Behaviour of Cement Concrete Containing Marble Waste as Coarse Aggregate in Aggressive Environment

Ph.D. Thesis

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CANDIDATE'S DECLARATION

I hereby certify that the thesis entitled "**Behaviour of Cement Concrete Containing Marble Waste as Coarse Aggregate in Aggressive Environment"** submitted in partial fulfillment of the requirements for the award of **Doctor of Philosophy** in **Civil Engineering** to the **Malaviya National Institute of Technology, Jaipur** is an authentic record of research work carried out by me under supervision and guidance of Prof. Ashok K. Vyas. The work incorporated in this thesis has not been submitted elsewhere for the award of any degree.

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

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CERTIFICATE

This is to certify that the thesis entitled “**Behaviour of Cement Concrete Containing Marble Waste as Coarse Aggregate in Aggressive Environment”**, submitted by **Sudarshan Dattatraya Kore (2013RCE9008)** to **Malaviya National Institute of Technology, Jaipur**, is a record of bona fide research work under my supervision and I consider it worthy of consideration for the award of the degree of **Doctor of Philosophy** of the Institute.

Signature of Supervisor
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Abstract

A huge amount of waste is generated during quarrying and processing of marble stone. It has been reported that, approximately 50% of waste is generated during mining operation and about 15% during processing work. Majority of this waste is in the form of pieces stones of irregular shape and sizes dumped on open land and along roads, which create a lot of environmental as well as health problems. Management of huge quantity of waste to prevent such adversities is a major challenge to the society. In the present study the marble waste obtained from nearby area was crushed into crusher to obtain the desired gradation suitable for coarse aggregate. A detailed characterization of marble waste was carried out in this study. Mechanical and durability properties of concrete with marble waste as coarse aggregate were investigated.

The concrete mixes were designed as per BIS-10262-2009 and particle packing density method keeping the constant water-cement ratio 0.45. Conventional coarse aggregate was replaced in the range of 20%-100% by aggregate produced from marble waste in concrete mixes.

The results obtained in the study indicated that, the concrete mixes prepared with marble waste as coarse aggregate show improvement in workability and compressive strength. The concrete produced with marble waste as coarse aggregate does not have any adverse impact on compressive strength. The incorporation of marble waste as coarse aggregate in concrete mixes with packing density approach resulted in appreciable reduction in permeability by 7% to 8% as compared to that of control mixes. The marginal improvement in resistance against abrasion was noticed.

The chloride ion penetration depth in concrete mixes reduced by 4% and 8% as compared to that of respective control mixes after 91 days of exposure. Under the action of 10% magnesium sulphate solution, the concrete with marble waste performs better as compared to that of control mixes. In order to understand the possible mechanism the microstructural study was carried out and it indicated that, the dolomite mineral is formed in concrete mixes with marble waste, which is responsible for improving the bond between aggregate –cement paste.

When concrete specimens were also exposed to standard fire, concrete mixes designed by BIS code method showed increase in compressive strength by approximately 15% up to 400°C due to accelerated hydration of cement paste. On the contrary, mixes designed by packing density method showed loss in compressive strength due to excessive internal pore pressure. The results of flexural strength were on the same lines to that of compressive strength. All the concrete mixes showed increase in water absorption with increase in temperature levels after fire exposure.

Further, durability properties like carbonation, chloride ion penetration, resistance to acids and sulphates were evaluated for specimens subjected up to 400°C temperature. From the carbonation test it was observed that, depth of penetration of carbon di oxide in concrete mixes with marble aggregate increased significantly by 8% after 91 days of exposure. Even after exposure to fire the depth of chloride ion penetration was lesser by 22% after 91 days. Concrete mix with marble aggregate under the action of chemical attack such as acids and suphates showed significant reduction in compressive strength of concrete.

Overall the product becomes sustainable with saving of 14% in overall cost of concrete when designed by packing density approach. From the study it was observed that, it has a good potential for bulk consumption of marble waste in construction industry in an environmental friendly manner. The utilization of this waste not only solves the problem associated with disposal of marble waste but also conserve the natural resources.

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List of Abbreviations

BIS : Bureau of Indian Standards

CH : Calcium Hydroxide

CSH : Calcium Silicate Hydrate

E : Ettringite

V : Voids

CaO : Calcium Oxide

CO_2 : Carbon di Oxide

GGBS : Ground Granulated Blast Furnace Slag

GSI : Geological Survey of India

IIT : Indian Institute of Technology

MNIT : Malaviya National Institute of Technology

OPC : OPC- Ordinary Portland Cement

PPC : PPC- Portland Pozzolane Cement

SCC : SCC- Self Compacting Concrete

SEM : SEM- Scanning Electron Microscopy

TGA : TGA- Thermo Gravimetric Analysis

XRD : XRD- X-Ray Diffraction

1 INTRODUCTION

1.1 General

Concrete is the most versatile and widely used construction material. Huge amount of concrete is being used for building infrastructure facilities such as dams, bridges, under-water structures, water retaining structures, roads and buildings. The primary components of concrete are aggregates, cement/ binder and water. The global annual production of concrete in the year 2014 was estimated as 4.2 billion tons and which is expected to increase by 2.9 % in 2018 (Şahan Arel 2016). Approximately 60 to 75% constituents of concrete consist of aggregates. The large scale production of concrete consumes huge quantity of conventional coarse aggregate which strain the natural sources of raw material extraction resulting in ecological imbalance, climate change, effect on wild life, air pollution, etc.

In the light of the above adverse effects, many researchers started using industrial or mining waste as aggregate in concrete mixes. Fly ash, Ground Granulated Blast Furnace Slag (GGBS), copper and steel slag have been tried as replacement for fine aggregate in concrete mixes by many researchers in the past. Similarly Limestone, Sandstone mining waste and cutting waste have also been investigated as partial replacement of coarse aggregate in concrete mixes.

In a study carried out by Elçi et al. (2015), normal strength concrete was produced using limestone waste obtained from five different quarries in Turkey. The conventional coarse aggregates were completely replaced by aggregate produced from limestone waste. They concluded that, aggregate produced from lime stone waste can be effectively used as complete replacement of conventional coarse aggregate without any adverse effects on concrete strength.

Rana et al. (2015) in their study compared the rheology of aggregate produced from limestone waste with other conventional aggregates (Basalt, Quartz and Sandstone). They reported that, limestone and sandstone concrete mixes were more workable because these aggregates were more or less round shaped.

Kumar et al. (2016) used the waste produced from sandstone quarry as partial replacement for conventional coarse aggregate. They found that the desired workability can be easily achieved after complete replacement. At 40% substitution there was no significant fall in compressive strength.

In addition to the above mentioned stone waste, marble waste has also been used by various researchers to evaluate the feasibility of partial or complete replacement of conventional coarse aggregate.

A few studies have been carried out by past researchers on utilization of marble waste as replacement for conventional coarse aggregate in concrete mixes. A preliminary study on possible use of marble waste as replacement for conventional coarse aggregate was reported by Sadek M. et al. (2002). They reported that 75% replacement of coarse aggregate obtained from marble waste increases the compressive strength by 27.7% and flexural strength by 11.7%. Binici et al. (2008) reported that 100 % replacement of natural coarse aggregates by marble waste in concrete mixes increases the compressive strength, flexural strength and split tensile strength. They also reported improvement in sulphate resistance and reduction in chloride ion penetration. Hebhoub et al. (2011) reported that, 75% replacement of conventional coarse aggregate by aggregate produced from marble waste increases the mechanical properties of concrete. Another study by Ceylan, H., & Manca (2013) reported that, concrete produced by using marble aggregate as complete replacement for conventional aggregate achieved target mean strength at all curing ages. André et al. (2014) reported that, partial replacement of conventional coarse aggregate by marble waste in different percentages (20% to 100%) in concrete mixes improves workability and compressive strength. They also reported that, chloride ion penetration reduced but carbonation was almost same in both the mixes. Abdul et al. (2014) reported that, complete replacement of conventional coarse aggregate by marble waste improves the physical and mechanical properties of concrete.

A long term comprehensive study on mechanical and durability properties of concrete mixes using marble waste as coarse aggregate was needed to justify its use in infrastructure works and eliminate associated problems regarding waste disposal. Following paragraphs give details of its production in the state of Rajasthan.

1.2 Marble Stone industry

In India the state of Rajasthan is known as mineral majestic State as it produces more than 50 types of mineral and rocks, a few important ones amongst them are Granite, Marble, Sandstone, Limestone, Slate and Quartzite. Marble – generally a white based elegant looking stone, geologically a thermally metamorphosed rocks belonging mainly to Precambrian rock formations of Rajasthan, spread over in 16 belts in 15 districts of the state is much sought after stone. The occurrence of marble is in districts of Nagaur, Uadaipur, Banswara, Jaipur, Sirohi, Bhilwara, Jaisalmer, Bundi, Pali, Dungarpuer, Ajmer, Chittorgrah and Sikar.

The world famous Taj Mahal and Victoria Memorial are built of marble produced from the mines of Makrana (Rajasthan).

1.3 Origin of Problem

The stone industries are the backbone to the economy of Rajasthan state, India. Approx. 95% of production of marble in India is from Rajasthan state alone (MSME-Development Institute Govt. of India, Ministry of Micro, Small & Medium Enterprises 2009). Marble is widely used in buildings, monuments and sculptures. Its utility value lies in its beauty, strength and resistance to erosion.

Rajasthan has around 4000 marble mines and about 1100 marble gang saws. The waste generated during the mining operation is approximately 50% of the overall production (MSME-Development Institute Govt. of India, Ministry of Micro, Small & Medium Enterprises 2009). According to Jain et al. (2015) in India the solid waste generation per annum is around 48 million tons. The waste produced during mining and processing operation is huge in quantity and a little part of it is used in small scale in manufacture of tiles and statues and rest is dumped on open land, roadsides forestland, pasture lands & agricultural fields leading to widespread environmental pollution and loss of fertile top soil. Such wastes adversely affect vegetation in nearby area and create lot of environmental and health problems to the people in nearby area as shown in Figure 1.1.

Figure 1.1 Marble waste dumped in open lands

The marble quarries and industries impose significant environmental impacts on the surrounding areas. It is generated in the form of solid marble waste i.e. odd size and shapes of marble pieces, slurry and powder which create environmental pollution in nearby areas of mining and processing industries.

Table 1.1 Quantum of quarry waste produced

Stage of Marble Industry	Type of marble waste	Nature of Marble Waste	Mechanized mines & Processing units using gang saw cutting machines	With mechanized mines using blasting	With semi mechanized mines using blasting	Weighted Average Waste
Mining & trimming up to the stage of dispatch to processing units	Mine waste	Odd shaped & sized blocks & fragments of various sizes (mine slurry not recoverable)	30%	55%	65%	50%

(Source : MSME-Development Institute Govt. of India, Ministry of Micro, Small & Medium Enterprises 2009)

It can be seen from the Table 1.1 that, quarry waste produced is minimum in mechanized mines and processing units. The quantum of waste increases in semi mechanized mines using blasting. However, the average waste generation during the mining operation is about to 50% to 60%. That is a huge amount and needs to be disposed with minimum cost. The best way is to use this waste in some infrastructure works. Such utilization is going to reduce burden on disposal system and cost of the infrastructure shall be reduced.

1.4 Need for Further Research:

In view of the findings by past researchers it can be observed that, workability and mechanical properties slightly improve compared to control mixes. Some researchers have also studied a few durability aspects as reported in previous paragraphs. But long term mechanical and durability properties are still not known when marble waste is used as coarse aggregate in concrete mixes. Moreover, behavior of concrete using marble waste in aggressive environment like exposure to fire and acid has still not been traced. Savings in cost and energy data have not yet been reported.

Hence this research work was taken up for long term detailed study on use of marble waste as coarse aggregate with the following specific objectives;

- Characterization of Marble Waste and Conventional Aggregate
- To study the influence of marble waste as partial replacement of conventional coarse aggregate on mechanical and durability properties of concrete.
- To study the behavior of concrete containing marble waste as partial replacement of conventional coarse aggregate, exposed to aggressive environments like fire and acid solution.

1.5 Organization of Thesis

The thesis has been divided in to six chapters. A brief introduction highlighting the marble waste generated during the quarrying and processing stages, their impact on the surrounding environment is written in the Chapter 1. A comprehensive review of use of marble waste as a partial replacement for fine aggregate and partial replacement for conventional coarse aggregate in concrete mixes along with their impact on the properties of concrete such as workability, strength and durability has been described in Chapter 2. The characterization of all the materials used in the study has been included in Chapter 3. The methodology adopted for the design of concrete mixes and testing of properties of concrete has been written in detail in chapter 4. The detailed analysis of the test results in the form of graphs and tables and discussion of the entire study along with suitable reasoning is included in Chapter 5. In Chapter 6 the overall conclusion drawn from the entire study along with the future scope for the work is incorporated.

2 LITERATURE REVIEW

2.1 General

A huge amount of waste is generated during quarrying and processing of marble stone. Approximately 50% of waste is generated during mining operation and around 15% during processing (Status Report ,2009). This waste is in the form of stones of irregular shape and size and slurry that are dumped on open land and along roads, which create a lot of environmental as well as health problems. These include water pollution, air pollution, degradation of aquifers, de-vegetation of the area, visual impacts, and loss of flora & fauna. Scarcity of available vacant lands adds to these woes. Management of huge quantities of waste to prevent such adversities is a major challenge for the society. Many researchers in the past have attempted to use these wastes in one form or another in construction activities.

2.2 Marble waste in concrete

Various researchers carried out studies on use of marble waste as a construction material in concrete mixes. Some of the researchers used fine powder as a partial replacement for cement in concrete mixes, others used it as a partial replacement for fine aggregate and coarse aggregate in concrete mixes.

2.2.1 Use of Marble Powder as a partial replacement for cement in concrete

A number of studies have been carried out using marble powder as a partial replacement of cement in concrete mixes by many researchers.

Ergün (2011) reported that, replacement of cement by marble powder in different percentages 5%, 7.5% and 10% enhances the mechanical properties of concrete mixes. It was reported that, compressive strength increased by 11.29%, 12.72% at 5% and 7.5% replacement but at 10% replacement it decreased by 13.82%

as compared to that of control mixes. On the contrary flexural strength decreased by 3.92% and 6.07% at replacement level of 7.5% and 10%, respectively. However at 5% replacement flexural strength, was found to be equal to that of control concrete. He observed that, the marble powder acts as a filler which resulted in dense matrix of the concrete mixes. Another important finding reported by him was that marble powder had a binding property which developed chemically by the hydration of calcite and C_3A which reduces the porosity in concrete matrix.

Another study carried out by Shirule et al. (2012) used marble powder as a partial replacement for cement in concrete mixes with a constant water-cement ratio of 0.5. They reported that, compressive strength of concrete mixes increased by 13.2% and 17.7% at 5% and 10% replacement levels whereas at 15% and 20% replacement compressive strength decreased by 13.3% and 17.8%, respectively. The same study reports that, split tensile strength of concrete mixes increased by 5.44%, 11.52%, and 5.44% at 5%, 10% and 15% replacement levels, respectively. But at 20% replacement, it decreased by 5.75%. Finally they also suggested that, the optimum percentage for replacement of cement by marble powder was 10% by weight.

In a study carried out by Shelke et.al. (2012) marble powder was used as 8%, 12% and 16% replacement of cement and a constant 8% silica fume was added to concrete mixes with a water-cement ratio 0.45. They reported that, workability decreased as the replacement of cement by marble powder increased. It was also reported that, compressive strength increased by 3.92% at 8% replacement level and at 12% and 16% replacement, the compressive strength decreased by 10.3% and 20.83%, respectively. The optimum percentage for replacement of cement by marble powder was recommended by authors to be 8%.

Another study carried out by Omar et al. (2012) on concrete mixes with partial replacement of cement by marble powder (5%,10% and 15%). It was reported that, compressive strength of concrete mixes increased by 5%, 16% and 21% at 5%, 10% and 15% replacement of cement by marble powder. A similar trend was observed in split tensile strength and flexural strength of concrete mixes. They also reported, enhancement in properties of concrete such as compressive strength, split tensile strength and flexural strength was due to the presence of active SiO_2 in marble powder which is capable of reacting with $Ca(OH)_2$ in concrete to form secondary calcium silicate hydrate, making it structurally dense.

Mishra et al. (2013) used marble dust as partial replacement for Portland cement in concrete mixes with a constant water-cement ratio of 0.46 in different percentages 0% to 10% by weight. Marble dust was ground together with Portland cement clinker. They reported that, compressive strength of the resulting blended cement concrete mixes increased by 19% at10% replacement level as compared to that of control concrete. The increase in compressive strength was due to increase in fineness of blended cements which helps in development of uniform pore spaces and products of hydration.

Noha Soliman et al (2013) carried out studies where marble powder was used as a partial replacement for cement in different percentages 0% to 20% at an increment of 2.5% in concrete mixes with a constant water-cement ratio of 0.45. They reported that, replacement of cement by marble powder up to 10% increased the workability. Further replacement of cement by marble powder decreases workability due to increased surface area of fine particles. It was reported that, compressive strength of concrete mixes increased by 10% to 25% at replacement 2.5% to 5% by weight. However further increase of replacement of marble powder in concrete resulted in fall in compressive strength. The tensile strength of concrete was increased by 5.6% at 5% replacement level. The modulus of elasticity of concrete increased by 2.8% to 11.2% at replacement 2.5% and 5% by weight. Higher replacement of cement by marble decreases the compressive strength, tensile strength and modulus of elasticity of concrete. It was reported that, marble powder acts as an inert material, which enables dispersion of cement paste which resulted in increase in strength of concrete mixes. Finally it was suggested that, the optimum percentage for replacement of cement by marble powder was 5%.

In a study by Vaidevi (2013), concrete mixes were prepared by replacing cement by marble powder (finer than 250 µm) in different percentages 5%, 10%, 15% and 20% with constant waster-cement ratio of 0.47. It was reported that, up to 10% replacement of cement by marble powder the compressive and tensile strengths improved by 9.75% and 13.64%, respectively. Whereas 20% replacement of cement by marble powder, led to reduction in compressive and tensile strength by 22.64% and 13.63%, respectively. It was also reported that, the reduction in mechanical properties of concrete was due to lack of binding material because marble powder does not contribute in hydration mechanism.

Rana et al. (2015) reported that, marble sludge can be used as a partial replacement for ordinary Portland cement up to 10% in concrete mixes. The dense microstructure of concrete formed due to the inclusion of marble, enhanced the concrete mixes’ durability properties namely resistance to permeation, chloride migration and corrosion, but at the expense of reduced workability. Large surface area of marble powder was attributed to the cause of this reduction in workability of the concrete mixes. It was also reported that, 10% marble sludge as partial replacement of cement does not affect the formation of hydration products.

In a study taken up Talah et al. (2015) it was reported that, replacement of ordinary Portland cement by 15% marble powder in concrete with a water-cement ratio of 0.50 enhanced the mechanical and durability performance. The study showed that, there is a slight decrease in workability, air content and increase in density of concrete mixes as compared to that of control concrete. The compressive strength of concrete containing 15% marble powder was found to increase from 52 MPa to 65 MPa, whereas the compressive strength of reference concrete increased by 39 MPa to 48 MPa, as curing age increased from 28 days to 365 days, respectively. They also reported that, resistance to chloride ion penetration increased because marble powder cannot degrade the concrete under hydrochloric media.

2.2.2 Use of marble waste as a partial replacement for fine aggregate

Hameed et al (2009) in their study reported that, replacement of natural sand by marble powder and quarry rock dust in equal proportion (50% quarry rock dust and 50% marble powder) in concrete mixes showed improvement in compressive and split tensile strengths by 9.5% and 8.65%, respectively. The permeability of concrete reduced by 19% as compared to that of control specimens. The same study showed that, replacement of sand by marble powder from 25% to 100% in 1:3 mortar mixes increased the resistance to sulfates and acids as compared to that of control mortar mixes. It was also reported that, the compressive strength of mortar specimens immersed in magnesium sulphate solution marginally increased by 0.75% to 1.06% as replacement of fine aggregate by marble powder increased from 25% to 100% as compared to that of control specimens.

Corinaldesi et al. (2010) conducted studies on mortar mixes (1:3) with marble powder as replacement for natural sand up to 10% by weight. They reported that, 10% replacement of sand by marble powder, reduces compressive strength by 10% which was insignificant and workability can be maintained by using a dose of acrylic based super-plasticizers.

Hebhoub et al. (2011) conducted studies on concrete mixes with crushed marble waste as a partial replacement for sand. They reported here that, workability of concrete mix decreased as the percentage of replacement increased due to higher water absorption of natural aggregates. The compressive strength of concrete mix increased by 17.2%, 23.65% and 16.1% at 25%, 50% and 75% replacements, respectively but at 100% replacement level the compressive strength was reduced by 23.29%. With regard to tensile strength the same trend as that of compressive strength was observed. It was also stated that, the desired workability could be achieved by applying proper dose of water to compensate the absorbed water by marble waste and natural aggregate during mixing.

Rai et al. (2011) also prepared concrete mixes by replacing fine aggregate using marble waste in proportions from 0% to 20% with a constant water-cement ratio of 0.44. They reaffirmed that, compressive and flexural strength of all mixes was 5% to 10% higher than that of control mixes.

Complete replacement of fine aggregate by crushed marble waste for a concrete mix with constant water-cement ratio of 0.45 was done by Uygunolu et al. (2012). Here ordinary Portland cement was also replaced by fly ash in different percentages 10%, 20%, 30%, and 40%. Their results indicated that, pre-fabricated concrete interlocking blocks achieved sufficient quality. It was also reported that, porosity, resistance to abrasion and water absorption increased marginally due to filling effect of marble waste and hydration of fly ash which make the concrete chemically stable and much denser than that of control concrete. The concrete paving blocks prepared with replacement of sand by marble powder up to 30% and cement by fly ash up to 30%, had higher resistance to alkali silica reaction due to lower quantity of alkali silica gel generated with marble waste during hydration.

Gencel et al. (2012) carried out a study to evaluate the potential use of crushed marble waste as a partial replacement for conventional fine aggregates in different percentages 0% to 40% for production of concrete paving blocks. They reported that, compressive strength, density and water absorption decreased by 22%, 5% and 28% as compared to that of control concrete at 10% to 40% replacements of fine aggregate by marble powder. It was also reported that, resistance to abrasion increased by 20.14% and relative strength loss after freeze –thaw resistance decreased by 4.43% at 40% replacement.

Gameiro et al (2013) investigated the effect of partial replacement of conventional fine aggregate by marble mining waste 20%, 50%, and 100% on the physical and mechanical properties of concrete with a constant water-cement ratio 0.55. They concluded that, replacement of sand by marble mining waste up to 20% in concrete mixes improved the mechanical and durability properties. They also reported that, the workability decreased due to increase in surface area of fine particles which can be adjusted by increasing the water-cement ratio. Compressive strength decreased as the replacement ratios of fine aggregate by marble mining waste increased due to increase in water-cement ratio.

Silva et al. (2014) investigated the influence of replacement of fine aggregate by marble mining waste on concrete mixes. The replacement level of fine aggregate by marble powder was kept at 20%, 50% and 100% without changing the water-cement ratio of 0.43. They reported that, workability of concrete mixes decreased as replacement level for fine aggregate by marble mining waste increased and this can be maintained by adjusting the water-cement ratio. The same study reports that, the compressive strength decreased by 2.5% to 10.2%, split tensile strength increased by 1.5% to 3.6%, modulus of elasticity increased by 1.9% to 3.5%, abrasion resistance decreased by 10.1% to 31.9% as replacement level for fine aggregate by marble mining waste increased from 20% to 100%, respectively. The cause behind marginal decrease in properties of concrete mixes was, increase in water-cement ratio and poor wear resistance of fine aggregate from marble mining waste. Finally they concluded that, at higher replacement levels the mechanical properties of concrete mixes were slightly reduced and these aggregates could be used in structural concrete mixes.

Ural et al. (2014) conducted studies on concrete mixes with marble dust (finer than 300 μm) as a partial replacement for conventional fine aggregate from 5% to 15%. They reported that, the addition of marble dust requires more water for lubrication due to increased surface area of marble dust. The compressive strength at 5% replacement was increased by 16.74% and at higher replacement levels marginal decrease in compressive strength was reported. The ultrasonic pulse velocity values increase by 1.8%, 9.18% at 5% and 15% replacement of conventional fine aggregate by marble dust. It was concluded that, the replacement of fine aggregate by marble dust in concrete mixes reduced the porosity by filling of macro pores and enhanced the performance of concrete from the durability aspects.

2.2.3 Use of marble waste as a partial replacement for conventional coarse aggregate

In a study by Sadek M. et al. (2002), conventional coarse aggregate were replaced by marble waste in different proportions 0% to 100% by weight in manufacturing of cement concrete bricks. It was reported that, 75% replacement of coarse aggregate obtained from marble waste increases the compressive strength by 27.7%, flexural strength by 11.7% and water absorption of concrete reduced by 53.7% as compared to that of control concrete. They also reported that, the solid concrete bricks produced by using marble waste as coarse aggregate satisfy the required criteria of Egyptian standards.

Binici et al. (2008) investigated the influence of 100 % replacement of natural coarse aggregates by marble waste, in concrete mixes with a constant water-cement ratio of 0.4. River sand and ground blast furnace slag (GBFS) were used as fine aggregate. They reported that, concrete mixes prepared with marble waste were more workable than that of control mixes due to low water absorption and smooth surface texture of marble waste. The average compressive strength, flexural strength and split tensile strength of concrete mixes increased by 27%, 7.25% and 3.75%, respectively when compared to that of control concrete. It was also reported that, these concrete mixes showed considerably better performance under chemical attack. The concrete mixes containing marble aggregate showed nearly about 30% reduction in compressive strength as compared to 50% in control concrete when immersed in 10% sodium sulphate solution. The depth of chloride penetration was also reduced by 70% as compared to that of control concrete.

Hebhoub et al. (2011) conducted studies on use of marble waste as partial and complete replacement (0 to 100%) in the form of fine aggregate and coarse aggregate in concrete with 0.5 as water-cement ratio. It was reported that, workability decreased as replacement level increased and insignificant change in density of concrete mix was observed. Compressive strength of all concrete mixes increased as percentage for replacement of conventional coarse aggregate by marble aggregate increased. It was reported that, an increase in compressive strength was 16.84% for mixes, prepared with 75% replacement of fine and coarse aggregate.

Gencel et al. (2012) described the potential use of aggregate produced from marble waste as a partial replacement for conventional coarse aggregate in different percentages 0%, 10%, 20% and 40% for preparation of concrete paving blocks. It was reported that, for all the concrete mixes density and water absorption decreased by 3.9% and 20%, respectively for 40% replacement. The ultra-sonic pulse velocity also decreased to 3.95 km/sec, as replacement of conventional coarse aggregate by marble aggregate increased to 40% by weight. Marginal reduction in compressive strength from 5.7% to 14% at 10% to 40% replacement level was reported. Also the same trend was reported in split tensile strength of concrete. It was reported that, the average relative strength loss of concrete blocks prepared by replacing conventional coarse aggregate by marble waste (0% to 40%) after freeze-thaw cycles was approximately 41.65% less than that of control concrete. Finally they concluded that, marble waste can be used as a replacement for conventional coarse aggregate up to 40% to produce the concrete paving blocks.

Martins et al. (2014) reported that, coarse aggregate from marble waste can be used as a partial replacement for conventional coarse aggregate to improve the mechanical properties of concrete. They also reported that, the workability of all concrete mixes increased by 4.16% to 9.34% and density marginally decreased by 0.28% to 4.21% at 20% to 100% replacement level. With regard to compressive and tensile strength, marginal reduction by 5.2% to 6.2% and 1% to 10.4%, at replacement of 20% to 100%, respectively was reported. This decrease was approximately10% and found to be insignificant. It was also reported that, concrete mixes prepared using marble waste as coarse aggregate showed reduction in abrasion resistance by 26.8% as compared to that of control concrete mixes.

In a study by André et al. (2014), marble waste was used in different percentages (20% to 100%) as replacement for conventional coarse aggregate. The improvement in workability of concrete mixes containing marble aggregate was reported, due to plain surface and low water absorption of marble aggregates as compared to that of conventional coarse aggregates. With regard to compressive strength it was stated that, concrete mixes showed downward trend with increasing incorporation ratio from 20% to 100%, but this decrease was considered almost insignificant with variations up to 10.3%. They also reported that water absorption and depth of carbonation of concrete containing marble aggregate showed similar results to that of control concrete. They also reported, the chloride penetration resistance of concrete containing marble aggregate increased due to presence of alumina in marble aggregate.

Another study by Ceylan et al (2013) reported that, concrete produced by using marble aggregate as complete replacement for conventional aggregate achieved target mean strength at all curing ages as per Turkish standards. It was also reported that, higher values of ultrasonic pulse velocity were obtained and Schmidt surface hardness of waste marble aggregate was more than that of conventional aggregate.

In a study by Abdul et al. (2014) marble waste was used as a complete replacement for conventional coarse aggregate. They reported that, the physical and mechanical properties of concrete improved. The workability of concrete mixes increased by 50% due to smooth flat surface and low water absorption of marble aggregate. It was also reported that, the compressive and flexural strength of concrete containing marble aggregate increased by 29.62% and 11.44% as compared to that of control mix.

In view of the above findings it can be observed that, strength of concrete with 75% marble waste as coarse aggregate and 25% natural aggregate gives maximum compressive strength and flexural strength. Properties like water absorption, suphate resistance, and resistance to chemical attack were improved by use of marble waste as coarse aggregate in concrete mixes.

2.3 Packing Density Approach for Design of Concrete Mixes

There are various methods available for the design of concrete mixes. In general practice the concrete mixes are designed by using the national standards.

Around 75% of constituents of concrete consist of coarse and fine aggregates. In packing density approach these aggregates are mixed in a proportion to arrive at maximum packing density and minimum void content. Cement paste is added in the selected mixture of aggregates to achieve desired workability and strength. In the design of concrete mixes by packing density approach the quantity of aggregates (coarse and fine) increases when compared to the conventional method of mix design. The increase in packing density and minimum voids content in the aggregate mixture results in reduced amount of binder required to give the desired strength of concrete mix. The concrete mix becomes stiff and hence desired workability is not achieved. Therefore, super plasticizers are required to be added in the mix to attain desired slump (Wong et al. 2013). Reduced usage and hence reduced production of cement content in concrete results in lesser emission of carbon dioxide in the atmosphere. Overall the concrete becomes cost effective. Work done by a few researchers is described briefly in subsequent paragraphs.

Powers T. C. (1968) suggested that, the void ratio of the binary particulate system is minimum at a particular combination of aggregate fractions.

Lecomte et al (2005) in their study reported that, higher coarse aggregate content would results in significant decrease in compressive strength due to bleeding which resulted in alteration of bond between cement paste and aggregate.

Obla et al. (2007) in their study reported, the broad distribution of aggregate particles in combined aggregate gradation led to lower voids content. It was also reported that, addition of fine aggregate and extending the range of aggregate from 2.36 mm to 37.5 mm decreases the void content approximately by 19%.

A study by Kwan & Wong (2008), depicts the effect of blending cement with various supplementary materials like pulverized fuel ash and condensed silica fume on packing density. They reported that, addition of ultrafine particles in the cement increases the packing density and addition of super plasticizer always increases the packing density of cementetious materials.

Fennis et al. (2008) in their study reported, how centrifugal consolidation can be used to measure the packing density of powders. They also reported, the replacement of ordinary Portland cement by fly ash content 30%, 50% and 70% will increase the packing density of paste. It was also reported that, the particle packing density method can be used to lower cement content in concrete for economical design.

De Larrard (2009) reported that, workability and packing density can be improved by using gap graded aggregates. It was also stated that, fine particle in the aggregate mixture plays an important role for improving the packing density and their rheology when water was added in the mix.

Another study by Fennis et al. (2009) conducted studies on concrete mixes designed by packing density method and using three types of cement replacing materials such as fly ash, quartz powder and ground incinerator bottom ash. The compressive strength, flexural strength and modulus of elasticity of concrete increased by 23.75%, 20% and 7% as compared to that of control concrete. With regard to creep and shrinkage the concrete produced using fly ash and quartz powder showed lower values as compared to that of control concrete. It was also reported that, the concrete mixes can be designed by using packing density approach and it is possible to reduce the cement content up to 50% and the CO_2 emission by 25%.

In a study by Fung et al. (2009), the comparison between theoretical models and experimental packing density with compacted and un-compacted condition on cement mortar with different sizes of aggregate fractions was investigated. They reported that, the packing density of blended fine aggregates is higher than that at un-compacted condition and less sensitive to compaction. It was also reported that, the packing density values obtained by experimental methods are closer to that of theoretical models.

In a study conducted by Shekarchi et al. (2010), different methods to determine the packing density of aggregates were used. It was reported that, packing density of aggregate mixture obtained by dry packing methods with compaction is slightly more by 1% to 3% and by loose packing methods was 7% to 12%, less than that of Toufer model. It was suggested that, the dry packing method with compaction is more suitable for packing density measurement.

Study by Marc Rached et al. (2010) reported that, shape and size of aggregates affect the packing density and required paste volume to achieve the desired strength and workability. Poorly shaped or poorly graded aggregates can increase the paste volume and this can be minimized by using the gap graded aggregates. The compressive strength of all concrete mixes show nearly similar trend, but it was adversely affected when mixture containing low paste volume and high fine aggregate ratio used. They finally concluded that, decreased paste volume results in decreased shrinkage and permeability of concrete mix.

A study by Sobolev et al (2010) reported that, for obtaining dense structure and optimum packing density the aggregates falling in the range of 0.7 times maximum size of aggregate should not be less than 40%. Another important fact reported was, higher or denser packing can be achieved by the use of wide range of aggregate particles.

A study conducted by Anson-cartwright (2011) reported that, by inclusion of an intermediate sized aggregate in the concrete mix, a reduction in cement paste by up to 16% was possible.

Kwan et al. (2012) in their study used fine and coarse aggregates, blended together to determine the packing density under compacted and un-compacted conditions with addition of super-plasticizer. They reported that the blending of fine and coarse aggregate together increases the packing density of aggregate mixture. The use of 1% super plasticizer by weight of cement and compaction by tamping rod would always increase the packing density of aggregate. They concluded that, increasing the size range of aggregate fraction from 1.18 to 20 mm and blending them together resulted in significant increase in packing density by 40%.

In a study carried out by Eenu et al. (2012) high performance concrete was designed by using packing density approach. They reported, aggregate combinations with high packing density yielded higher values of compressive strength and higher values of compressive strength was observed when the coarse aggregate content was not less than 50%. It was also reported that, the aggregate combinations having high angularity exhibited lower values of packing density.

The presence of more coarse particles and high packing density would reduce the required paste content and improve the volume stability resulting in better durability. This fact was reported by Mangulkar & Jamkar (2013) .

Kwan et al. (2014) in their study used different size class of fine aggregate and were blended together to determine the effect of fine contents on packing density and void content. They reported, addition of 15% size class finer than 75µm increased the packing density and reduced void content. It was also reported that, the presence of size class finer than 75µm and 150µm particles of fine aggregates would increase the packing density by 14% and reduce void content by 33% when all size class of fine aggregate were blended together and compacted by tamping rod.

In a study carried out by Li et al (2014), concrete mixes were designed by using packing density method in which ordinary Portland cement (OPC) was partially replaced (0% to 30%) by pulverized fuel ash (PFA) condensed silica fume (CSF). They reported, blending of OPC by 20% PFA decreased void ratio by 13% and 14% at cementitious materials content of 15% and 20%, respectively. Whereas the blending of OPC by 20% PFA and 5% CSF decreased void ratio by 16% and 19% at cementitious materials content of 15% and 20%, respectively. It was also reported that, the use of cement in the range of 15% to 20% by volume in concrete mixes gives minimum void content.

Bhattacharjee et al. (2014) in their study reported, the optimum packing density can be obtained by combining the coarse and fine aggregates in a definite ratio. They also reported that, the compressive strength obtained by packing density approach is comparable to that obtained from Bureau of Indian Standards (BIS) code method for a given water-cement ratio. The co-relation curves plotted between compressive strength vs. water-cement ratio and compressive strength vs paste content could be used to decide the water-cement ratio and paste content for specified grade of concrete in packing density method.

In the light of the above mentioned research work it can be concluded that, strength properties of concrete mixes can be improved by selecting adopting packing density approach.

2.4 Effect of elevated temperature on performance of concrete

When concrete is exposed to elevated temperatures, mechanical and durability properties of concrete are also altered (Phan 1996). Partial damage of cement paste takes place due to dehydration at temperature of 110ºC, which is the standard temperature for drying of materials. Progressive deterioration of cement paste starts above this temperature (Feldman & Sereda 1970). During heating slight expansion in cement paste take place at temperature of 200°C which results in increase in total pore volume of cement paste (Piasta 1984). Approximately 65% to 75% of concrete volume remains occupied by aggregates (Hager 2013) and are stable up to a temperature range of 300ºC to 350ºC. The chemical changes occur in the cement paste and aggregates during exposure to elevated temperature above 500ºC (Short et al. 2001). When the temperature reaches about 500ºC to 550ºC, dissociation of calcium hydroxide $Ca(OH)_2$ take place according to following equation (Hager Gaweska 2013),

$Ca(OH)_2 \rightarrow CaO + H_2O\uparrow$

Rehydration of CaO in the presence of moisture turns in to calcium hydroxide $Ca(OH)_2$ above temperature of 550ºC which increases the volume and induces pore pressure within the concrete resulting in formation of cracks and finally concrete loses its strength. The most of non-siliceous aggregates are stable up to temperature of 600ºC whereas at higher temperature the calcareous materials such as calcite, magnetite and dolomite dissociate into an oxide and carbon dioxide is released. The dissociation of calcium carbonate takes place at around 898ºC and the partial dissociation occurs at temperatures as low as 700ºC. The aggregates originated from igneous rock (basalt) shows degassing and expansion within the temperature range of 1200ºC to 1300ºC (Hager 2013). The effect of elevated temperature or fire are visible in the form of surface cracks, change in color and spalling of concrete (Chen et al. 2009).

Li et al. (2004) reported, the effect of specimen size, strength grade and high temperature profiles on concrete performance. The residual compressive strength of C70 grade concrete after exposure to fire at 200, 400, 600 and 800ºC was 82%, 63%, 58% and 27% whereas the residual split tensile strength was 86%, 82%, 52% and 16%. The strength loss after exposure to fire of large size specimens was less as compared to that of small size specimens. It was also stated that, more reduction in strength of high grade concrete was due to dense microstructure which induces the internal pore pressure resulting in formation of cracks and spalling of concrete.

Another study carried out by M.V. Krishna Rao (2005) reported the effect of elevated temperature on performance of high strength concrete (HSC). They reported that, initially the compressive strength of high strength concrete mix up to 250ºC was increased by approximately 49%, 41% and 33% at 1, 2 and 3hr of exposure durations. It was also reported that, above 400ºC temperature compressive strength decreased gradually. The retained compressive strength of high strength concrete at 600, 800 and 950ºC was 91%, 48% and 40%, respectively. This rapid decrease was due to induced pore pressure developed within concrete trying to thrust out which leads to formation of cracks on the surface of concrete results in spalling and deterioration of concrete.

Sakr et al (2005) in their study used different types of cooling regimes and coating material to investigate the behavior of concrete under exposure to elevated temperature. They reported that, compressive strength of unprotected concrete was reduced by 9%, 50%, 68% and 87% whereas for protected concrete it was reduced by 5%, 41%, 67% and 84% at 250ºC, 500ºC, 750ºC and 950ºC, respectively. It was stated that, this reduction in compressive strength of concrete was due to removal of free water and dehydration of cement matrix in the concrete at higher temperature. The same study also reported that, the compressive strength of water cooled unprotected concrete specimens was reduced by 15%, 70%, 88% and 0% whereas in case of protected concrete the reduction was 13%, 67%, 84% and 0% at 250ºC, 500ºC, 750ºC and 950ºC, respectively. Finally they concluded that, use of FR2 viscous colorless fire protection material as coating on concrete has significant effect on the performance of concrete below temperature of 500ºC.

Behavior of ordinary micro concrete (OMC) and high performance micro concrete (HPMC) to elevated temperature with different cooling types was studied by Husem

(2006). He reported that, compressive strength of OMC was reduced by 7%, 12%, 27% and 47% cooled by air and 27%, 29%, 44% and 100% when cooled in water, at temperatures 200,400, 600 and 800ºC. Whereas the reduction in compressive strength of HPMC was 32%, 23%, 26% and 51% cooled by air and 33%, 29%, 34% and 56% cooled by water, in the above mentioned temperature range. It was also stated that, the cooling by water reduces more compressive strength than that cooling by air because, disintegration of concrete takes place in presence of water.

Yin et al. (2000) performed studies on concrete at high temperature with the effect of different cooling regimes on compressive strength and pore properties of concrete was reported. They reported that, the residual strength of normal strength concrete (NSC) under gradual and rapid cooling was 43% and 26%. On the other hand, the residual strength of high strength concrete under gradual and rapid cooling was 32% and 22% at temperature of 800ºC. The porosity of NSC was increased by 94% and 56% while HPC was increased by 170% and 315%, for gradual and rapid cooling. It was also reported that, the denser pore structure was observed in rapid cooling rather than gradual cooling because of re-hydration of $Ca(OH)_2$. From this study it was concluded that, the different cooling regimes have remarkable effect on concrete properties at elevated temperature.

Various researchers used different types of pozzolonic materials such as fly ash, blast furnace slag, silica fume, etc. to enhance the performance of concrete under fire exposure. They found that, these pozzolonic materials show better performance to retain maximum strength and durability of concrete after exposure to higher temperature.

Another study conducted by Poon et al. (2001) reported, strength and durability properties of concretes at elevated temperature up to 800ºC using different pozzolanic materials such as fly ash and blast furnace slag. The replacement levels of cement by fly ash and blast furnace slag were in the range of 0% to 40%. They reported that, pozzolanic concrete mixes showed better performance as compared to that of non-pozzolanic concrete at elevated temperatures. The residual strength of non-pozzolanic concrete at 200, 400, 600 and 800ºC was 93%, 74%, 30% and 10%. The residual strength of concrete containing 40% fly ash was 106%, 84%, 45% and 18% in this temperature range. On the other hand, the residual strength of concrete containing 40% blast furnace slag was 92%, 81%, 54% and 20% in the above mentioned temperature range. Finally they suggested that, the optimum replacement level of cement by fly ash and blast furnace slag were 30% and 40%, to achieve better performance of concrete at elevated temperature.

A study carried out by Arioz (2007) reported, the effect of elevated temperature on the properties of concrete using different types of aggregates crushed lime stone and river gravel. He reported that, when concrete was subjected to temperature of 200ºC to 1200ºC, the loss in weight was increased by 5% to 45%, respectively. The relative strength of concrete prepared by using crushed lime stone aggregate was 105%, 96%, 90%, 30%,13% and 6% and relative strength of river gravel aggregate was 98%, 70%, 40%, 35%,10% and 0% at 200, 400, 600, 800, 1000 and 1200ºC, respectively. It was also reported that, the decomposition of siliceous took place at 570ºC causing volume expansion whereas the lime stone aggregate decompose at 800 to 900ºC and expands with increase in temperature.

A study carried out by Toumi et al. (2009) reported the effect of elevated temperature on the properties of normal strength concrete (NSC), high strength concrete (HSC) and high strength polypropylene concrete (HSPC). They reported that, slight increase in compressive strength by 2%, 3% and 5% of NSC, HSC and HSPC was observed when temperature reaches to 300ºC and maintain for 1 hr. This increase was due to evaporation of free water and removal of crystallized water within the cement paste. It was also reported that, further increase in temperature at 500ºC and 700ºC the loss in compressive strength of NSC was 22% and 42%, for HSC 38% and 71% and for HSPC 6% and 38%. They concluded, the addition of 1.5% polypropylene fibers in HSC improves residual strength because it melts at higher temperature which provides a passage of voids to reduce the explosive spalling.

Noumowe et al. (2009) investigated the permeability of high strength concrete (HSC) with and without addition of polypropylene fibers subjected to elevated temperature for 200 and 600°C. They reported that, the permeability of HSC at 200°C and 600°C increased from $5.5\times10^{-16}\text{m}^2$ to $3600\times10^{-16}\ \text{m}^2$ and in high strength concrete containing 2% polypropylene fibers the values increased from $10.2\times10^{-16}\text{m}^2$ to $2890\times10^{-16}\text{m}^2$. It was also reported that, the permeability of concrete containing 2% polypropylene fibers was 85% higher than that of control concrete. This increase was caused by the melting of polypropylene fibers around 170°C by increasing the porosity in the concrete.

Kizilkanat et al. (2013) reported, the type of aggregate used in concrete and its influence on thermo physical properties of concrete at elevated temperature. They reported that, the thermal conductivity of concrete containing calcareous aggregates was lower than the concrete containing siliceous aggregates at elevated temperature. The calcareous aggregates showed higher moisture resistance factor values among all other types of aggregates. It was also reported, there was no significant change observed in thermal conductivity of concrete and moisture resistance factor values were reduced significantly up to 300ºC. It was also reported, the thermal conductivity and moisture resistance factor showed lower values at 600ºC.

In the light of literature described above it can be summed up that normal strength concrete perform better as compared to high strength mixes when subjected to fire. Concrete prepared with calcareous aggregate performs better in high temperature environment because of its low thermal conductivity.

2.4.1 Exposure of concrete to Fire

A lot of studies were carried out by the researchers in the past to study the behavior of concrete under elevated temperature or fire for different types of concrete containing conventional / natural coarse aggregate (Hager 2013, Li et al 2004). But no such specific study has been carried out by the researchers to study the behavior of concrete containing marble waste as coarse aggregate in concrete mixes exposed to real fire. This might be due to the fact that marble is susceptible to serious explosive spalling when exposed to fire temperature above 600ºC (Hager 2013). This observation is well expected when marble as a single material is exposed to fire. In case marble is used as coarse aggregate in concrete, it will be bonded by surrounding fine aggregate, coarse aggregate with cement paste. Thus there will be limited exposure of marble coarse aggregate to fire. Impact of fire on coarse aggregate in interior part of mix is expected to be mild. Relevant research data have not been traced so far in the available literature. Therefore, in order to study behavior of concrete mixes with marble waste as coarse aggregate exposed to fire, relevant experimental data shall be required.

2.4.2 Exposure of Concrete to Acid

Similarly, marble is susceptible to acid attack and gets dissolved after prolonged exposure in acidic environment (Beatrix Kerkhoff 2002). As mentioned earlier marble shall be protected by aggregate and cement paste when used as coarse aggregate in concrete mixes. Such protection gives limited access to acid to attack. Hence performance of concrete attacked by acid is expected to be better than that for raw marble. Experimental data can only project real behavior of concrete containing marble waste in acidic environment.

2.5 Need for further study

As stated in the origin of the problem in section 1.3 that, marble industry produces enormous amount of waste during mining and processing operation. In India the solid waste generation is around 48 million tons. The waste produced during mining and processing operation of marble is huge in quantity and a small part can be utilized for manufacturing of tiles and statues and rest part is dumped in open lands. The dumping of this waste creates a lot of environmental as well as health issues in nearby areas. During processing operation huge quantity of waste is generated and this is thrown on the open land. The waste is in the form of irregular stones and slurry. This slurry settles at site which results in ugly appearance and also causes dust in the air in summers and imposes threat to agriculture and health (Binici et al. 2007).

Hence, many countries have been working on how to reuse the waste material so as to reduce hazards to the environment (Karasahin and Terzi, 2007). However, wastes can be used to in development of new products so that natural resources are used more efficiently and the environment is protected from waste deposits.

A few studies have been conducted by the researchers in the past to resolve the issues of marble waste generation. In these studies a few mechanical and some durability properties of concrete using marble waste as coarse aggregate in concrete were evaluated. These studies illustrate that, the use of marble waste as coarse aggregate in concrete mixes does not have adverse impact on mechanical properties such as workability, compressive strength, ultrasonic pulse velocity, etc. as compared to that of conventional concrete mixes. In case of durability properties of concrete, no clear trend was reported by the past researchers. The results obtained for durability properties are for short duration of time; no long term study was conducted in the past. Structures are many times subjects to fires or acid rain especially in industrial towns. Behavior of concrete containing marble waste has not yet been reported in aggressive environment like exposure to fire or acidic environment. Such evaluation is necessary before arriving at conclusion for deciding use of marble waste in construction work

Therefore, to resolve the issues of disposal of marble waste and to understand the behavior of concrete using marble waste as coarse aggregate needs more justification and clarification. Because of these reasons a detailed and long term assessment of mechanical and durability properties in normal and aggressive environment has been taken up.

3 Materials

3.1 General

For the preparation concrete mixes basic materials required are cement, fine aggregate, coarse aggregate and water. The Portland pozzolana cement (PPC) was used as binding material for the concrete. Locally available river sand and quartzite were used as conventional fine aggregate and coarse aggregate, respectively for concrete test specimens. Marble waste was brought from nearby area of Rajasthan, India and crushed in to the crusher to use as a partial replacement for conventional coarse aggregate.

3.2 Cement

Pozzolona portland cement (PPC) used in this study fulfills the requirement of BIS: 1489-part 1-1991 (1991). The properties of cement were determined in the laboratory of Malviya national institute of technology (MNIT), Jaipur as per Indian standards BIS: 4031, are shown in Table 3.1.

Table 3.1 Physical properties of cement

Initial Setting Time	47 minute
Final setting time	483 minute
Compressive strength	
3 days	20MPa
7 days	24MPa
28 days	39MPa
Consistency	27%
Specific gravity	3.11

3.3 Fine Aggregate

The sand used in the study conforms to grading zone II as per BIS: 383-1960 (1997). The properties of sand are presented in Table 3.2

Table 3.2 Physical properties of sand and particle size distribution of sand

<table>
<tr><th colspan="4">Physical properties of sand</th></tr>
<tr><td>Sr. No.</td><td colspan="2">Physical property</td><td>Test Value</td></tr>
<tr><td>1</td><td colspan="2">Specific gravity</td><td>2.66</td></tr>
<tr><td>2</td><td colspan="2">Water Absorption (%) by weight</td><td>2.00</td></tr>
<tr><th colspan="4">Particle size distribution of sand</th></tr>
<tr><td>Sr. No.</td><td>Sieve size</td><td>percentage passing</td><td>Cumulative % Retained</td></tr>
<tr><td>1</td><td>10 mm</td><td>100</td><td>0</td></tr>
<tr><td>2</td><td>4.75 mm</td><td>95.45</td><td>4.55</td></tr>
<tr><td>3</td><td>2.36 mm</td><td>89.25</td><td>10.75</td></tr>
<tr><td>4</td><td>1.18 mm</td><td>79.45</td><td>20.55</td></tr>
<tr><td>5</td><td>600 µ</td><td>59.4</td><td>40.6</td></tr>
<tr><td>6</td><td>300 µ</td><td>14.35</td><td>85.65</td></tr>
<tr><td>7</td><td>150 µ</td><td>1.2</td><td>98.8</td></tr>
<tr><td colspan="2">Grading Zone as per BIS 383</td><td>Zone II</td><td>Cumulative % retained=261.9</td></tr>
<tr><td colspan="2">Fineness Modulus</td><td>-</td><td>2.62</td></tr>
</table>

The sand was free from silt and deleterious matter. From the Table 3.2 it can be seen that, the sand falls in Zone II of BIS: 383-1960.

3.4 Coarse Aggregate

The coarse aggregate used in the study conforms to BIS: 383-1960 (1997). The nominal maximum size of coarse aggregate used was 20 mm. The properties of coarse aggregate are presented in Table 3.3. The chemical composition of conventional and marble coarse aggregate are presented in Table 3.5.

Table 3.3 Physical properties of coarse aggregate

Physical properties of coarse aggregate		
Sr. No.	Physical property	Test Value
1	Specific gravity	2.61
2	Water Absorption (%) by weight	0.54
3	Los Angeles Abrasion Value (%)	25.88
Particle size distribution of coarse aggregate		
Sr. No.	Sieve size (mm)	percentage passing
1	40	100
2	20	95
3	10	54.88
4	4.75	6.8
Gradation as per BIS 383		Nominal maximum size of aggregate 20 mm

The coarse aggregate used in this study was hard, angular and free from impurities.

3.5 Marble Waste

The marble waste used in this study was from Rajnagar area of Rajasthan state. The waste dumped on the road side or on the open lands was taken and crushed to obtain desired gradation of coarse aggregate. The properties of marble waste are shown in Table 3.4

Table 3.4 Physical properties of Marble coarse aggregate

Physical properties of coarse aggregate		
Sr. No.	Physical property	Test Value
1	Specific gravity	2.70
2	Water Absorption (%) by weight	0.05
3	Los Angeles Abrasion Value (%)	34.87
Particle size distribution of Marble coarse aggregate		
Sr. No.	Sieve size (mm)	percentage passing
1	40	100
2	20	95.28
3	10	37.28
4	4.75	0.14
Gradation as per BIS 383		Nominal maximum size of aggregate 20 mm

Table 3.5 Chemical compositions of Marble Waste and Natural Aggregate

Component	**Aggregate produced from Marble Waste (%)**	**Conventional Aggregate (%)**
LOI	45.07	5.08
SiO_2	3.75	53.70
CaO	33.12	4.83
MgO	17.91	2.01
Fe_2O_3	0.13	10.66
Al_2O_3	Traces	Nil
Sulphate content	Nil	Nil

It can be seen from Table 3.4 that, fraction finer than 4.75 mm in marble waste is negligible as compared to that in natural coarse aggregate.

The values obtained in the Los Angeles Abrasion test are within the limits as per BIS: 2386 part IV -1963. Hence the marble aggregates can be used in concrete pavement works.

4 Methodology

4.1 General

The concrete mix designs by packing density method and as per BIS were carried out by in the laboratory. Test specimens were cast for investigations in the laboratory. Properties of concrete in fresh and hardened states such as workability, compressive strength and flexural strength were determined. In the second stage the concrete cubes of different sizes were cast for durability properties. Concrete specimens were also tested after exposure in aggressive environment, such as resistance to chloride ion penetration, carbonation, resistance to sulfate attack and acid attack. Further the microstructure properties of concrete mixes were studied.

4.2 Concrete Mix Design Methods

The concrete mixes were designed by BIS code method and Packing Density method. Coarse aggregate was taken as mix of 75% marble waste and 25% conventional coarse aggregate. This selection was based on the best performance obtained in trial mixes by author and past researchers. Conventional coarse aggregate was used for control concrete. Therefore, test specimens of following four mixes were cast,

1) BIS code method
    i. Control concrete
    ii. Concrete with marble waste
2) Packing density method
    i. Control concrete
    ii. Concrete with marble waste

The water-cement ratio in all the mixes was fixed as 0.45. The mix proportion obtained are shown in Table 4.1 and Table 4.2, respectively.

4.2.1 Concrete mix Designed by Packing Density Approach

For the design of concrete mix by packing density approach various formulations of aggregate fractions were prepared. Firstly two size fractions of coarse aggregate 20 mm and 10 mm were mixed in a definite proportion by weight, such as 90:10, 80:20, 70:30 and 60:40, etc., and the bulk density of each mixture was determined. Initially the bulk density starts increasing with increase in finer fraction; however, a stage was reached when the bulk density of the coarse aggregate mixture, instead of increasing, decreases. The mixture giving highest bulk density was mixed with fine aggregate in the ratio of 90: 10, 80: 20, 70: 30, 60: 40, 50: 50, etc. By increasing the finer content, the bulk density increases up to a peak value after which it starts reducing. Thus, the proportion obtained for maximum bulk density was fixed for total aggregates, i.e., coarse aggregates 20 mm: coarse aggregates 10 mm: fine aggregates was 36: 24: 40 by weight. The bulk density, packing density and voids contents plotted against the weight fraction of all in aggregate are presented in the Figure 4.1, Figure 4.2 and Figure 4.3, respectively.

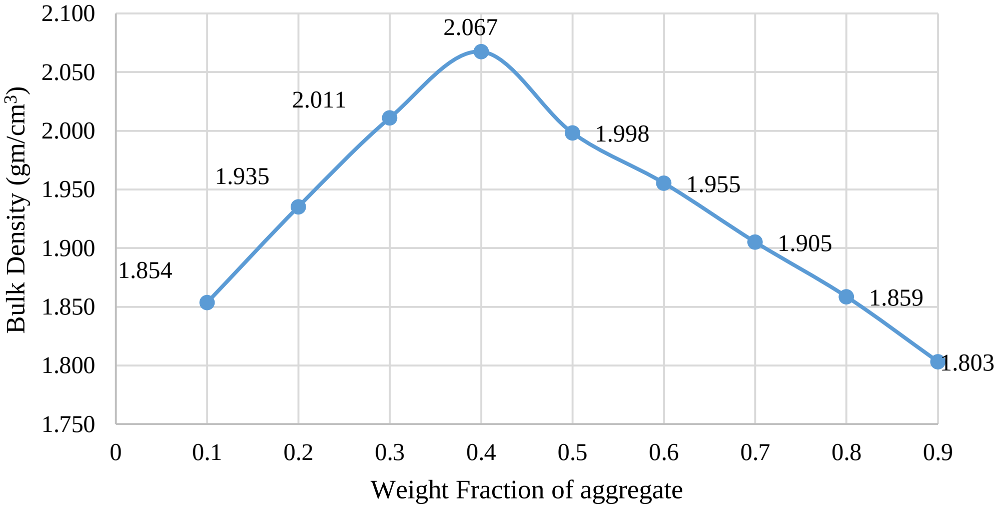


Figure 4.1 Bulk Density of all in aggregate

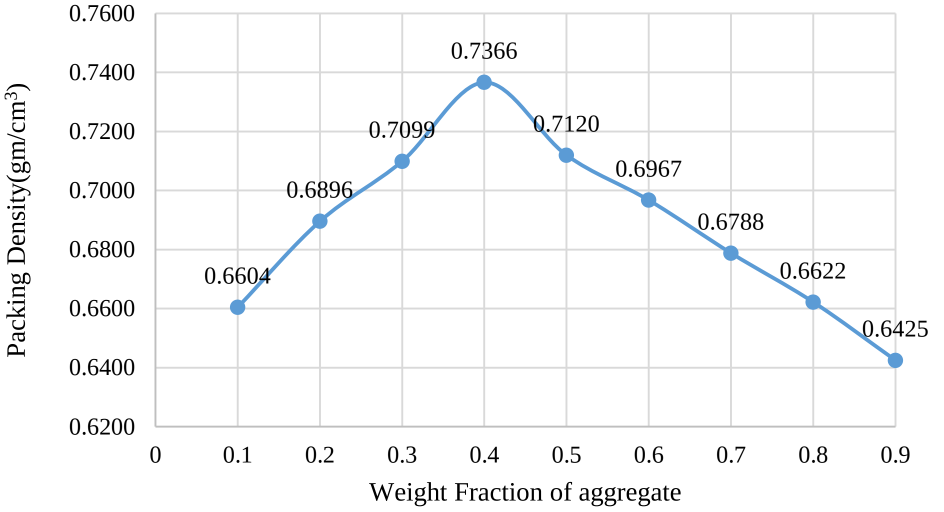


Figure 4.2 Packing Density of all in aggregate

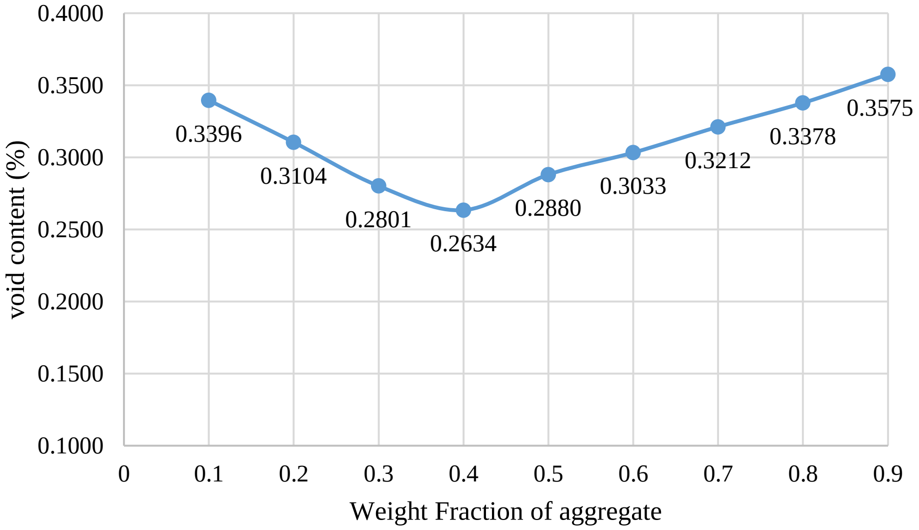


Figure 4.3 Void content of all in aggregate

After getting all the values of bulk density, packing density and void contents the mix proportion of concrete was calculated. The concrete mixes was prepared by taking cement paste content 5%, 10%, 15%, and 20% in excess of void content in the aggregate mix in order to achieve desired workability. Workability and compressive strengths were determined. It was found that maximum strength (refer Figure 4.4) was obtained with desired slump of 75 mm for mix prepared with paste content 10% in excess of voids.

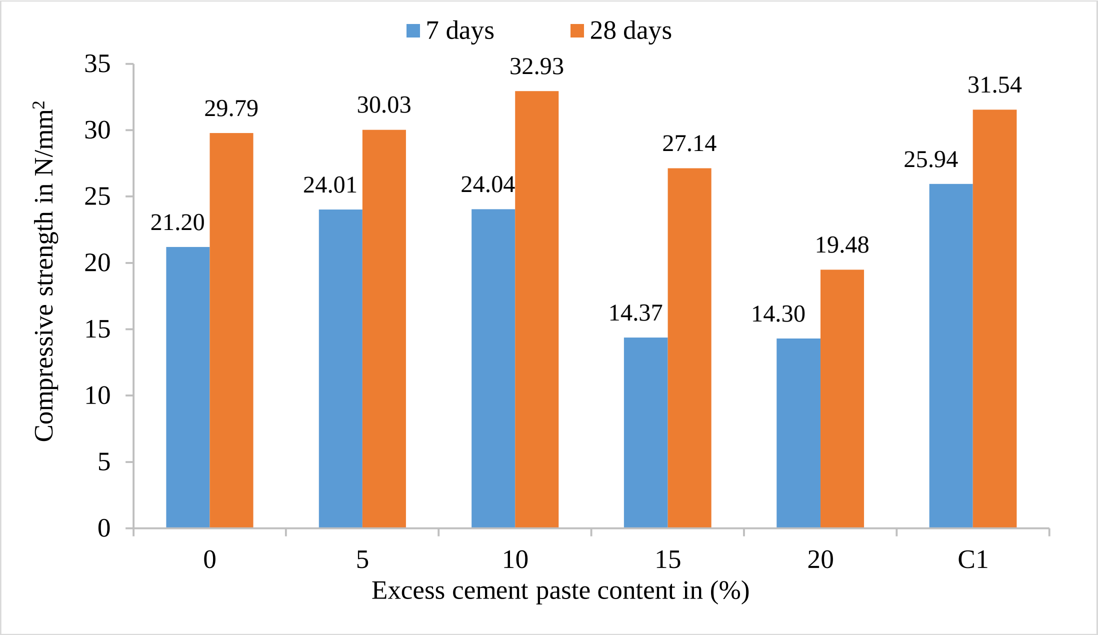


Figure 4.4 Variation in compressive strength of concrete mixes

Therefore, all the concrete mixes were prepared by adding cement paste 10% in excess of voids and a constant water-cement ratio of 0.45. The detailed calculation for the design of concrete mix by packing density approach is given in **Annexure I**. The mixture proportion of concrete designed by packing density approach are shown in Table 4.2**.**

Table 4.1 Mix Proportion of concrete mix designed by BIS code Method

Mix	Water (lit.)	cement (kg)	sand (kg)	Coarse Aggregate (kg)			
				Natural Aggregate		Aggregate produced from Marble waste	
				20 mm	10 mm	20 mm	10 mm
C 1	192	426	663	531	649	-	-
C2	192	426	663	133	162	239	646

Table 4.2 Mix Proportion of concrete designed by Packing Density Approach

Mix	Paste content in excess of voids (%)	Water (lit.)	cement (kg)	sand (kg)	Coarse Aggregate (kg)			
					Natural Aggregate		Marble Aggregate	
					20 mm	10 mm	20 mm	10 mm
C3	10	157	348	800	720	480	-	-
C4	10	154	343	805	181	121	544	362

Note:

C1 - shows control mix designed as per BIS 10262-2009.

C2 - shows concrete mix containing 75% marble aggregate and 25% conventional coarse aggregate designed as per BIS 10262-2009.

C3 - shows control mix designed by Packing Density approach

C4 - shows concrete mix containing 75% marble aggregate and 25% conventional coarse aggregate designed by Packing Density approach

From the mix proportion designed by packing density approach it can be seen that, the sand content was increased as compared to that of concrete mixes designed as per BIS 10262-2009.

4.3 Preparation of Concrete specimens

The ingredients of concrete were mixed in a mixer and cubes of size 150 ×150×150 mm were cast as shown in Figure 4.5 for determining the compressive strength and water permeability test. The cubes of 100×100×100 mm were cast for the durability tests.

Figure 4.5 Casting of specimens

The molds used in this study for casting of concrete specimens confirms to BIS: 10086 (1999b). All the specimens were de molded after 24±1h and were cured in water tank at room temperature up to the specified age of test.

4.4 Workability of Fresh Concrete

The slump cone test was used to measure the workability of fresh concrete mix as per BIS 1199 -1959 (Bureau of Indian Standards (BIS) 1999a). To achieve the desired workability of 75 mm the third generation poly-carboxylic base super plasticizer was used. The freshly mixed concrete were poured in to the slump cone apparatus in three successive layers. Each layer was tamped 25 times by a tamping rod. Then the cone was lifted up slowly and concrete was allowed to settle down. The height of concrete was measured with the help of measuring scale.

4.5 Density of Concrete

To study the effect of partial replacement of conventional coarse aggregate by marble aggregate on the self-weight of concrete the density of hardened concrete was measured. This test was conducted on the 28 days cured concrete specimens of size 150×150×150 mm. The concrete specimens were kept in oven for 24±1h to achieve the constant weight. The weight of the concrete specimens after cooling was recorded. The density of concrete specimens is calculated by following formula,

$$\text{Density of concrete} = \frac{\text{mass of specimen}}{\text{volume of specimen}} \tag{4.1}$$

4.6 Water Absorption

Water absorption test a simple test to determine the quality of concrete in terms of porosity. This test was carried out as per BIS: 15658 (Bureau of Indian Standards 2006) on 100 mm cube size specimens. Three 100×100×100 mm cube size specimens were kept in oven for 24±1h and then taken out to cool at room temperature. There after these specimens were completely immersed in water for 24±1h. These specimens were taken out from the water and allowed to drain of the surface water for 1 minute by placing on the wire mesh. The visible water on the surface of the specimens was removed by damp cloth. Then the weight of the specimens measured and this weight was W1. Subsequent to saturation, the specimens were dried in a ventilated oven at 107 + 7°C for not less than 24 h±1h and until two successive weighing at intervals of 2 h show an increment of loss not greater than 0.2 percent of the previously determined mass of the specimen and dry weight was noted W2.

The percent water absorption of each specimens was calculated from the following equation,

$$\text{Water absorption} = \frac{(W_1\text{-}W_2)}{W_2} \times 100 \tag{4.2}$$

4.7 Compressive Strength of Concrete

Compressive strength of concrete specimens was determined at 7 days, 28 days, 90 days and 180 days curing age as per BIS: 516-1959 (Bureau of Indian Standards (BIS) 2002). The test setup for compressive strength test is shown in Figure 4.6.

Figure 4.6 Compressive strength test set up

4.8 Flexural Strength

Flexural strength of concrete specimens was determined after 7 days and 28 days of curing as per BIS: 516-1959 (Bureau of Indian Standards (BIS) 2002). The digital flexural testing machine of 100KN capacity was used to test the beam specimens of size 100×100×500 mm.

Figure 4.7 Flexural strength test setup

The two point loading set up as shown in Figure 4.7was applied on beam specimens with a span of 500 mm. The specimen was loaded on the setup and load applied till the specimens failed and the maximum load applied was recorded.

4.9 Permeability

In order to assess the porosity in concrete, water permeability test was conducted as per German standard DIN -1048 part 5 (1991) (Anon 1991). The concrete specimens were kept in oven at 107 + 7°C to achieve the constant weight. The concrete specimens were taken out from the oven after 24 h ±1hr and cooled down to room temperature. The weight of the specimens was measured. These specimens were exposed to a constant water pressure of 0.5 N/mm^2 acting normal to the mould-filling direction, for a period of 72 hr. The pressure in the pressure head is maintained for 72 hr. with periodic monitoring. The test set up for the water permeability test is shown in Figure 4.8.

Figure 4.8 Water Permeability test set up

The specimens were removed from the apparatus after 72 hr. and the weight of the specimens was measured. Then these specimens were split in to two pieces. After 5–10 min drying, the maximum depth of penetration was measured from the 3 specimens and mean was reported as the depth of penetration.

4.10 Ultrasonic Pulse Velocity

A non-destructive test method was conducted on the concrete specimens as per BIS: 13311 part 1; 1992 (Bureau of Indian Standards (BIS) 1999c) to assess the homogeneity and quality of concrete. Cube specimens of size 100×100×100 mm were used. The underlying principle of assessing the quality of concrete is that comparatively higher velocities are obtained when the quality of concrete in terms of density, homogeneity and uniformity is good. In case of poorer quality, lower velocities are obtained. Density and modulus of elasticity of aggregate also significantly affect the pulse velocity.

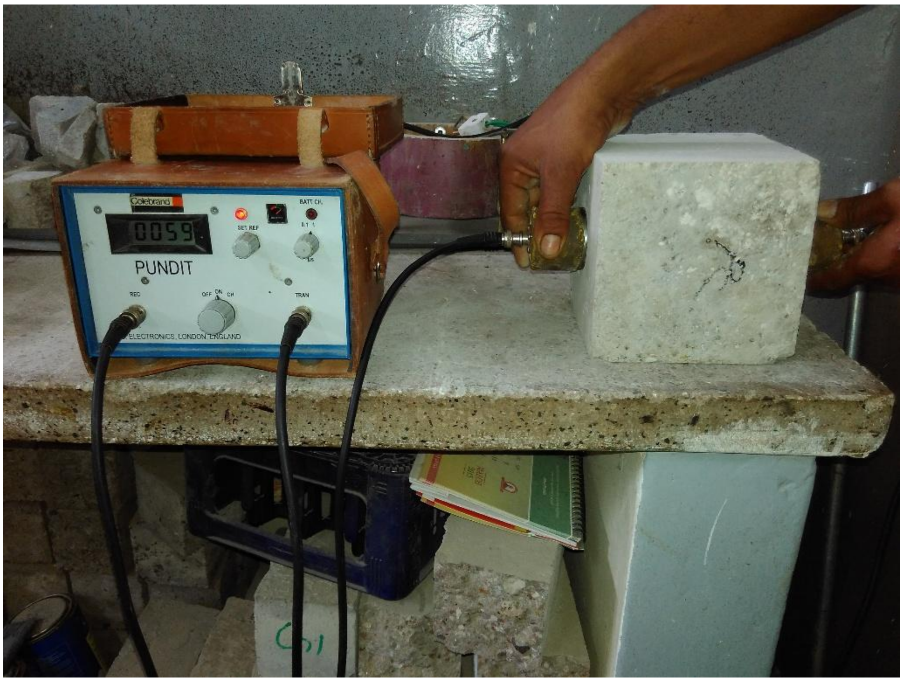


Figure 4.9 Ultra Sonic Pulse Velocity Tests set up

For this test, a commercial ultrasonic pulse velocity (UPV) measurement device and two ultrasonic transducer with a center frequency of 54 kHz were used for measuring the propagation time of a sonic wave through the specimen. To provide a proper contact between transducer and surface of the concrete cube sufficient amount of gel was applied. The pulse velocity (V) is calculated by dividing the length of the specimen (L) by transit time (T).

Ultra sonic Pulse Velocity was determined by following equation,

$$V = \frac{L}{T} \tag{4.3}$$

4.11 Abrasion Resistance

The abrasion test was conducted to measure the resistance to wear. Deterioration of concrete may take place due to abrasion caused by various exposures (skidding, rubbing and sliding off an object) on the concrete surface. Abrasion resistance was measured in term of depth of wear of concrete under standard testing conditions. It was performed according to BIS 1237: 1980 (Bureau of Indian Standards 1237 2001) on 28 days cured 100 mm size concrete cubes. The test specimens were dried at 100 ± 5°C for 24 hours and then weighed to the nearest 0.1 g. The concrete specimens were fixed in the holding device of the abrasion testing machine with the surface to be ground facing the disc and loaded at the center with 300 N. A suitable abrasive powder of 20 g was spread evenly on the rotating disc. Then the disc was rotated with a speed of 30 rev/min and the abrasive powder was spread continuously on the rotating disc. After every 22 revolutions the disc was stopped, the abraded powder of the specimens and the abrasive powder remain on the disc is removed. Now the specimen is rotated about its vertical axis through an angle of 90^0 in clockwise direction and fresh 20 g abrasive powder is spread evenly on the disc. Procedure was repeated 9 times there by giving a total number of 220 revolutions. After competition of 220 revolutions specimen was removed from the holding device of the machine. Then abraded surface of the specimen was cleaned with the help of brush and weight was measured.

Figure 4.10 Test set up for Abrasion test

As per the code, in general, purpose tiles, the average maximum wear shall not exceed 3.5 mm and wear on any individual specimen shall not exceed 4 mm. For heavy-duty floors, it is 2 mm and 2.5 mm, respectively. The depth of wear was calculated by the average loss in thickness of the specimens after abrasion by using the following equation,

$$t = \frac{(W_1 - W_2) \times V_1}{W_1 \times A} \tag{4.4}$$

Where,

t = average loss in thickness in mm,

W_1 = initial mass of specimens in g,

W_2 = final mass of abraded specimens in g,

V_1 = initial volume of specimens in mm^3,

A = surface area of specimens in mm^2,

4.12 Carbonation

The carbonation test was conducted as per CPC 18 RILEM guidelines (Rilem & Matt 1988). The concrete cubes of 100×100×100 mm were cut with the help of cutter in to four pieces of prisms of size 50 × 50 ×100 mm. After cutting these specimens were kept in oven at a temperature of 100 ± 5°C for 24 hours. These specimens were removed from the oven after 24 hours and allowed to cool down to room temperature. Three coats of epoxy paint was applied on the cut specimens by keeping top and bottom side of prism non- painted. These specimens were kept aside for drying the paint. After drying these specimens were kept in carbonation chamber as shown in Figure 4.10at 5% controlled CO_2 concentration with preset relative humidity (50 ± 5%) and temperature (27 ± 1 ^{0}C).

Figure 4.10 Carbonation chamber

After desired CO_2 exposure (14, 21, 28, 35, 42, 56, 90,120,150 and 180 days), three prisms from each representative concrete mix were taken out and split in to two pieces. An aqueous alcoholic solution of 1% phenolphthalein was sprayed on the freshly broken pieces of the specimens. In carbonated areas the solution remain colorless and in non-carbonated areas the phenolphthalein indicator turns purple red as shown in Figure 4.11.

Figure 4.11 Splitting of specimens after testing

The depth of carbonation at the top and bottom face of the specimen was recorded for each of the three specimens.

4.13 Acid Attack

Resistance of concrete specimens to acid attack were evaluated as per ASTM C 267-01 (ASTM 2012). The concrete specimens were immersed in 5% dilute Sulfuric acid solution to determine the resistance against adverse environment. The solution was replaced once in a month to maintain the pH of the solution.

Figure 4.13 Specimens immersed in sulfuric acid solution

These specimens were kept in the 5% dilute sulfuric acid solution for 28 days, 56 days and 91 days to determine the gain or loss in compressive strength and loss in mass of specimens respectively.

4.13.1 Visual Inspection

The visual inspection of the concrete specimens attacked by dilute sulphuric acid was carried out to study the variation in shape, surface erosion and roughness of the surface. The inspection was carried out after 28 days, 56 days and 91 days of immersion in dilute sulfuric acid solution. Before the inspection the specimens were taken out of the solution and allowed to drain of the solution. The specimens were washed with tap water and then allowed to dry.

4.13.2 Loss in mass

After 28 days of curing the concrete cube specimens of 100×100×100 mm size were kept in oven at a temperature of 100 ± 5 °C for 24 ± 1hr These specimens were taken out from the oven and cooled down to room temperature. The oven dry weight of the specimens was taken as initial weight. These were immersed in 5% dilute sulfuric acid solution. These specimens were taken out from the solution after 28 days, 56 days and 91 days and kept on the 10 mm coarse wire mesh to drain off the water. The water on the surface was wiped by using damp cloth and weight of the surface dry specimens was noted.

The percentage change in the mass of the specimens was calculated from the following equation,

Loss in mass after immersion in acid solution,

$$\text{Loss in mass (\%)} = \frac{W_{\text{(initial)}} - W_{\text{(t days)}}}{W_{\text{(initial)}}} \times 100 \tag{4.5}$$

Where,

$W_{\text{(initial)}}$ = weight of specimens before immersion in acid solution,

$W_{\text{(t days)}}$ = weight of specimens at (t) days after immersion in acid solution.

4.13.3 Compressive strength of acid attacked specimens

The concrete cube specimens of 100×100×100 mm size were immersed in 5% dilute sulfuric acid solution. These specimens were taken out from the solution and kept on 10 mm coarse wire mesh to drain off the solution. The water on the surface was wiped by using damp cloth and weight of the surface dry specimens was noted as W2. The compressive strengths of concrete specimens were determined after 28 days, 60 days and 90 days.

Reduction in compressive strength after immersion in acid solution,

$$\text{Reduction in compressive strength (\%)} = \frac{fc_{(initial)} - fc_{(tdays)}}{fc_{(initial)}} \tag{4.6}$$

Where,

$fc_{(initial)}$ = 28 days compressive strength of the concrete mixture before immersion in acid solution

$fc_{(t\ days)}$ = compressive strength at (t) days after immersion in acid solution

4.14 Sulphate Attack

Resistance of concrete specimens to sulfate attack was evaluated as per ASTM C 267-01 (ASTM 2012). The concrete specimens were immersed in 10 % dilute magnesium suphate solution to determine the resistance against adverse environment.

Figure 4.12 Specimens immersed in Magnesium Sulfate solution

The solution was replaced once in a month to maintain the pH of the solution. These specimens were immersed in the solution for 28 days, 56 days 91 days, 180 days and 300 days to determine the gain or loss in compressive strength and loss in mass of specimens respectively.

4.14.1 Loss in mass

After the 28 days of curing the concrete cube specimens of 100×100×100 mm size were kept in oven at a temperature of 100 ± 5 °C for 24 ± 1hr These specimens were taken out from the oven and cooled down to room temperature. The oven dry weight of the specimens is taken as initial weight. These were immersed in 10% dilute magnesium sulphate solution. These specimens were taken out from the solution after 28 days, 56 days, 91 days, 180 days, 300 days respectively and kept on the 10 mm coarse wire mesh to drain off the water. The water on the surface was wiped by using damp cloth and weight of the surface dry specimens was noted.

The percentage change in the mass of the specimens was calculated from the following equation,

Loss in mass after immersion in acid solution,

$$\text{Loss in mass (\%)} = \frac{W_{(\text{initial})} - W_{(\text{t days})}}{W_{(\text{initial})}} \times 100 \qquad (4.7)$$

Where,

$W_{(\text{initial})}$ = weight of specimens before immersion in acid solution,

$W_{(\text{t days})}$ = weight of specimens at (t) days after immersion in acid solution.

4.14.2 Compressive strength of silphate attacked specimens

The concrete cube specimens of 100×100×100 mm size were immersed in 10% magnesium sulfate solution. These specimens were taken out from the solution and kept on the 10 mm coarse wire mesh to drain off the solution. The water on the surface was wiped by damp cloth and weight of the surface dry specimens was noted as W2. The compressive strengths of concrete specimens were determined after 28 days, 56 days, 91 days, 180 days and 300 days.

Reduction in compressive strength after immersion in sulphate solution,

$$\text{Reduction in compressive strength (\%)} = \frac{fc_{(initial)} - fc_{(tdays)}}{fc_{(initial)}} \qquad (4.8)$$

Where,

$fc_{(initial)}$ = 28 days compressive strength of the concrete mixture before immersion in sulphate solution

$fc_{(t\ days)}$ = compressive strength at (t) days after immersion in sulphate solution

4.15 Chloride Ion Penetration

This test was conducted on 28 days cured concrete cube specimens of 100 mm ×100 mm ×100 mm. These specimens were kept in oven at a temperature of 100 ± 5°C for 24 ± 1 hour. The specimens were removed from the oven after 24 ± 1 hour and allowed to cool down at room temperature. The dry concrete cube specimens were soaked in 4% NaCl solution.

Figure 4.13 Chloride ion penetration of concrete mix

These specimens were taken out from the solution after 28 days, 56 days and 91 days of interval and allowed to drain of the excess water. The water on the surface of the specimen was wiped with the help of damp cloth. These cubes were broken in to two pieces and the 0.1N silver nitrate solution was sprayed on the broken pieces. The sprayed silver nitrate reacts with the free chloride ions present on the concrete surface and forms a white precipitate of silver chloride. In the places where free chlorides are absent, $AgNO_3$ reacts with hydroxide to form a brown precipitate of silver oxide (AgO). Thus, the boundary of color change indicates the depth of chloride penetration as shown in Figure 4.13.

4.16 Microstructural analysis

To study the probable phases formed in the concrete mixes after partial replacement of conventional coarse aggregate by marble aggregate, the microstructural analysis was carried out. The X-Ray diffraction (XRD) analysis was carried out on the powdered sample of control concrete and concrete containing marble aggregate. The

XRD analysis was carried out at Material Research Centre (MRC) laboratory Malviya National Institute of Technology, Jaipur.

To study morphology of concrete mix Scanning Electron Microscope (SEM) analysis was carried out on control concrete and concrete containing marble aggregate. The prism of size 10 mm ×10 mm ×20 mm was cut from the concrete specimens at a depth of 50 mm. The surface of the specimen was polished by platinum layer to improve the conductivity of electrons through them.

4.17 Effect of elevated temperature on concrete properties

To study the behavior of concrete under fire the concrete cube specimens of 100 mm×100 mm ×100 mm and beam specimens of size 100 mm×100 mm × 500 mm were cast. These casted specimens were de-molded after 24±1 h and transferred to water tank for curing at room temperature up to 28 days. After curing of 28 days these specimens were taken out from the water tank and allowed to drain off the surface water. The compressive strength of these concrete cube specimens was tested as per BIS: 516-1959 (Bureau of Indian Standards (BIS) 2002).

The gas fired furnace was used to expose the concrete specimens to elevated temperature under standard fire as shown in Figure 4.14. The temperature range of 200ºC, 400ºC, 600ºC and 800ºC was set to study behavior of concrete at different temperature levels. Before exposure to fire weight of the each specimens was noted. Now the specimens were kept in the fire chamber and specimens were allowed to heat. The temperature was controlled to vary as per standard fire curve as shown in Figure 4.15.

Figure 4.14 Details of Gas Fired Furnace

The K- type thermocouple was used to measure the temperature inside the furnace and this thermocouple was in contact with the surface of the concrete. As the temperature reached to the desired level the fire was immediately stopped. Then these specimens were allowed to cool down to a room temperature.

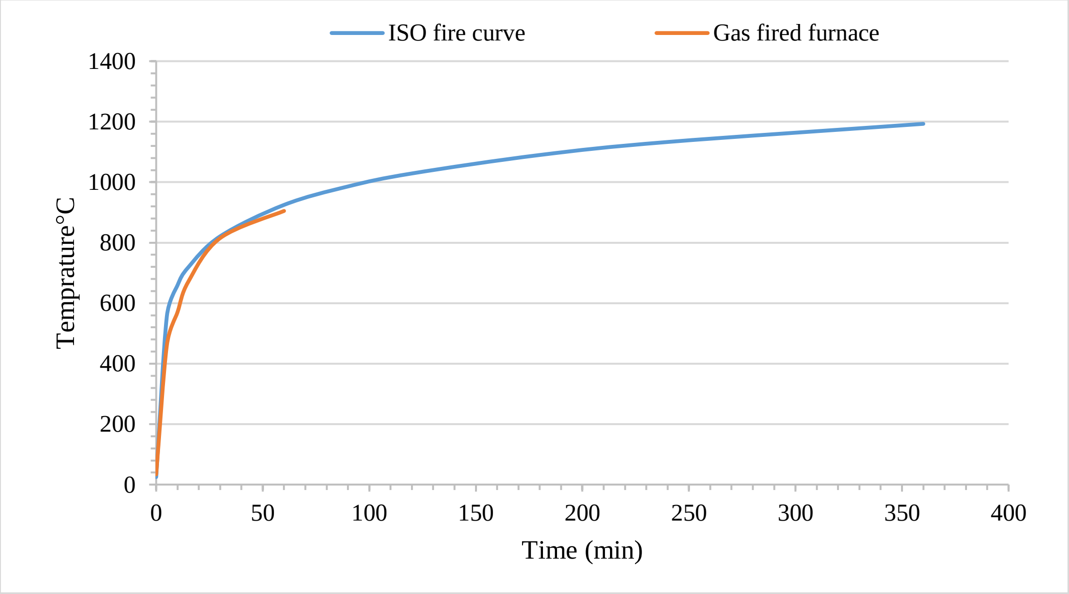


Figure 4.15 Temperature-time curve of furnace

The cooled specimens were then weighed to know the change in mass of the specimens due to fire. After weighing the compressive strength of the specimens was determined. The above mentioned procedure was repeated to study the flexural behavior of beams exposed to fire.

5 Results and Discussion

5.1 General

Initially lean concrete mixes were designed using marble waste in different percentages. Trial investigations were carried to study the behavior of concrete using marble waste as a partial and complete replacement of conventional coarse aggregate. Based on results regular concrete mixes with 0.45 water-cement ratio were cast with varying percentages of marble waste as coarse aggregate. Mechanical and long term durability properties were studied. Behavior of concrete in aggressive environment was also studied as described in subsequent paragraphs.

5.2 Preliminary Study on lean concrete mixes

For this purpose lean concrete mixes M10 and M15 with water-cement ratios 0.60 and 0.55 were designed as per BIS: 10262-2009 (2009) code method. The concrete mixes were prepared by replacing the conventional coarse aggregate by marble waste in different percentages 20%, 40%, 60%, 80% and 100% by weight. Concrete mixes without marble waste have been referred as control concrete.

For the above mentioned concrete mixes workability, compressive strength and permeability properties were studied and the results of the study are discussed below.

5.2.1 Workability

Workability of all concrete mixes was determined by slump test. The variation in workability of concrete mixes with partial and complete replacement of conventional coarse aggregate by marble aggregate are shown in Figure 5.1.

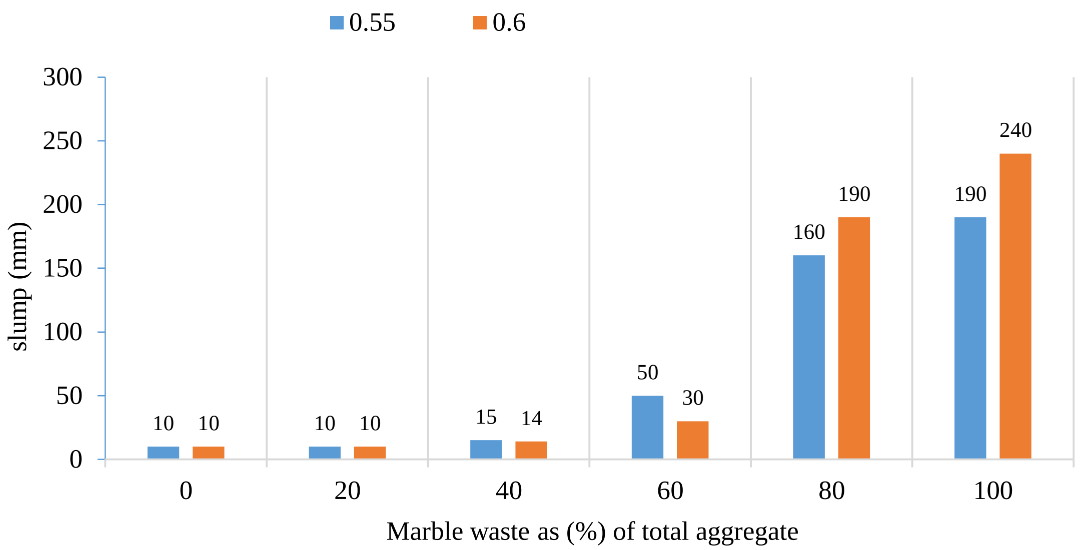
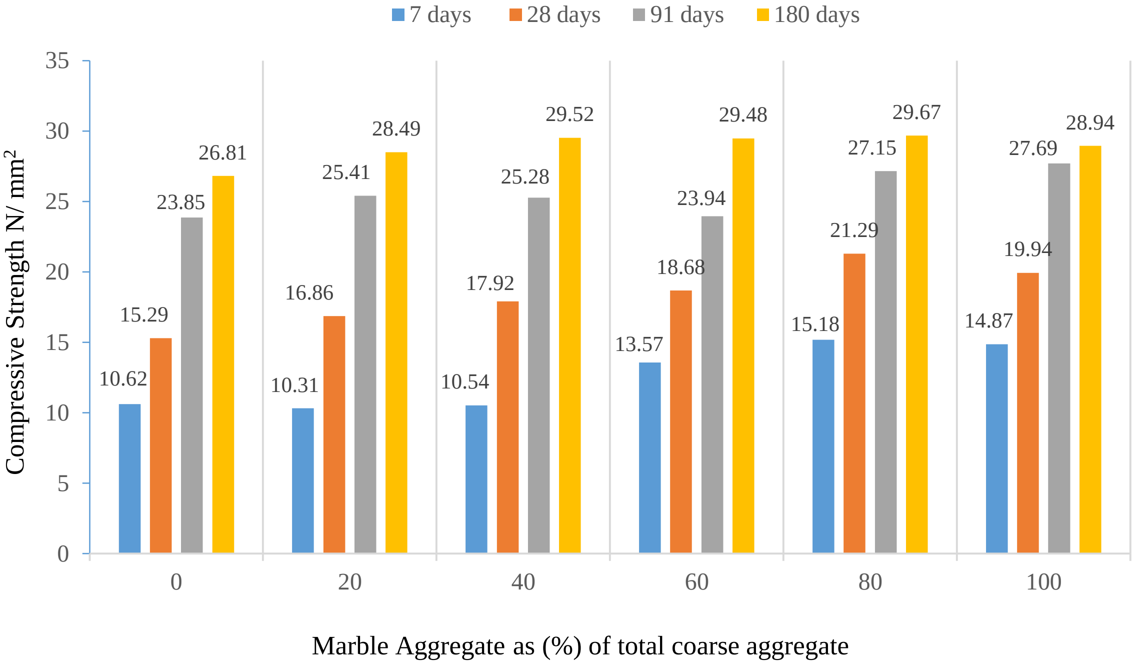


Figure 5.1 Variation in workability of lean concrete mixes

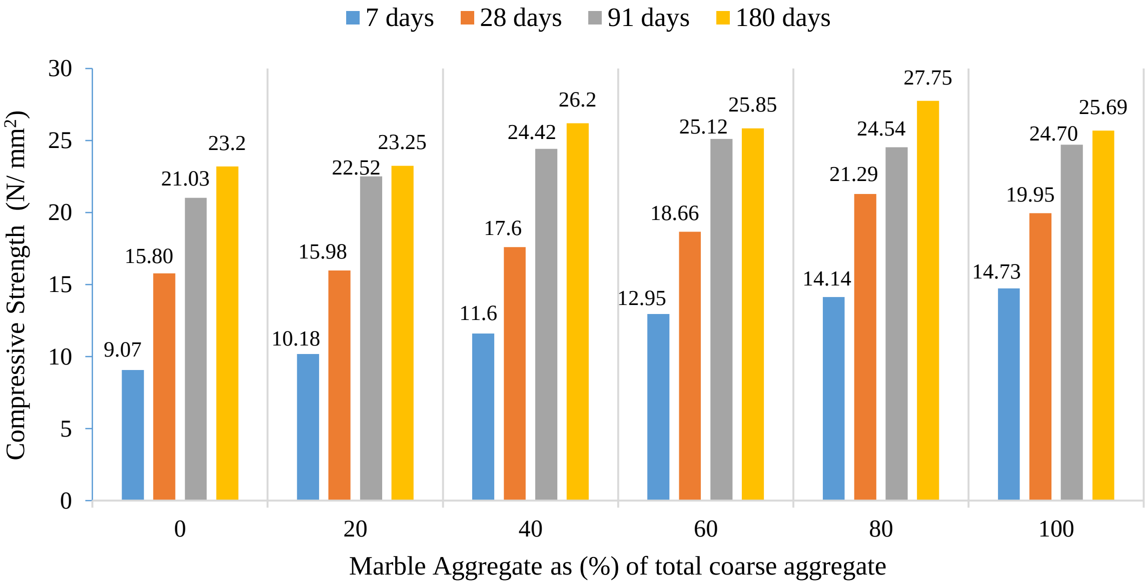
From the Figure it can be seen that, for all the water-cement ratios workability of all the concrete mixes increased with increase in replacement levels as compared to that of control mixes. At higher replacement levels i.e. at 100% replacement the workability of concrete prepared with water-cement ratio of 0.55 and 0.60 was 19 to 24 times higher as compared to that of control concrete. The increase in workability, was due to smooth surface and round shape of marble aggregate. Another reason could be lack of finer fraction of aggregate in mixes with higher replacement levels.

5.2.2 Compressive strength

The behavior of control concrete and concrete containing marble aggregate with different water-cement ratios were studied. The compressive strength of concrete mixes were determined at 7, 28, 91 and 180 days. The results of the compressive strength are shown in Figure 5.2.



(a) Compressive strength at 0.55 water-cement ratio



(a) Compressive strength at 0.60 water-cement ratio

Figure 5.2 Variation in compressive strength of lean concrete mixes

It can be seen from the above Figure 5.2 that, the compressive strength of concrete mixes prepared with partial and complete replacement of conventional coarse aggregate by marble aggregate increases with increase in marble waste content in mixes. The increase was prominent at all curing ages. At 80% replacement level with

0.60 water-cement ratio, the 28 days compressive strength was 60% higher as compared to that of control concrete. On the other hand, at 0.55 water-cement ratio with 80% replacement level the 28 days compressive strength was 40% higher than that of control concrete. Reason for increase in strength of concrete mixes is, marble aggregate contains higher carbonate content than that of conventional aggregate which improves the aggregate- cement paste bond (Hebhoub et al. 2011). In fact formation of calcium carbo-aluminate in inter transitional zone strengthens bond between cement paste and aggregate. A marginal decrease was observed in both the mixes at 100% replacement level but still it was higher than that of control concrete. The marginal decrease at 100% replacement level was probably due to lack of finer fraction in marble aggregate.

5.2.3 Permeability

To study the porosity of concrete the permeability test was conducted as per German standard DIN-1048-part 5 (1991) on the concrete specimens. The permeability was measured in terms of depth of water penetration in concrete samples. The variation in depth of penetration of the concrete mixes are presented in Figure 5.3.

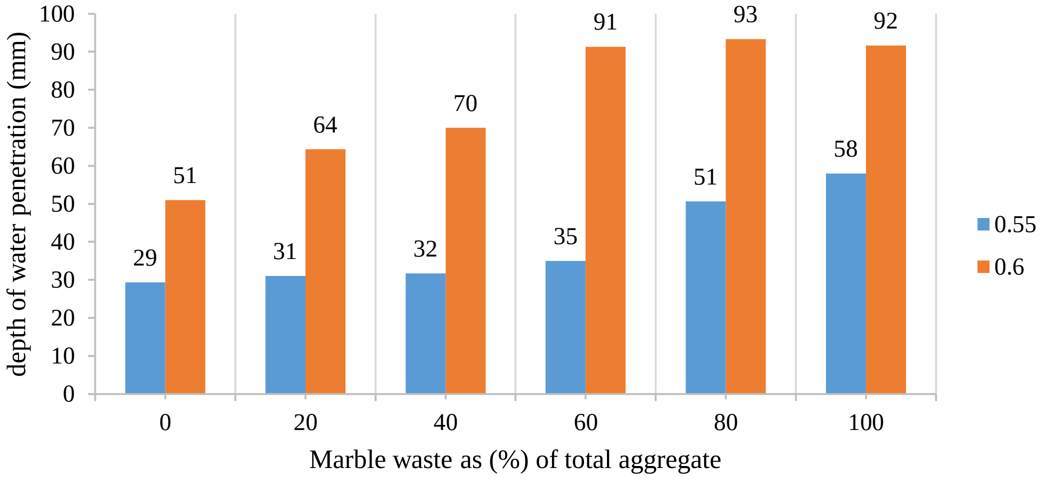


Figure 5.3 Variation in permeability of lean concrete mixes

.From the Figure 5.3 it can be observed that, the permeability of concrete mixes increases with increase in replacement levels of conventional coarse aggregate by marble aggregate. For the concrete prepared with 0.55 water-cement ratio the permeability increased by 7% to 50% at replacement levels of 20% to 100%. Whereas at 0.60 water cement- ratio permeability increased by 26% to 45% at above mentioned replacement level. The permeability of the concrete depends upon various factors, the most important factor is interconnectivity of pores. The results showed that, the interconnectivity of pores in the concrete prepared with marble aggregate was more than that of concrete prepared with conventional aggregate. This increase in permeability was probably due to deficiency of particles finer than 10 mm in marble aggregate as seen in particle size distribution of marble aggregate in Table 3.4.

Permeability tests indicate that, use of marble waste as coarse aggregate helps improve workability and compressive strength of concrete mixes. The peak value of compressive strength was obtained at 80% replacement. The depth of water penetration increases in permeability tests with higher marble aggregate contents. This fact goes against durability of concrete. Therefore, in order to gain strength and achieve better durability, packing density method was opted for design of concrete mixes for further study.

5.3 Study on concrete mixes with different design methods

In further study, the concrete mixes were designed by BIS code method (BIS: 10262-2009) and Particle Packing Density methods. The conventional coarse aggregate was replaced by 75% marble aggregate.

The mechanical and durability properties of concrete mixes were studied. In durability study, the properties of concrete related to porosity and behavior under aggressive environment were investigated. Durability of all the concrete mixes with regard to the exposure in aggressive environmental conditions was studied by assessing the resistance to acid attack, sulphate attack, carbonation and chloride penetration. The resistance to acid and sulphate attack was investigated over a period of 91 to 300 days. Further the study was extended to examine the performance of concrete exposed to standard fire at different temperature levels.

5.3.1 Workability

The workability of all concrete mixes was determined by slump test. The results of slump test (refer Table 5.1) of concrete mixes show the required dose of super-plasticizer to achieve the desired slump in the range of 75 to 100 mm.

Table 5.1 Variation in workability of concrete mixes

Mix	Excess Cement Paste	Slump (mm)	dose of super-plasticizer (%) by weight of cement
C1	-	78	0.25
C2	-	90	0.25
C3	10	90	0.9
C4	10	95	1.3

Note:

C1 - control mix designed as per BIS 10262-2009

C2 - concrete mix containing 75% marble aggregate and 25% conventional aggregate designed as per BIS 10262-2009.

C3- control mix designed by Packing Density approach.

C4-concrete mix containing 75% marble aggregate and 25% conventional aggregate designed by Packing Density approach.

The increase in quantity of super-plasticizer in mixes C3 and C4 was more as compared to that of control mix due to increased amount of fine aggregate (sand) mixed to achieve higher packing density. The workability of concrete mix depends upon various factors, i.e. maximum size and shape of aggregate, grading, water absorption. The most important factor which affects the workability of concrete is the shape of aggregate particles. The round shaped aggregates are more workable than that of angular shaped aggregates (Shetty, M.S., Gambhir 2013). Here in concrete mix C4 aggregates produced from marble waste were used and these were round in shape whereas the conventional aggregate were angular in shape. The increase in value of slump in mix C2 and C4 was due to presence of round shaped marble aggregates.

It can be concluded that, use of aggregate produced from marble waste as partial replacement for conventional coarse aggregate results in improved workability of concrete mixes.

5.3.2 Density

The influence of different design approaches and replacement of conventional coarse aggregate by aggregate produced from marble waste was assessed by measuring the weight of concrete specimens. The variation in density of concrete mixes are shown in Figure 5.4.

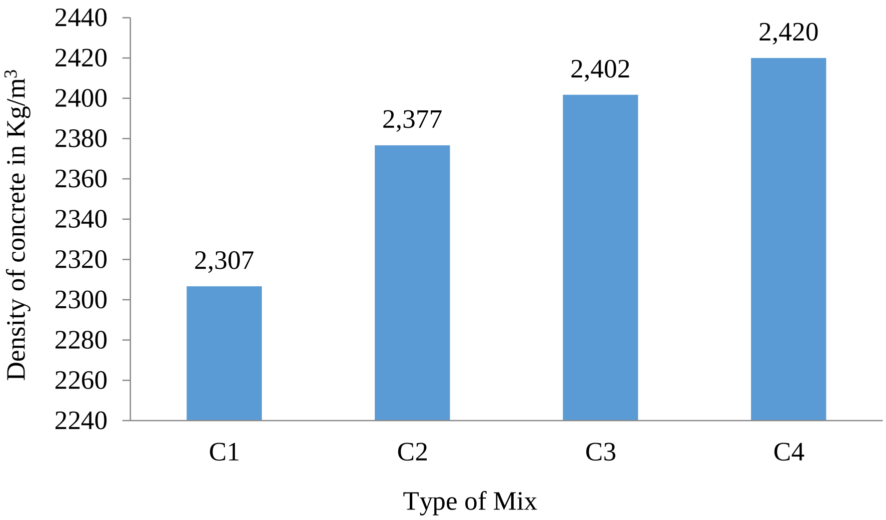


Figure 5.4 Variation in Density of concrete mixes

From Figure 5.4, it can be observed that, density of concrete mixes increases with partial replacement of conventional coarse aggregate by marble aggregate as seen in C2 and C4 mixes. Marginal increase in the density of concrete mixes was observed and this value ranged from 0.7% to 3%. The density of concrete depends upon volume fractions of constituent materials and their densities, and the volume of voids present in the concrete. Concrete density is inversely proportional to its porosity. The increase in density of mix C2 was due to higher specific gravity of marble aggregates. Due to Improved packing of particles in concrete mix C4 the porosity of concrete decreases and hence density increases by 3%.

It can be concluded from the above results that, the density of concrete mixes increases marginally with the use of marble aggregate following packing density approach for design of concrete mixes.

5.3.3 Water Absorption

To assess the porosity in concrete the tests such as water absorption and permeability were conducted. The influence of partial replacement of conventional coarse aggregate by marble aggregate on water absorption of concrete mixes was studied and results are presented in Figure 5.5.

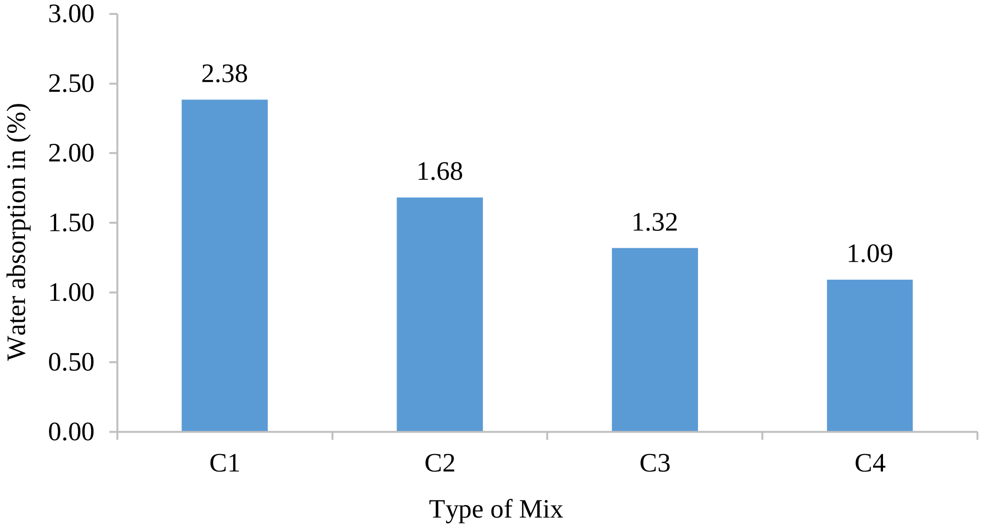


Figure 5.5 Variation in water absorption of concrete mixes

It can be seen from Figure 5.5, that the water absorption of all the concrete mixes decreased significantly. The water absorption of concrete mix C2 decreased by 29% as compared to that of control mix C1. This is because of high porosity of conventional coarse aggregate as compared to marble aggregate. On the other hand the concrete mixes (C3 and C4) designed by packing density approach show 44% and 54% reduction in water absorption as compared to that of control mix C1 which was designed by BIS code method. The major cause behind decrease in water absorption of concrete mixes C3 and C4 was due to filling of pores by cement paste and finer aggregate particles. Overall benefit of using marble aggregate and packing density method would be reduction in water absorption from 2.38% to 1.09%.

5.3.4 Compressive strength

The compressive strengths of concrete mixes were determined at 7, 28, 90, 180 and 365 days. The results of the compressive strength are shown in Figure 5.6. All the concrete mixes were designed by keeping constant water-cement ratio of 0.45. In mixes C1 and C2 the concrete mix was designed to meet the target strength of M25.

On the other hand, concrete mixes C3 and C4 were proportioned with maximum packing density approach keeping same water-cement ratio of 0.45.

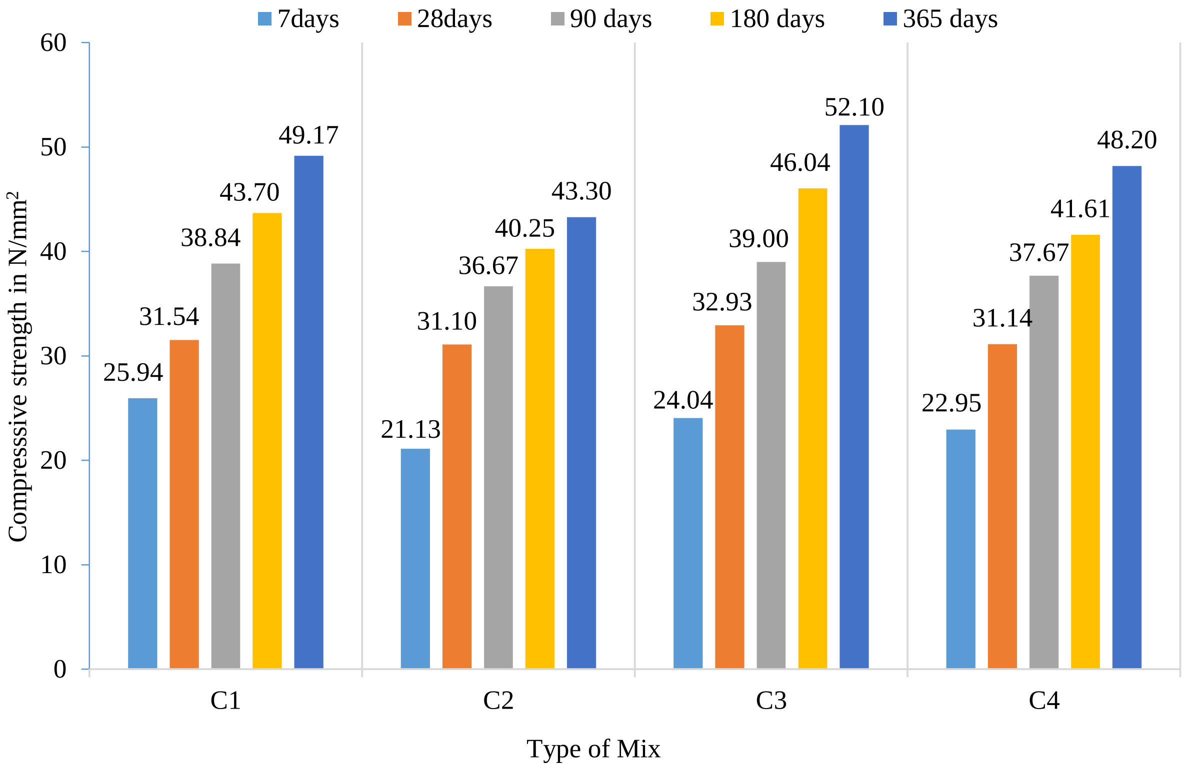


Figure 5.6 Variation in Compressive strength of concrete mixes

It can be seen from Figure 5.6 that, the compressive strength of concrete mixes C2 and C4 show marginal reduction by approximately 5% to 10% as compared to that of control concrete. The loss in compressive strength of concrete mix C2 and C4 at 28, 90,180 and 365 days curing age was within 10%. This reduction in compressive strength was considered almost insignificant up to 10% variation as per the provisions of Bureau of Indian Standards BIS: 516 -1991(2002). The minute loss in compressive strength of concrete mix C2 and C4 might be due to lack of finer content in marble aggregates. The compressive strength of concrete mixes designed by packing density approach show better performance as compared to that of concrete mixes designed by BIS code method. The major advantage of the concrete designed by packing density approach was saving in cement content by 24% as compared to that of concrete mixes designed by BIS code method.

5.3.5 Flexural Strength

The flexural strength test was conducted on the concrete specimens and results are shown in Figure 5.7.

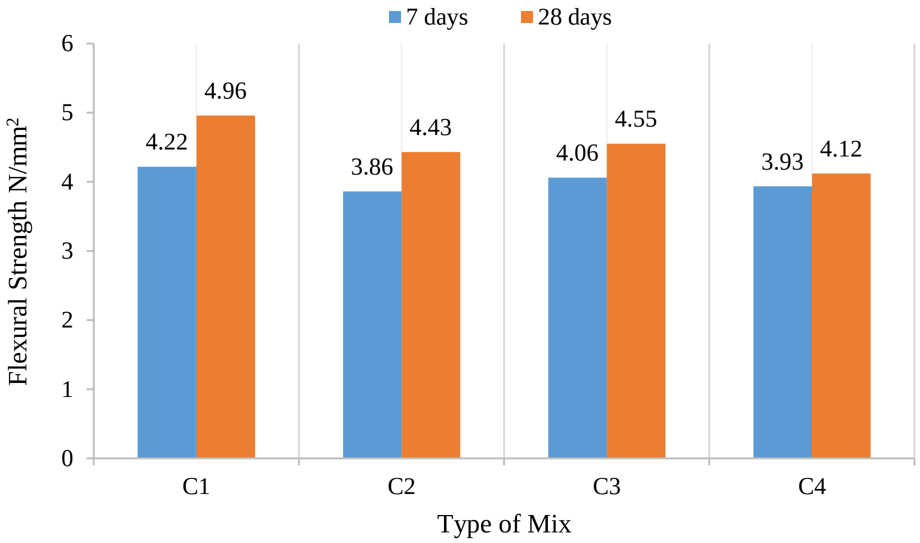


Figure 5.7 Variation in Flexural strength of concrete mixes

Form the Figure 5.7, a negligible reduction in flexural strength of concrete C2, C3 and C4 was observed. The flexural strength of 28 days concrete mix C2 was reduced by 10% as compared to that of control mix C1, mix C4 reduced by approximately 9% as compared to that of mix C3. As we compare the results of control mixes designed by both the methods, mix C3 shows 8% reduction in flexural strength as compared to that of mix C1. The overall loss in flexural strength of concrete mixes was within 10%.

The concrete mixes designed by packing density approach with partial replacement of conventional coarse aggregate by marble aggregate does not show any major loss in flexural strength. This decrease is almost insignificant up to 10% variations and this fact was also reported by Binici et al. (2008) in their study.

5.3.6 Permeability

To examine the porosity of concrete the permeability test was conducted as per German standard DIN-1048-part 5 (1991). The permeability was measured in terms of depth of water penetration in concrete samples. The variation in depth of penetration of various concrete mixes are shown in Figure 5.8.

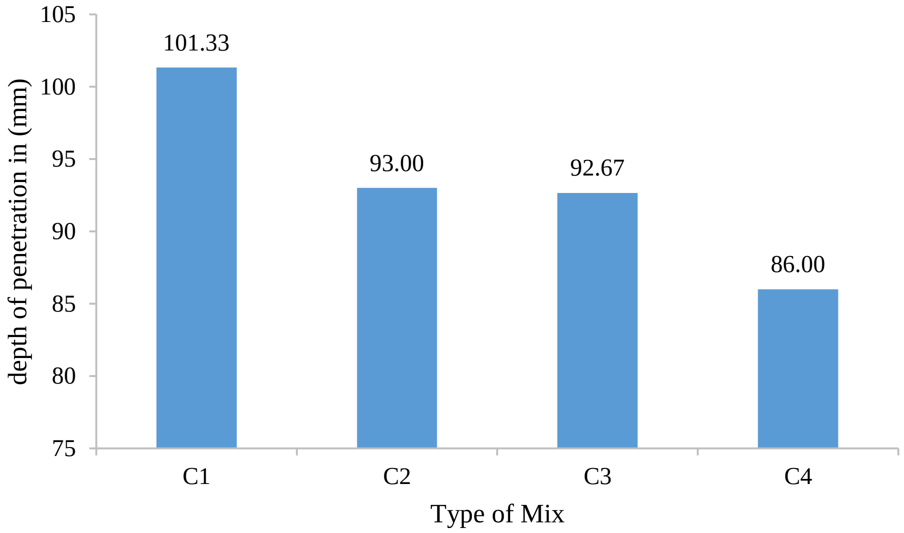
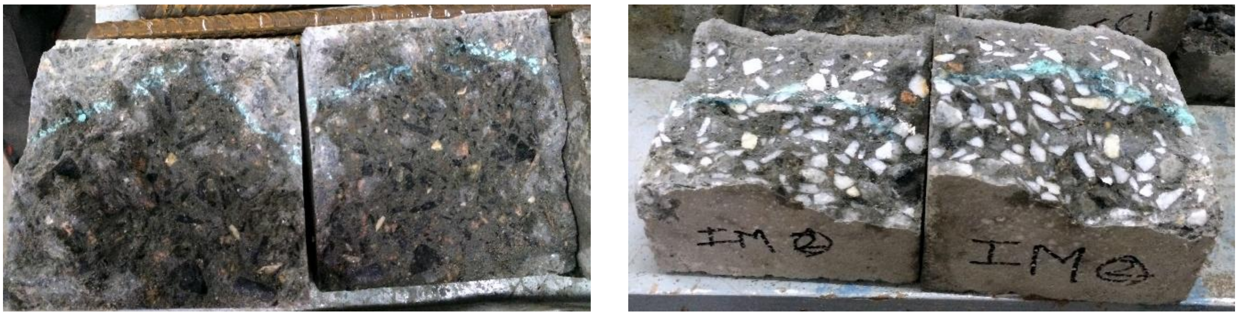


Figure 5.8 Variation in water permeability of concrete mixes

From the results of permeability test it was observed that, the permeability of concrete mixes decreases with the use of aggregate produced from marble waste. There are number factors on which the permeability depends. The most important factor which influences the permeability of concrete is interconnectivity of pores in the concrete. The depth of water penetration of concrete mix C2 reduced by 8% as compared to that of control mix C1. This reduction is possibly due to less porosity of marble aggregate as compared to that of conventional aggregate. It can be seen from Table 3.5 that water absorption of marble aggregate is about 25% of that of normal aggregate. On the other hand, the change in design approach in mix C3 and C4 shows reduction in depth of water penetration in concrete mixes. The fine aggregate content in mix C4 was increased by approximately 20% as seen from Table 4.2. The increase in sand content plays an important role for achieving dense packing of particle within the concrete. The reduction in depth of water penetration was attributed to the filling of pores. Another reason for decrease in depth of water penetration in mix C2 and C4 is round shaped marble aggregate which leaves minimum voids resulting in permeability (De Larrard, 2009 and Quiroga et al, 2004).



Mix C1 Mix C2

Mix C3 Mix C4

Figure 5.9 Images of water permeability test specimens

5.3.7 Ultra Sonic Pulse Velocity (UPV)

A non-destructive test was conducted on concrete specimens as per BIS: 13311 part 1; 1992 to assess the homogeneity of concrete. The underlying principle of assessing the quality of concrete is that comparatively higher velocities are obtained when the quality of concrete in terms of density, homogeneity and uniformity is good. In case of poor quality, lower velocities are recorded. The results of ultra-sonic pulse velocity test are shown in Figure 5.10.

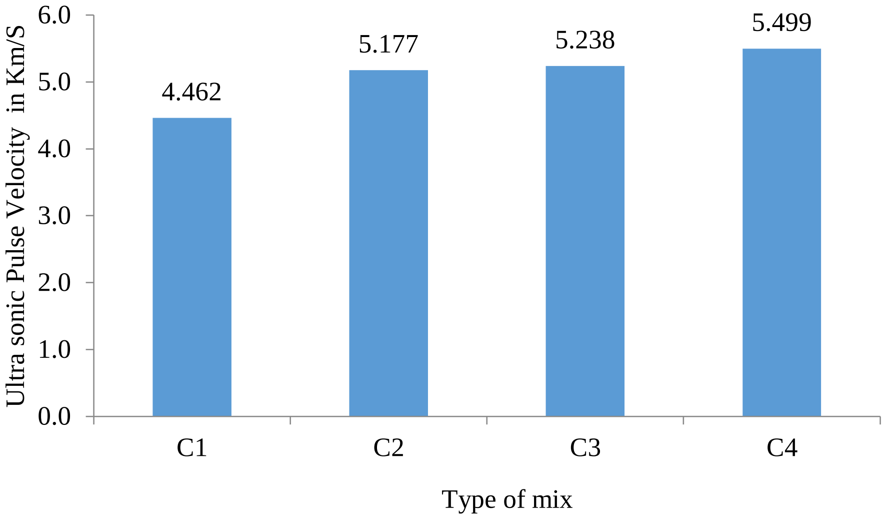


Figure 5.10 Variation in Ultra sonic pulse velocity of concrete mixes

It can be seen from Figure 5.10 that all the concrete mixes fall in the excellent category as per BIS: 13311 part 1; 1992 shown in Table 5.2. The ultrasonic pulse velocity values range from 4.462 km/s to 5.499 km/s. The values obtained in concrete mixes C2 and C4 increased as compared to that of respective control mixes.

Table 5.2 Velocity criteria for concrete quality grading

Sr. No	Pulse velocity by cross probing	Concrete quality grading
1	Above 4.5	Excellent
2	3.5 to 4.5	Good
3	3.0 to 3.5	Medium
4	Below 3.0	Doubtful

The highest value of ultra-sonic pulse velocity was obtained in mix C4. Mixes C3 and C4 were designed by the same method hence pore filling effect was same for both the mixes. Aggregate was changed as marble waste in C4 mix. Marginal increase in UPV value for C4 is due to less porosity of marble as coarse aggregate as compared with that of conventional aggregate. The increase in the value of mix C2 and C4 indicates better quality in terms of density, homogeneity and uniformity as compared to those of control mixes.

5.3.8 Abrasion Resistance

Resistance to abrasion was measured in terms of depth of wear of concrete under standard testing condition as per BIS 1237: 1980 (Bureau of Indian Standards 1237 2001). The average depth of wear of concrete specimens is shown in Figure 5.11.

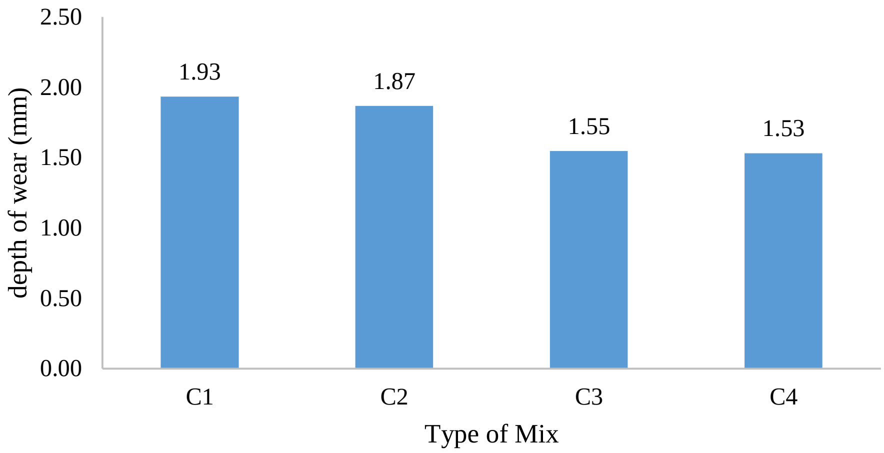


Figure 5.11 Variation in depth of wear of concrete mixes

The depth of wear range between 1.53 mm to 1.93 mm. The highest value of abrasion was for control concrete and the lowest value for concrete prepared using marble waste. As per the provisions given by the BIS 1237: 1980, in general purpose tiles the average maximum wear shall not exceed 3.5 mm and for heavy-duty floors, it is 2.5 mm. However the results obtained in this test depict that, depth of wear in all the concrete mixes are within the permissible limits specified by the code and can be used for heavy duty flooring tiles.

5.3.9 Carbonation

On exposure to carbonation carbon dioxide from the atmosphere reacts with calcium hydroxide to form calcium carbonate. On further reaction with carbon dioxide, calcium bicarbonate is formed. The reaction is as follows:

$$CO_2 + Ca(OH)_2 \quad \rightarrow \quad CaCO_3 + H_2O \quad \xrightarrow{CO_2} \quad Ca(HCO_3)_2 \qquad (5.1)$$

To judge this chemical change, carbonation test was carried out on the concrete mixes as per CPC: 18- RILEM guidelines. An aqueous alcoholic solution of 1% phenolphthalein was sprayed on the freshly broken pieces of the specimens. In carbonated areas the solution remain colorless and in non-carbonated areas the phenolphthalein indicator turns purple red. The results of the carbonation test for all the concrete mixes at different exposure durations are presented in Figure 5.12. From Figure 5.13 the carbonated areas can be clearly differentiated from the non-carbonated areas due to the color change.

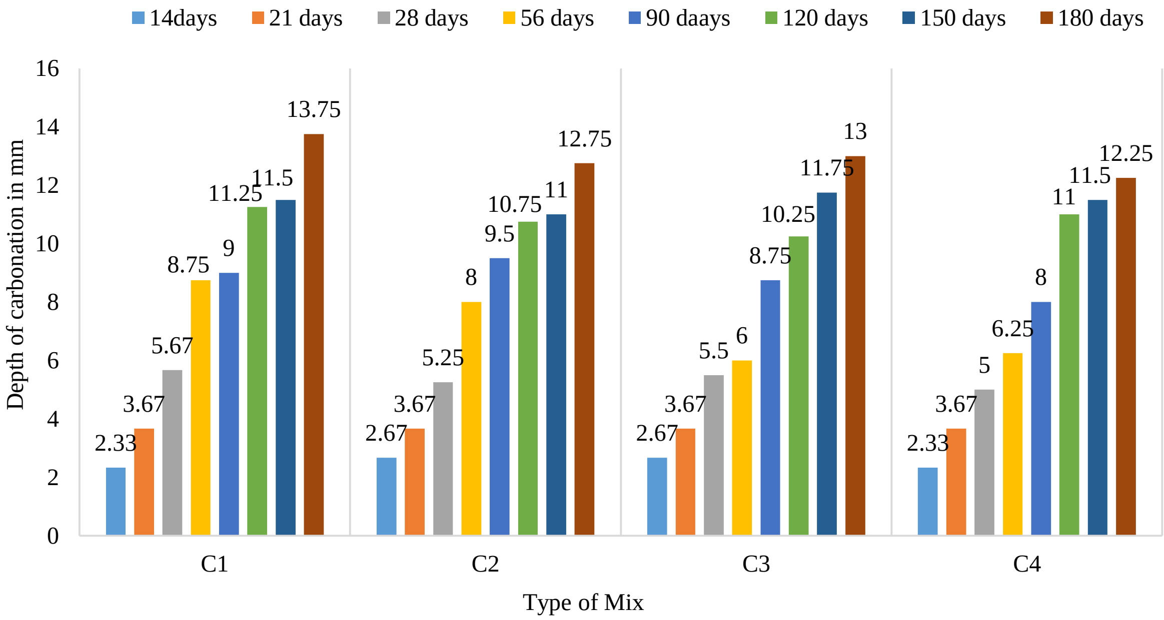
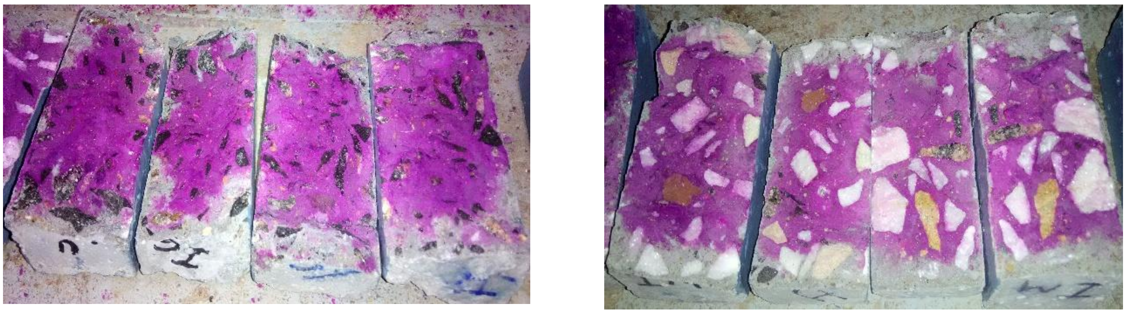


Figure 5.12 Variation in depth of carbonation of concrete mixes

From Figure 5.12 it can be seen that, the depth of carbonation of concrete mixes increases with increase in exposure duration. In the initial days of exposure up to 21 days the carbonation depth of mix C2 and C4 was almost equal to that of respective control mixes. At 28, 56 and 90 days the depth of carbonation of concrete mix C2 reduced by 7%, 8% and 5% as compared to that of control mixes. In case of mix C4 the depth of carbonation reduced by approximately 9% at 28 and 90 days as compared to that of control mix. The results obtained for mix C2 and C4 are closer and slightly lower than that of control mixes at all exposure durations. Also the incorporation of marble aggregate as replacement for conventional coarse aggregate does not have adverse effect on carbonation resistance. The carbonation resistance of concrete mainly depends on its porosity. The porosity in the concrete mix C4 was reduced due to dense microstructure resulting in favorable results. This fact was clearly seen from the result of the ultra-sonic pulse velocity and water absorption tests.



Mix C1 Mix C2

Mix C3 Mix C4

Figure 5.13 Images showing depth of carbonation after 180 days of exposure

5.3.10 Acid Attack

The durability of concrete structures which are situated in the industrial areas is likely to be affected by acids present in the environment. Impact of acid on concrete mixes under study is described in following paragraph.

5.3.10.1 Visual Observation of Acid Attacked concrete specimens

The images of the concrete specimens after exposure to acid attack are shown in Figure 5.14. It can be clearly seen that, with the increase in duration of exposure of concrete specimens the deterioration level increases. This fact is attributed to removal of cement paste and mortar by the attack of acid. After 56 and 91 days of immersion in acid, the concrete specimens showed higher loss of cement matrix and white spots due to formation of gypsum are clearly seen on concrete specimens as shown in Figure 5.14. The equation 5.2 shows the reaction mechanism for the formation of Gypsum and Ettringite due to action of sulphuric acid.

$$\begin{aligned}
&Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4 .2H_2O \\
&CaSiO_2 \, . \, 2H_2O + H_2SO_4 \rightarrow CaSO_4 + Si\,(OH)_4 + H_2O \qquad (5.2)\\
&3CaO. \, Al_2O_3 \, .12H_2O + 3\,(CaSO_4 \, . \, 2H_2O) + 14H_2O \rightarrow \underset{\text{(Ettringite)}}{3CaO. \, Al_2O_3 . \, 3CaSO_4 . \, 32H_2O}
\end{aligned}$$

(a) after 28 days of immersion in acid solution

C1 C2 C3 C4

(b) after 56 days of immersion in acid solution

C1 C2 C3 C4

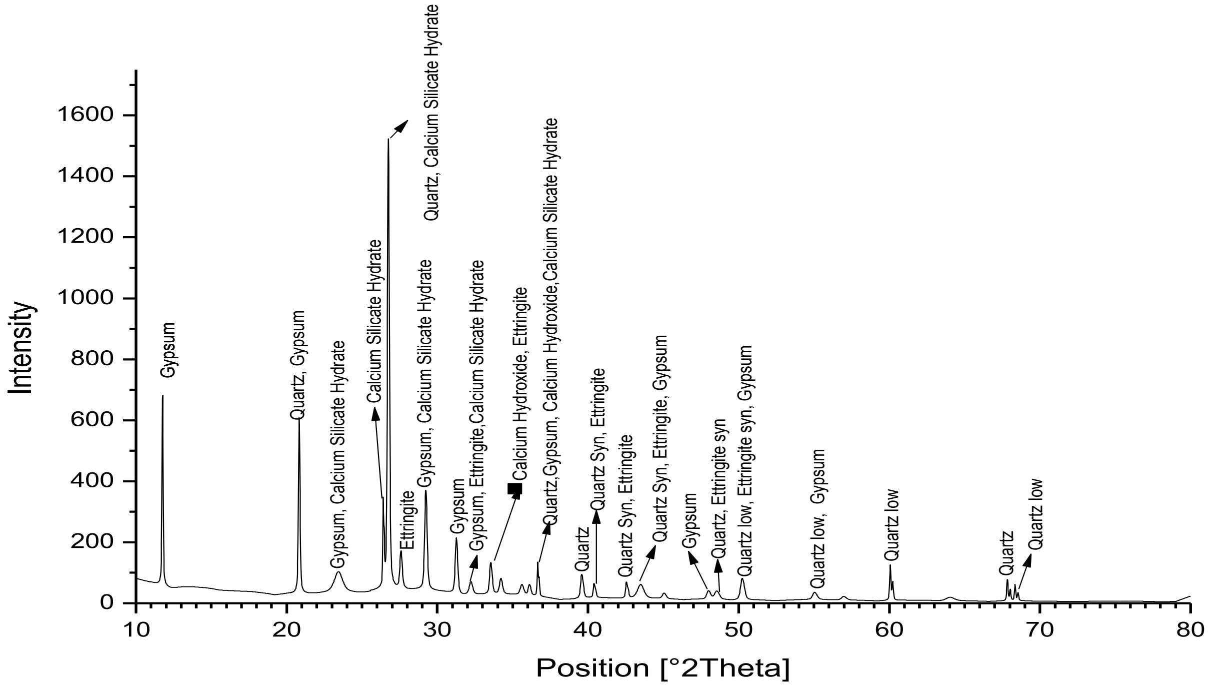
(c) after 91 days of immersion in acid solution

C1 C2 C3 C4

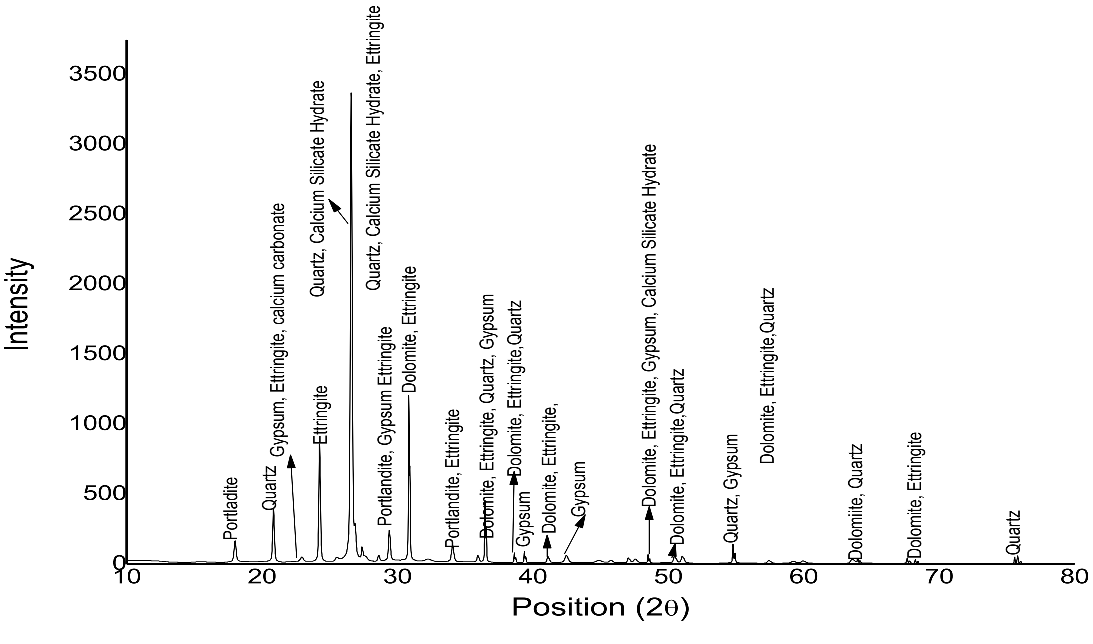
Figure 5.14 Images of Acid attacked concrete specimens at different exposure durations

5.3.10.2 X-Ray diffraction analysis of acid attacked samples

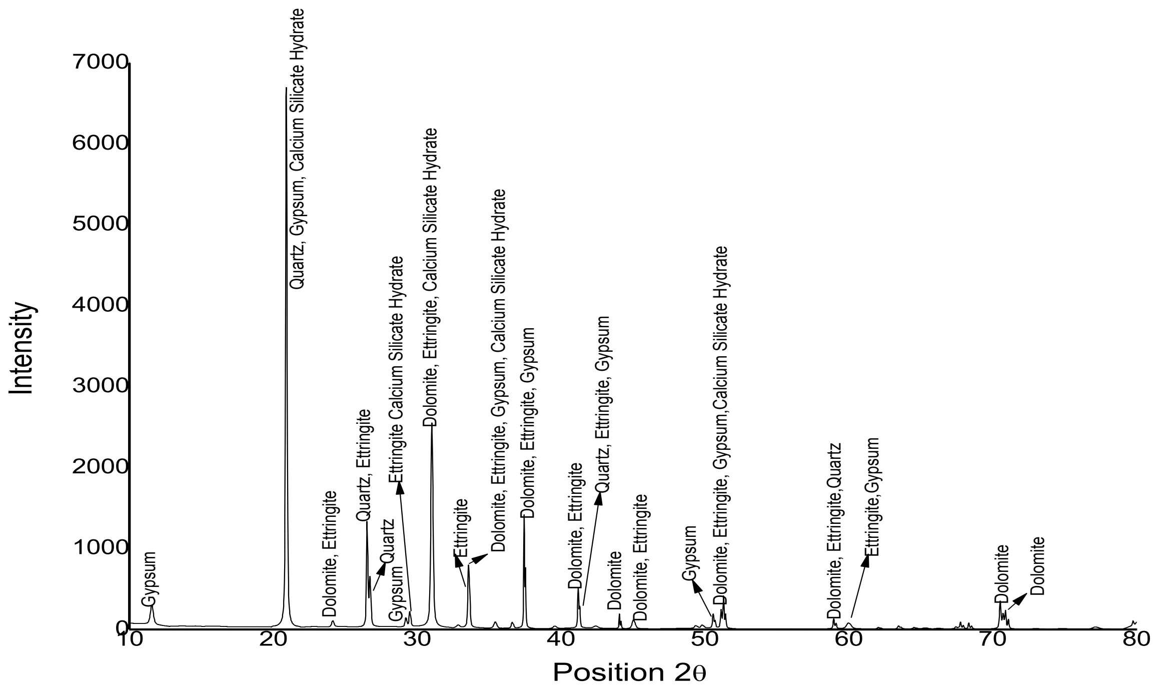
The X-ray diffraction analysis was carried out on the powder samples of concrete specimens exposed to 5% dilute sulfuric acid solution after 91 days. The results of the study are shown in Figure 5.15.



(a) XRD of control concrete Mix C1 exposed to 5% H_2SO_4 solution after 91 days



(b)XRD of concrete mix C2 exposed to 5% H_2SO_4 solution after 91 days



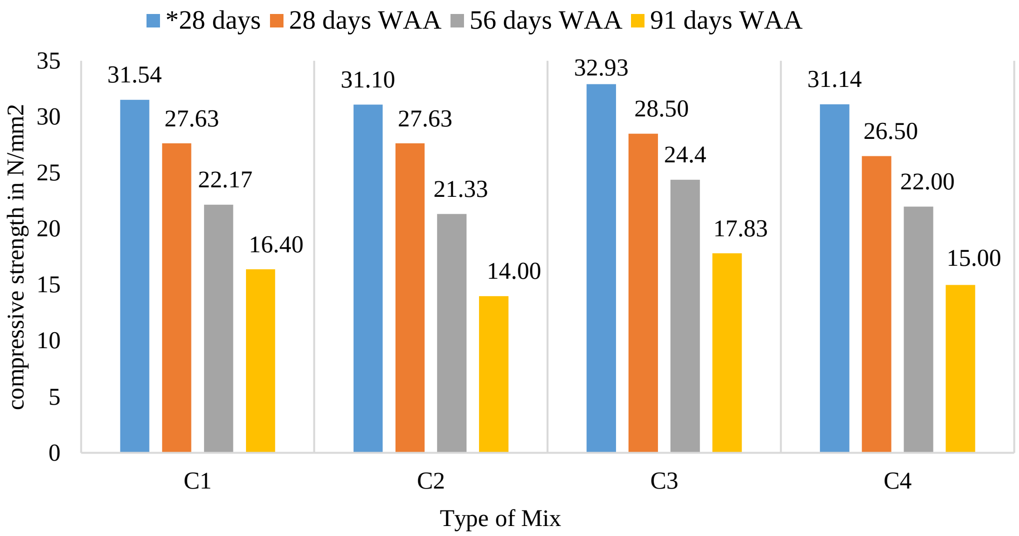
(c)XRD of concrete mix C4 exposed to 5% H_2SO_4 solution after 91 days

Figure 5.15 X-Ray diffraction analysis of powder samples of concrete specimens after 91 days of exposure in sulphuric acid

The X-Ray diffraction analysis was carried out on the powder samples of concrete specimens after 91 days of exposure in sulphuric acid and it was observed that, the concrete mix C2 and C4 showed more number of peaks of dolomite present in marble and products like Gypsum and Ettringite as compared to that of control mix. The formation of these products was due to presence of carbonate in the marble aggregate which are very prone to sulphuric acid reaction (Mehta & Monteiro 2001a).

5.3.10.3 Loss in compressive strength

The results of the compressive strength test after acid attack are shown in Figure 5.16.



*Without acid attack, WAA: - With Acid Attack

Figure 5.16 Variation in compressive strength of acid attacked concrete specimens

It can be seen that, approximately 15% fall in compressive strength was recorded for all concrete mixes after 28 days exposure to sulphuric acid. All these values are higher than characteristic compressive strength of M25 grade concrete for which design mix C1 was attempted. With increase in duration of exposure, the compressive strength reduced. The reduction in compressive strength values was approximately 50%of that of control concrete after 91 days exposure. The basic reason behind the decrease in compressive strength was reaction between portlandite [$Ca\ (OH)_2$] in the cement paste with sulphuric acid which resulted in formation of gypsum ($CaSO_4.\ 2H_2O$). This was followed by a reaction between earlier formed gypsum crystals and calcium aluminate which led to formation of ettringite. The peaks of ettringite are seen in X-ray diffraction pattern shown in Figure 5.15.

In particular the loss in compressive strength of concrete mixes C2 and C4 was comparatively more than that of respective control mixes C1 and C3. The reason behind this is presence of calcium carbonate content in marble waste. Calcium carbonate is also prone to sulphuric acid attack and forms gypsum readily. The peaks in X-ray diffraction pattern shows significant formation of gypsum and ettringite minerals as seen in Figure 5.15 (b) and Figure 5.15 (c). Ettringite increases its own volume by 2.2 times as reported by K & Rizkalla (1989). The increase in volume induces internal pressure within the voids resulting in formation of cracks, which allow the entry of solution into the voids and attack the cement paste and aggregate further. Replacement of siliceous aggregate by calcareous aggregates containing dolomite having a minimum calcium oxide concentration 50% aids in neutralizing the acid as reported by (Beatrix Kerkhoff 2002). The acid attacks the entire exposed surface more uniformly, reducing the rate of attack on the paste and preventing loss of aggregate particles at the surface. Therefore, appreciable loss in strength due to presence of marble aggregate in C2 and C4 mixes is not seen. The compressive strengths of C3 and C4 mixes are slightly higher than that of C1 and C2 mixes because of dense structure created by increased packing density. Also the reduced paste contents in mixes are less susceptible to damage the structure of concrete. Thus adopting packing density approach for concrete mixes even with marble waste as coarse aggregate has no significant detrimental effect on compressive strength even after exposure to acidic environment. This also depicts that, the packing density approach followed for design of concrete mixes results in better durability aspects.

It is worth to mention that such study of concrete mixes with marble waste exposed to acid has been reported first time. The results are not discouraging as these are almost at par with those of control concrete.

5.3.11 Sulphate Attack

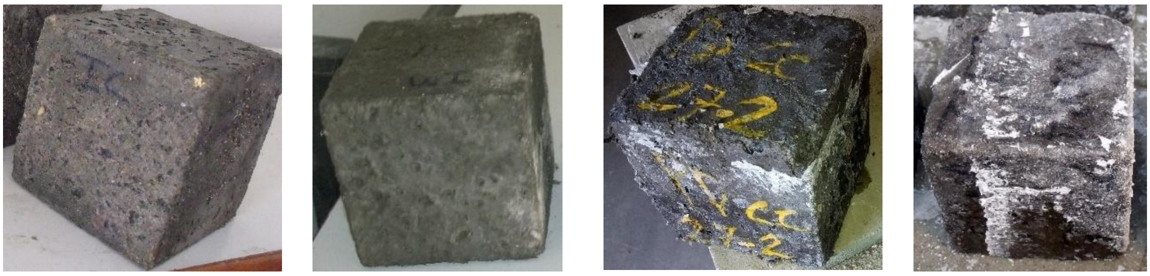
The concrete specimens were immersed in 10% magnesium sulpahte solution for a period of 300 days. The visual observation and impact on compressive strength are discussed in next paragraph.

5.3.11.1 Visual observation of concrete specimens

The images of concrete specimens after exposure to 10% $MgSO_4$ solution are shown in Figure 5.17. During the test period up to 91 days of immersion no spalling or cracks were observed on the concrete specimens. Only white spots of sulphate were seen on the specimens as shown in Figure 5.17 (a). As the exposure duration extended to 180 days, the concrete specimens showed signs of spalling and cracking on the surface. This was due to formation of ettringite as seen in scanning electron microscope images shown in Figure 5.15.Continued formation of ettringite fill the voids and finally concrete specimen burst leading to formation of wide cracks and spalling of concrete as seen in Figure 5.17 (c).

(a) after 91 days of immersion in sulphate solution

C1 C2 C3 C4



(b) after 180 days of immersion in sulphate solution

C1 C2 C3 C4

(c) after 300 days of immersion in sulphate solution

C1 C2 C3 C4

Figure 5.17 Images of Sulphate attacked concrete specimens at different exposure durations

5.3.11.2 Loss in mass

The change in the mass of concrete specimens after exposure to 10% magnesium sulphate solution over a period of 28, 56, 91, 180 and 300 days is shown in Figure 5.18.

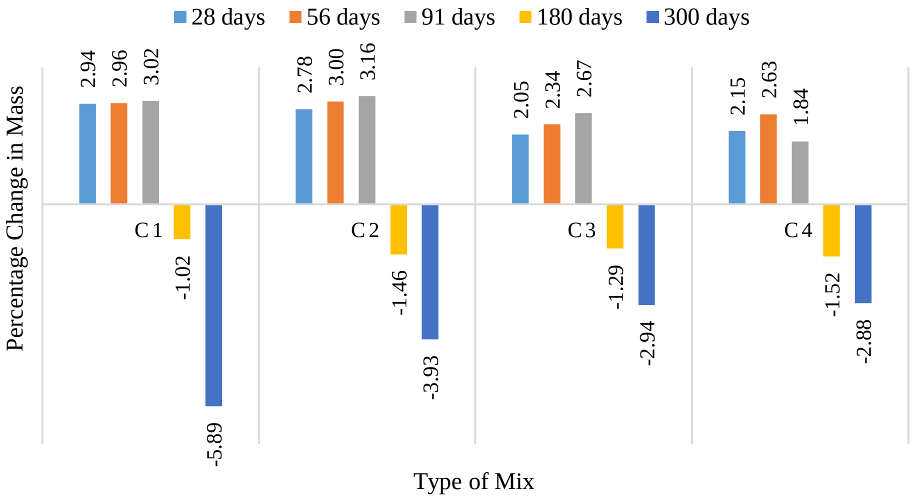
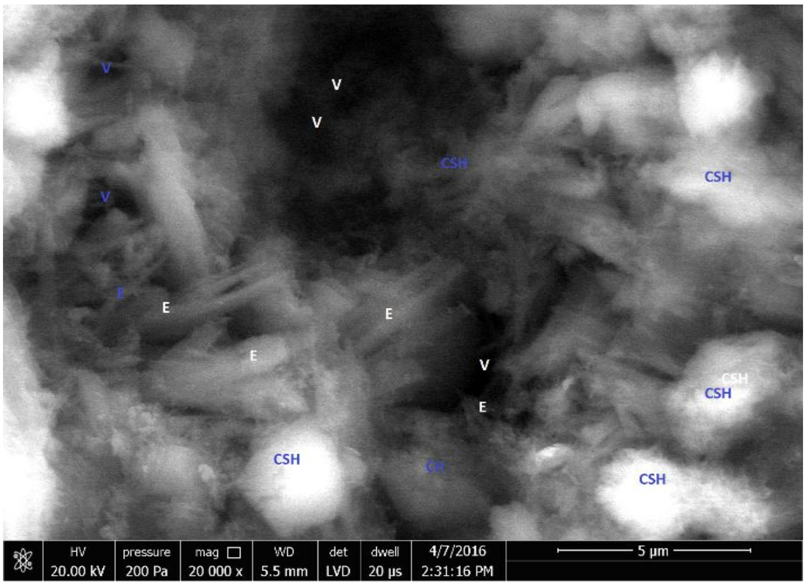


Figure 5.18 Variation in mass of concrete specimens after exposure to sulphate solution

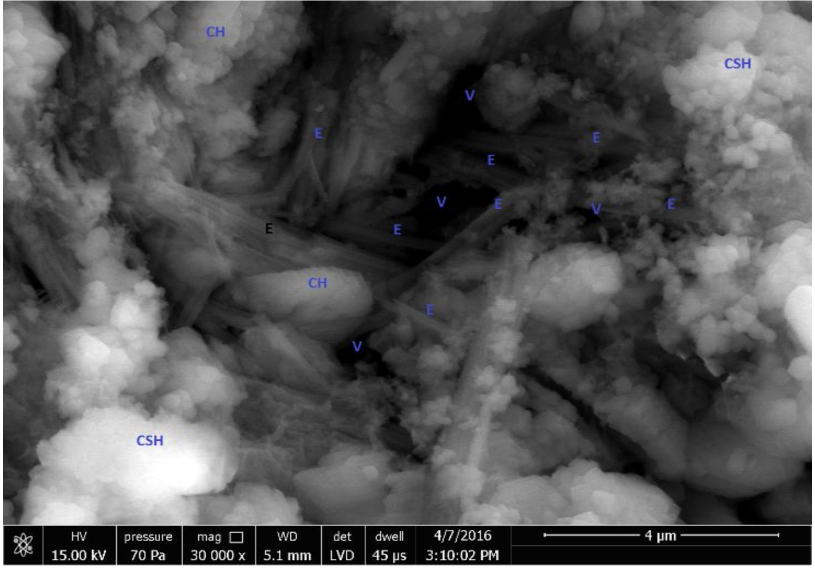
It can be seen from the Figure 5.18 that, all concrete mixes show increase in mass of concrete up to exposure period of 91 days. This increase in mass was due to formation of expansive product such as gypsum and ettringite minerals within the pores by the reaction of magnesium sulphate with the free lime in the cement paste. The filling of pores by the ettringite needles as seen in scanning electron microscope images of Figure 5.19 resulted in increased mass of concrete specimens. On the other hand the increase in exposure period beyond 91 days, concrete mixes showed loss in mass. This decrease in mass was associated with the excessive formation of ettringite product due to action of magnesium sulphate with the cement paste. The crystals of ettringite formed within the voids exert pressure on the surrounding area, also the absorption of solution cause swelling of concrete. The swelling and cracks formed on the surface of concrete are clearly seen in Figure 5.17 (c). It is interesting to note that initial increase in mass of concrete mixes C1 and C2 was higher than that of concrete mixes C3 and C4. This is due to availability of higher cement paste content in C1 and C2 mixes which after chemical reaction with $MgSO_4$ produce increased amount ettringite.

$$\begin{aligned} &C_4AH_{13}+3CSH_2+14H \longrightarrow C_6AS_3H_{32}\ _{\text{(Ettringite)}}+CH \\ &C_4ASH_{(12\text{-}18)}+2CSH_2+{}_{(10\text{-}16)}H \longrightarrow C_6AS_3H_{32}\ _{\text{(Ettringite)}} \\ &C_3A+3CSH_2+26H \longrightarrow C_6AS_3H_{32}\ _{\text{(Ettringite)}} \end{aligned} \tag{5.3}$$

Note: C-CaO, A- Al_2O_3, H-H_2O, S- SO_4, CH- $Ca(OH)_2$



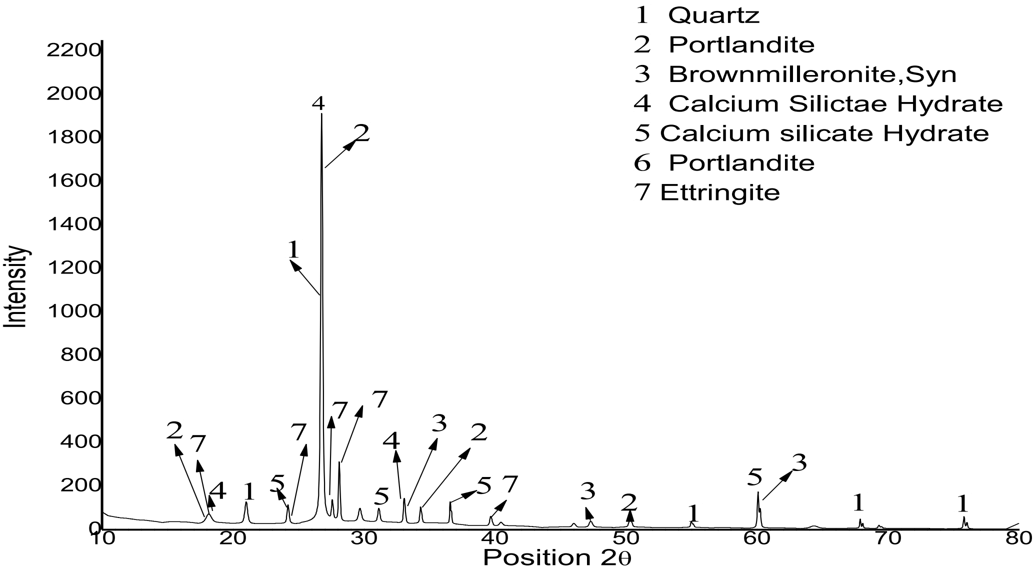
(a) Mix C1



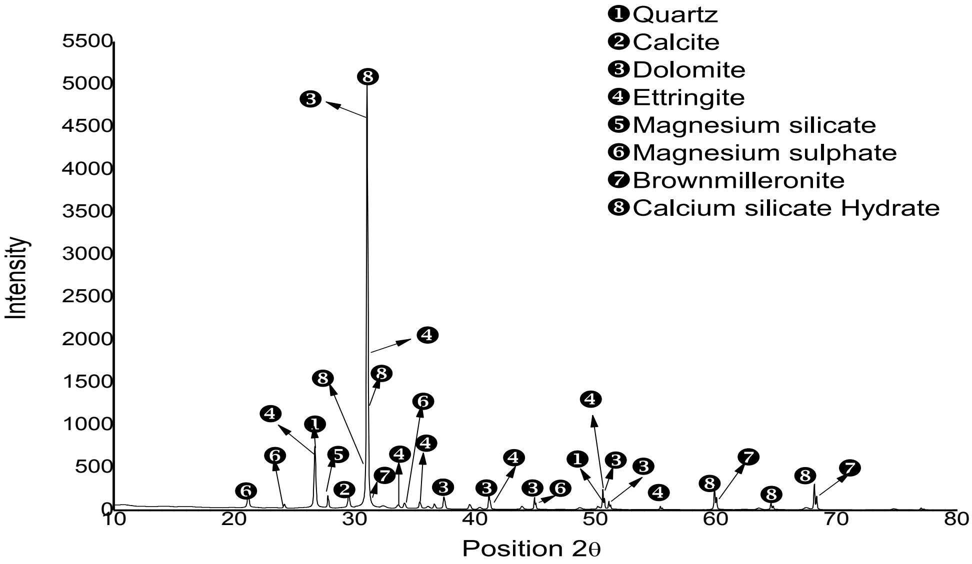
(b) Mix C4

[E=Ettringite, CH= Calcium Hydroxide, CSH =Calcium Silicate Hydrate, V= voids]

Figure 5.19 SEM micrographs of concrete mixes after 180 days of immersion in 10% magnesium sulphate solution



(a) Mix C1

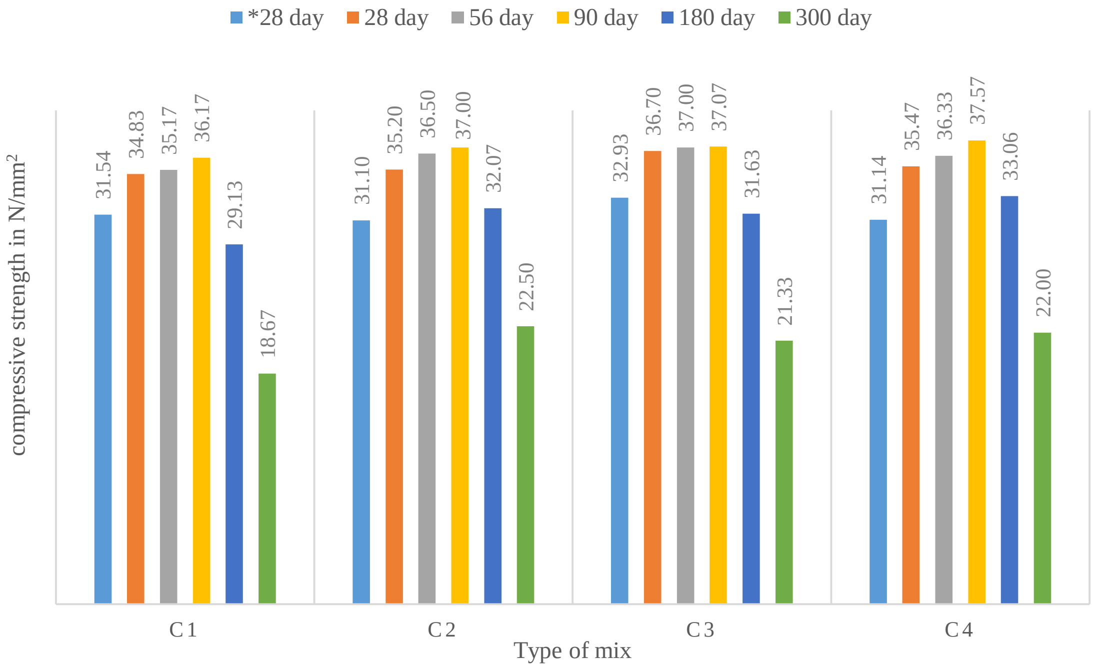


(b) Mix C4

Figure 5.20 XRD of concrete mixes after 180 days exposure to 10% MgSO4 solution

5.3.11.3 Loss in compressive strength

Figure 5.21 shows the variation in compressive strength of concrete mixes exposed to 10% magnesium sulphate solution over period of 300 days.



Note: *- without sulfate attack

Figure 5.21 Variation in compressive strengthof concrete specimens after exposure to sulphate solution

From the Figure 5.21 it can be seen that, the compressive strength of all concrete mixes increased with increase in exposure duration up to 91 days. The compressive strength of concrete mixes C1, C2, C3 and C4 increased by approximately 11%, 18%, 13% and 20% after 91 days of exposure. The initial increase in compressive strength of sulphate attacked concrete specimens was caused by the ettringite crystals formation (refer Figure 5.19) within the pores present in the concrete mixes. These ettringite crystals fill up the voids within the concrete.

As the exposure period extended beyond 91 days the concrete showed loss in compressive strength. It was due to excessive formation of expansive products like gypsum and ettringite in the concrete as identified in X-ray diffraction analysis shown in Figure 5.20. The formation of ettringite causes volume expansion within the voids resulting in spalling as reported by Mehta & Monteiro, (2001) and finally cracks were formed on the surface of the concrete. These cracks increase the permeability of concrete and allow solution to enter the voids. The entry of sulphates in pores starts attacking the cement paste and deteriorate the bond between aggregate and cement paste. Finally deterioration of cement paste causes loss in compressive strength of concrete. The loss in compressive strength of concrete mix C2 and C4 was 27% and 29% whereas concrete mix C1 and C3 show 41% and 35% loss in compressive strength after exposure of 300 days. The results clearly indicate that, the concrete mixes containing aggregate produced from marble waste show better performance as compared to that of control mixes because use of calcareous aggregates retard expansion resulting from sulfate attack caused by acid ($MgS0_4$) solution Beatrix Kerkhoff (2002).

One more dominating characteristic feature of mixes C2 and C4 is the capability of marble aggregate to strengthen the interfacial transition zone around them. This strengthening improves the long-term durability of concrete (P. Kumar 2003). As a result, the fall in strength is relatively lesser than for mixes without marble aggregates when subjected for a prolonged exposure of 300 days.

5.3.12 Chloride Ion Penetration

Silver nitrate spray test was used to measure the depth of chloride ion penetration. The color change at the surface of the broken sample helps differentiate the regions of free chloride (colorless) and the region where chloride is bound by C_3A in the form of calcium chloroaluminate hydrate (brown) in the cement matrix (P. Kumar 2003). The depth of penetration (i.e. where chloride content is in excess than the capacity of up to which C_3A can react and bind it) where chloride is left unbound was obtained by measuring the average depth in three samples. The results of depth of chloride ion penetration are shown in Figure 5.22.

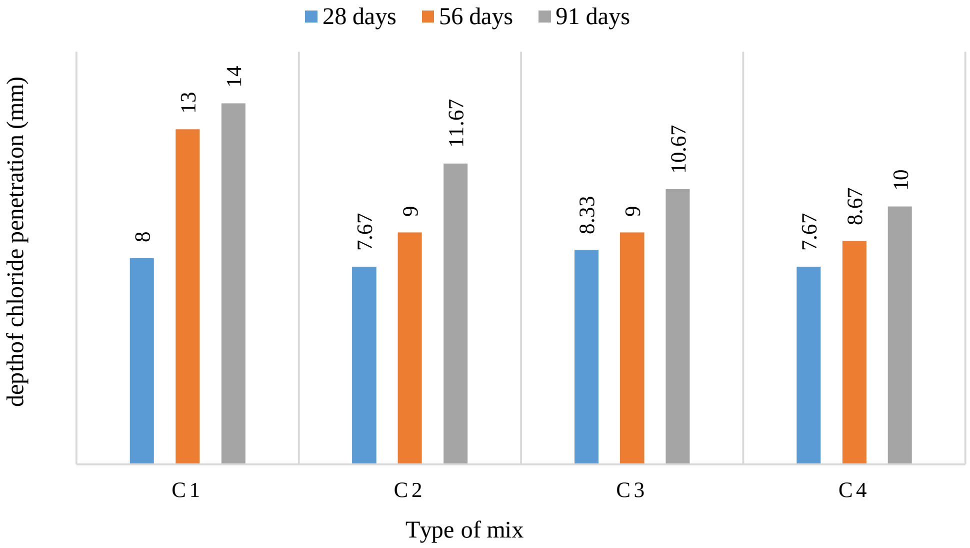
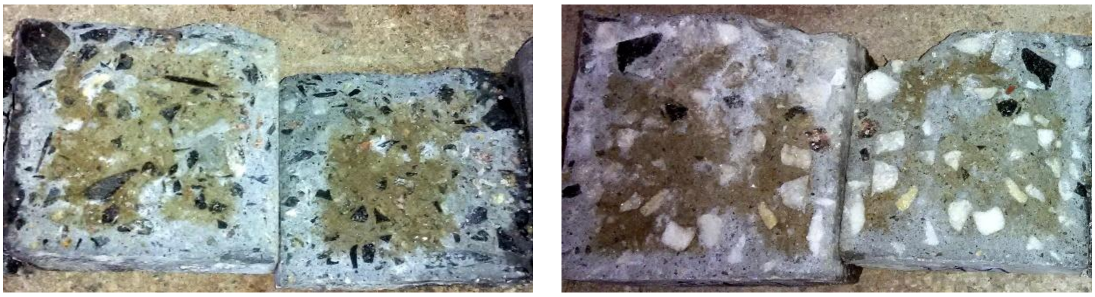


Figure 5.22 Variation in depth of chloride penetration of concrete mixes

It can be observed that, the depth of chloride penetration of concrete containing marble aggregates show better performance. The mix C4 shows lowest values of depth of penetration as compared to that of control mix. The depth of chloride penetration of concrete mainly depends on the porosity of concrete. The concrete mixes designed by packing density approach showed lesser porosity as compared to that of concrete mixes designed by BIS code method. Marble aggregate with less porosity also restricts flow through coarse aggregate. This has been already proved from the results of water absorption and permeability tests. Therefore, the possible reason for decrease in penetration depth is reduced porosity of marble aggregate and concrete mix due to better packing of particles within the concrete mix.

It is worth mentioning that, though C_3A reacts with marble aggregates to form calcium carbo-aluminates, on exposure to a chloride environment, the capacity of C_3A to fix the free chlorides has not been affected significantly. The chloride penetration results assert the same.

Another reason for this reduction is presence of Al_2O_3 in the marble aggregates. The presence of alumina favors the formation of tricalcium aluminate (C_3A), which fixes the chloride ions and forms insoluble compounds. Binici et al. (2008) also reported that, concrete prepared by replacing primary aggregates by marble aggregates enhances the resistance against chloride penetration.



Mix C1 Mix C2

(c) Mix C3 (d) Mix C4

Figure 5.23 Images of chloride ion penetration of concrete specimens after 91 days

5.3.13 Cost Analysis and CO2 Emission

The cost analysis of the concrete mixes designed by both the methods is given in Table 5.3. From the Table 5.4 it was observed that, the change in design methodology resulted in saving in cement content. Designing the concrete mixes by packing density method resulted in approximately 20% reduction in cement consumption as compared to that of mix C1. Overall concrete mix designed by packing density method and use of marble waste in concrete reduces the overall production cost around 14%.

Table 5.3 Total material cost for production of Concrete Mix

Cost of material for production of 1 m^3 of concrete						
Mix	Water	Cement	Sand	Natural Aggregate	Marble Aggregate	Total material cost (Rs.)
C1	-	2386/-	355/-	479/-	-	3220/-
C2	-	2386/-	355/-	120/-	310/-	3171/-
C3	-	1949/-	425/-	488/-	-	2862/-
C4	-	1920/-	425/-	98/-	317/-	2760/-

Table 5.4 Saving in cement content

Mix	Cement content (kg/m^3)		Saving in cement by Packing Density method (kg/m^3)	Percentage saving in cement
	BIS code method	Packing Density method		
Control Mix	426	348	78	18.30
Mix with marble waste	426	343	83	19.48

The Carbon dioxide emission from the production of cement and emission generated during the transportation of aggregates is shown in Table 5.5.

For production of 1 Ton of cement around 0.94Ton of carbon dioxide is liberated in to the environment (Şahan Arel 2016). The CO_2 emission factor for road transport i.e. for truck or lorries is considered as 512.2 g/Km (Ramachandra & Shwetmala 2009).

Table 5.5 Caron di Oxide Emission from Cement and Aggregate source

Carbon di Oxide Emission per m^3 of concrete					
Mix	Cement (Kg)	CO_2 emission (Kg)	CO_2 emission (Kg)		
			Source of Natural Aggregate	Source of Marble Aggregate	Total
C1	426	401	2.75	-	2.75
C2	426	401	0.7	0.26	0.95
C3	348	327	2.65	-	2.65
C4	343	322	0.68	0.25	0.93

Form the above table it can be observed that, the cement content required for the design of concrete mix C4 reduced by 20% as compared to that of control mix C1 which resulted in 20% lesser emission of carbon dioxide.

Adopting packing density approach for design of concrete mixes would reduce the annual global cement production and CO_2 release by 20%. The concrete produced using packing density approach is not only cost effective but durable and sustainable product mitigating environmental pollution to a large extent.

5.4 Impact of Fire on properties of concrete

Impact of fire on concrete mixes with marble waste as coarse aggregate has been studied first time. The mechanical and durability properties have been studied in depth and results have been supported by optical microscopy and XRD analysis.

5.4.1 Visual observations of concrete after exposure to fire

The test specimens were exposed to real standard fire at temperatures 200, 400, 600,800ºC in a gas fired furnace. Up to a temperature up to 400°C, not much change was observed on the surface of all the concrete specimens. When temperature was raised beyond 400ºC, the exterior surface turned light red [refer Figure 5.24(c) and Figure 5.26(c)] in C1 and C3 mixes. This was due to presence of Fe_2O_3 in quartzite used as conventional coarse aggregate. This red color dominated on surface at 600ºC and later converted into dark patches at 800ºC exposure. The cracks also started appearing on surface of test specimens [refer Figure 5.24 (d) and Figure 5.26 (d)] at 600ºC due to pore pressure built up by conversion of moisture into vapors.

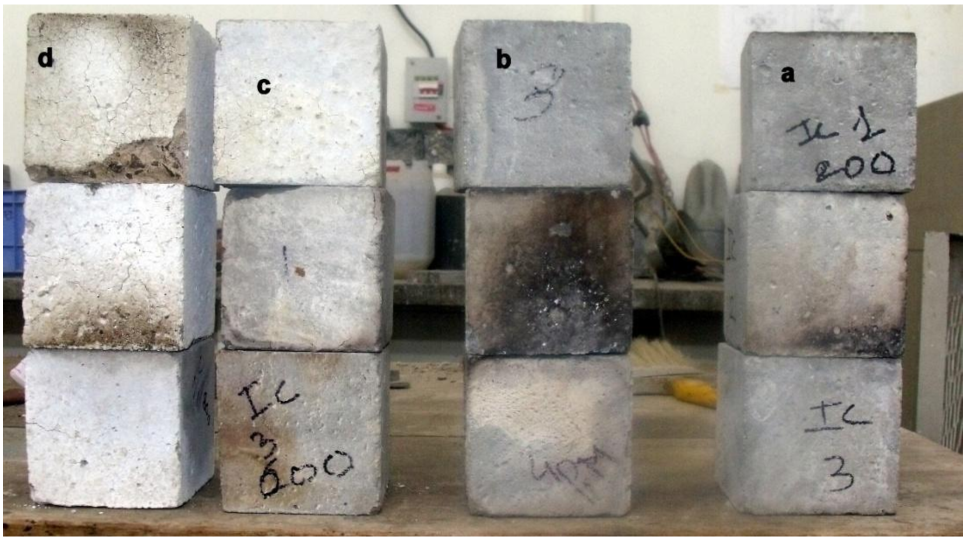


Figure 5.24 Concrete mix C1 after exposed to (a) 200°C, (b) 400°C, (c) 600°C and (d) 800°C

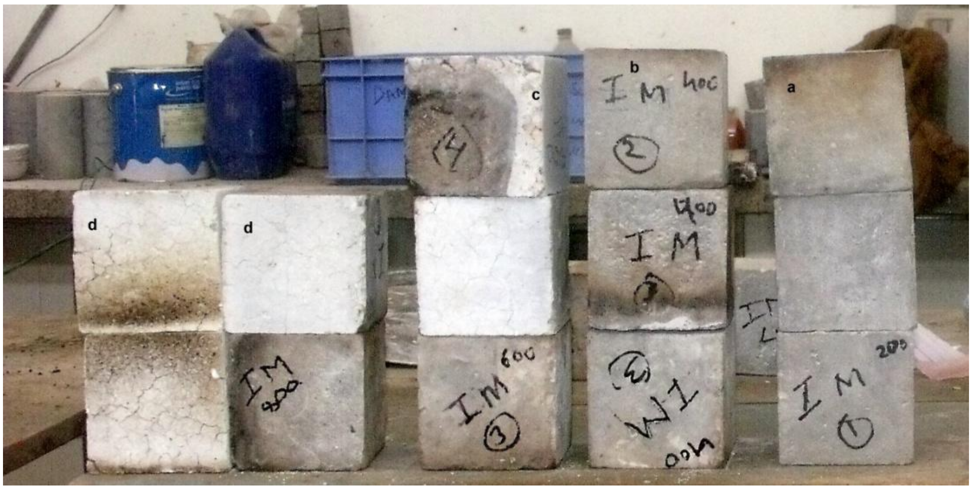


Figure 5.25 Concrete mix C2 after exposed to (a) 200°C, (b) 400°C, (c) 600°C and (d) 800°C

Figure 5.26 Concrete mix C3 after exposed to (a) 200°C, (b) 400°C, (c) 600°C and (d) 800°C

Figure 5.27 Concrete mix C4 after exposed to (a) 200℃, (b) 400℃, (c) 600℃ and (d) 800℃

Concrete specimens of C2 and C4 with marble waste were observed with pale shades of white and grey [refer Figure 5.25(c) and Figure 5.27(c)] when exposed to 600ºC with appearance of random cracks on the surface. The color change was due to decomposition of $CaCO_3$ into CaO and CO_2 at elevated temperature. All the types of specimens show spalling of edges and corners at temperature beyond 600ºC. Another possible reason for cracks according to Hager (2013) is that, additionally formed CaO during process of de-carbonation at high temperature expands its volume by 44% and thereafter while cooling results in formation of cracks and spalling of concrete.

5.4.2 Loss in mass

Loss in mass of concrete specimens after exposure to fire is shown in Figure 5.28.

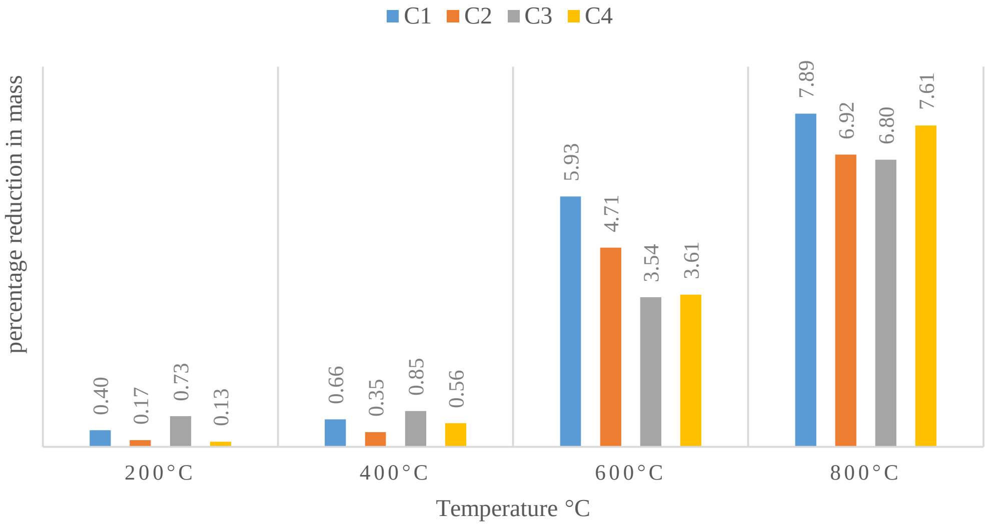


Figure 5.28 Variation in mass of concrete after exposure to fire

It can be seen that, loss in mass of concrete increases with increase in temperature of all the mixes. Higher the temperature more is the loss in mass of concrete. It was insignificant up to temperature of 400ºC but rose abruptly when temperature was raised to 600ºC. Initially weight was lost due to conversion of free moisture to vapors. Later when temperature was raised to about 500-600°C, the portlandite content rapidly drops, as decomposes according to following reaction:

$$Ca(OH)_2 \quad = \; CaO + H_2O \qquad (5.3)$$

At higher temperature of 800ºC major loss in weight of concrete was attributed to second phase of the C-S-H decomposition and dissociation of cement paste followed by spalling of concrete mixes in some instances. In fact first phase transformation of sand and conventional coarse aggregate in C1 and C3 mixes takes place at 574ºC due to BETA-ALPHA quartz inversion as reported by Hager Gaweska (2013). On the other hand $CaCO_3$ present in C2 and C4 mixes is stable up to 600ºC and it starts decomposing into CaO and CO_2 beyond 700ºC.

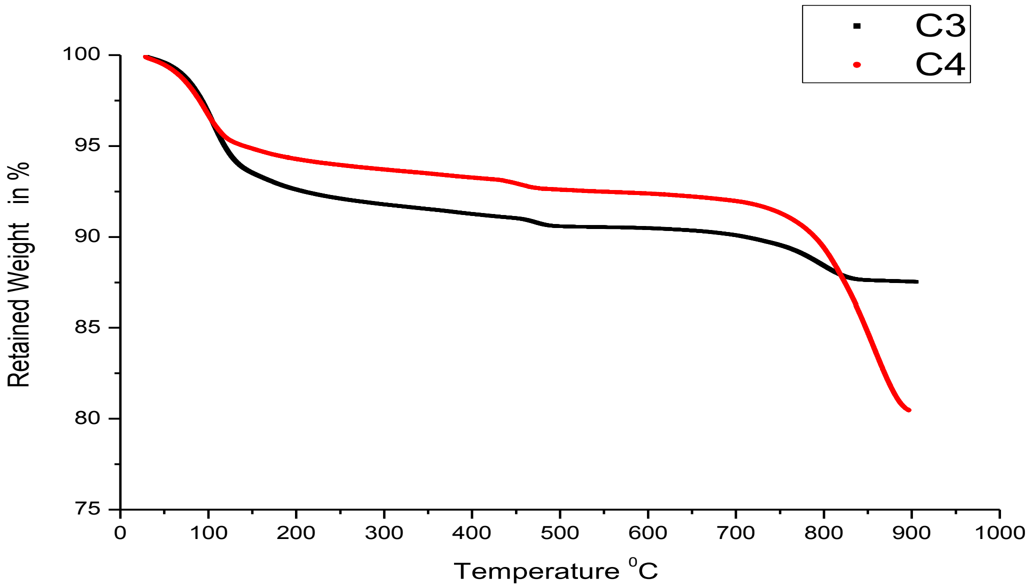


Figure 5.29 Thermo Gravimetric Analysis of concrete mixes

It abruptly starts melting around 800ºC and appreciable loss in mass of concrete can be noted. This fact has also been observed in Thermo Gravimetric Analysis (TGA) (refer Figure 5.29) conducted by author.

5.4.3 Compressive strength

The effect of fire on the compressive strength of concrete is shown in Figure 5.30. The compressive strength of concrete mixes C1 and C2 increases with increase in temperature up to 400ºC. This initial increase in strength is attributed to acceleration of hydration process of cement paste. Another possible reason for increase in compressive strength is due to reduction of internal pore pressure within the voids as repoted by Kodur (2000). The concrete mixes C3 and C4 indicate downward trend in strength with increase in temperature. There is about 15 -16% fall in the strength of both the mixes at exposure of 200ºC. This may be due reduction in cohesive forces with expansion of water at this temperature. When temperature rises to approximately 350ºC break up of some siliceous aggregates takes place (Hager Gaweska 2013) resulting in further fall in strength. The strength obtained at 400ºC is almost equivalent to characteristic strength of M25 mix that is expected from the water cement ratio of 0.45. Further increase in temperature in the range of 460-540°C leads to portlandite decomposition and showing whitish patches indicating formation of CaO (Piasta 1984).

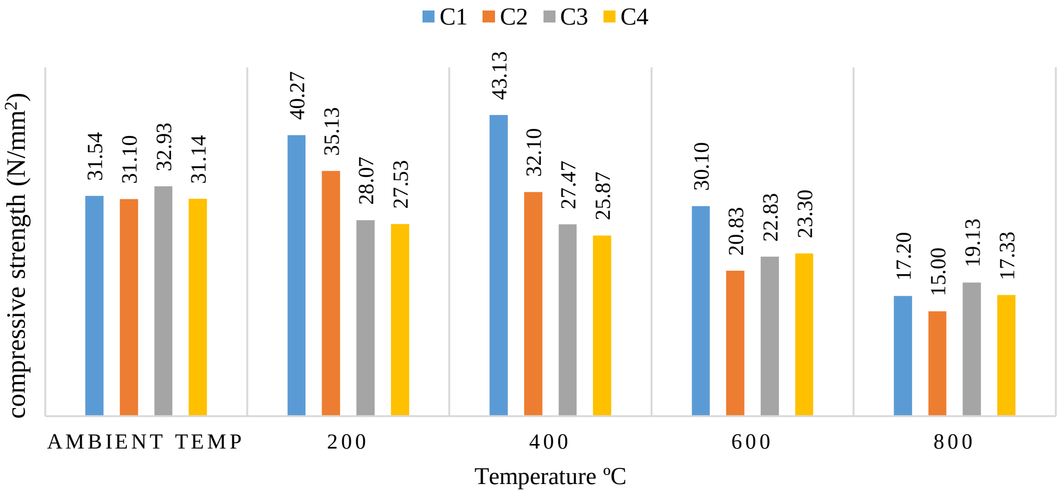


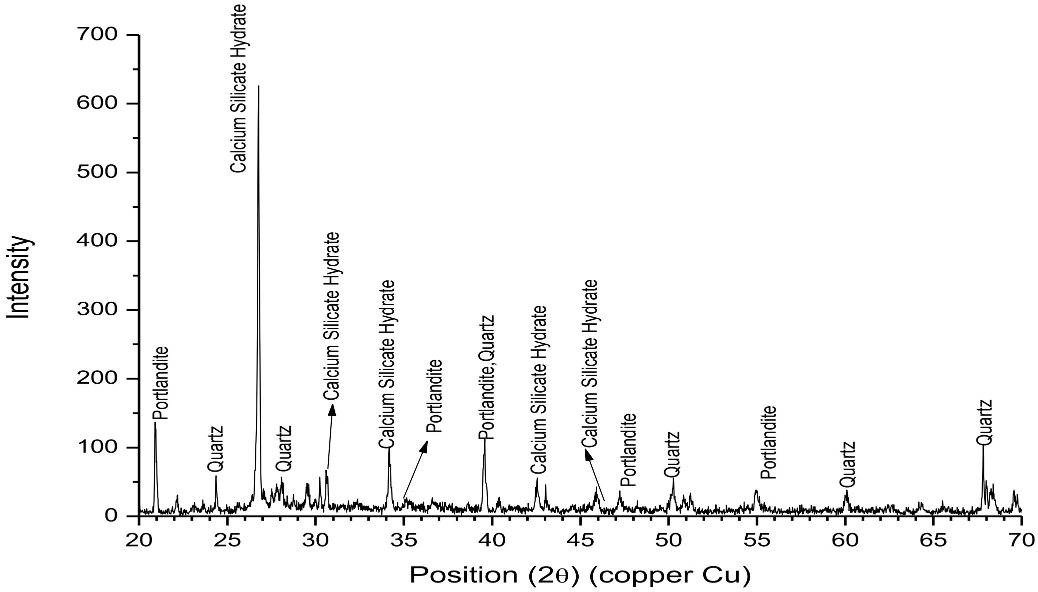
Figure 5.30 Compressive strength of concrete after exposure to fire

Marble aggregates remain relatively stable (refer Figure 5.30) at 600ºC showing slightly better strength of C2 and C4 mixes than that of C1 and C3 mixes. Continuous heating of test specimens cause further fall in strength of C2 and C4 mixes around 800ºC as marble aggregates start melting as observed in TGA (refer Figure 5.29). This phenomenon is also visible in optical microscope image shown in Figure 5.34(b) and (d).

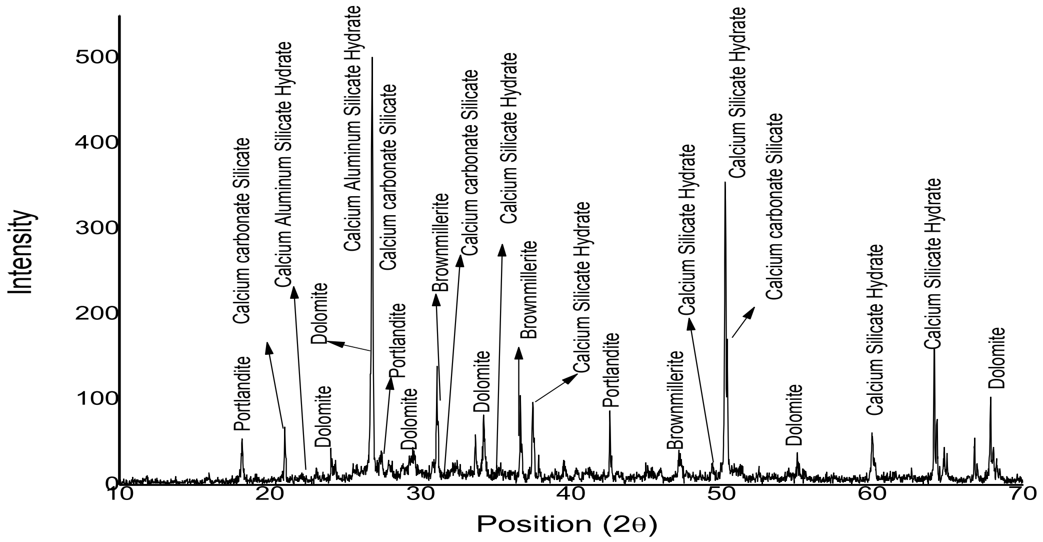
Reduction in compressive strength of all the concrete specimens around 800ºC was due to dehydration of C-S-H gel and decomposition of calcium hydroxide resulting in formation of calcium oxide. Peaks of calcium oxide are uniformly seen in XRD pattern [refer Figure 5.32(b)] of concrete mix C4 after exposure to fire. At higher temperatures around 800ºC the formation of gases within the concrete does not have enough space to release the stress, which results in crack formation, bursting of concrete or explosive spalling.

5.4.3.1 X-Ray Diffraction (XRD) Analysis

The X-ray diffraction analysis was carried out on the concrete specimens designed by packing density approach are shown in Figure 5.31 and Figure 5.32

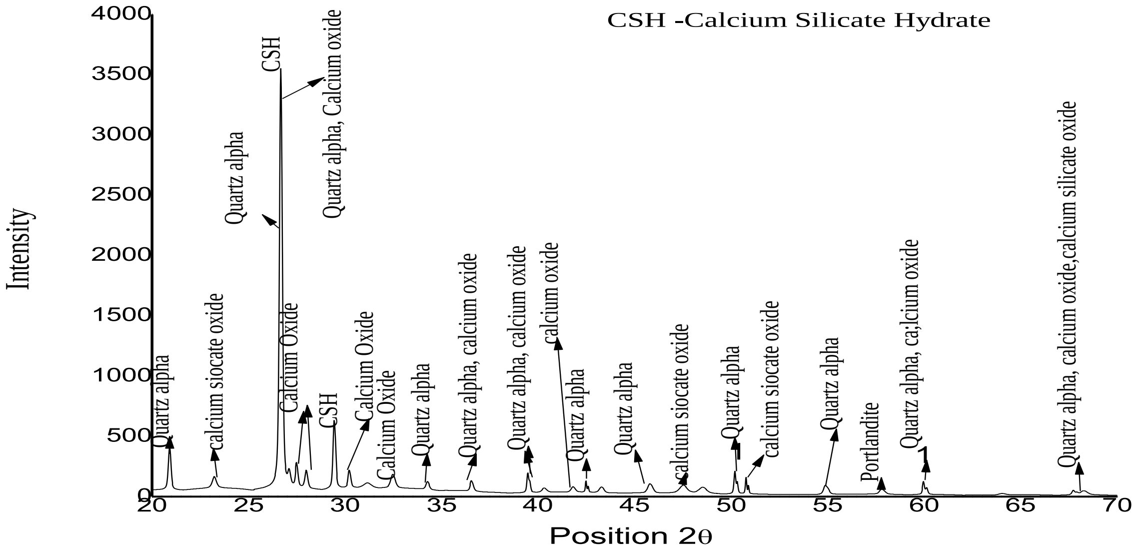


(a) XRD pattern of concrete mix C3

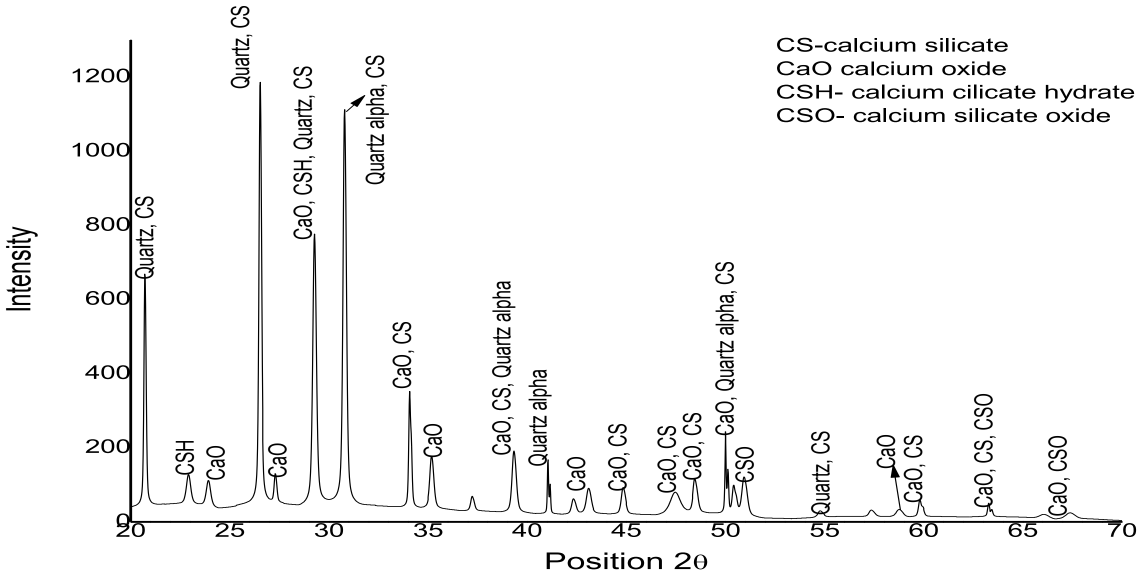


(b) XRD pattern of concrete mix C4

Figure 5.31 XRD of concrete samples at ambient temperature condition



(a) XRD pattern of concrete mix C3



(b) XRD pattern of concrete mix C4

Figure 5.32 XRD of concrete mixes after fire (at 800 °C)

From the XRD analysis it was confirmed that, after exposure to fire concrete undergoes physical and chemical changes. The decomposition of CSH gel and calcium hydroxide turns into calcium oxide (CaO) due to removal of chemically bound water. The alpha-beta conversion of quartz aggregate takes place at 573°C. A number of peaks obtained as a result of alpha-beta conversion of quartz aggregate after exposure to 800°C are seen in Figure 5.32 (a). Also the peaks of the CSH gels are comparatively very low as compared to that of ambient temperature samples as shown in Figure 5.31 and Figure 5.32. In the concrete mix C4, a number peaks of calcium oxide (CaO) were seen. This was due to dissociation of calcium carbonate. Other major peaks of calcium silicate were observed due to dissociation of calcium silicate hydrate as shown Figure 5.32 (b).

The decomposition of calcium hydroxide, calcium carbonate and calcium silicate hydrate resulted in formation of calcium oxide and calcium silicate. Due to dissociation of calcium silicate hydrate the loss in mechanical properties of concrete takes place which has been already discussed in above sections.

5.4.4 Water absorption

Water absorption data of concrete samples exposed to fire at various temperatures are shown in Figure 5.33.

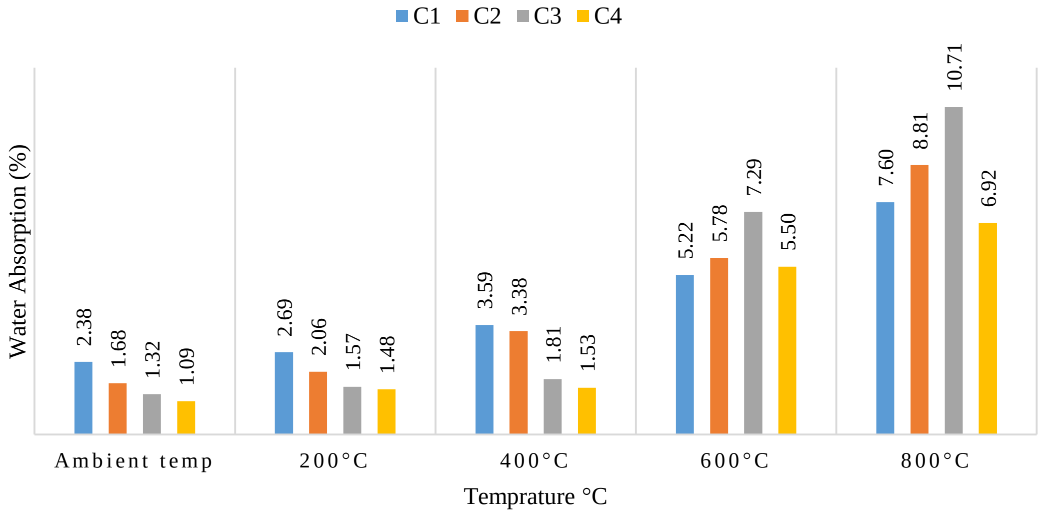


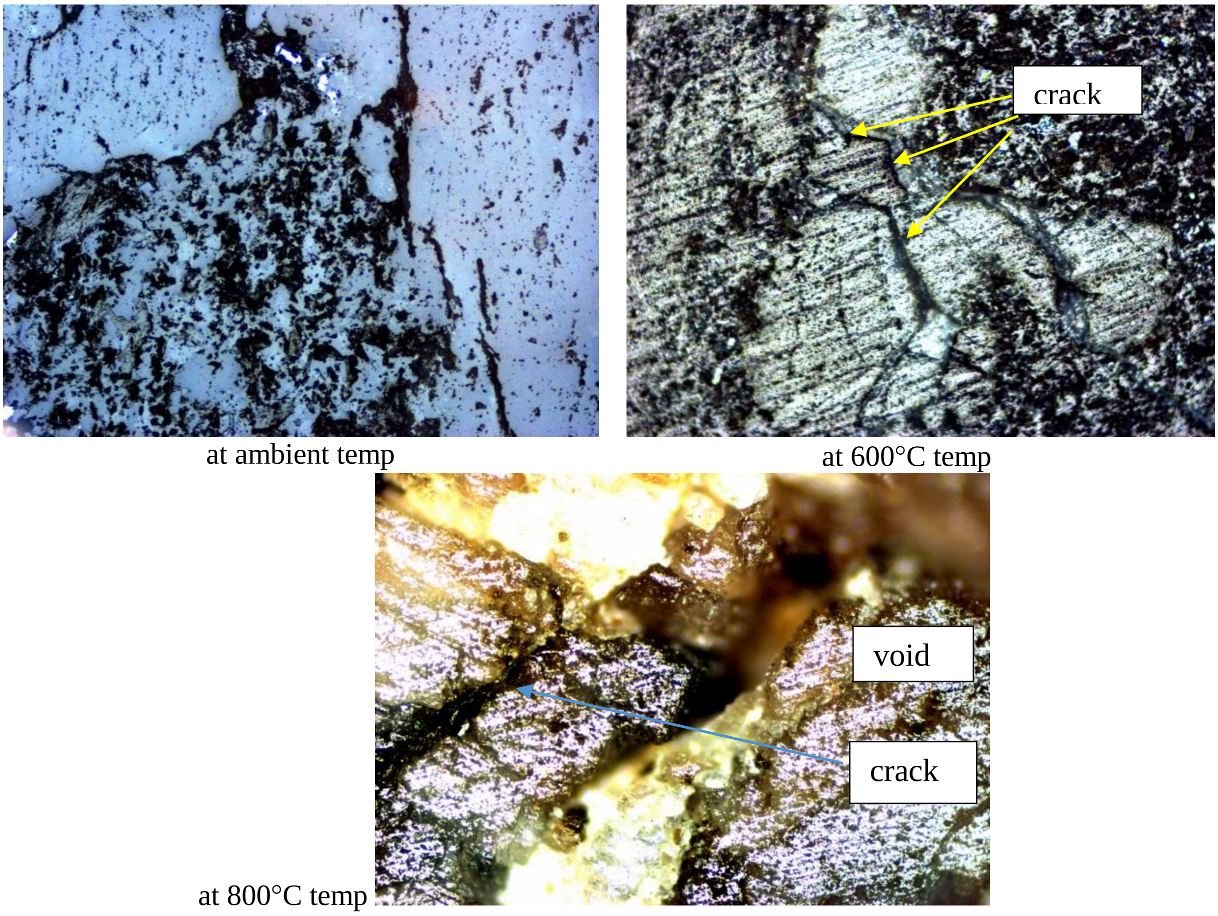
Figure 5.33 Water absorption of concrete after exposure to fire

It can be seen that water absorption increases with increase in temperature of fire exposure of test specimens. It increases gently till temperature of 400ºC is reached. Further increase in temperature shows higher water absorption at 600ºC and 800ºC. This is because of formation of excessive voids due to dehydration of C-S-H gel in both the mixes. The voids formed within concrete are clearly seen in the optical microscopic images as shown in Figure 5.34.

It is also observed that C2 and C4 mixes containing marble waste absorb less water than control mixes C1 and C3 due to the difference in water absorption of both the coarse aggregates. Sand and quartz aggregate undergo Alpha-Beta conversion around 600ºC in test specimens as observed in XRD pattern shown in Figure 5.31(a). Mixes designed by packing density method require approximately 20% extra sand. Thus more quantity of sand in C3 mixes are undergoing Alpha-Beta conversion and hence large volume of water absorption is seen at 600 and 800°C in such mixes.

5.4.5 Optical Microscopy

The optical microscopy study was carried on concrete samples exposed to fire at different temperatures as seen in Figure 5.34. This study was carried out to notice the changes in microstructure of concrete samples after exposure to fire.

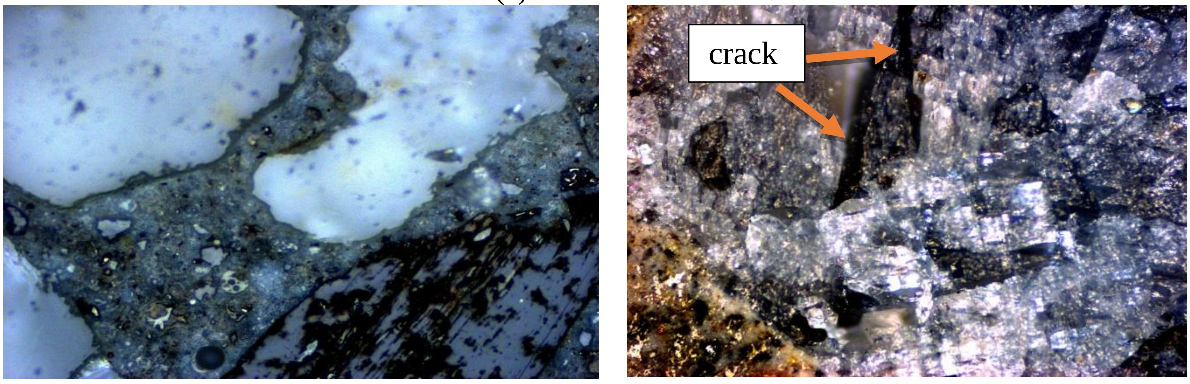


at ambient temp

at 600°C temp

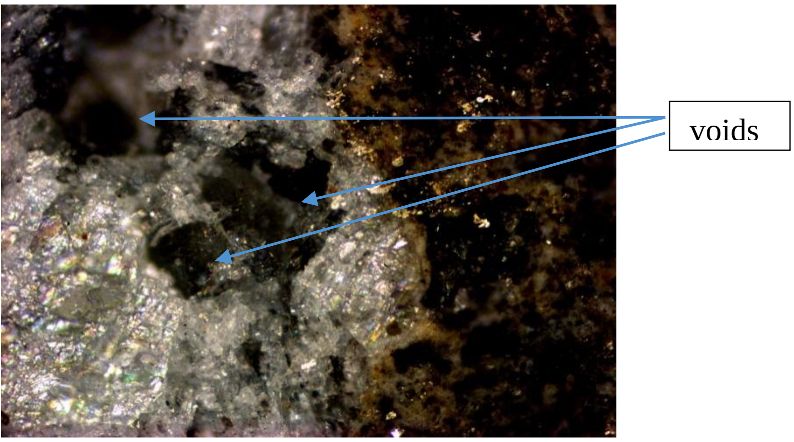
at 800°C temp

(a) Concrete mix C1



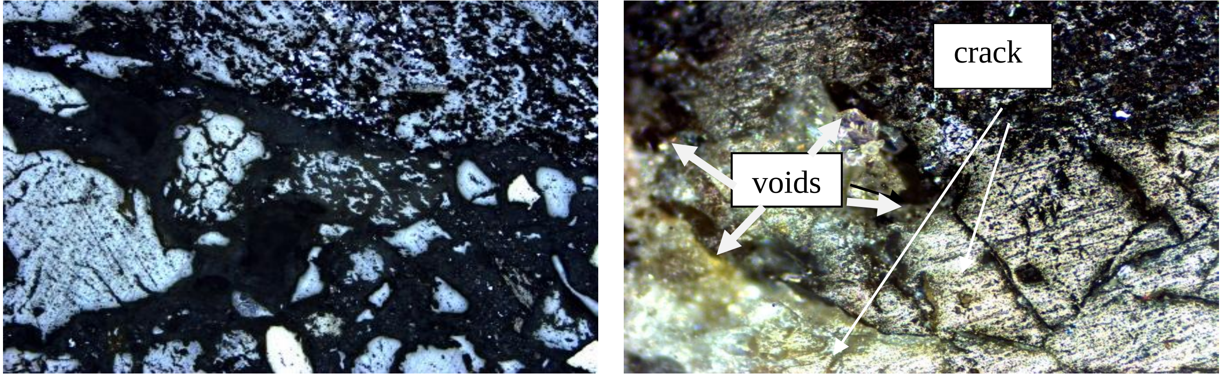
at ambient temp

at 600°C temp



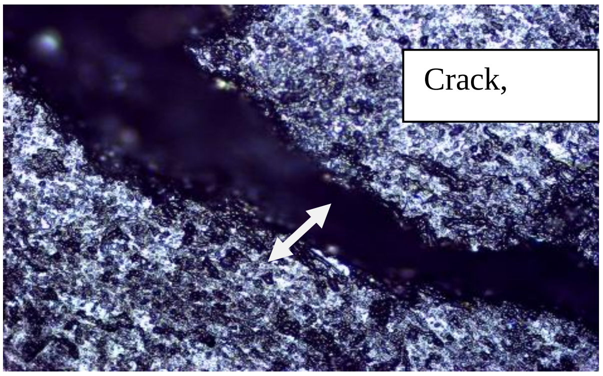
at 800°C temp

(b) Concrete mix C2



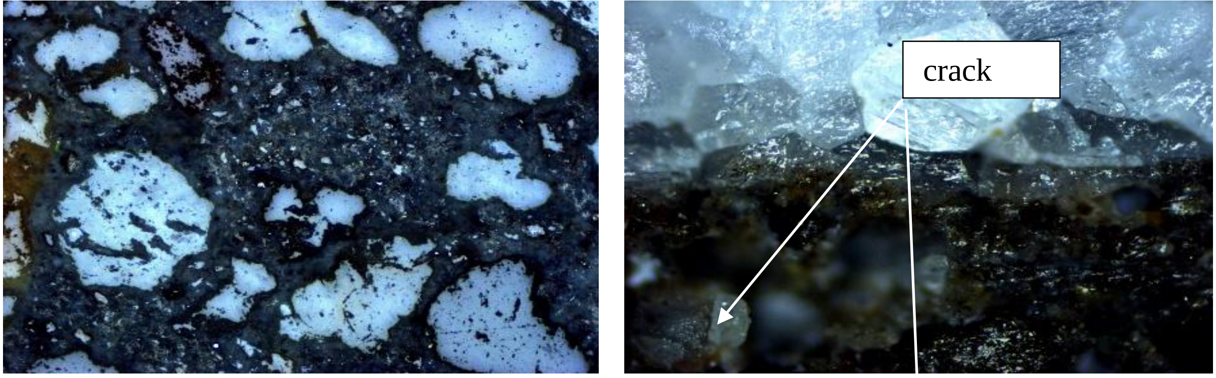
at ambient temp

at 600°C temp



at 800°C temp

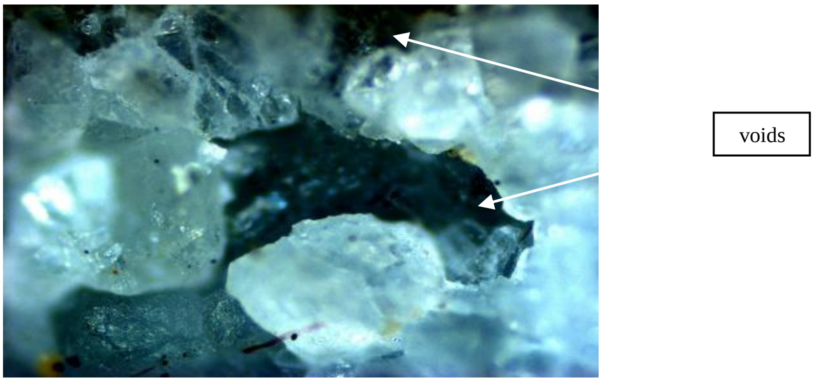
(c) Concrete mix C3



at ambient temp

at 600°C temp

at 800°C temp



(d) Concrete mix C4

Figure 5.34 Optical microscopy of concrete mixes at different temperature levels

The heating of concrete cause expansion of aggregate and contraction of cement paste around the aggregates. Cement-aggregate bond is the weakest point in concrete when exposed to fire. Majority of concrete structures are damaged due to thermal incompatibility of cement and aggregate within the concrete after exposure to fire. Figure 5.34 shows images taken from optical microscopy of control concrete made of siliceous aggregate and concrete containing marble aggregate after exposure to 600°C and 800°C. After exposure to 600°C the control concrete shows the crack passing through siliceous aggregate. The breakup of siliceous aggregate takes place around 600°C due to alpha-beta conversion of quartz and sand. The red color is seen in control mix after reaching to 800°C due to melting of sand and quartz aggregate. After reaching to a temperature of 800°C, aggregates start melting and cement paste decomposes which creates voids within the concrete. The increase in voids in the concrete is also confirmed from the results of water absorption test as presented in Figure 5.33. The formation of voids within the concrete weakens the aggregate-cement paste bond and finally concrete loses its strength as presented in Figure 5.30.

In concrete mixes C2 and C4 the cracks and voids are formed at 600°C and 800°C, respectively as shown in Figure 5.34 (b) and (d). The partial dissociation of calcium carbonate starts near about 600°C. After reaching to a temperature of 800°C decomposition of dolomite takes place and turns in to calcium oxide with release of carbon di oxide. Due to calcination process of $CaCO_3$ it gives pale shades of white as clearly seen in Figure 5.34 (b) and (d). The melting of marble aggregate is seen in Figure 5.34 (b) and (d) at 800°C. Overall the concrete containing carbonate aggregate perform better up to 800°C as compared to that concrete containing siliceous aggregate. Better performance of carbonate aggregate is due to higher heat capacity than siliceous aggregate as reported by Kodur (2000).

5.4.6 Ultrasonic Pulse Velocity (UPV)

Variation in Ultrasonic pulse velocity (UPV) values of concrete mixes after exposure to fire can be seen in Figure 5.35.

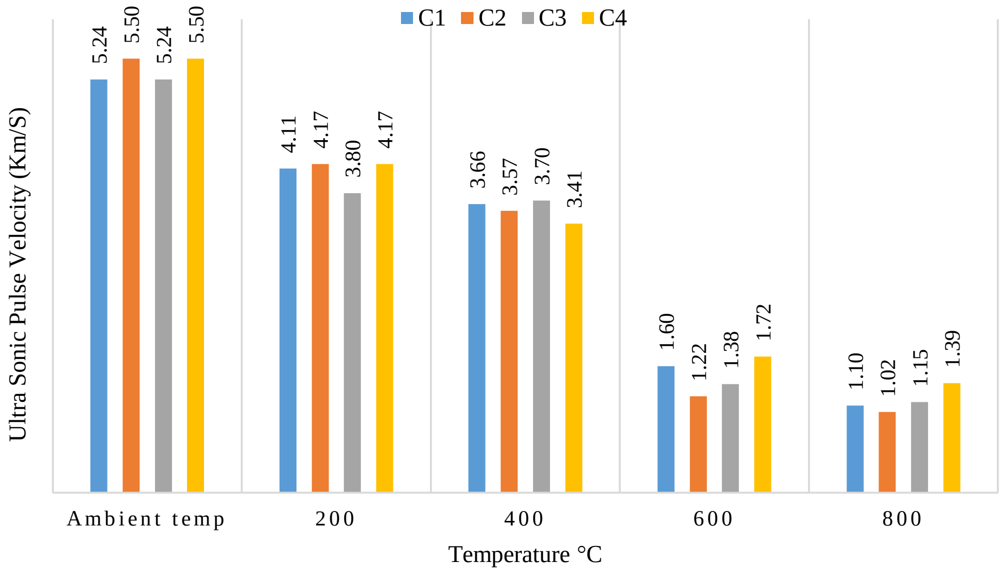


Figure 5.35 Variation in Ultrasonic Pulse velocity of concrete after exposure to fire

The results of the tests show that, UPV of concrete mixes decrease with increase in temperature levels. The reasons for decrease in UPV of all mixes is, increased porosity due to rising temperatures. It can be seen that UPV values at 200ºC fall by one third of that at ambient temperature due to capillary loss of water and ettringite dehydration. A steep fall was again observed from temperature 400ºC to 600ºC. This phenomenon could be due to progressive dehydration of C-S-H gel and phase transformation of aggregates around 600ºC. UPV values of 3.5 Km/sec are good for concrete and all the concrete mixes up to 400ºC have shown satisfactory performance as per BIS 13311 (Part 1):1999 (1999c). All the concrete mixes above 400ºC could not perform up to the standard.

5.4.7 Flexural strength

Flexural strengths of concrete mixes exposed to fire are shown in Figure 5.36.

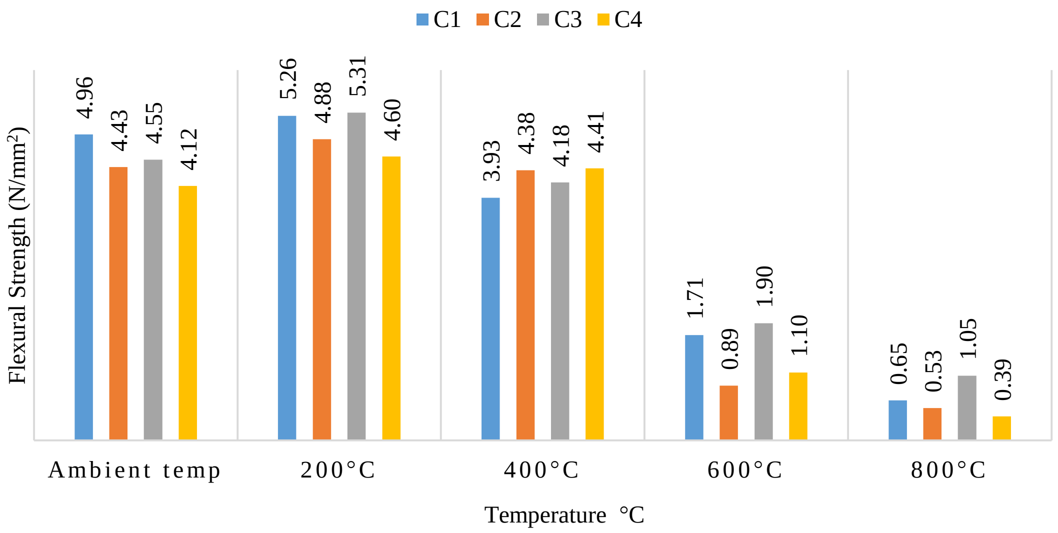
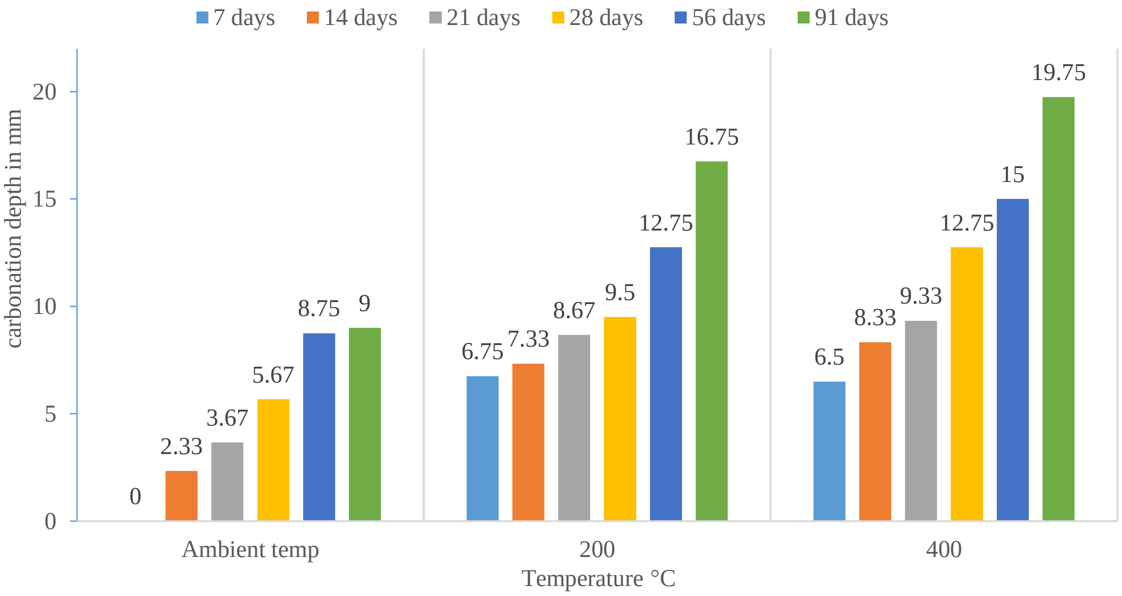


Figure 5.36 Flexural strength of concrete after exposure to fire

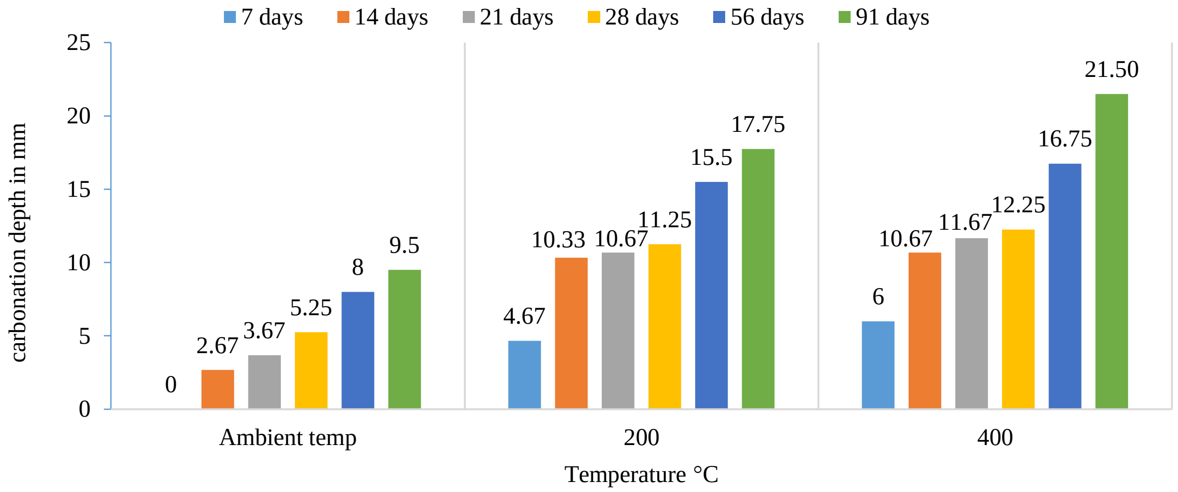
It can be seen from Figure 5.36 that, the flexural strength of all concrete mixes initially increase at a temperature of 200ºC by about 15%, and at 400ºC it remains almost same as that on ambient temperature. This increase in flexural strength might be due to accelerated hydration process and evaporation of free water from the cement paste. At 400ºC when the specimens after heating are cooled in air some of the evaporated water is regained from moisture present in the environment. This phenomenon restricts further fall in strength. This fact was also reported by Husem (2006), Toumi et al. (2009). At 600°C and 800°C temperature the flexural strengths of all concrete mixes drastically dropped. Sudden decrease in flexural strength is due to changes in the physical and chemical properties of cement and aggregates discussed earlier. Thus it can be summed up that up to 400ºC, there is no adverse effect on strength parameters of concrete mixes.

5.4.8 Carbonation

The carbonation test was carried out on the concrete mixes as per CPC: 18- RILEM guidelines. The results of the carbonation test for all the concrete mixes designed by packing density approach at 200ºC and 400ºC exposure conditions are presented in Figure 5.37.



(a) Mix C3



(b) Mix C4

Figure 5.37 Variation in depth of carbonation in concrete mixes

It can be seen from Figure 5.37 that, the depth of carbonation of concrete mixes increases with increase in durations of exposure to fire. At ambient temperature, the results obtained for mix C4 are closer and slightly lower than that of control mix at all exposure durations. The reason for this might be due to lower value of thermal coefficients of marble than that of conventional aggregate quartzite. Also the incorporation of marble aggregate as replacement for conventional coarse aggregate does not affect the resistance to carbonation. As the temperature was increased, the depth of carbonation also increased for all the concrete mixes. After exposure to 200°C and 400°C the depth of carbonation of concrete mix C3 increased by 86% and 119% at 91 days as compared to that of ambient temperature results. On the other hand, the depth of carbonation in mix C4 increased by 87% and 126% at 91 days for above mentioned temperatures as compared to that of ambient temperature specimens. The depth of carbonation mainly depends on porosity in concrete mix. The cause for increase in carbonation depth was increased porosity into the concrete mixes due to higher temperature conditions.

5.4.9 Acid Attack

The durability of concrete structures which are situated in the industrial areas is likely to be affected by acids present in the environment. Impact of acids on concrete mixes has been discussed in subsequent paragraphs.

5.4.9.1 Visual Observation of Acid Attacked concrete specimens

The images of the concrete specimens after exposure to fire followed by acid attack are shown in Figure 5.38

at 28 days at 56 days at 28 days at 56 days

Mix C3 after 200°C Mix C3 after 400°C

at 28 days at 56 days at 28 days at 56 days

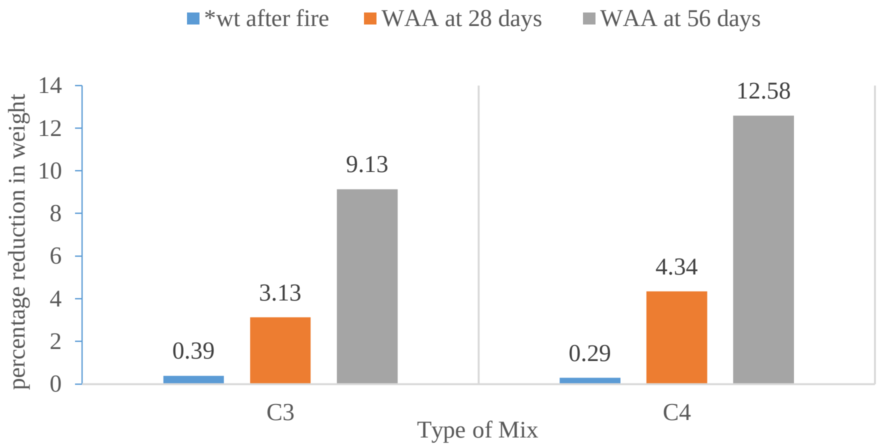
Mix C4 after 200°C Mix C4 after 400°C

Figure 5.38 Images of Acid attacked concrete specimens at different exposure durations after fire

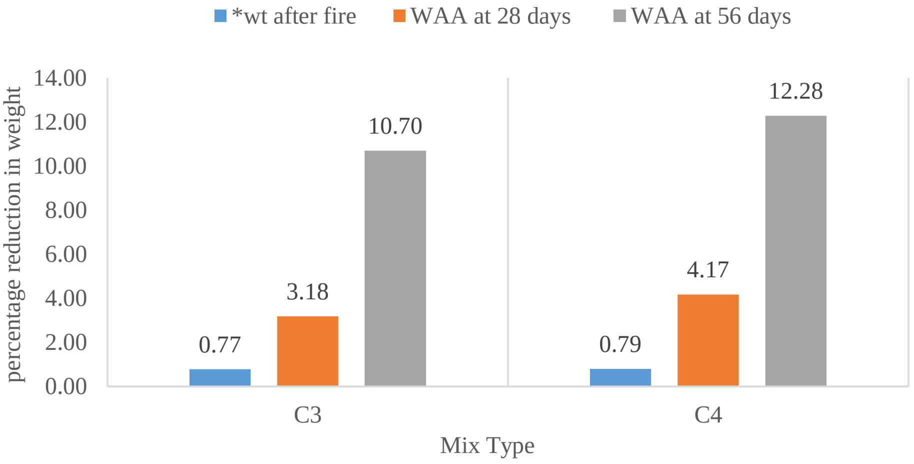
From the Figure 5.38 it is clearly seen that, after exposure to fire at different temperature conditions and increase in duration of concrete specimens the deterioration level increases. After 28 days of exposure the top layer of cement paste vanished. After 56 days of immersion in acid, the concrete specimens showed more loss of cement matrix and white spots of the formation of gypsum are clearly seen on surface of concrete specimens. The deterioration of concrete specimens after exposure to fire at 400°C was more as compared to that at 200°C. This fact was due to increase in pores in the concrete mixes due to higher temperature. The pores formed within the concrete allows the solution to enter deep which results in progressive deterioration of concrete.

5.4.9.2 Loss in weight

The concrete specimens showed loss in mass after immersion in 5% sulphuric acid. The loss in mass of concrete specimens was observed and shown in Figure 5.39.



(a) at 200°C



(b) at 400°C

Note: *- Without acid attack, WAA- With Acid Attack

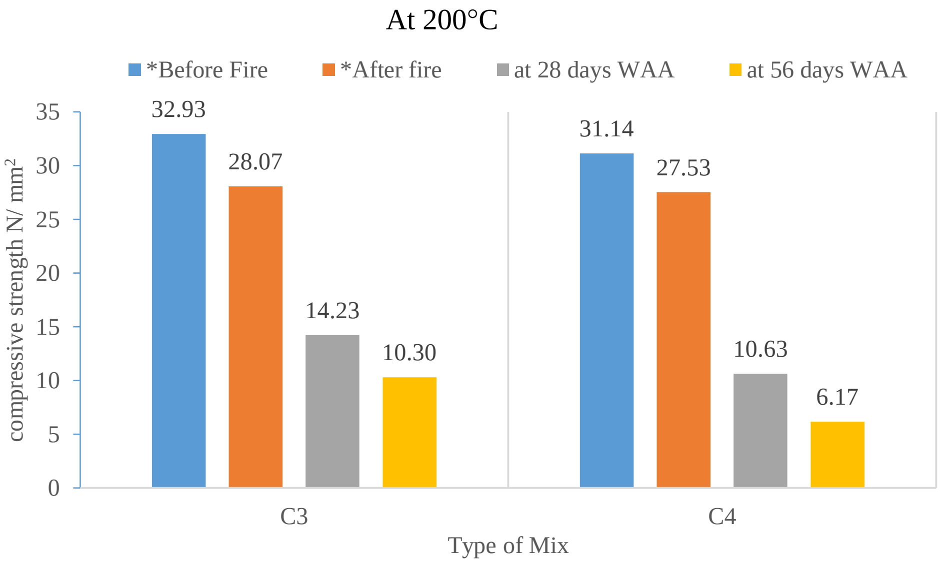
Figure 5.39 Variation in weight of concrete specimens after exposure to fire at different exposure durations

The results shown in Figure 5.39 showed that, the percentage loss in mass of concrete specimens after exposure to fire and then immersed in 5% sulphuric acid solution increased with increase in exposure durations. At 200°C and after 56 days of immersion in sulphuric acid solution, the concrete mix C4 showed 38% more loss in mass as compared to that of control mix. The more or less same trend was observed in mix C4 at 400°C exposure. This increased loss in mass in concrete mix C4 was due to excessive erosion of cement paste from concrete and formation of gypsum during reaction with sulphuric acid. The detailed chemical reaction is shown below.

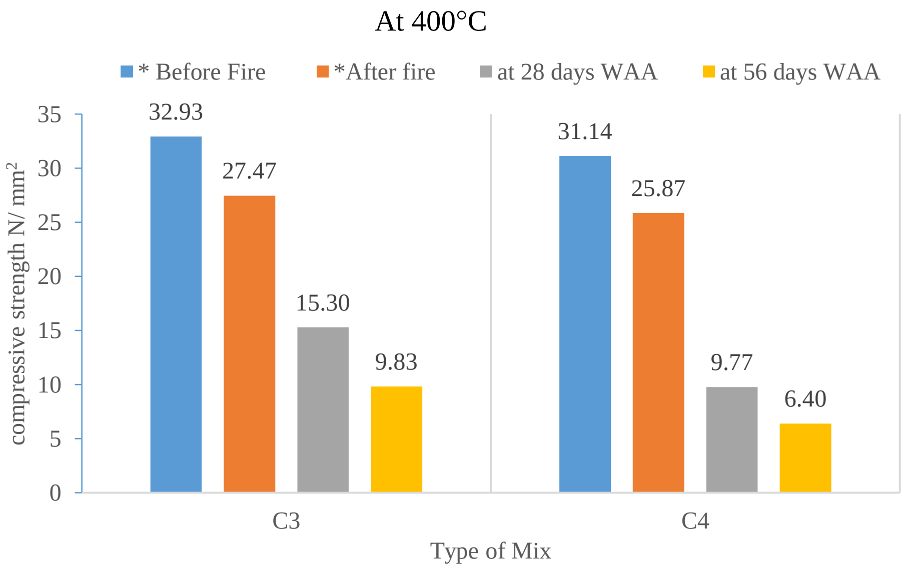
$$\begin{aligned}
&Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4.2H_2O \\
&CaSiO_2 \ . \ 2H_2O + H_2SO_4 \rightarrow CaSO_4 + Si\ (OH)_4 + H_2O \\
&3CaO.\ Al_2O_3\ .12H_2O + 3\ (CaSO_4\ .\ 2H_2O) + 14H_2O \rightarrow 3CaO.\ Al_2O_3 .\ 3CaSO_4 .\ 32H_2O
\end{aligned} \tag{5.4}$$

5.4.9.3 Compressive strength

The variation in compressive strength of concrete exposed to fire followed by acid attack was studied. The results of the compressive strength were compared with the specimens which were not affected by the acid attack and not exposed to fire. The results of the compressive strength test after acid attack are shown in Figure 5.40.



(a) at 200°C



(a) at 400°C

Note: *- Without acid attack, WAA- With Acid Attack

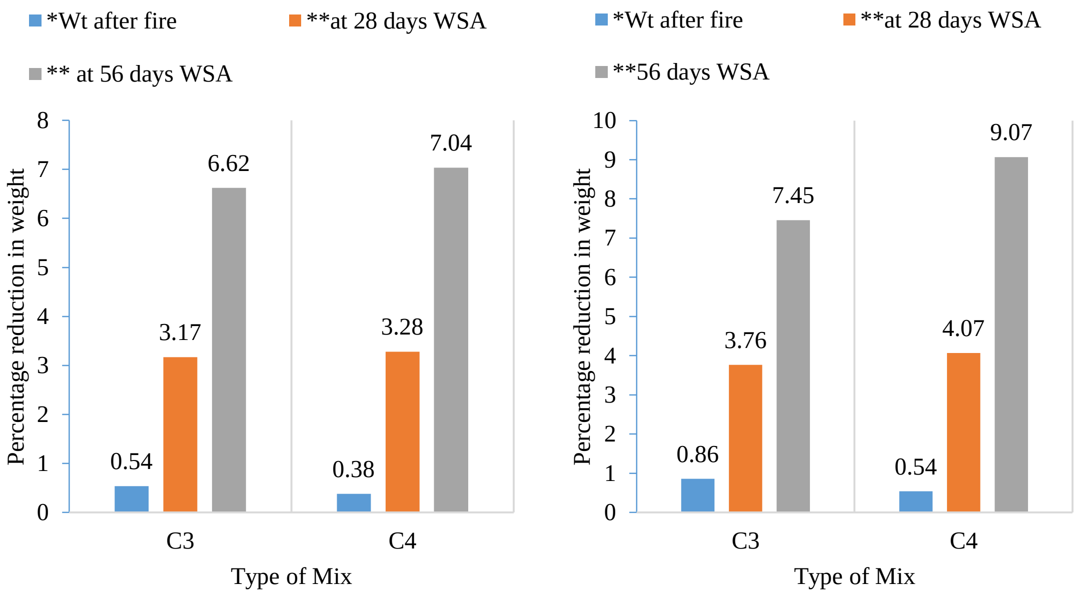
Figure 5.40 Variation in compressive strength of concrete mixes at different exposure durations

It can be seen that, simple exposure of concrete to standard fire up to 400ºC of test specimen designed by packing density method, loose compressive strength marginally. When such concrete is exposed to 5% sulphuric acid solution for 28 days, then appreciable loss in compressive strength (approximately 50%) can be noticed due to appreciable increase in porosity and formation of gypsum. Extended contact with acid causes gypsum to react with calcium aluminate leading to formation of errtingite. Excessive formation of ettringite is detrimental to compressive strength of concrete. Comparison of results of C3 and C4 mixes after 28 days of contact with acid solution shows that approximately 33% more loss in strength of C4 mixes took place in comparison to C3 mixes. This was probably due to lack of finer fractions in marble aggregate resulting in more percentages of voids. Such voids could give access to acid to attack CSH gel resulting in excessive formation of gypsum and ettringite ultimately leading to loss of strength. Overall it can be concluded that concrete mixes designed by packing density approach, when exposed to fire and followed by 5% sulphuric acid do not yield intended results.

5.4.10 Sulphate Attack

5.4.10.1 Loss in Mass

The changes in the mass of concrete specimens after exposure to fire and immersed in 10% magnesium sulphate solution over a period of 28 and 56 days are shown in Figure 5.41.



(a) at 200°C (b) at 400°C

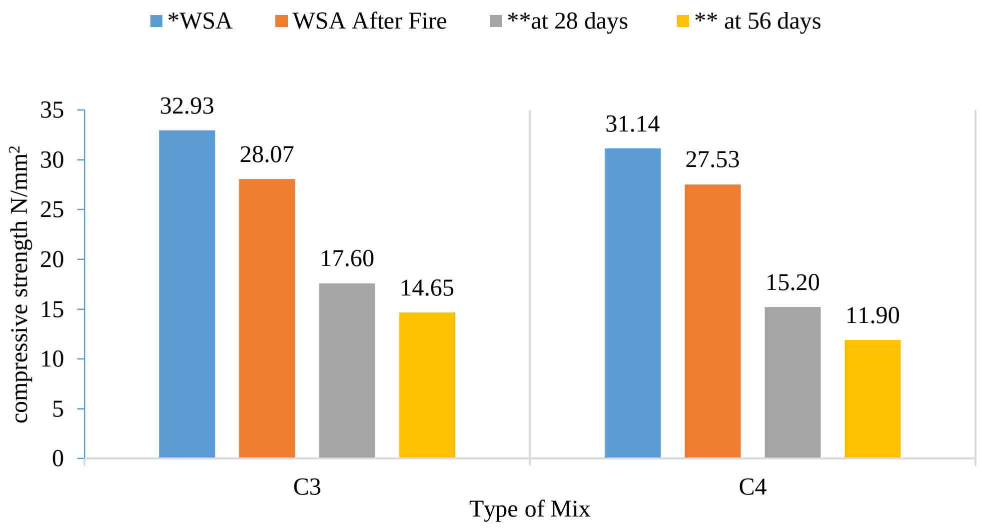
Note: *- Without sulphate attack, **-after fire, WSA- With Sulphate Attack

Figure 5.41 Variation in weight of concrete specimens after exposure to fire at different exposure durations

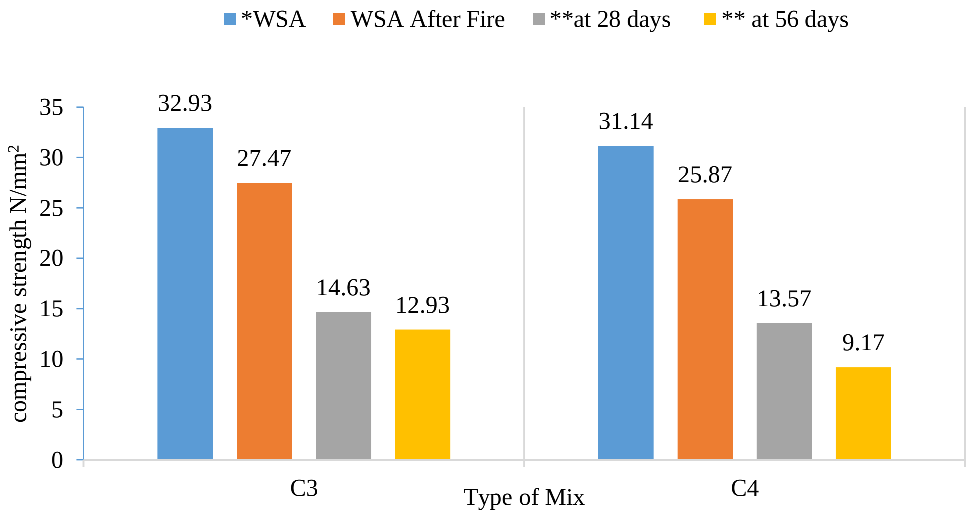
From the Figure 5.41 it can be seen that, the percentage loss in mass of concrete mixes increased with increase in exposure duration. At 200°C and after 56 days of immersion in 10% magnesium sulphate solution, the loss in mass of concrete mixes C3 and C4 increased by 92% and 95%, and at 400°C the loss in mass of concrete mixes C3 and C4 increased by 88% and 94% as compared to that of control samples. Due to high temperature conditions, aggregate and cement paste experiences a change in physical and chemical reactions which result in formation of voids in concrete. Excessive formation of ettringite product due to action of magnesium sulphate with the cement paste can be held responsible for significant loss in mass.

5.4.10.2 Compressive strength

The variation in compressive strength of concrete specimens after exposure to fire followed by immersion in 10% magnesium sulphate solution is given in Figure 5.42.



(a) at 200°C



(b) at 400°C

Note: *WSA- Without sulphate attack, **-after fire, WSA- With Sulphate Attack

Figure 5.42 Variation in compressive strength of concrete mixes at different exposure durations

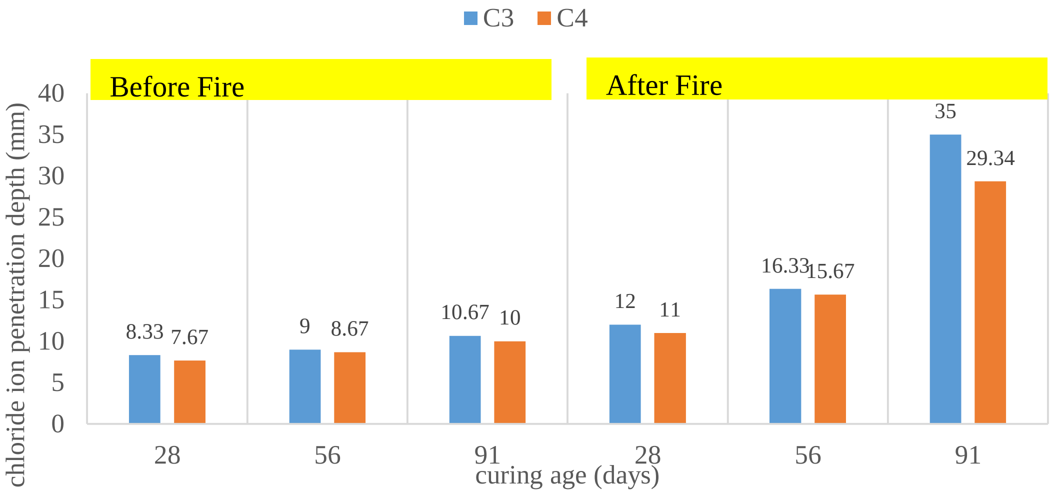
It can be seen from Figure 5.42 that, compressive strength of all the concrete mixes decreased with increase in exposure durations. The compressive strength of concrete mix C3 decreased by 47% and 56% while that of mix C4 decreased by 51% and 62%, respectively, at 56 days of immersion in magnesium sulpahte solution after exposure to 200°C temperature. On the other hand, the compressive strength of concrete mix C3 decreased by 56% and 61% while that of mix C4 decreased by 57% and 71%, respectively at 56 days of immersion in magnesium sulpahte solution after exposure to 400°C temperature. These data show that fall in compressive strength follow the same trend as seen in acid exposure condition.

It may be noted that the compressive strength of all the test specimens exposed to sulpahtes without fire exposure only increased marginally up to 91 days exposure as seen in Figure 5.21. This was because of filling of pores by ettringite. Later on extended exposure duration the excessive ettringite bursts the concrete structure leading to reduction in compressive strength.

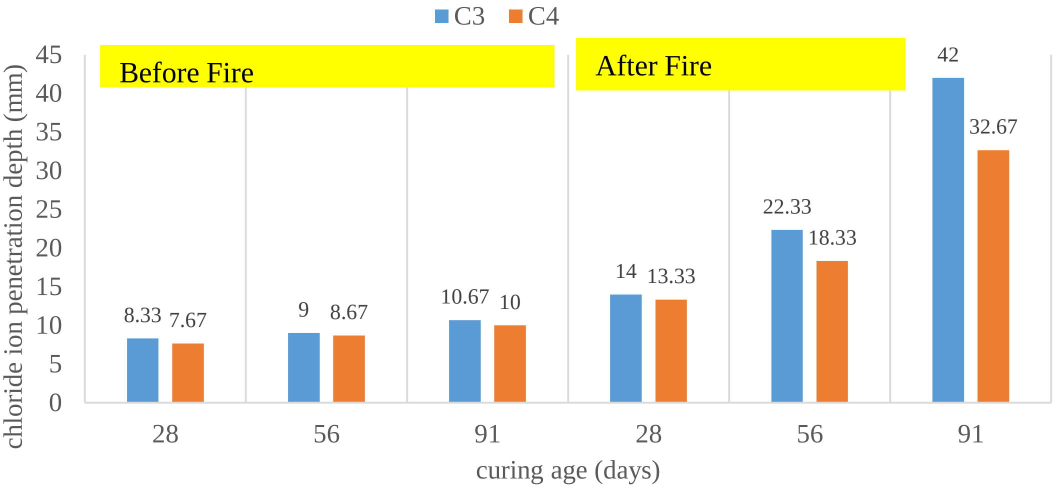
In the present circumstances, the excessive formation of voids due to fire exposure does not allow increase in compressive strength. On the contrary the compressive strength comes down since very beginning.

5.4.11 Chloride Ion Penetration

The variation in the results of depth of chloride ion penetration after exposure to fire at different temperatures and immersed in sodium chloride solution are shown in Figure 5.43.



(a) at 200°C



(b) at 400°C

Figure 5.43 Variation in depth of chloride ion penetration of concrete mixes

From the results it can be seen that, both the concrete mixes show an increase in chloride ion penetration depth with increase in fire exposure durations at different temperatures. After exposure to 200°C temperature, the chloride ion penetration depth of concrete mix C3 increased by 44%, 81% and 228% whereas in mix C4 it increased by 43%, 81% and 193% at 28 days, 56 days and 91 days respectively, as compared to that of ambient temperature samples. After exposure to 400°C, the chloride penetration depth of concrete mix C3 increased by 68%, 148% and 294% whereas mix C4 increased by 74%, 111% and 227% respectively, as compared to that of ambient temperature samples. The depth of chloride penetration of concrete mainly depends on the porosity of the concrete. The basic reason behind the increased chloride penetration depth was increased porosity in the concrete mixes due to higher temperature conditions when exposed to fire. The concrete containing marble aggregate show little bit better performance due to presence of Al_2O_3 in the marble aggregates. The presence of alumina favors the formation of tricalcium aluminate (C_3A), which fixes the chloride ions and forms insoluble compounds. When an aqueous solution of silver nitrate ($AgNO_3$) is added to the aqueous solution of sodium chloride (NaCl), a white precipitate of silver chloride (AgCl) is formed that is indicated by the following chemical reaction.

$$AgNO_3 + NaCl = AgCl + NaNO_3 \tag{5.4}$$

6 Conclusions

A comprehensive study on use of marble waste as coarse aggregate in concrete mixes has been carried out first time. Especially exposure of such concrete mixes in acid environment and in fire has not yet been reported prior to this study. Results of concrete mixes prepared with packing density approach using marble waste and control mixes designed as per BIS: 10262-2009 have been compared in successive paragraphs.

- Incorporation of marble waste as coarse aggregate improves the workability of concrete mixes.
- The compressive strength of concrete mixes is not affected by use of marble waste as coarse aggregate. The results are nearly close to that of control concrete.
- The permeability is reduced by 15% as compared to that of control concrete. The reduced permeability is an indication of enhanced durability properties of concrete.
- Use of marble waste as coarse aggregate in concrete mixes increases the ultra-sonic pulse velocity value by approximately 19% which indicates that quality of concrete in terms of density and homogeneity is better as compared to that of control mixes.
- The resistance to abrasion of concrete produced using marble aggregate improved by 21% as compared to control mixes. It indicates that, marble waste can be used as coarse aggregate in pavement construction.

- The carbonation depth of concrete containing marble aggregate shows nearly same trend as that of control concrete.

- Reduction in the depth of chloride ion penetration was approximately 29% as compared to control mixes.

- Compressive strength of concrete in 5% sulphuric acid solution for 28 days was found to be only 5% lower than that of control concrete specimens. Such insignificant loss in compressive strength proves that marble waste can be used as coarse aggregate in concrete mixes without much botheration.

- Compressive strength of concrete exposed to 10% magnesium sulphate solution shows loss in strength in long term (beyond 91 days exposure) in all the mixes. Reduction in compressive strength of concrete with marble waste was less as compared to control concrete. It shows no adverse effect of use of marble waste in concrete.

- After exposure to standard fire, the compressive strength of all concrete mixes maintain their characteristic strength of M25 grade concrete till 400°C. Exposure beyond 400°C shows significant loss in compressive strength ofall the mixes.

- The results of flexural strength after exposure to fire are on the same line as that of compressive strength.

- The water absorption of concrete mixes containing marble waste at all the temperatures was less than that of control mixes. Appreciable difference was noted at 600ºC and 800ºC.

- The ultrasonic pulse velocity showed that, the porosity in concrete mixes increased with increase in temperatures due to evaporation of water, decomposition of calcium hydroxide and dissociation of calcium carbonate in to CaO and CO_2. All the concrete mixes fall under Good concrete quality grading up to a temperature of 400°C as per BIS: 13311: part-1, 1992.

- Concrete mixes with marble waste exposed to fire up to 400°C followed by acid attack loose approximately 15% more compressive strength as compared to control mixes.

- At temperatures 200°C and 400°C the durability properties such as carbonation and chloride penetration of concrete containing marble waste show nearly same trend as that of control concrete mixes.

From the above study it can be concluded that, the use of aggregate produced from marble waste can be used as replacement of conventional coarse aggregate in concrete mixes. The increase in resistance against abrasion and significant reduction in depth of chloride penetration are added benefits ensuring enhanced life of concrete structure without sacrificing strength.

Quantity and cost estimates of concrete made with marble waste as coarse aggregate have been worked out first time. The quantity estimates of materials show that 24% cement is saved using packing density approach of design. Thus making concrete overall cost effective and reduced consumption of cement also results in 24% less emission of CO_2 in the environment. Thus the product becomes sustainable with saving in 14% overall cost of concrete designed by packing density approach. The results obtained in this study clearly indicates that, the optimum percentage for replacement of conventional coarse aggregate by marble aggregate is 75 %.

At higher temperature concrete produced from marble waste does not have any significant detrimental effect on properties of concrete as compared to control concrete.

Recommendations for Future Work

- A study can be carried out on high strength concrete mixes with different water-cement ratios.
- The long term durability properties of concrete such as bond with reinforcement, shrinkage, corrosion and freeze-thaw can be carried out on concrete mixes using marble waste as coarse aggregate.

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List of Publications

International Journals:

1. **S.D. Kore**, A.K. Vyas, Impact of Marble Waste as Coarse Aggregate on properties of lean cement concrete, Case Studies in Construction Materials 4 (2016) 85–92. doi:10.1016/j.cscm.2016.01.002
2. **S.D. Kore**, A.K. Vyas, Cost Effective Design of Sustainable Concrete Using Marble Waste as Coarse Aggregate, Journal of Materials and Engineering structures, Vol 3, No 4 (2016) (Research Review of Science and Technology) (ESCI)
3. **S.D. Kore**, A.K. Vyas. Durability of concrete using marble mining waste, Journal of Building Materials and Structures, [S.l.], v. 3, n. 2, p. 55-67, dec. 2016. ISSN 2353-0057. Available at: <http://journals.oasis-pubs.com/index.php/jbms/article/view/25>. Date accessed: 12 Mar. 2017. doi:10.5281/zenodo.242638.
4. **S. D. Kore**, A.K. Vyas, Performance Evaluation of Concrete using Marble Mining Waste, SSP - Journal of Civil Engineering, Vol. 11, Issue 2, 2016, DOI: 10.1515/sspjce-2016-0018
5. **S.D. Kore**, A.K. Vyas, Impact of Fire on Mechanical Properties of Concrete Containing Marble Waste, Journal of King Saud University Engineering Sciences
6. **Kore S.D**. and Vyas A. K., Particle Packing Density Approach for Design of Concrete Mixes Using Marble Waste, Journal of Civil Engineering and Urbanism, 2016-6 (4): 78-83. Journal homepage: www.ojceu.ir
7. **S. D. Kore**, Hasan sahan arel, Ageing Management and Life Extension of Concrete in Nuclear Power Plants, Powder Technology, Volume 321, November 2017, Pages 390-408, https://doi.org/10.1016/j.powtec.2017.08.053 (SCI)
8. **S.D. Kore**, A.K. Vyas, Packing Density Approach for Sustainable Development of Concrete, Slovak Journal of Civil Engineering Journal of Materials and Engineering structures, (Research Review of Science and Technology) (ESCI) [Accepted]

9. **S. D. Kore**, A.K. Vyas, Syed Ahmed Kabeer K.I., A Brief Review on Sustainable Utilization of Marble Waste in Concrete Mixes, International Journal of Sustainable Built Environment (Elsevier) [Under Review]
10. **S. D. Kore**, A.K. Vyas, Syed Ahmed Kabeer K.I., "Influence on properties of concrete with ISF slag and marble waste as fine aggregate" Engineers Australia Technical Journals Taylor and Fransis [Under Review]
11. **S.D. Kore**, A.K. Vyas, Hasan sahan arel, Evaluation of mechanical properties of concrete for its sustainable development using Packing Density Approach, Environment, Development and Sustainability (Springer) [Under Review]
12. **S.D. Kore**, A.K. Vyas, "Use of marble waste and ISF slag for production of Environmental friendly durable concrete", Environmental Processes (Springer) [Under Review]

International Conferences:

1. **S.D. Kore**, A.K. Vyas, Behavior of Concrete Using Marble Waste as Coarse Aggregate, UKIERI Concrete Congress –Concrete Research Driving Profit And Sustainability, Nov. 2015, NIT Jalandhar (Punjab) India.
2. **S.D. Kore**, A.K. Vyas, Packing Density Approach for Production of Cost Effective and Durable Concrete, International Conference on Advances in Concrete Technology Materials & Construction Practices (CTMC-2016), Goa Engineering College, Farmagudi – Goa, India
3. **S.D. Kore**, A.K. Vyas, Influence of Particle Packing Density Approach on Strength and Durability properties of Concrete Containing Marble Waste, International Conference on Trends and Recent Advances in Civil Engineering – TRACE 2016, Amity University Noida, Uttar Pradesh, India

Annexure I

Detailed mix design procedure of concrete by Packing Density Approach

To design the concrete mix by packing density approach, the bulk density, packing density and void content in the all in aggregate mixture is required. These values can be determined from the laboratory trials by mixing and combining the different size of aggregates. The detailed concrete mix design calculations and steps are given below.

Table 1 Physical Properties of Aggregates

Aggregate Type	Specific gravity	Water Absorption (%) by weight	Grading Zone
Conventional Coarse aggregate	2.78	0.54	As per Table 2 of BIS 383
Fine Aggregate	2.66	2.0	Zone II As per Table 4 of BIS 383
Coarse Marble Aggregate	2.88	0.05	As per Table 2 of BIS 383

1. Determination of bulk density of combined coarse aggregate 20 mm, coarse aggregate 10 mm and fine aggregate i.e. all in aggregate.

$$\text{Bulk Density} = \frac{\text{W2 - W1}}{\text{volume of mould}}$$

Where,

W1= weight of empty mold

W2= weight of mold filled with aggregate

$$\text{Bulk Density} = \frac{17.420 - 9.200}{3976.07} = 2.067\ \text{gm / cm}^3$$

2. Determination of Void content of all in aggregates

$$\text{Void content} = \frac{\text{specific gravity of aggregate - bulk density of aggregate}}{\text{specific gravity of aggregate}}$$

$$\text{Void content} = \frac{2.74 - 2.067}{2.74} \times 100$$

Void content = 24.55%

3. Determination of Packing density of aggregates

$$\text{Packing Density} = \frac{\text{bulk density} \times \text{weight fraction of aggregate}}{\text{specific gravity of aggregate}}$$

$$\text{Packing density of 20 mm aggregate} = \frac{2.067}{2.78} \times 0.36 = 0.2677$$

$$\text{Packing density of 10 mm aggregate} = \frac{2.067}{2.78} \times 0.24 = 0.1785$$

$$\text{Packing density of fine aggregate} = \frac{2.067}{2.66} \times 0.40 = 0.3109$$

Total packing density of all in aggregate = 0.2677 + 0.1785 + 0.3109

= 0.7571 gm/cm^3

This value of packing density is used for further calculations.

4. Determination of paste content

The paste content is at least equal to the void content and paste content in excess of void content is utilized to coat the aggregate particles and give sufficient flowabilty with desired strength.

Void content = 1- packing density =1 - 0.7571 = 0.2429

Selecting the paste content 10% in excess of void content (refer methodology)

Paste content = 0.2429 + (0.1× 0.2429)

= 0.2672

Volume of aggregate = 1- 0.2672

= 0.7328 cc

Total solid volume of aggregate =

$$\frac{\text{weight fraction of 20 mm aggregate}}{\text{specific gravity of 20 mm aggregate}} + \frac{\text{weight fraction of 10 mm aggregate}}{\text{specific gravity of 10 mm aggregate}} + \frac{\text{weight fraction of fine aggregate}}{\text{specific gravity of fine aggregate}}$$

Total solid volume of aggregate

$$= \frac{0.36}{2.78} + \frac{0.24}{2.78} + \frac{0.40}{2.66}$$

= 0.3662 cc

Weight of 20 mm aggregate = $\frac{0.7328}{0.3662} \times 0.36 \times 1000 = 720.39$ Kg/ cum

Weight of 10 mm aggregate = $\frac{0.7328}{0.3662} \times 0.24 \times 1000 = 480.26$ Kg/ cum

Weight of fine aggregate = $\frac{0.7328}{0.3662} \times 0..40 \times 1000 = 800.44$ Kg/ cum

Water-cement ratio is 0.45

W/C = 0.45

W = 0.45 × C

Total paste = C + W = $\frac{C}{3.15}+\frac{0.45}{1}$

Total paste = 0.7675 × C

Cement content = $\frac{\text{paste content}}{\text{Total paste}}$

= $\frac{0.2672}{0.7675}\times 1000$

= 348.16 Kg / cum

Water content = 0.45× C

= 0.45 ×348.46

= 156.67 Kg / cum

Total quantity of materials for one cubic meter concrete mix for water-cement ratio 0.45 is as follows,

Water	= 156.67 Kg / cum
Cement	= 348.16 Kg / cum
Fine aggregate	= 800.44 Kg / cum
Coarse aggregate	
10 mm	= 480.26 Kg / cum
20 mm	= 720.39 Kg / cum

Above mentioned quantities were taken to design the mixes by packing density approach.

Vitae of the Author

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