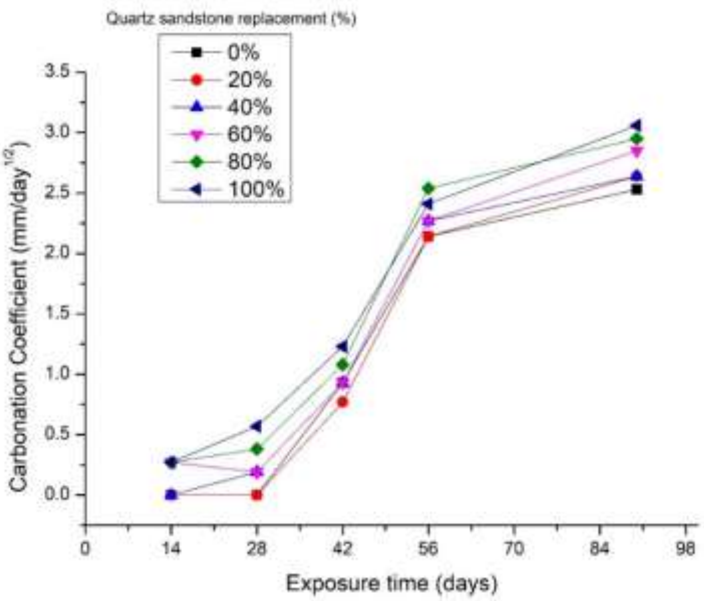

Figure 4.21: Carbonation Coefficient for the series with w/c 0.40

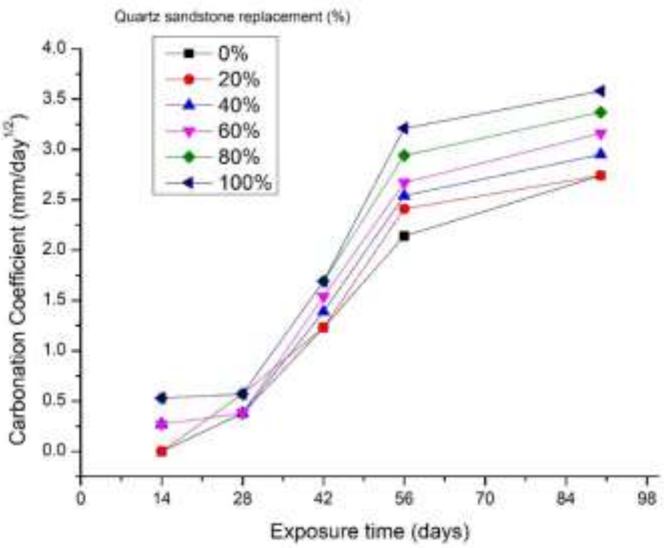

Figure 4.22: Carbonation Coefficient for the series with w/c 0.45

In the series with water-cement 0.35, the signs of carbonation started showing by 28 days of testing. The increase in carbonation was gradual and almost equal for all the replacement levels at 14 days at 0.35 and 0.40 water-cement ratio. However at the same water-cement ratio, the carbonation coefficient showed an increasing trend as the days of exposure increased. The substitution levels of 20% and 40% at 0.35 w/c showed a similar trend of increasing pattern. Similarly, the substitution levels of 40% and 60% at 0.40 w/c showed an identical increase expect the 90 days exposure. However, a clear increasing trend of carbonation coefficient was noted down at 0.45 water-cement ratio.

Results indicated that the concrete mixes with lower w/c ratios offered better resistance to carbonation. The rate of increase in carbonation was marginal till 40% substitution level at lower water-cement ratios. However, at higher replacement levels (above 40%) the carbonation depth increased. The resistance to carbonation decreased as the percentage of Dholpur stone (quartz sandstone) increased.

4.4.4 Chloride Ion Penetration

The graphs showing the change in the depth of penetration of chloride ions with respect to the change in percentage of Dholpur stone (quartz sandstone) are given in Figures 4.23-4.25.

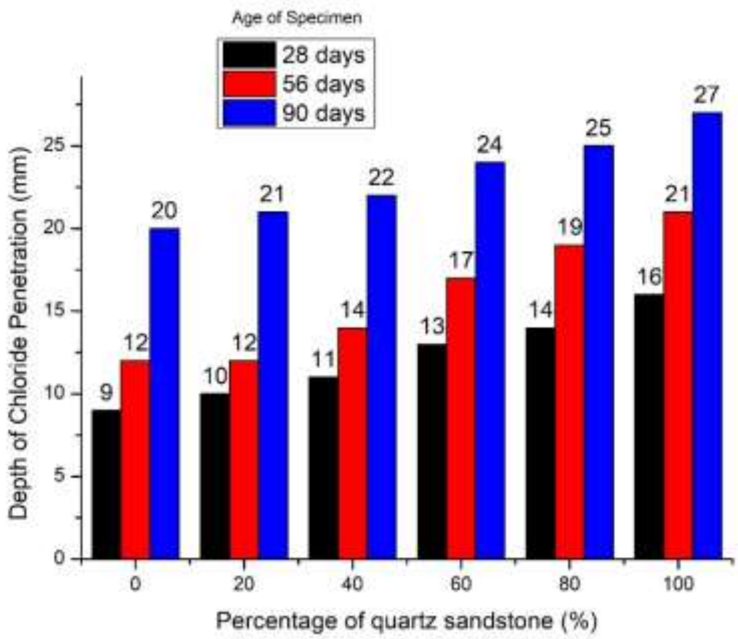

Figure 4.23: Chloride Ion Penetration of concrete mixes with w/c 0.35

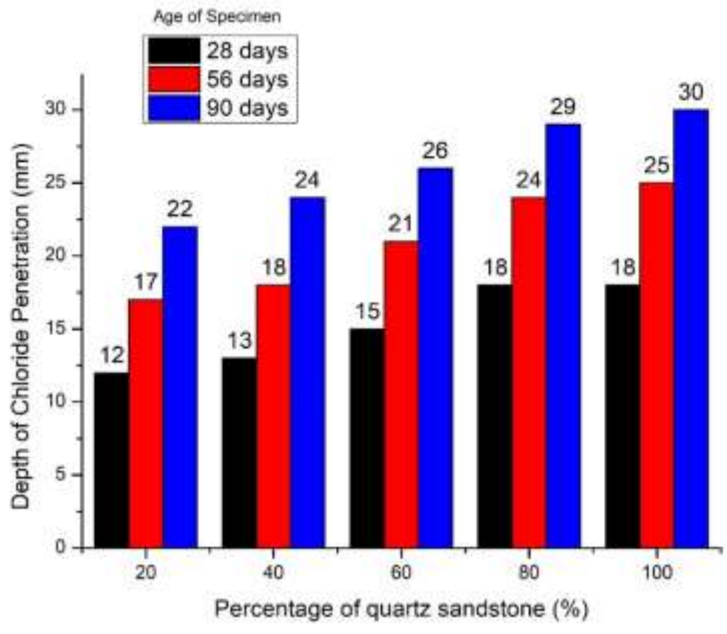

Figure 4.24: Chloride Ion Penetration of concrete mixes with w/c 0.40

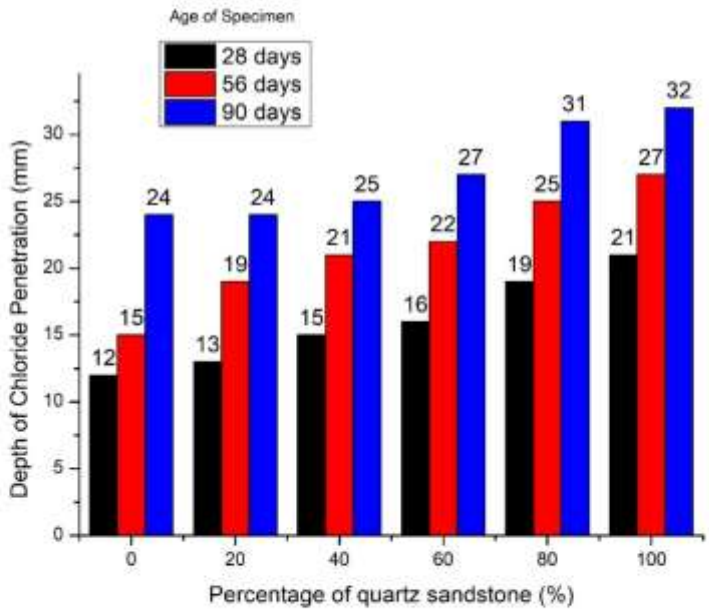

Figure 4.25: Chloride Ion Penetration of concrete mixes with w/c 0.45

Figure 4.26: Spraying of 0.1N Silver Nitrate Solution on freshly split concrete specimens

Figure 4.27: Freshly Split Concrete Specimens showing depth of Chloride Penetration after 28 days (Arrows indicate the depth of chloride penetration)

The chloride ion content in concrete produces an acceleration in cement setting and early hardening of concrete. However long term studies have shown contradictory results and negative influences on the durability properties in concrete (Etxeberria et al., 2016).

While considering the concrete mix with water-cement ratio of 0.35, the depth of penetration of chloride ions increased as the dosage of Dholpur stone (quartz sandstone) increased. The chloride penetration of the control concrete tested at various days of exposure were similar to that concrete with 20% quartz sandstone. A gradual increase in chloride penetration was noticed in the specimens beyond 20% replacement. At 90 days, the penetration depth was 20 mm for control concrete and 27 mm for concrete mix with 100% quartz sandstone. For series II, a similar gradual increase of penetration was observed as the quartz sandstone dosage increased. At 90 days, the control concrete recorded a penetration of 22 mm and the concrete with 100% quartz sandstone showed 30 mm penetration. The concrete specimens with 0.45 water-cement ratio showed a similar increasing trend of penetration depth. However at 90 days, the control concrete and 20% quartz sandstone concrete showed a depth of penetration of 24 mm. A maximum depth of penetration of 32 mm was recorded for the concrete with 100% quartz sandstone at 0.45 w/c. The gradual increase in chloride penetration can be related to reduced packing capacity of concrete as the quartz sandstone replacement increased. However at lower water cement ratios (0.35 and 0.45), control concrete and concrete with 20% substitution showed a similar penetration depth.

4.4.5 Acid Attack

For testing the concrete mixes resistance to acid attack, the specimens were immersed in dilute sulphuric acid for a period of 90 days and the following tests were carried out.

4.4.5.1 Reduction in Compressive Strength of Acid Attacked Specimens

The compressive strength of specimens subjected to attack by acid were recorded at 28, 56 and 90 days of immersion and the results were related with the compressive strength of non-acid attacked specimen at 28 days of curing. The amount of loss was calculated and expressed in percentage in the graphs given in Figures 4.28-4.30.

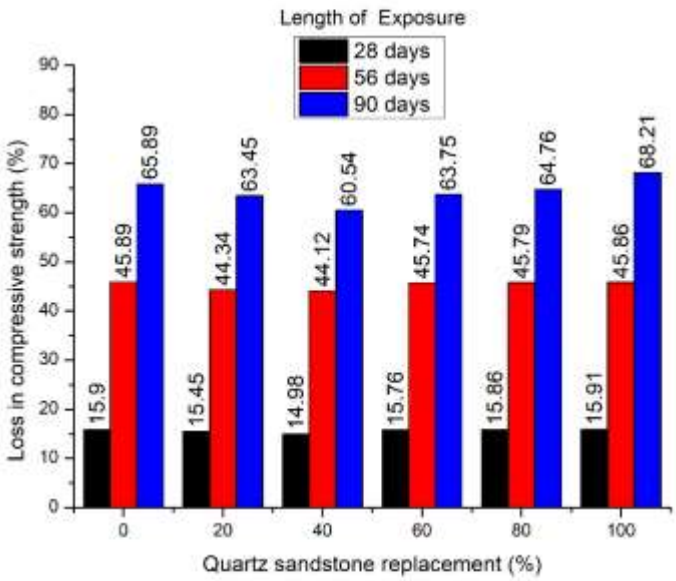

Figure 4.28: Reduction in compressive strength of acid attacked specimens with w/c 0.35

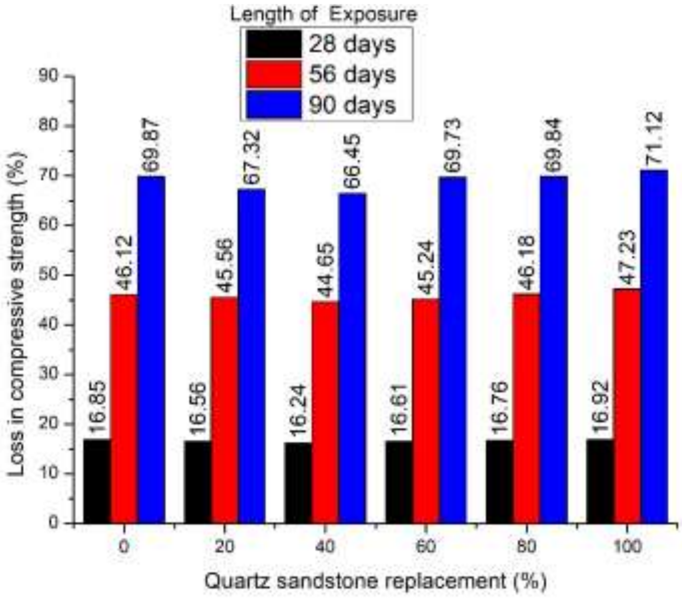

Figure 4.29: Reduction in compressive strength of acid attacked specimens with w/c 0.40

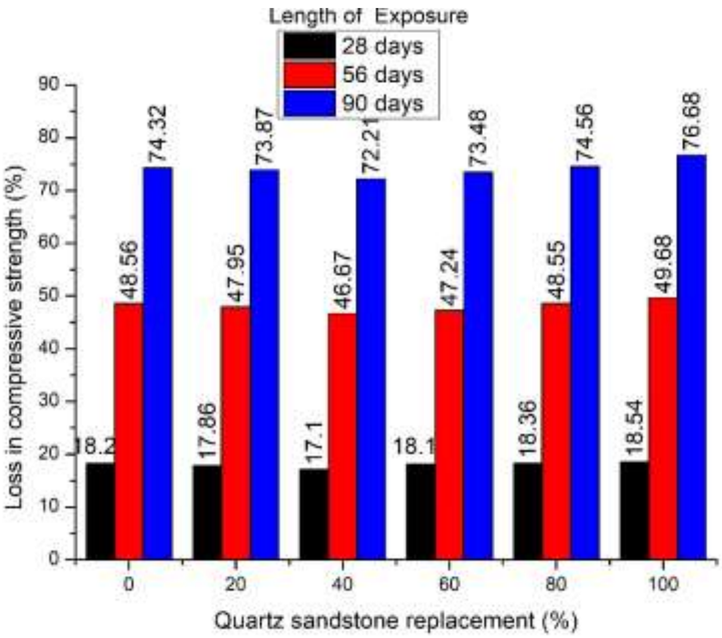

Figure 4.30: Reduction in compressive strength of acid attacked specimens with w/c 0.45

There was considerable reduction in resistance to compression in all the concrete samples irrespective of the quartz sandstone replacement and water-cement ratios. On comparing the reduction in control concrete's compressive strength; there were observed a reduction in loss till 40% quartz sandstone addition in all the concrete mixes with water cement ratios of 0.35, 0.40 and 0.45. However for mixes at 0.35 and 0.40, this loss in compressive strength of concrete samples was less than control concrete samples up to 80% substitution of quartz sandstone aggregates. At 0.45 water-cement ratio, the maximum loss in compressive strength of acid attacked specimen was observed at 76.68% at 100% replacement of quartz sandstone. Minimum loss of 44.1% with 0.35 water-cement ratio was recorded at 40% substitution of quartz sandstone aggregates.

The loss in compressive strength pattern upon sulphuric acid was found to be similar to that of mass loss model. Sandstone aggregates being siliceous in nature, resistance against acid attack is expected to better since quartz being damaged at a lower rate. The reason for an increase in compressive strength loss can be due to increased porosity at higher replacement levels (≥60%) of quartz sandstone. The other reason can be inherited due to alumina content (as observed by XRF) if not practically inert like corundum or mullite, can promote ettringite formation if all other required components (Ca, SO_4 and H_2O) are present.

Figure 4.31: Image of acid attacked specimens after 28 days of acid immersion

Figure 4.32: Acid attacked specimen at 90 days. Control concrete (left) and 100% (right) quartz sandstone.

The figures 4.31 and 4.32 shows the images of specimens subjected to acid attack at 28, 56 and 90 days of acid attack exposure. It was noticed that the particles in the concrete samples after 40% replacement were breaking away by material separation as the duration of exposure increased. This breaking away might be the primary cause for the reduced loss in compressive strength upto 40% replacement of Dholpur stone (quartz sandstone).

4.4.5.2 Variation in Weight of Acid Attacked Specimens

The weight of acid attacked specimens were calculated at 28, 56 and 90 days of immersion and the results were compared with that of non-acid attacked after 28 days of water curing. The amount of loss in weight of concrete samples were calculated and expressed in percentages in the graphs shown in Figures 4.33-4.35.

In all the design mixes, more weight loss was recorded in the control combinations and it was found that the weight loss decreased in concrete containing quartz sandstone aggregates up to 40% replacement. Maximum weight loss of 8.42 % was recorded at 100% replacement for series I. Measured weight loss in control concrete was similar to that of concrete with 80% quartz sandstone coarse aggregates. Minimum loss of 2.87% was recorded at 40% replacement for the water-cement ratio of 0.35.

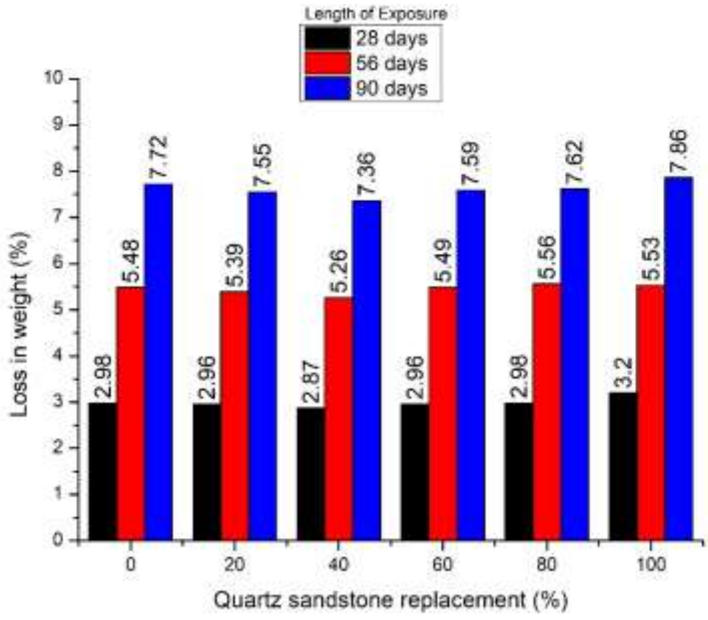

Figure 4.33: Weight loss (%) of Acid attacked specimens, water-cement ratio 0.35

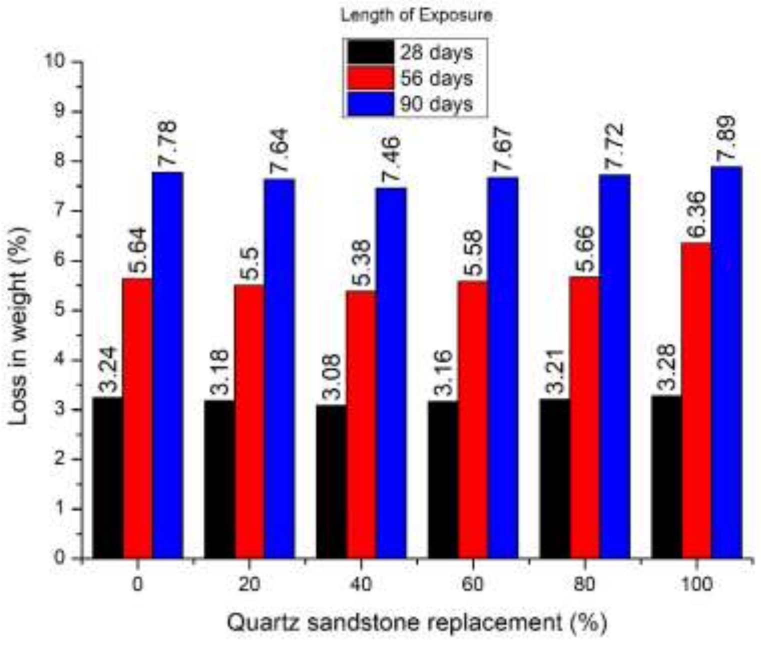

Figure 4.34: Weight loss (%) of Acid attacked specimens, water-cement ratio 0.40

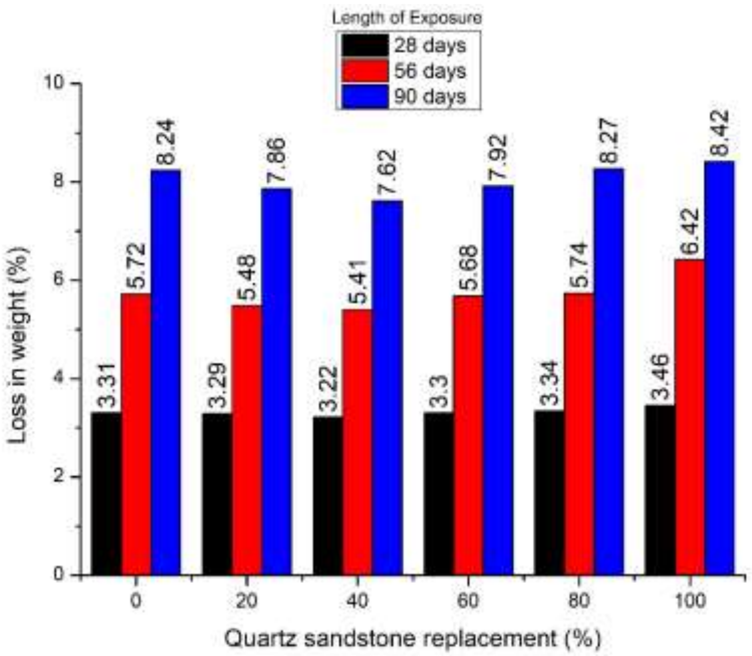

Figure 4.35: Weight loss of Acid attacked specimens, water-cement ratio 0.45

The resistance of sandstone concrete against concrete sulphuric acid attack may be due to highly siliceous nature of sandstones as quartz is being damaged by acid at a much slower rate than hydrated cement stone. However, this decrease in mass loss was observed only till 40% replacement level due to increasing in micro voids and porosity of concrete samples at higher replacement levels of quartz sandstone.

4.4.5.3 Water Absorption of Acid Attacked Specimens

Figures 4.36-4.38 shows the variation in the amount of water absorbed by acid attacked specimens (expressed in percentage) with respect to amount of Dholpur stone (quartz sandstone) added.

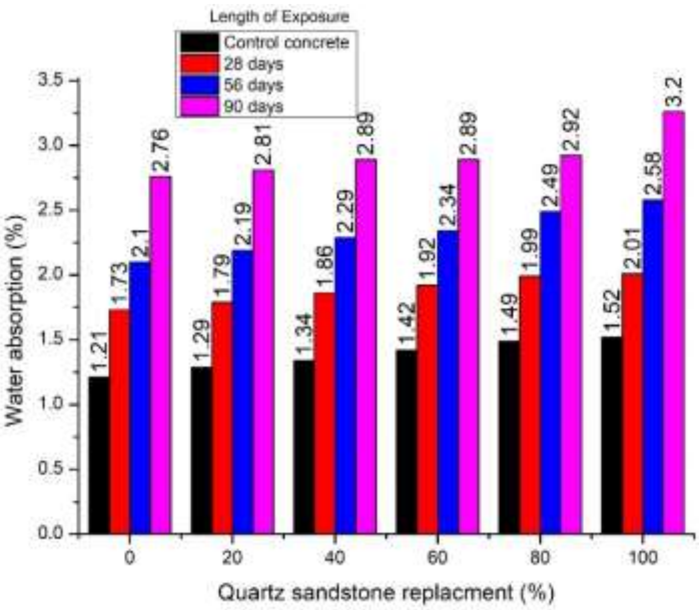

Figure 4.36: Water absorption of Acid attacked specimens, water-cement ratio 0.35

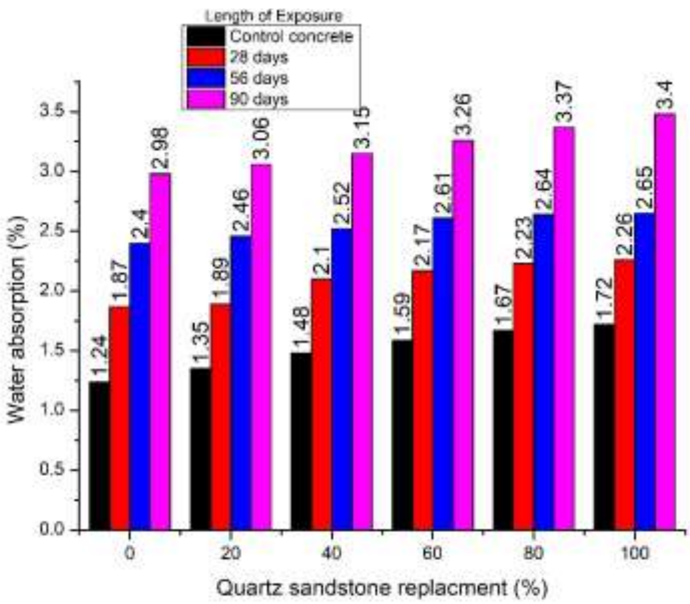

Figure 4.37: Water absorption of Acid attacked specimens, water-cement ratio 0.40

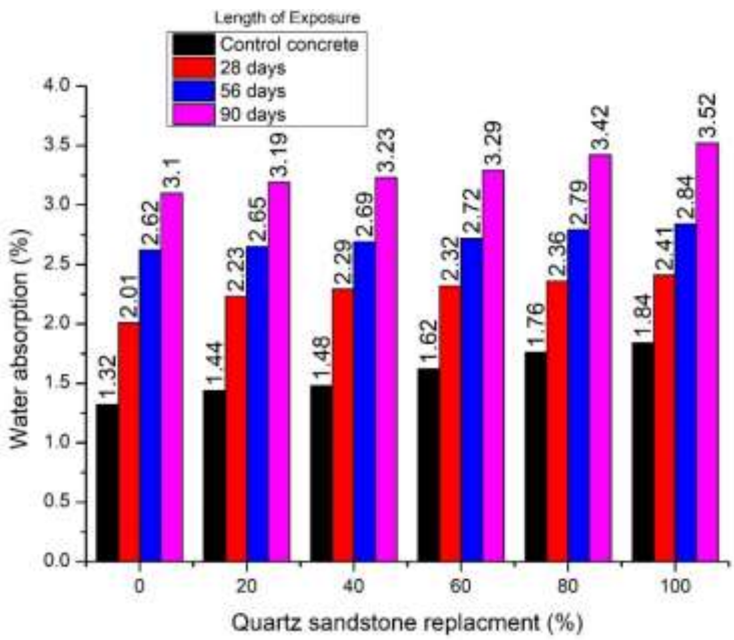

Figure 4.38: Water absorption of Acid attacked specimens, water-cement ratio 0.45

For the concrete mixes with water cement ratio of 0.35; gradual increase in the amount of water absorbed was noticed at 28 days of acid attack when quartz sandstone coarse aggregates were replaced. A similar trend was noted at 56 days and 90 days. At 90 days, the water absorption of control concrete was 2.76%, for the mix with 60% quartz sandstone it was 2.89% and for 100% quartz sandstone mix it was 3.26%. A similar pattern of increasing water absorption values was observed in mixes of 0.40 and 0.45 water-cement ratios.

The amount of water absorbed by all concrete mix specimens increased in comparison to control concrete made solely from conventional coarse aggregates irrespective of water cement ratios and age. This increase in water absorption in all the mixes may be due to the excessive micro voids as the sandstones were added. With increase in water-cement ratio, the internal voids might have increased resulting in a gradual increase in the absorption values. The other reason can be inherited from the formation process of sandstone itself (i.e. sedimentation) which results in voids within the material.

Visual examination of affected samples (Figure 4.32) showed that the top face was completely removed exposing the aggregates of control concrete. However, this removal of the top layer was less in concrete having 100% quartz sandstone. Discolouration of samples was also noticed on the specimens with quartz sandstones which were due to the colour of sandstones itself. On continuous exposure of samples against sulphuric acid, the top layer of concrete specimens was severely eroded revealing the sedimentary stones which increased the water absorption.

4.4.5.4 X-ray diffraction observation of acid attacked specimen

Development of compact, lightweight, rugged, portable, and economically viable equipment for implementation of some analytical chemistry methods such as XRD has facilitated their growing semi destructive or non-destructive field applications (Peyvandi et al., 2015). Inorder to establish the formation of ettringite by XRD, 3-4 grams of powdered representative samples were taken from acid attacked specimens. The selection of specimens was based on shortest and longest exposed to magnesium sulphate solution. A pair of control concrete (0% quartz sandstone) and 100% quartz sandstone concrete at 28 days and 90 days were taken for the study. To attain a clear understanding a pair from halfway (i.e. 60% quartz sandstone concrete) were also included. A28 and A90 denote control concrete at 28 and 90 days exposure to sulphuric acid attack. Similarly, D28 and D90 denotes concrete with 60% quartz sandstone at 28 days and 90 days exposure. F28 and F90 denote concrete with 100% quartz sandstone at 28 days and 90 days exposure to sulphuric acid.

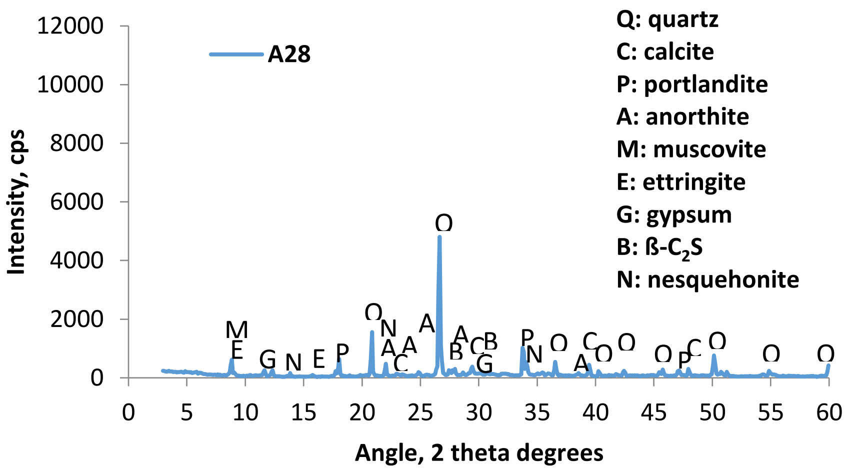

Figure 4.39: X-ray diffractogram of concrete after sulphuric acid exposure. (A28: Control concrete at 28 days exposure)

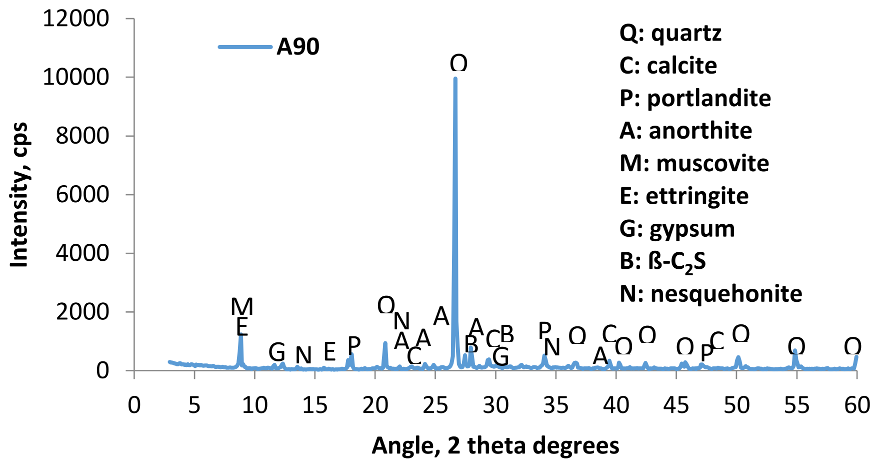

Figure 4.40: X-ray diffractogram of concrete after sulphuric acid exposure. (A90: Control concrete at 90 days exposure)

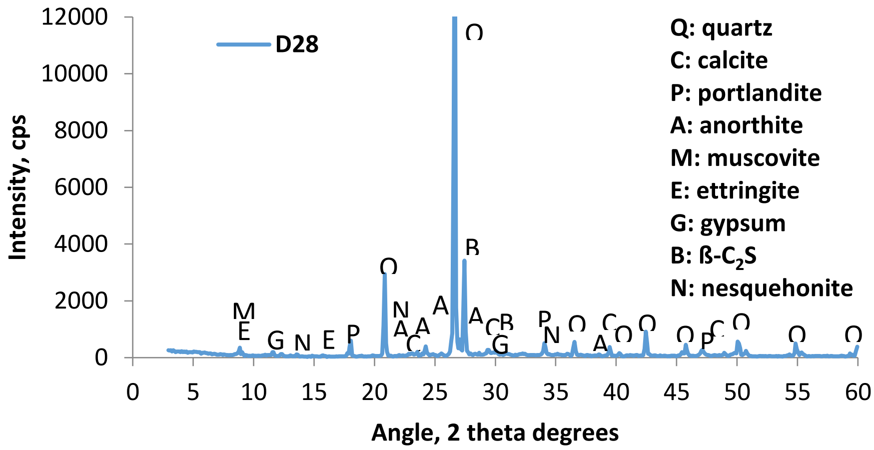

Figure 4.41: X-ray diffractogram of concrete after sulphuric acid exposure. (D28: Concrete with 60% Dholpur/quartz sandstone at 28 days exposure)

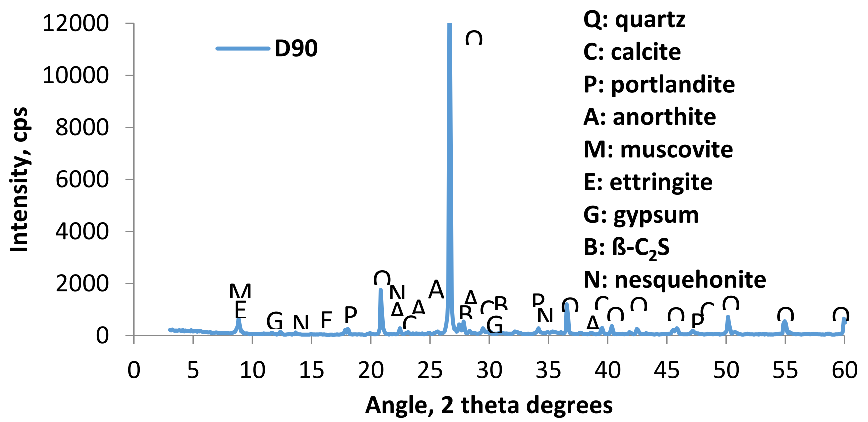

Figure 4.42: X-ray diffractogram of concrete after sulphuric acid exposure. (D90: Concrete with 60% Dholpur/quartz sandstone at 90 days exposure)

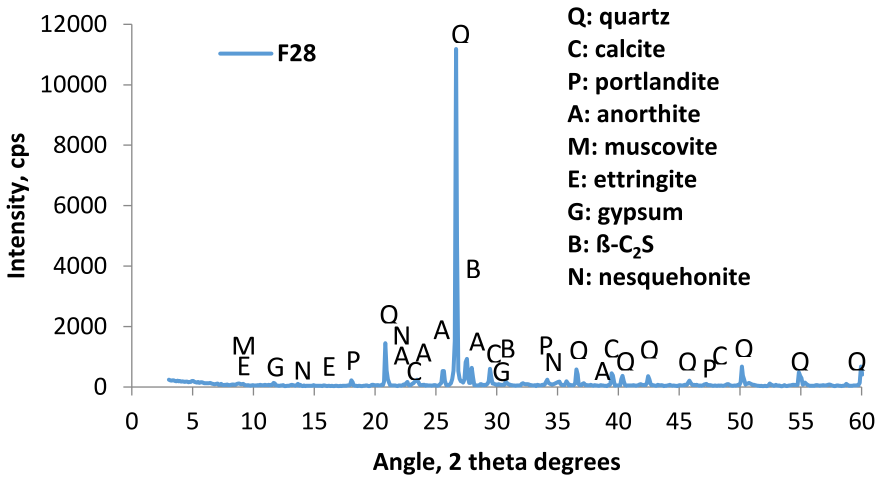

Figure 4.43: X-ray diffractogram of concrete after sulphuric acid exposure. (F28: Concrete with 100% Dholpur/quartz sandstone at 28 days exposure)

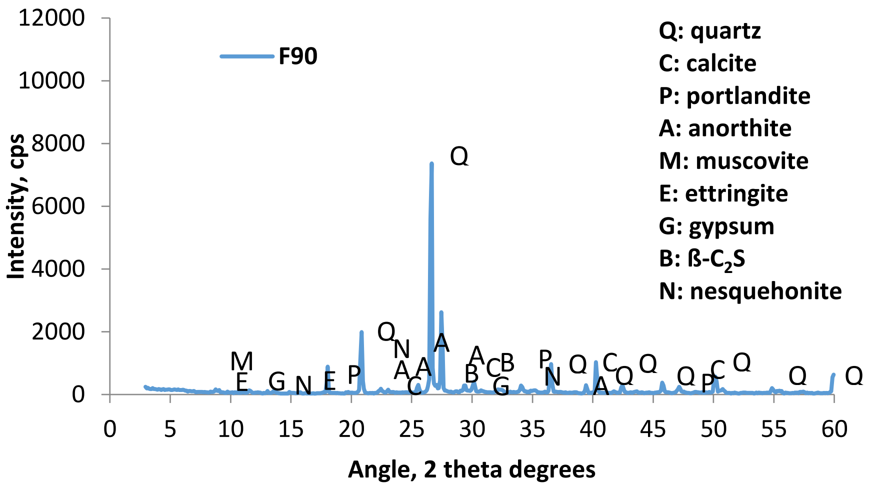

Figure 4.44: X-ray diffractogram of concrete after sulphuric acid exposure. (F90: Concrete with 100% Dholpur/quartz sandstone at 90 days exposure)

X-ray diffractogram revealed the presence of ettringite and also nesquehonite ($MgCO_3.3H_2O$) which can be related to the magnesium sulphate exposure. Irrespective of in what compounds calcium is present, it is alkaline in nature, so the more of it being present better the resistant to acids. With sulphuric acid calcium reacts to form gypsum (Figures 4.39-4.44) and by salvaging itself it stops further damage by acids referring to the pH buffering capacity of concrete. Another aspect of concrete exposed to sulphuric acid being a physical one i.e. gypsum is not as high as those phases calcium used to be before the reaction. Also, reaction product such as ettringite may be more voluminous than the overall volume of the components it formed from; hence expansion and appearance of cracks can be seen, which reduces strength and make concrete less resistant to attack (e.g. acid can migrate deeper in cracks). This expansion, micro cracks and the increased porosity of the concrete at higher replacement level of quartz sandstone might have increased the loss in compressive strength of concrete against sulphuric acid attack.

4.4.6 Sulphate Attack

After 28 days of water curing specimens were oven dried weight after which they were subjected to continuous soaking for 3 to 6 months in a solution containing 3% $MgSO_4$. Three different tests comprising the weight loss, compressive strength variation and water absorption was carried out.

4.4.6.1 Variation in Compressive Strength of Sulphate Attacked Specimens

The graphs showing the percentage drop in compressive strength of sulphate attacked specimens are shown in Figures 4.45-4.46.

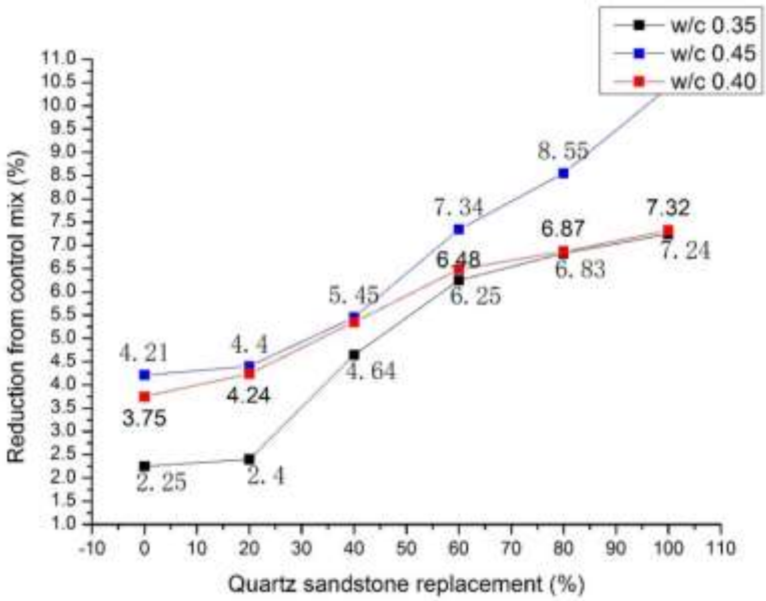

Figure 4.45: The difference in compressive strength (%) at 90 days of specimens subjected to sulphate attack.

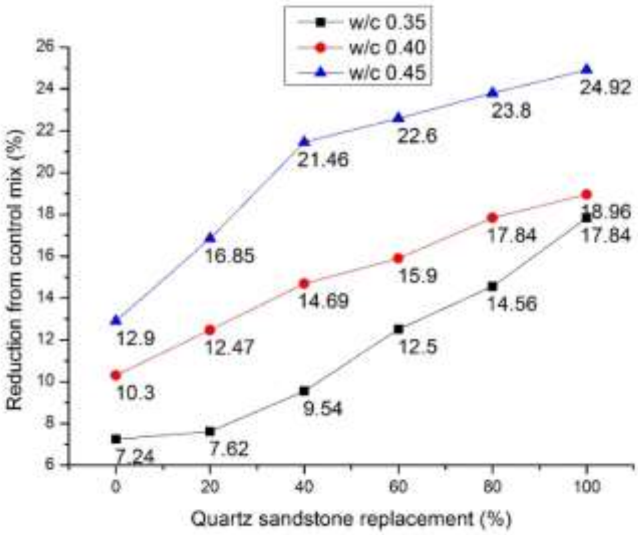

Figure 4.46: The difference in compressive strength (%) at 180 days of specimens subjected to sulphate attack.

Figure 4.47: Concrete Specimens after 28 days curing.

Figure 4.48: Control Concrete Specimens and Dholpur/quartz sandstone concrete samples after 180 days Sulphate Attack Test

Figure 4.49: Sulphate attacked specimens after compressive strength testing at 90 and 180 days

Observation of test results showed that the compressive strength loss increased as the quantity of quartz sandstone increased. Minimum loss of 2.25% was observed for the control mix at 90 days for 0.35 water-cement ratio, and maximum loss of 8.55 % was seen at 100% substitution of quartz sandstone aggregates. A similar pattern of compressive strength loss was observed after 180 days of sulphate attack. The specimen images after the compressive strength testing of sulphate attacked specimens are shown in Figures 4.47- 4.49. From the visual inspection of broken samples, it is evident that the bond between aggregates and cement paste is affected by the continuous immersion in $MgSO_4$ solution. This led to the increase in loss of compressive strength and cracks at the interfaces of aggregate cement paste when the quartz sandstone was increased. Depending on the cementation materials of sandstones, the sandstone can have a higher or lower workability. The binding agent can be either quartz, calcium carbonate or clay.

Quartz sandstones tend to have higher durability than that of sandstone having calcite and the sandstones with clay has the least durability (Sankaran and Sobhi 2014; Yongan et al., 2012; Siegesmund and Török 2014). It was well observed by many researchers that sandstones containing calcarenite degrade at a faster rate. The degradation of calcarenite increases as the solubility of it increases. The solubility is less in pure water and it increases as the CO_2 percentage increases in pure water. Due to this phenomenon, building materials having sandstones can crumble and degrade causing increase in porosity (Samaouali et al., 2008; Schaffer

1932; Duane 2016). However, the sandstone with quartz studied here showed a considerable performance to sulphate attack upto 40% substitution at lower water-cement ratios (0.35 and 0.40).

4.4.6.2 Variation in Weight of Sulphate Attacked Specimens

The results for the change in weight of specimens subjected to a sulphate environment after 28, 90 and 180 days for all the three series are shown in Figures 4.50-4.52.

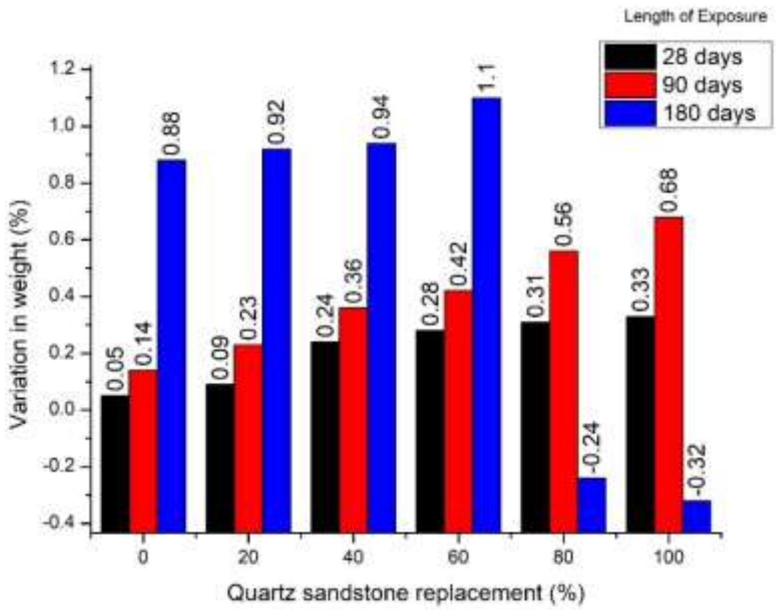

Figure 4.50: Variation in weight of specimens subjected to sulphate attack with water-cement ratio 0.35.

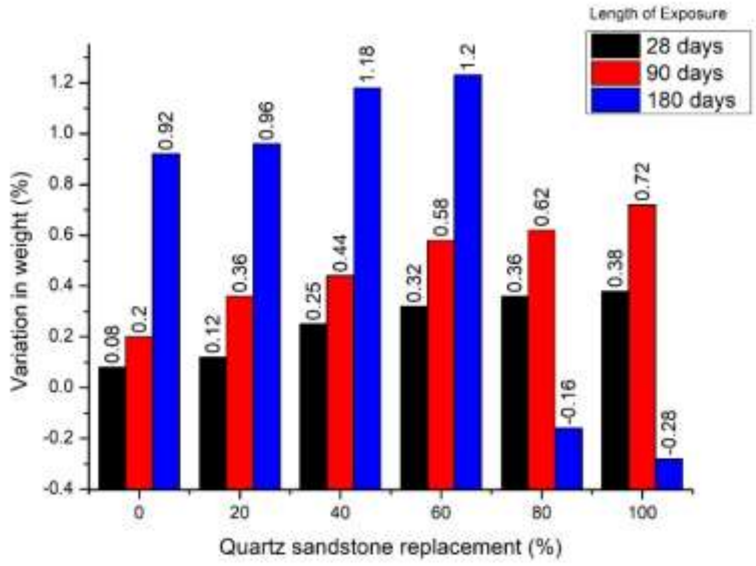

Figure 4.51: Variation in weight of specimens subjected to sulphate attack with water-cement ratio 0.40.

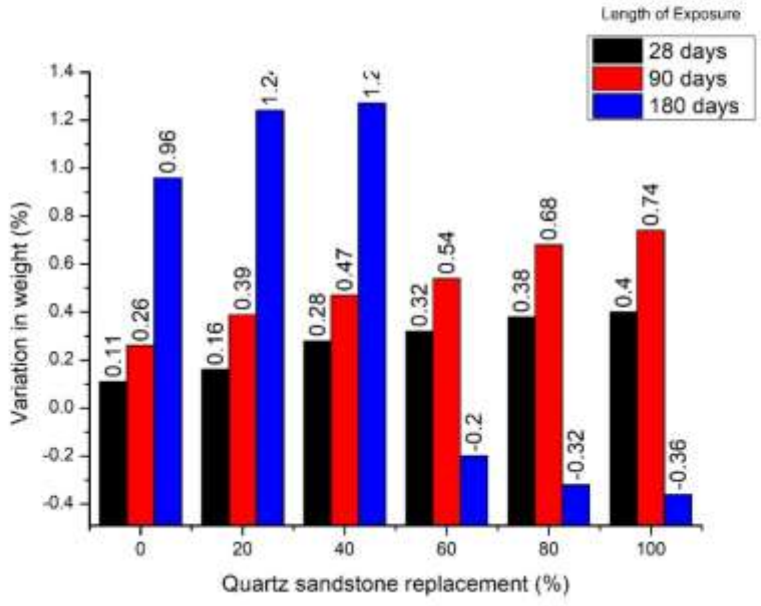

Figure 4.52: Variation in weight of specimens subjected to sulphate attack with water-cement ratio 0.45.

The results showed a consistent increase in weight upto 90 days of sulphate attack. The weight variation of sulphate attacked specimens at 180 days were also studied. For all the water-cement ratios, a continuous gain in weight of the specimens was noticed upto a period 90 days. The specimens with 80% and 100% quartz sandstone showed a decrease in weight after 180 days of sulphate attack. For series III, concrete with 60% quartz sandstone also showed a decrease in weight. This was due to the chipping out of concrete/ suspended in solution due to the continuous exposure of sandstone aggregate at higher replacement levels. The test results show that concrete with greater amount of quartz sandstone would be more affected by attack of sulphate compounds.

4.4.6.3 Water Absorption of Sulphate Attacked Specimens

Water absorption test for the sulphate attacked specimens were performed at 90 and 180 days of immersion. The results are given in Figures 4.53-4.55.

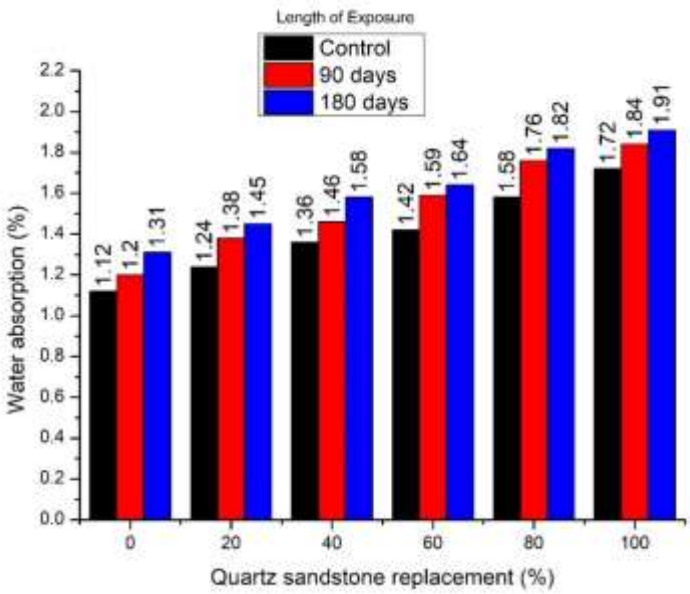

Figure 4.53: Water absorption in weight of specimens subjected to sulphate attack with water-cement ratio 0.35.

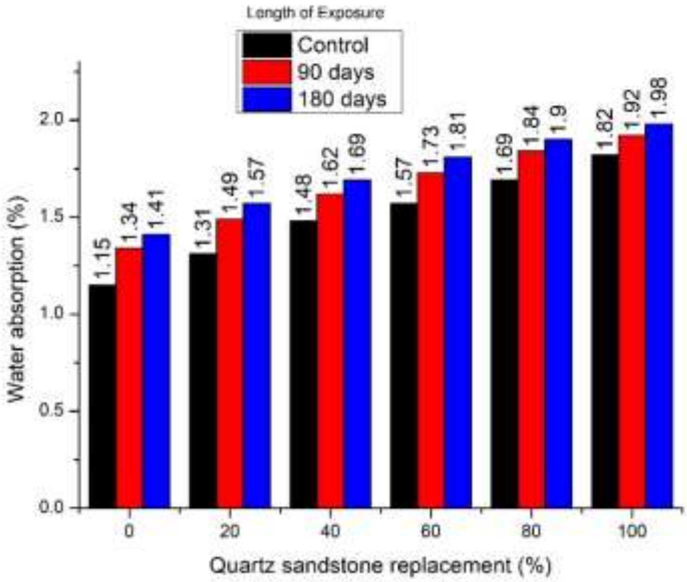

Figure 4.54: Water absorption in weight of specimens subjected to sulphate attack with water-cement ratio 0.40.

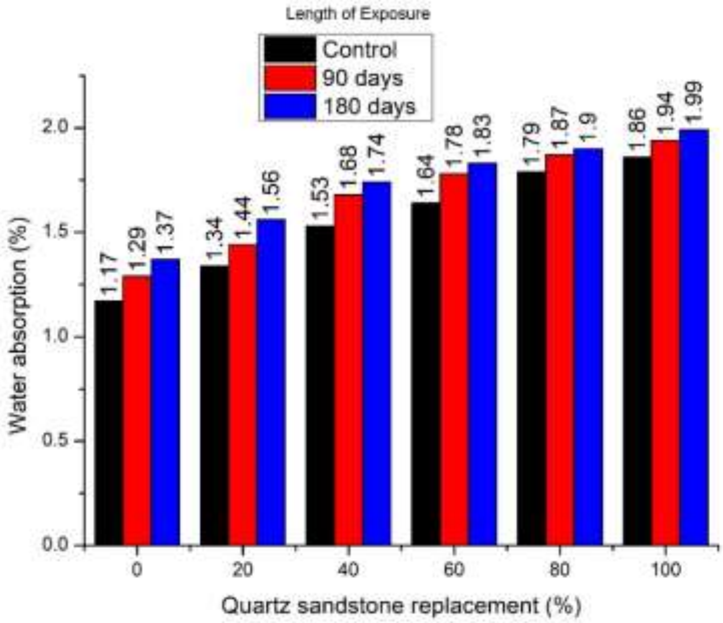

Figure 4.55: Water absorption in weight of specimens subjected to sulphate attack with water-cement ratio 0.45.

From the results, the water absorbed by sulphate attacked specimens showed an increasing trend which was similar to that water absorption of control specimens made with conventional aggregates. A minimum water absorption (1.12%) was noticed for the control mix at 28 days of exposure at 0.35 water- cement ratio and a maximum water absorption (1.86%) was observed at 100% substitution of quartz sandstone at 0.45 water-cement ratio. At 90 d, a minimum water absorption (1.2%) was observed for control mix at 0.35 water-cement ratio and a maximum (1.94%) water absorption was observed at 100% quartz sandstone concrete at 0.45 water-cement ratio.

When comparing to control mix, the amount of water absorbed by sulphate attacked specimens increased at 180 days. It can be observed in all the mixes, the amount of water absorbed by specimens increased with respect to time. At the end of 180 days, more disruption of concrete was observed which led to the overall increase in water absorption.

4.4.7 Comparison of Acid and Sulphate Attack.

Acid attack and Sulphate attack are two different exposure conditions. The former damages the entire mortar or concrete body, whereas magnesium sulphate shall affect the cementitious phases only, not aggregates. Different performance of samples with different aggregates when exposed to $MgSO_4$ may still be noticed, but that is not a direct effect, but the choice of aggregate may affect the physical properties of the sample (e.g. permeability, strength [via absorbing or not absorbing mix water at certain extent]). The simplest comparison between the performance of the aggregates within otherwise identical condition (mix composition, cement content, w/c ratio, curing history etc.) is that we show the mass change (loss) after certain time periods. If loosing less mass preferably after a long period, it can be claimed more durable/resistant.

Acid attacks cement stone and aggregate alike, with the weight losses showing the difference in damage accounted for the type of aggregate (presumably the cement type, cement content and w/c were the same in both sets of experiments). Sandstones without much quartz is relatively easy to attack with H_2SO_4, its $CaCO_3$ is turned into $CaSO_4$ and water, with simultaneous release of CO_2 gas. If part of the aggregate is quartz, which does not react with sulfuric acid, the damage is obviously smaller as observed in the results here. In the magnesium sulphate attack, it dominantly affects cement paste, so the sort of aggregate used is of secondary importance, perhaps only through influencing the transport properties of the matrix and affecting the access of magnesium sulphate to the core of the specimen.

4.4.8 Corrosion of Steel Reinforcements

The macro cell corrosion for various concrete mixes were calculated at initial day (0 day), 28 days, 56 days, 90 days and 180 days for the series with water-cement ratios of 0.35, 0.40 and 0.45. The results are shown in Figures 4.56-4.58.

According to ASTM G 109-99a, a minimum value of 10 µA has been considered to guarantee the presence of sufficient corrosion. A positive macrocell current indicates that corrosion is in progress and vice versa.

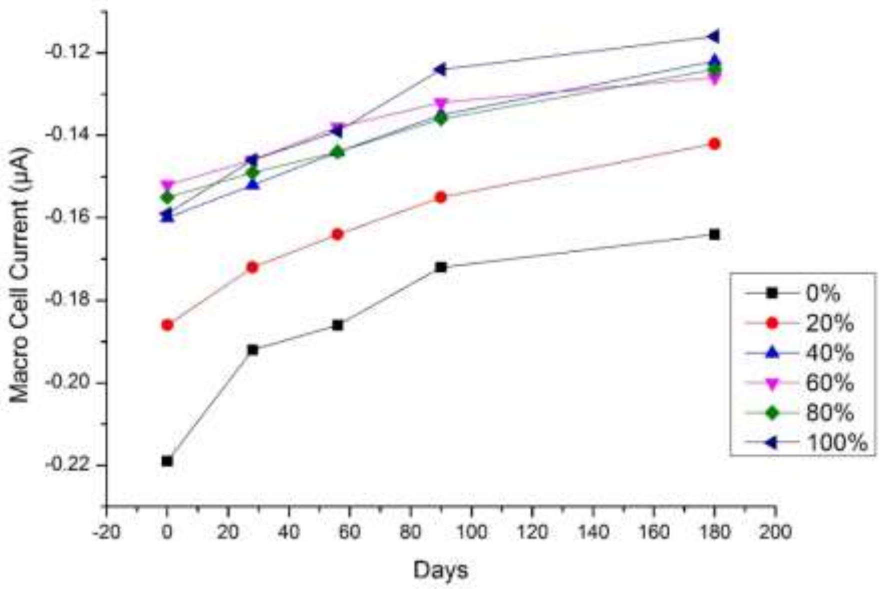

Figure 4.56: Macrocell Current of Specimens with water-cement ratio 0.35

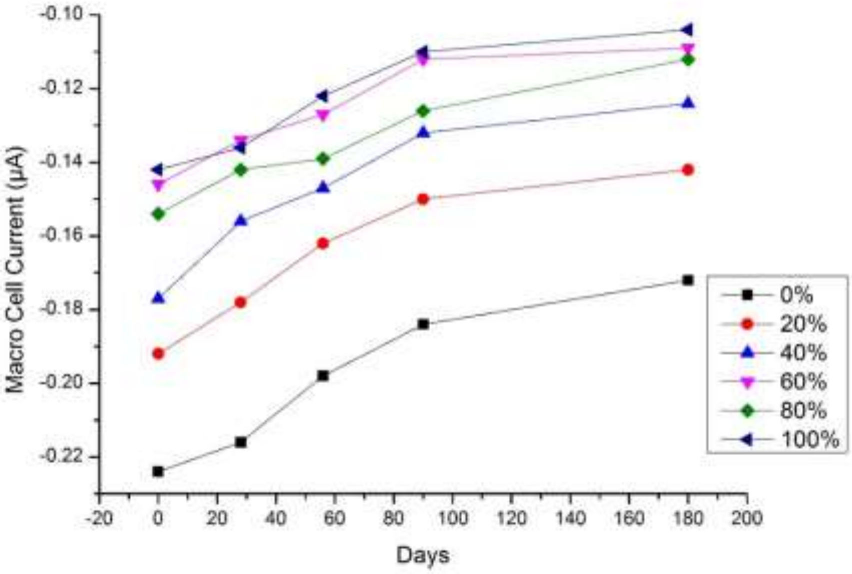

Figure 4.57: Macrocell Current of Specimens with water-cement ratio 0.40

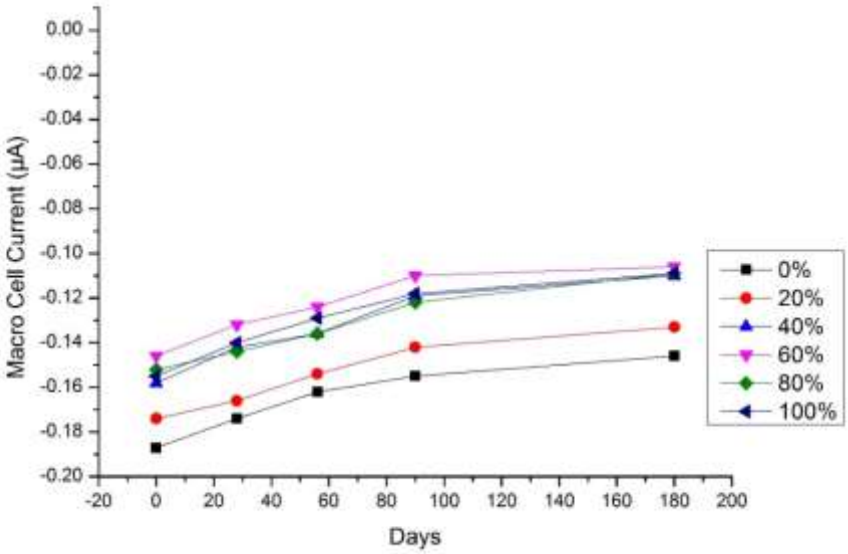

Figure 4.58: Macrocell Current of Specimens with water-cement ratio 0.45

In series I, initially the readings of the control mix were -0.219 µA and the readings at 180 days were -0.164 µA. For the mix with 40% quartz sandstone, the readings were -0.160 µA and -0.122 µA respectively and in the mix with 100% quartz sandstone it were -0.159 µA and -0.116 µA respectively. All the readings were found to increase gradually from 0 day to 180 days. From the above observations, it can be said that the control mix in all the water-cement ratios showed better resistance to corrosion. At 0.35 water-cement ratio, the substitutions 40%, 60% and 80% showed almost a similar trend with very little deviation. At 0.40 water-cement ratio, the control mix showed a macro cell current of -0.224 µA at 0 day and -0.172 µA at 180 days. At 100% replacement it was -0.142 µA and -0.104 µA respectively. At 0.45 water-cement ratio, it was -0.187 µA at 0 day and -0.146 µA at 180 days for the control mix and -0.155 µA at 0 day and -0.109 µA at 180 days for 100% substitution.

From the observations, it was clear that the readings were showing a trend of changing to positive from negative for all the mixes. Since all the observed readings were less than 10 µA, we could conclude that there is absence of corrosion in the specimens after 180 days of ponding. The same conclusion can be drawn for total corrosion calculated as per ASTM G 109-99a. For all the concrete mixes, the total corrosion was less than 150 C which is the threshold value as per the standard. So we can conclude that there is absence of sufficient corrosion for any of the specimens at 180 days.

4.4.9 Microstructure by SEM

A preliminary study was done using scanning electron microscope to know the compatibility of Dholpur/quartz sandstone for the partial replacement of natural aggregates. The accelerated electrons having large amount of kinetic energy is made to dissipate into variety of signals by electron-sample interactions. Secondary electron produces SEM images that are used to study the crystal structure and mineral orientations. These electrons are important as it shows morphology and topography of samples. Non-destructive tests like SEM serves as an important study for analysing the use of sandstones in concrete since there is no volume loss in material and the same sample can be used repetitively. S-4700 Field Emission Scanning Electron Microscope (FE-SEM) was used to image the samples.

It was evident from the images that the sandstone particles have a dense structure and presumably low porosity / low water absorption on comparison to other regular sandstones, which is beneficial from technical point of view (i.e. the water demand would be low).

On the other hand, there are fairly sharp cleavages and the smaller particles show some elongation, which adversely affect the rheology / flow characteristics of fresh concrete and can be mitigated by increasing the w/c ratio or applying super plasticizers. The two effects (low water absorption and angular shape) compensate each other, so all in all, based on its morphology, this material appears to be suitable for concreting. The SEM images of natural aggregate and quartz sandstone in shown in Figure 4.59.

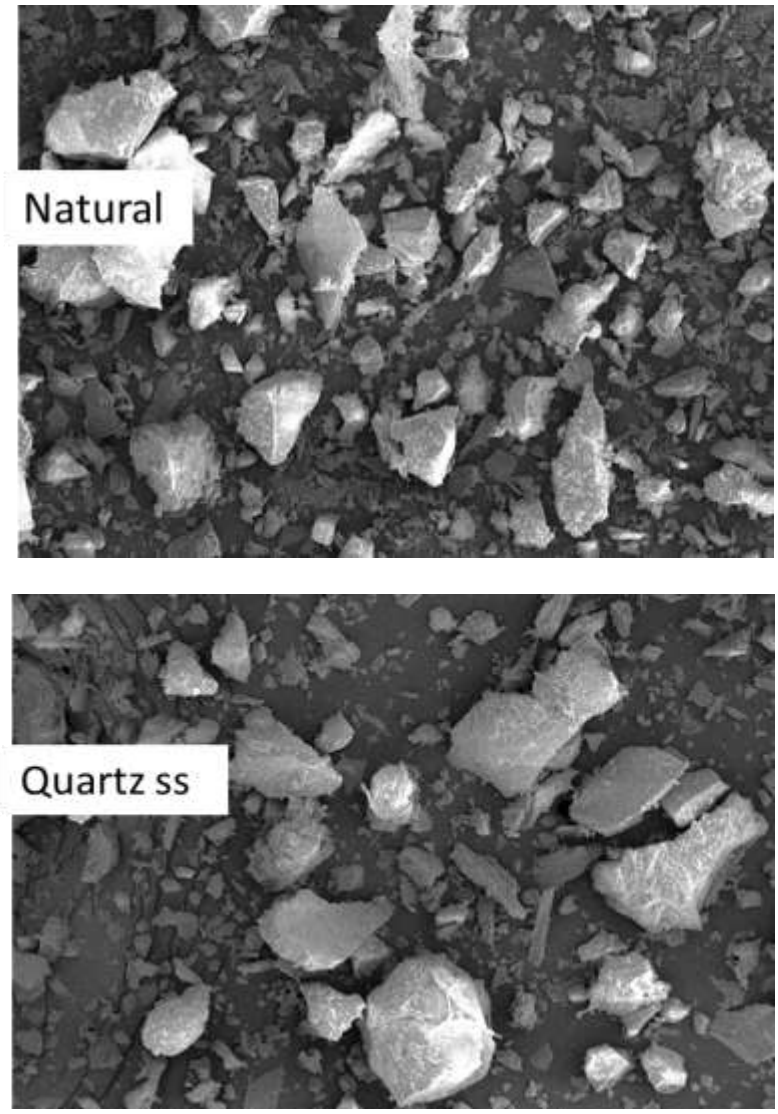

Figure 4.59: SEM images of natural and quartz sandstone aggregates, × 500 magnification

The microscopic study was further studied after using Dholpur stone/quartz sandstone as a substitute for coarse aggregate in concrete. From the microstructure study, it can be said that sandstones are poorly sorted i.e. grains of various sizes occur together and the framework is dominated by quartz, whose particles are generally rounded to sub-rounded. The interstitial matrix of sandstone consists of all silt-sized quartz, mica and probably also sub-microscopic clay minerals at grain surfaces. In general, it can be said that sandstone is held together by phyllosilicate minerals (clay and fragments of rock) due to local compaction and rarely by chemical cementation. By sedimentology, sandstones can be categorised into mature, sub-mature, super mature and immature (Table 4.3).

Table 4.3: The textural maturity of sandstone based on clay content, minerals present and type of grains (Folk 1951)

Maturity	Clay content	Minerals present	Grain type
Immature	Greater than 5% clays or silt.	A Large proportion of unstable minerals such as feldspar and lithic fragments.	Angular and diverse grain sizes.
Submature	Less than 5% clays or silt.	Some unstable minerals and lithic fragments.	Moderately sorted grains (Mostly angular and a few rounded grains).
Mature	Less than 5% clay or silt	Stable minerals like lithics and chert.	Clasts are sub angular to sub-round.
Supermature	Less than 5% clays or silt	Exclusively stable minerals.	Well sorted grains. Clasts are sub-rounded to round.

From the preliminary study on quartz sandstone waste, clay content in the aggregate was below 5% having some unstable and lithic fragments such as feldspar and the grains were moderately sorted (mostly angular and few rounded grains). From the SEM, X-ray diffraction and methylene blue observations (as shown in section 3.2.4), it was found that the Dholpur stone/quartz sandstone belong to submature category. From the SEM tests on 28 days cured sample, there appears slightly greater porosity and also finer grain texture when moving from the control sample towards higher replacement levels with sandstone. Control concrete was made with magmatic rock aggregate as opposed to the sedimentary sandstone which absorbs more water, leaving gaps at interfaces between sandstone and cement paste which reduces compressive strength as the quartz sandstone content is increased (Figures 4.60). The other reason for higher porosity is inherited from the formation process of sandstone (i.e. sedimentation), resulting in voids within the material.

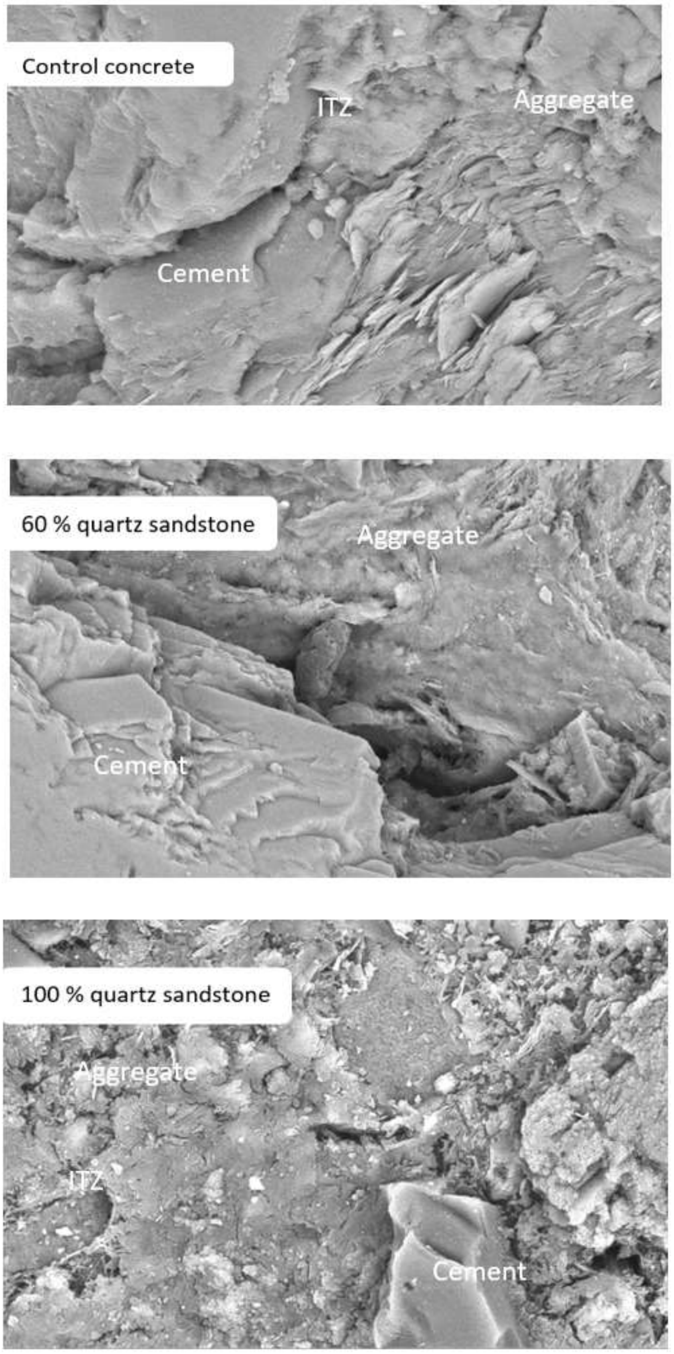

Figure 4.60: SEM images of concrete samples containing quartz sandstone, (x 1000 magnification)

4.4.10 X-ray fluorescence (XRF) observation

The concrete samples having various percentages of quartz sandstone substitution was analysed (Table 4.4). Concrete samples having 20% and 60% quartz sandstone coarse aggregates had almost similar amounts of quartz. Quartz is strong and non-porous as granite which is good for acid resistant properties. The presence of CaO was more in control concrete having only conventional coarse aggregates and similar compositions were found in concrete samples having 40%, 80% quartz sandstone substitutions. Since XRF detects only the elements, not compounds, calcium in concrete is most likely to be present in C-S-H gel, CaOH, C_3S, C_2S, unhydrated clinker minerals and even in ettringite. Calcium being alkaline in nature and irrespective of the compound it is present, so the more of it being present more the resistant to acid attack.

Alumina (Al_2O_3) content was found to be more in concrete having quartz sandstone replacements. This is due to the presence of them in quartz sandstone itself. All the other minerals like iron oxide, potassium oxide, sodium oxide, magnesium oxide and sulphur trioxide were found to be in very less quantity.

Table 4.4: X-ray fluorescence results showing the elemental composition of concrete with various replacements of quartz sandstone aggregates.

Elemental composition (%)	Concrete coarse aggregate replacement percentages					
	0 % quartz sandstone	20 % quartz sandstone	40 % quartz sandstone	60 % quartz sandstone	80 % quartz sandstone	100 % quartz sandstone
SiO_2	60.21	63.70	60.54	63.51	61.62	62.71
CaO	15.31	10.94	14.87	11.24	14.59	13.91
Al_2O_3	6.95	8.41	7.37	7.69	6.98	7.47
Fe_2O_3	1.58	1.64	1.69	1.19	1.32	1.16
K_2O	1.46	2.01	2.71	2.97	2.92	3.54
Na_2O	0.83	1.35	0.74	0.61	0.38	0.42
SO_3	0.73	0.51	0.66	0.53	0.69	0.59
MgO	0.58	0.65	1.00	0.57	0.74	0.68
TiO_2	0.28	0.34	0.25	0.25	0.27	0.24
P_2O_5	0.090	0.093	0.073	0.065	0.056	0.050
Sr	0.058	0.043	0.056	0.043	0.062	0.061
Cl	0.033	0.023	0.027	0.017	0.026	0.019
MnO	0.031	0.018	0.031	0.018	0.025	0.023
Ba	0.023	0.013	0.088	0.029	0.043	0.049
Zr	0.019	0.021	0.000	0.023	0.024	0.021
Cr	0.012	0.009	0.012	0.014	0.012	0.012
Sum	88.20	89.76	90.11	88.75	89.75	90.95

4.4.11 Thermogravimetric Analysis

Simultaneous Thermal Analyser (STA 6000) with Pyris Instrument Viewer software was used for analysing the samples. The sample was checked to be compatible with ceramic crucible at temperature, atmosphere and time and the nitrogen gas was connected to the analyser in the balance port with sample purge gas flow rate at 30 ml/min. The temperature was applied at the rate of 20°C and Pyris manager was used to get the math and smoothen the curves obtained after the run was complete with furnace returning to room temperature. Some of the researchers have witnessed loss of bound water at 50°C to 600°C and correlated with hydration rate changes taking place in the concrete samples containing other stone wastes in concrete (Singh et al., 2016; Rana et al., 2015).

Mass loss of hydration products assigned to specific temperature ranges by several researchers are as follows: 30–105°C due to evaporation of free (capillary) water; 110-170°C due to decomposition of ettringite; 180–300°C due to partial dehydroxylation of C–S–H (possibly indicating partial transformation of C–S–H gel to xonotlite); 400–500°C dihydroxylation of portlandite ($Ca(OH)_2$); 700–900°C due calcination of $CaCO_3$ and dehydroxylation of xonotlite (Cheyrezy et al., 1995; Ruiza et al., 2005; Helmi et al., 2016). A similar weight loss pattern was obtained for the concrete samples containing quartz sandstone (Figure 4.61). The pattern showed a decrease in loss of weight as quartz sandstone was added in concrete as a replacement for coarse aggregates. However, at 40% and 60% replacements, the pattern was almost the same with very little deviation and it is evident from the pattern that there is loss of moisture content upon heating with major events occurring at 150°C, 450°C and 750 °C (Figure 4.62). Ettringite starts to lose water and transform to monosulphate at as low as 67°C, the event at 150°C may be the decomposition of monosulphate. The 450°C event shall then be linked to portlandite, whereas the 750°C event to $CaCO_3$ (present as carbonated portlandite or as part of the constituent materials (e.g. aggregate)).As the quartz sandstone dosage increases there is less portlandite available in the mixes due to more moisture absorption. For calcite the reason may be due to a low amount of it being present in the sandstone aggregate / in concrete containing different percentages of sandstone aggregate when compared to conventional coarse aggregates.

The plots were further studied in detail to analyse the decomposition of portlandite and calcite as the quartz sandstone dosage was increased in cement concrete (Figure 4.63).

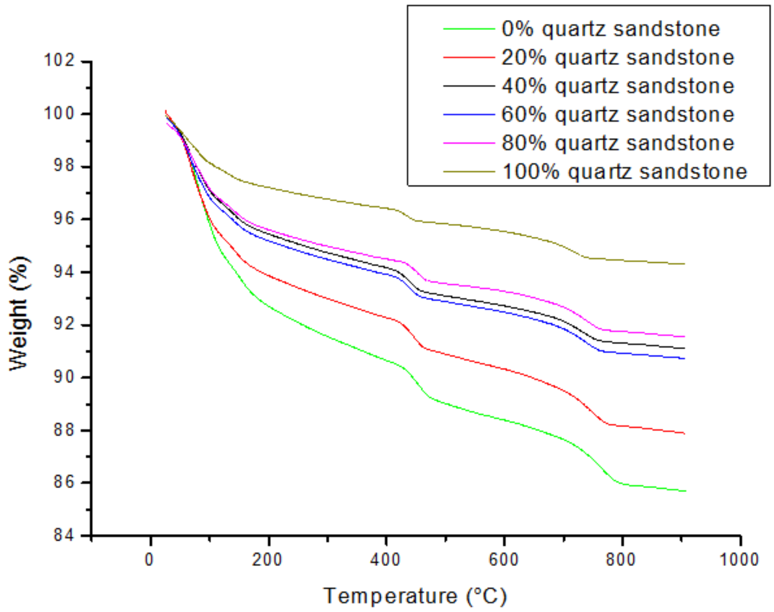

Figure 4.61: Weight % vs Temperature for various substitutions of quartz sandstone

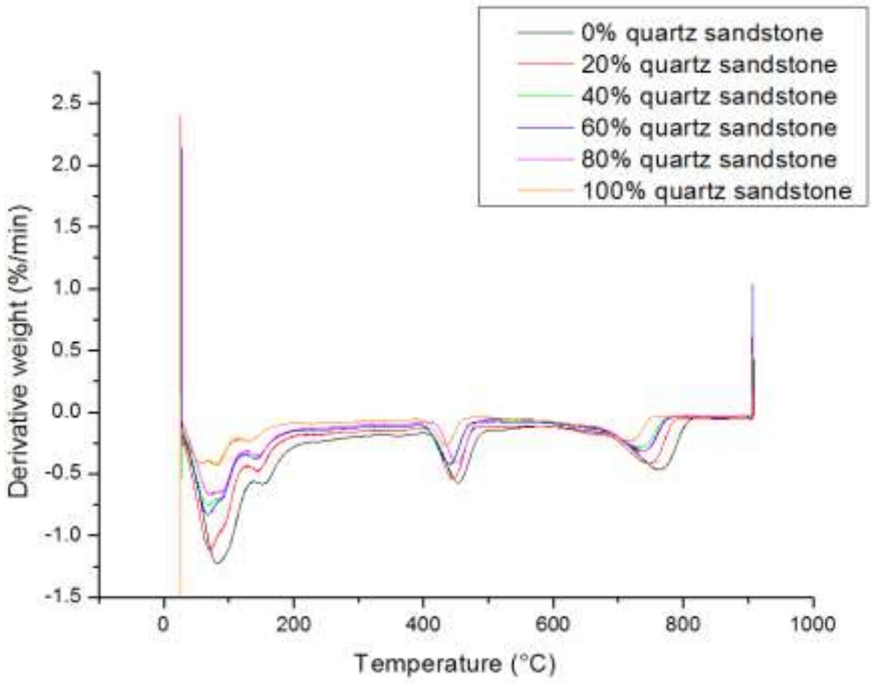

Figure 4.62: DTG curves showing major events upon temperature change

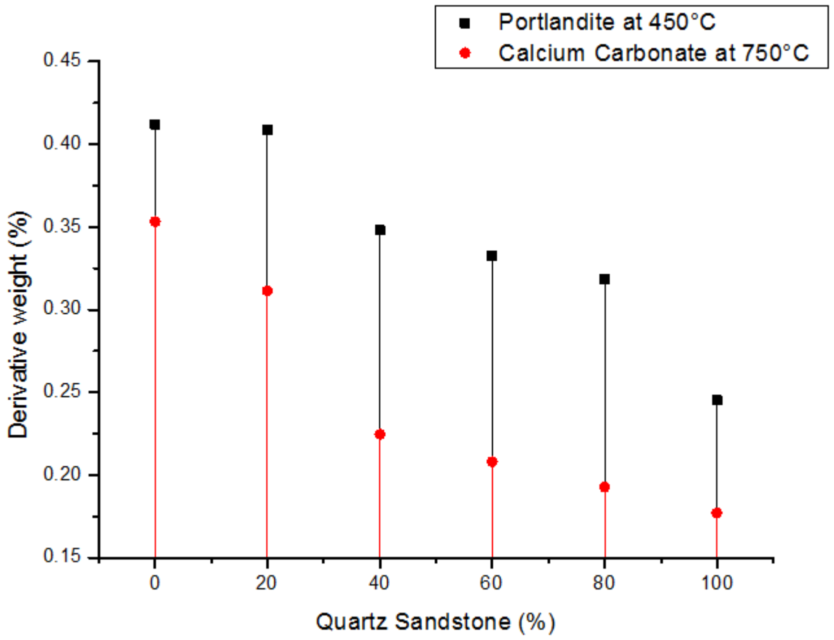

Figure 4.63: Vertical drop plot showing portlandite and calcite decomposition upon quartz sandstone replacement

4.4.12 Action of Fire

It is important to understand the fire endurance of a construction material inorder to ensure safety for life and property (Sarker et al., 2014). Concrete members generally exhibit good fire resistant properties due to its low thermal conductivity, high thermal capacity and slow degradation of mechanical properties of concrete with temperature (Kodur and Agrawal, 2016). The author couldn't find any standard procedure for high temperature testing in concrete. Most of the researchers have employed testing concrete at higher temperatures and finding its residual strength after its exposure (Cree et al., 2013). The concrete containing 0%, 20%, 60% and 100% of quartz sandstones were tested against fire with an increasing temperature upto 800° C. The loss in compressive strength of specimens at 200° C, 400° C, 600° C and 800° C were observed (Figures 4.64-4.67). A maximum loss in compressive strength (80%) was observed at 800° C for the control mix at 0.45 water-cement ratio and a minimum loss (66%) was observed for the mix with 100% quartz sandstone concrete at 0.35 water-cement-ratio at the same temperature. At 600° C, greatest loss in compressive strength was observed for the control mix (56%) at 0.45 water-cement ratio and minimum loss was observed for mix with

100% quartz sandstone concrete at 0.35 water-cement ratio. Similar results were also observed at 400° C and 200° C testing temperatures. A maximum loss of 40% and 28% was observed for the control mixes at 400° C and 200° C. The minimum loss in compressive strength was about 23% and 18% for the mixes with 100% quartz sandstone at 400° C and 200° C. The residual compressive strength of concrete with lower w/b ratio are higher than the concrete with higher w/b (Qianmin Ma et al., 2015). . The residual compressive strength observed was similar to that of Mohamedbhai (Mohamedbhai, 1986) results in which he studied the effects of exposure time and rates of heating and cooling on residual strength of heated concrete. Also, the sandstones exhibit very different thermal properties basis on their mineral composition and granularity. Hence it becomes really important to know the specific properties of sandstone and also its origin. From the observed results of thermal tests, it can be said that the concrete mix with increased percentages of quartz sandstone performed better than that of mix with control concrete made with conventional coarse aggregates.

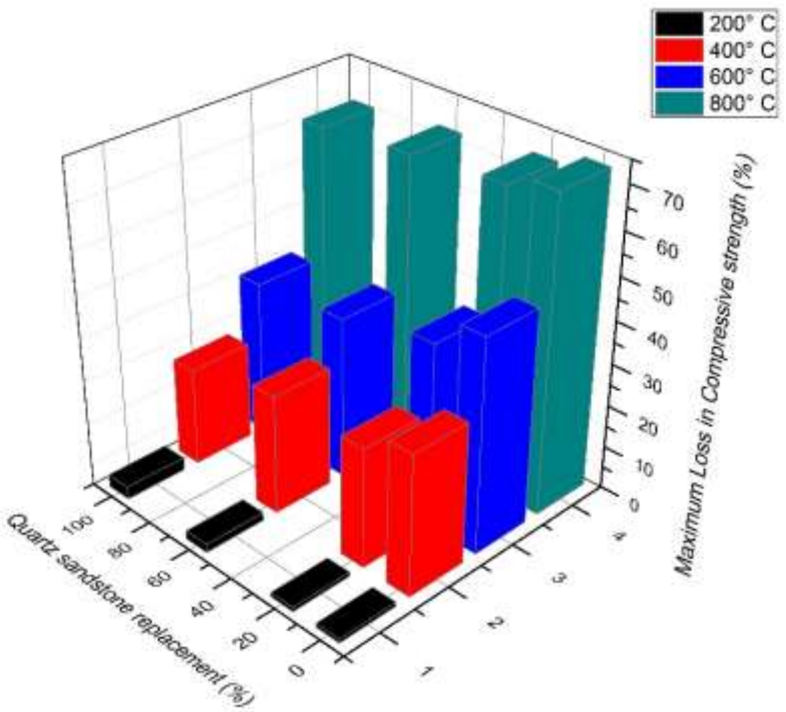

Figure 4.64: Maximum compressive strength loss of the specimens for water-cement ratio of 0.35.

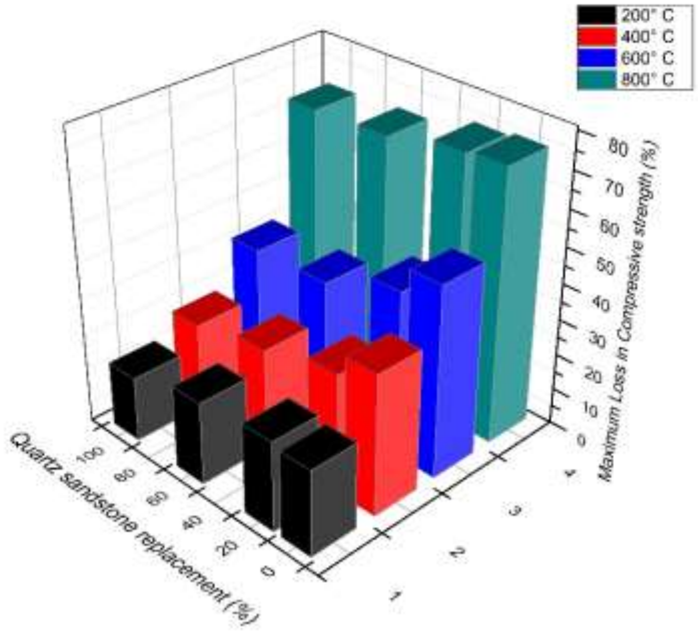

Figure 4.65: Maximum compressive strength loss of the specimens for water-cement ratio of 0.40.

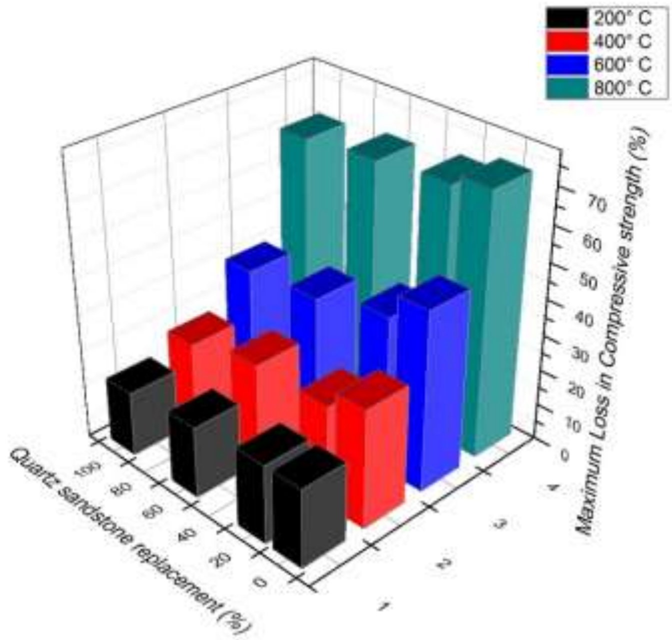

Figure 4.66: Maximum compressive strength loss of the specimens for water-cement ratio of 0.45.

Figure 4.67: Concrete image after exposure to fire at 800°C.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

5.1 Introduction

This experimental study was based on the effective utilisation of Dholpur stone/ quartz sandstone wastes as a substitute for natural coarse aggregates in cement concrete. In this research work, three series of concrete mixes were casted. M30 concrete grade was prepared with a water-cement ratios of 0.35, 0.40 and 0.45 respectively. Dholpur stone/quartz sandstone was substituted for coarse aggregates from 0% to 100% in multiples of 20%.

Mix Design with 0.35 water-cement ratio was considered as Series I and water-cement ratio of 0.40 as Series II and 0.45 water-cement ratio as Series III. In order to obtain consistent workability for concrete having both the aggregates (natural and Dholpur/quartz sandstone), super plasticiser was used for maintaining the compaction factor between 0.85-0.90. The properties of concrete such as compressive strength, flexural tensile strength, abrasion resistance, pull-off strength, water permeability, water absorption, resistance against acid attack and sulphate attack, carbonation, depth of chloride penetration, corrosion of steel reinforcements, thermogravimetric analysis and action of fire on compressive strength were tested. SEM test was also performed to study the microstructural properties.

5.2 On the basis of the results and discussions from this study, the following conclusions/findings can be drawn

Experiments were conducted to study potential use of quartz sandstone as partial replacement of coarse aggregates in concrete. The following important conclusions/findings may be drawn from this study are as follows.

5.2.1 The density of the concrete containing quartz sandstone was found to be decreasing as the replacement level increased. This is due to increasing use of lower specific gravity of quartz sandstone compared to conventional coarse aggregates.

5.2.2 From the results of compressive strength test for water-cement ratios of 0.35, 0.40 and 0.45, the compressive strength shows a decreasing trend as the quartz sandstone aggregate percentage was increased. At 28 days, for M30 grade of concrete, the target strength of 38.25 N/mm^2, was achieved for all replacements levels of quartz sandstone for water-cement ratios at 0.35 and 0.40 and till 40% substitution at w/c 0.45. A similar decreasing trend was noticed in flexural strength results. As the time passes i.e. at 90 days & 365 days, the compressive and flexural strength increases, this is similar to the traditional concrete.

5.2.3 Combined gradation of different sized aggregates and its compressive strength test results served as a good tool for finding out the ratios in which aggregates can be mixed together. This study was done for both parent and substitute aggregate individually before using the substitute aggregate as a partial replacement of parent aggregate. The exact ratio for mixing conventional coarse aggregates was found to be 55:45 (20mm: 10mm) and for quartz sandstone aggregates was 60:40 (25mm: 10mm) in order to achieve maximum compressive strength.

5.2.4 Resistance to abrasion decreased with the increase in the percentage of Dholpur aggregate (quartz sandstone aggregate) irrespective of the water-cement ratios. A maximum abraded depth of 1.89 mm was obtained for concrete with 100% quartz sandstone replacement at 0.45 water-cement ratio, which is within the permissible limit.

5.2.5 The sorptivity and permeation increased as the quartz sandstone aggregate replacement increased for all the water-cement ratios and a maximum sorptivity of 0.46 mm was observed for 100% replacement level.

5.2.6 A peak pull off strength of 3.9 N/mm^2 was observed for the control mix with a water-cement ratio of 0.35. A gradual reduction in pull-off strength results was recorded as the quartz sandstone percentage of substitution increased. Almost a same trend of strength variation was recorded in the other mix-designs with of 0.40 and 0.45 water-cement ratios. It was noted that the pull-off strength plots followed the similar trend as the variation in compressive strength.

5.2.7 The water absorption percentage was 1.12% for control mix at 0.35 water-cement ratio and a gradual increase in water absorption was observed as quartz sandstone substitution increased. The maximum water absorption (1.86%) was observed at 100% quartz sandstone substitution at 0.45 water-cement ratio.

5.2.8 The observations indicated that the concrete mixes with lower w/c ratios offered better resistance to carbonation. The rate of increase in carbonation was marginal till 40% substitution level at lower water-cement ratios. However, at higher replacement levels (above 40%) the carbonation depth increased.

5.2.9 The depth of Chloride Ion penetration increased as the Dholpur stone (quartz sandstone) replacement was increased. At 90 days of exposure to NaCl, the control concrete and 20% quartz sandstone concrete experienced a similar depth of penetration. A maximum depth of penetration of 32 mm was recorded for the concrete with 100% quartz sandstone at 0.45 w/c. The gradual increase in chloride penetration can be related to reduced packing capacity of concrete as the quartz sandstone replacement increased.

5.2.10 A considerable loss of compressive strength was observed in all the concrete samples irrespective of the quartz sandstone replacement and water-cement ratios for the acid attack test i.e. the effect of H_2SO_4. At 0.45 water-cement ratio, the maximum loss in compressive strength of acid attacked specimen was observed at 76.7% at 100% replacement of quartz sandstone. Minimum loss of 44.1% with 0.35 water-cement ratio was recorded at 40% substitution of quartz sandstone aggregates. Siliceous nature of quartz sandstones was found to be beneficial towards its resistance to sulphuric acid attack. Accurately graded concrete with quartz sandstone aggregates up to 40% substitution would be beneficial in areas of severe sulphuric acid exposure provided it has lesser clay content and up to 60% of conventional coarse aggregates can be replaced at areas with a minor sulphuric acid exposure.

5.2.11 It was found that the bond between aggregates and cement paste is affected by the continuous immersion in $MgSO_4$ solution. However, the sandstone with quartz studied here showed a considerable performance to sulphate attack upto 40% substitution at lower water-cement ratios (0.35 and 0.40).

5.2.12 Since all the values of macrocell corrosion test for various replacement levels, upto 100% replacement, were less than 10 µA for all the three series. We could conclude that there is no significant evidence of corrosion in the specimens after 180 days of ponding.

5.2.13 Microstructure study on Dholpur aggregate showed fairly sharp cleavages and elongations which affected the rheology of concrete. This phenomenon was controlled by increasing the dosage of super plasticisers when quartz sandstone aggregates were used. The microstructural study of concrete revealed increased porosity and finer grain structure as the Dholpur stone/quartz sandstone aggregate was replaced with conventional coarse aggregates.

5.2.14 The results of thermogravimetric results showed a decrease in the percentage of weight loss as the quartz sandstone aggregate was replaced by conventional aggregates. This can be due to increases of void spaces that prevents the heat transfer phenomena as the dosage of sandstone aggregates increased.

5.2.15 DTG graphs showed major events occurring at 150°C, 450°C and 750°C. On further analysis, loss in portlandite and calcite contributed to this less as the quartz sandstone content was increased in cement concrete. Local water deficient regions e.g. interfacial transition zones might have reduced the rate of hydration reaction which would have reduced the amount of portlandite generated when cement clinker turns to C-S-H gel and portlandite. On another account, portlandite is basic (in the meaning of non-acidic), and its quantity might be affected by ‘acidic' constituents. Sandstone may be siliceous, which in theory acts like that, although the thermodynamics of the process may not make it a valid procedure in the time-frame observed here.

5.2.16 Concrete made with quartz sandstones performed well against the action of fire. Minimum loss in compressive strength was observed for samples with 100% quartz sandstone substitution thus making it a suitable material for fire resistance. In terms of thermal efficiency, depending on the preferred strength of concrete samples to be attained, dosages of quartz sandstone may be taken up to 60%.

5.2.17 Approximate Cost Analysis: There occurs an approximate saving of Rs. 845 for one cubic metre of concrete, if Dholpur aggregates are used. A huge benefit of sandstone waste utilisation and the use of large amounts of conventional coarse aggregates can be reduced if this is implemented.

On comparing the limits of Indian Standards, concrete having quartz sandstone is recommended for structural works and general purpose tiles with an adequate replacement upto a certain water-cement ratio. Also, by the use of these sandstone wastes, there occurs a reduced consumption of depleting natural aggregates and the landfilling problems which contribute to the overall environmental benefits and sustainability.

5.3 Recommendations for Future Work

The following recommendations for further work may be considered as follows:

5.3.1 The concrete with waste Dholpur stone/quartz sandstone as fine aggregate replacement may be tested to study the mechanical and durability properties.

5.3.2 In the present study, experiments were performed with the use of Ordinary Portland Cement (OPC) as a binder in concrete. Tests may be conducted with Portland Pozzolana Cement (PPC) as a binder instead or also with the use of Fly Ash and/or with GGBS.

5.3.3 The tests like Mercury Intrusion Porosimetry, shrinkage, microcell and half-cell corrosion can be carried out for a deeper study.

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Annexure 1

Cost Benefit Analysis

Cost Benefit Analysis for Optimum Replacement level

In this research work, M30 grade of concrete with water-cement ratios of 0.35, 0.40 and 0.45 were casted. Dholpur stone (quartz sandstone) was replaced for conventional coarse aggregate from 0% to 100% in multiples of 20%. The cost benefit analysis is done on the basis of optimum replacement level which was found to be 40%. This limit was fixed due to its achievement of target strength for all the water cement ratios.

1. Cost of coarse aggregates as per the retail rate in 2017 is Rs 4-6/kg (https://www.materialtree.com/sand-aggregates/coarse-aggregates/20mm-coarse-aggregates).
2. Cost of Dholpur waste aggregates used in this research work is Rs 1-2 /kg excluding the transportation charges.
3. To produce 1 cubic metre of concrete, the amount of coarse aggregate required is about 683.65 kg/m^3 (w/c 0.35) (As per the design mix given in Chapter 3).
4. If 40% of coarse aggregates are replaced with Dholpur aggregates, it requires only 261.12 kg/m^3 due to its lesser coarse aggregates when replaced to its volume. Thus the cost for conventional coarse aggregate accounts for Rs 3418 and for a 40% replacement of Dholpur stone wastes accounts for Rs 2572.
5. There occurs an approximate saving of Rs. 845 for one cubic metre of concrete, if Dholpur aggregates are used.
6. A huge benefit of sandstone waste utilisation and the use of large amounts of conventional coarse aggregates can be reduced if this is implemented.

Comparison of mixes with equal compressive strengths (28 days)

At 28 days, it was noticed that the strength of concrete mixes with water-cement ratios 0.35, 0.40 and 0.45 at substitution levels of 100%, 60% and 20% respectively were similar. On further evaluation of their quantities of constituent materials, it can be seen that, mix with w/c 0.35 having only Dholpur stone was the **most economical** of all three. The detailed cost analysis is shown below:

The total cost of producing concrete made with 100% natural aggregates are given below:

For water-cement ratio 0.35 = Rs. 9505.1

For water-cement ratio 0.40= Rs. 9411.5

For water-cement ratio 0.45= Rs. 9291.1

The total cost of producing concrete made with 100% Dholpur stone aggregates are given below:

For water-cement ratio 0.35 = Rs. 5799.5

For water-cement ratio 0.40= Rs. 5628.5

For water-cement ratio 0.45= Rs. 5370.4

Cost analysis on 28 days concrete with similar strength:

Table A1.1: Table for mix with water-cement ratio 0.35 at 100% Dholpur stone replacement (C.S. 41 MPa)

Constituents	**Quantities (kg)**	**Rate (Rs)**	**Cost (Rs)**
Cement	440	5.6	2464
Natural coarse aggregate	-	5	0
Dholpur coarse aggregate	1242	2	2484
Fine Aggregate	635	1.3	825
Total			**5773**

Table A1.2: Table for mix with water-cement ratio 0.40 at 60% Dholpur stone replacement (C.S. 42 MPa)

Constituents	**Quantities (kg)**	**Rate (Rs)**	**Cost (Rs)**
Cement	405	5.6	2268
Natural coarse aggregate	503	5	2515
Dholpur coarse aggregate	755	2	1510
Fine Aggregate	645	1.3	839
Total			**7132**

Table A1.3: Table for mix with water-cement ratio 0.45 at 20% Dholpur stone replacement (C.S. 41 MPa)

Constituents	Quantities (kg)	Rate (Rs)	Cost (Rs)
Cement	342	5.6	1915
Natural coarse aggregate	1036	5	5180
Dholpur coarse aggregate	260	2	520
Fine Aggregate	662	1.3	861
Total			**8476**

Cost analysis on 180 days concrete with similar strength:

A1.4: Table for mix with water-cement ratio 0.35 at 60% Dholpur stone replacement (C.S. 46.2 MPa)

Constituents	Quantities (kg)	Rate (Rs)	Cost (Rs)
Cement	440	5.6	2464
Natural coarse aggregate	497.2	5	2486
Dholpur coarse aggregate	745.8	2	1491.6
Fine Aggregate	635.47	1.3	826.1
Total			**7267.7**

A1.5: Table for mix with water-cement ratio 0.40 at 40% Dholpur stone replacement (C.S. 46.1 MPa)

Constituents	Quantities (kg)	Rate (Rs)	Cost (Rs)
Cement	405	5.6	2268
Natural coarse aggregate	756.6	5	3783
Dholpur coarse aggregate	504.4	2	1008.8
Fine Aggregate	645	1.3	838.5
Total			**7898.3**

A1.6: Table for mix with water-cement ratio 0.45 at 20% Dholpur stone replacement (C.S. 46.1 MPa)

Constituents	**Quantities (kg)**	**Rate (Rs)**	**Cost (Rs)**
Cement	342.22	5.6	1916.4
Natural coarse aggregate	1037.54	5	5187.7
Dholpur coarse aggregate	260.98	2	521.9
Fine Aggregate	662.05	1.3	860.6
Total			**8486.6**

Cost analysis on 360 days concrete with similar strength:

At 360 days, it was noticed that the strength of concrete mixes with water-cement ratios 0.35, 0.40 and 0.45 at substitution levels 60%, 40% and 20% respectively were similar. The corresponding strengths were 46.4Mpa, 46.5 Mpa and 46.5 Mpa. This cost benefit analysis is similar to that of 180 days.

Percentage of Cost Savings at 28 days

The costs occurring for similar strengths at 28 days are given below:

Table A1.7: Percentage of Cost Savings at 28 days

Water-Cement Ratio	**Percentage of Replacement**	**Compressive Strength at 28 days (MPa)**	**Cost of concrete with similar strength at 28 days**	**Percentage Savings (%)**
0.35	100	41	**5773**	**31**
0.40	60	42	7132	15
0.45	20	41	8476	0

On comparing, **31%** savings of cost can be achieved by replacing 100% Dholpur stone at 0.35 water-cement ratio inorder to achieve a compressive strength of 41 Mpa at 28 days.

Percentage of Cost Savings at 180 and 360 days

The costs occurring for similar strengths at 180 and 360 days occurs at same replacement levels which are given as follows:

Table A1.8: Percentage of Cost Savings at 180 and 360 days

Water-Cement Ratio	**Percentage of Replacement**	**Compressive Strength (MPa) At 180 days**	**Compressive Strength (MPa) At 360 days**	**Cost of concrete with similar strength at 28 days**	**Percentage Savings (%)**
0.35	60	46.2	46.4	7267.7	**15**
0.40	40	46.1	46.5	7893.3	7
0.45	20	46.1	46.5	8486.6	0

On comparing, **15%** savings of cost can be achieved by replacing 60% Dholpur stone at 0.35 water-cement ratio inorder to achieve a compressive strength of 46.2 Mpa and 46.4 Mpa at 180 and 360 days respectively.

For attaining a same compressive strength with varying water-cement ratio and increase in percentage of Dholpur Stone waste, the net saving may be attained upto 31% at 28 days and 15% at 180 and 360 days respectively (as shown in the Table A1.7 and A1.8)

Annexure II

Mix Design Guidelines

For a standard mix, M30 concrete grade was designed as with a water-cement ratio of 0.40. Also, to study the variation in different properties of concrete, the mixes with water-cement ratio of 0.35 and 0.45 were used. The design steps for a M30 concrete with 0.40 water- cement ratio is given below.

1. $F'_{ck} = F_{ck} + 1.65S = 30 + 1.65\ (5) = 38.25\ N/mm^2$
2. Water cement ratio = 0.40
3. Maximum Water Content is 186 litres. Adjusting the water content based on aggregate used and super plasticiser used. Here we have used 162 litres (Table 2: IS 10262:2009).
4. Cement Content = (162/0.40) = 405kg/m^3. Maximum cement content is 450 kg/m^3.
5. Calculation of Volume of coarse and fine aggregate is calculated from Table 3 of IS 10262: 2009. Applying for size and workability, Corrected Volume of coarse aggregate is 0.66 and fine aggregate is 0.34.

Mix Calculations

a. Volume of concrete = 1 m^3.
b. Volume of cement = 0.129 m^3.
c. Volume of water = 0.162.
d. Chemical Admixture = 0.0035 m^3.
e. Volume of All in aggregates = 0.7055 m^3.
f. Mass of Coarse aggregate
   Coarse aggregate (55% of total CA x Specific Gravity 2.64) = 693.55 kg/m^3.
   10 mm (45% of total CA x Specific Gravity 2.64) = 567.45 kg/m^3.

In case of replacing natural CA by Dholpur Stone (quartz sandstone), the above step has to modified accordingly.

g. Mass of Fine aggregate = 645 kg/m^3.

Final Mix proportions

Cement = 405 kg/m^3.

Water = 162 kg/m^3.

Fine aggregate = 645 kg/m^3.

Coarse aggregate, 20 mm = 693.55 kg/m^3.

Coarse aggregate, 10 mm = 567.45 kg/m^3.

Superplasticiser = 4 kg.

Annexure III

Concrete mix Proportions

Table 1: Mixture proportions and properties of fresh concrete for water-cement ratio of 0.4.

Ingredients per cu.m	Id					
	BA	BB	CA	DA	EA	FA
Cement, kg	405	405	405	405	405	405
Water, kg	162	162	162	162	162	162
20 mm CA, kg	693.55	554.84	416.13	277.42	138.71	-
10 mm CA, kg	567.45	453.96	340.47	226.98	113.49	-
FA, kg	645	645	645	645	645	645
Quartz Sandstone Aggregate 25 mm, kg	-	151.32	302.64	453.96	605.28	756.6
Quartz Sandstone Aggregate 10 mm, kg	-	100.88	201.76	302.64	403.52	504.4
Admixture, %	1	1.05	1.1	1.15	1.2	1.2
Compaction factor	0.89	0.9	0.88	0.88	0.86	0.86

Table 2: Mixture proportions and properties of fresh concrete for water-cement ratio of 0.35.

Ingredients per cu.m	Id					
	A	B	C	D	E	F
Cement, kg	440	440	440	440	440	440
Water, kg	154	154	154	154	154	154
20 mm CA, kg	683.65	546.92	410.19	273.46	136.73	-
10 mm CA, kg	559.35	447.48	335.61	223.74	111.87	-
FA, kg	635.47	635.47	635.47	653.47	653.47	653.47
Quartz Sandstone Aggregate 25 mm, kg	-	149.16	298.32	447.48	596.64	745.8
Quartz Sandstone Aggregate 10 mm, kg	-	99.44	198.88	298.32	397.76	497.2
Admixture, %	1	1.05	1.1	1.15	1.2	1.2
Compaction factor	0.9	0.9	0.89	0.88	0.88	0.87

Table 3: Mixture proportions and properties of fresh concrete for water-cement ratio of 0.45.

Ingredients per cu.m	Id					
	KA	KB	KC	KD	KE	KG
Cement, kg	342.22	342.22	342.22	342.22	342.22	342.22
Water, kg	153.9	153.9	153.9	153.9	153.9	153.9
20 mm CA, kg	713.29	570.63	427.97	285.31	142.66	-
10 mm CA, kg	589.60	466.91	350.16	233.44	116.72	-
FA, kg	662.05	662.05	662.05	662.05	662.05	662.05
Quartz Sandstone Aggregate 25 mm, kg	-	155.63	311.25	466.88	622.50	778.13
Quartz Sandstone Aggregate 10 mm, kg	-	105.35	207.5	311.25	415	518.75
Admixture, %	1	1.05	1.1	1.15	1.2	1.2
Compaction factor	0.89	0.9	0.88	0.88	0.86	0.86