Journal of Materials and Engineering Structures

Research Paper

Mechanical and Durability Characteristics of High Performance Concrete Using Copper Slag as Fine Aggregate

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ARTICLE INFO

Article history :

Received : 27 December 2019

Revised : 1 June 2020

Accepted : 2 June 2020

Keywords:

High Performance Concrete

Copper Slag

Mechanical

Durability

Fine Aggregate

ABSTRACT

This paper reports the results of an experimental study on the high performance concrete made with copper slag as fine aggregate. The percentage of Copper Slag(CS) added by weight in a range viz. 0, 25, 50, 75 and 100% as a replacement of sand used in concrete and cement was replaced with 15% Metakaolin. The properties studied include compressive strength, splitting tensile strength, Sorptivity, Rapid Chloride Permeability Test (RCPT), Accelerated Carbonation test and Microstructural properties. The test results showed that the compressive strength increases up to 50% copper slag as replacement of sand, beyond which decrease in strength was observed. The results of RCPT and sorptivity with different proportions of copper slag at 28, 90, 120 days of curing period showed the lowest value for the mix containing 25% and 50 % copper slag at each curing age. Carbonation results show that concrete mixes with 85% cement and MK 15% with increasing percentage of copper slag, the carbonation depth decreases slowly especially for 75% and 100%. The microscopic view from Scanning electron microscopy (SEM) demonstrated more voids, capillary channels, and micro cracks with the increment of copper slag as substitution of sand as compared to the control mix, profoundly visible at 100% replacement of sand which is due to the presence of free water.

1 Introduction

Concrete has the leading position amongst the construction materials due to its remarkable versatility, the ease of construction and certain durability properties [1]. Besides cement and water, aggregates occupy nearly 80% of the concrete volume remaining the main constituent materials in concrete. Both coarse aggregates [with particle size > 4.75mm] and fine aggregates [with particle size < 4.75mm] are obtained either from natural resources or by crushing of large rocks [2]. Natural resources are depleting at a very faster pace worldwide whereas at the same time, the industrial wastes generated are also

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e-ISSN: 2170-127X,

RESEARCH REVIEW of Sciences and Technologies

increasing rapidly affecting the environment posing health hazards and creating the problem of its dumping simultaneously [3]. Disposal of this large volume of industrial waste appears to be the need of an hour. The idea behind the sustainable development is to utilise existing resources optimally to satisfy the future demands with ease [1]. Large quantities of such industrial wastes called the by-products are being produced by various industries every year viz. Bottom ash [4-8], iron ore tailings [9], imperial smelting furnace slag [10], recycled glass [11] and many more, which can be effectively prove to be a good substitute of natural fine aggregate. Apart from by-products like Fly ash(FA),Silica fume(SF), Ground-granulated blast-furnace slag(GGBS), many new by products are being generated having promising future for replacing cement, fine aggregates and coarse aggregates [12].

Copper slag (CS) is such one of the by-product obtained from Copper industry during matte smelting and refining of copper. Researches [13, 14] reviewed that the copper slag is effectively used in the production of value added products like abrasive materials and tools, tiles, glass, cutting tools and roofing granules. It is also reported that copper slag can be effectively used as a partial substitute for cement and aggregates in concrete and asphalt mixers. Copper slag due to its coarser size as compared to cement cannot be used directly as cement replacement since it has to be grounded to lessen the size as required by ASTM C1709-18. Consequently, CS as a replacement of cement is not being very prevalent in concrete production as compared to its utilization as a substitute for fine aggregate. Approximately, three times copper slag is generating for every ton of copper production. Iran, Brazil, Japan and US produced approximately 0.36, 0.244, 2.0 and 4.0 million tons of copper slag respectively every year [15].Chile, a south American country is the largest producer of copper slag according to the latest studies[16]. The present researches showed consumption of nearly 40 million tons of CS all around the world and if used properly, it can be a great alternative substitute for natural aggregate satisfying the demands of natural aggregates [17-19].

High performance concrete is a concrete, which is designed to give best mechanical and durability properties as compared to conventional cement concrete(CCC). HPC with cement as a binder alone lead to large evolution of heat of hydration, excessive shrinkage and more cost. Introduction of mineral admixtures viz. silica fume, Metakaolin, GGBS, fly-ash as a partial replacement of the cement not only increases the durability of the concrete, but also resulted in additional benefits in terms of cost reduction, saving energy and conserving natural resources [20-23].Metakaolin is an important admixture for concrete applications that enhance the performance of cementitious composites because of its high pozzolanic reactivity. Comparing it with silica fume, MK give comparable properties at a lower price and with better workability [24]. MK's obtained from poor Greek kaolins has a tremendous effect on strength after 2 days specifically at 28 & 180 days. Addition of the MK to the concrete accelerated its pozzolanic reaction between 7 and 28 days with steep decrease of calcium hydroxide [25]. MK leads to significant effects on the pore structure of the concrete making it more impermeable and resistant towards the ingress of harmful ions [26]. HPC with the substitution of CS for fine aggregates has been studied by various authors with different % variations of CS substitution. Overall improvement in the mechanical and durability properties of HPC is observed for 40-60% substitution of CS[1-3]. The replacement of 40% unground CS was found to be optimum to get best strengthening and durability results for Ultra high strength concrete. The incorporation of 60% ground copper slag of size less than 600 microns in the place of quartz sand even proved superior to get compressive, flexural and splitting tensile strengths in the range of 235MPa,47 MPa and 31MPa respectively [27]. In another study, copper slag admixed steel fibre concrete with 45% and 40%CS replacement has the best compressive and flexural strengths after 28 days comparing it with plain steel fibre concrete [28]. Recent study on HPC with 55% replacement of CS with fine sand showed 20%-30% increase in the compressive, splitting tensile and flexural strengths as compared to conventional concretes [29]. Another study elucidates that 60% replacement of CS with low w/b ratio proved to be optimum for maximum compressive, splitting tensile and flexural strengths [30]. The use of CS has also proved beneficial by accommodating natural aggregate with industrial waste, reducing cost of concrete, solving the problem of dumping and taking care of environment by minimizing environmental hazards.

2 Research significance

Although, various studies have been done on the use of copper slag in the production of concrete during the past years. There are very few researches done on high performance concrete incorporating copper slag in different percentages along with the mineral admixtures. Further, little or no study has been reported on the durability aspects such as carbonation effects. Therefore, beside mechanical properties such as workability, compressive strength, tensile strength and durability properties namely capillary suction test, rapid chloride penetration test and accelerated carbonation are carried out by in this present experimental work. Microstructure of the concrete is also studied by SEM (Scanning Electron Microscope) and EDS (Energy-dispersive X-ray Spectroscopy) checking the effect of copper slag substitution as sand.

3 Materials

3.1 Cement

The cement used was OPC (53 grade) conforming to BIS 12269-2013[31] purchased from Ultratech cement Ltd., Jalandhar (Pb), India.

3.2 Fine Aggregates

Locally available coarse sand confirming to zone II as per IS 383-1970[32] has been used for experimental work. The physical properties of fine aggregate like fineness modulus and specific gravity was 2.79 and 2.6 respectively.

3.3 Coarse Aggregates

Coarse aggregate of different sizes 20mm, 12.5mm and 10mm, locally available crushed aggregates were used, having fineness modulus IS 2386 - PART III 1963[33]of 2.64 and specific gravity IS 2386 - PART III 1963[33]of 6.93.

3.4 Copper slag

Copper slag is a by-product material obtained from the copper manufacturing industry. Copper slag used in this work brought from Taj Abrasives Industries located in Sikar, Rajasthan.

3.5 Metakaolin

Metakaolin is a De-hydroxylated form of clay mineral kaolinite. Metakaolin used in the work was from METACEM 85C (industrial name) supplied by M/s 20 microns Ltd, Vadodara, India.

3.6 Superplasticizer

Superplasticizer Fosroc AURAMIX 200 used in the work is a unique combination of the latest generation super plasticizers based on special polymers. AURAMIX 200 complies with IS 9103[34], BS: 5075[35] & ASTM C-494[36] Type G. It allows the production of HPC with high workability retention.

4 Physical and chemical properties of materials

Chemical compositions of OPC, FA and MK are presented in Table 1. It can be observed from the Table 2 that the contribution of limes (free & combined) is around 63% in the case of OPC as compared to 6% in copper slag 1.1% in fine aggregate and 0.25% in Metakaolin. Less percentage of lime indicates that these materials are not very reactive to be used as cementitious materials since required rate of hydration, high early strength need high quantity of lime [2].

High concentrations of Silica, alumina & Iron oxide in copper slag, fine aggregate &Metakaolin exceeding 70% indicated good capability of being high quality pozzolans.

Specific gravity & water absorption tests of copper slag and fine aggregate revealed the results that the fine aggregate has specific gravity of 2.6which is lower than that of copper slag (3.51), whereas the water absorption values for fine aggregate and copper slag were about 0.8% and 0.36% respectively.

These physical results suggest that the copper slag admixed concrete would have larger density values as compared to concrete with fine aggregate only. On the other hand, free water content will rise with the increase of copper slag in concrete due to its low water absorption property. This will increase the workability of concrete blends with high percentages of copper slag.

Table 1- Chemical properties of OPC, Fine aggregate, Copper Slag and Metakaolin(in %)

Components	OPC	Sand	Copper slag	Metakaolin
SiO_2	20.99	57.6	30.53	59.1
Al_2O_3	5.98	30.5	2.80	38.1
Fe_2O_3	4.10	3.72	57.82	1.25
CaO	60.78	1.10	1.60	0.25
MgO	0.96	0.38	1.48	-
SO_3	2.86	0.22	1.59	0.10
K_2O	1.18	1.35	-	0.07
Na_2O	0.86	0.10	-	0.06

5 Experimental program

5.1 Sample Preparation

Cement concrete cubes of size (150mm x 150mm x 150mm) were cast for each mix with a fixed w/c ratio of 0.23. For compressive strength and tensile test three samples for each mix were tested for 7, 28, 90 and 120 days of curing. Three 100Φ x 200mm long cylinders were prepared for each mix in order to perform Capillary Suction Test (CST) and Rapid Chloride Penetration Test (RCPT). Three discs of size 100mm x 50mm thick were obtained from each cylinder for conducting tests after 28, 90 & 120 days. Similarly, three discs were obtained from each cylinder for conducting RCPT tests after 28, 90 & 120 days of curing. Ten concrete prisms of size 100mm x 50mm were split into lengths from a full beam of size 100mm x 500mm for determining the accelerated carbonation.

5.2 Testing Procedure

After curing, the following tests were carried out on the cement concrete specimens.

- Compressive strength and tensile strength tests on cement concrete cubes was conducted on 200t capacity compressive testing machine as per IS standards guidelines IS 516[37] at 7, 28, 60 and 120 days of curing period.
- Capillary suction test was conducted in accordance with ASTM C-1585- 04[38] at 28, 90 and 120 days of curing period.
- RCPT test was conducted in accordance with ASTM C1202:1991[39] at 28, 90 and 120 days of curing period.
- Accelerated carbonation test was conducted on concrete specimen in accordance with CPC-18[40], RILEM: 1988[41] after the exposure of CO2 given for 4, 8, 12 and 16 weeks after 28, 90 and 120 days of curing period.

Table 2- Mix Proportion and w/c ratio for high performance concrete

Mix proportions (kg/m^3)							
Cement	**Fine aggregate**	**20 mm**	**12.5 mm**	**10 mm**	**Water (1)**	**w/c ratio**	**SP (per kg of cement)**
545	650	456	194	500	125.35	0.23	1.5%

Table 3- Proportions of concrete mixes prepared

Mix	Cement (kg/m^3)	Metakaolin (kg/m^3)	Fine aggregate (kg/m^3)	Copper slag (kg/m^3)	Coarse Aggregate (kg/m^3)		
					20 mm	12.5 mm	10 mm
Control	545	0	650	0	456	194	500
R 17	463.25	81.75	650	0	456	194	500
R18	463.25	81.75	487.5	162.5	456	194	500
R19	463.25	81.75	325	325	456	194	500
R20	463.25	81.75	162.5	487.5	456	194	500
R21	463.25	81.75	0	650	456	194	500

***w/c ratio = 0.23**
***SP =1.5%per kg of cement**

6 Result &Discussions

6.1 Properties of fresh concrete

The influence of using copper slag in place of natural sand on the workability and the bulk density of concrete specimen resulted in the substantial increase in these parameters due to its glossy surface texture and low water absorption by the copper slag. The high specific gravity of the copper slag as compared to the natural sand results in the increase in bulk density due to which larger quantity of copper slag is needed to occupy the same volume as that of natural sand [1]. The workability of the high performance concrete using copper slag as a replacement of fine aggregate increases as more and more fine aggregate is replaced but the presence of MK in concrete has a reverse effect on workability. It decreases as the percentage of MK increases in concrete. Due to reduction in the fineness modulus of cementitious material, the quantity of the cement paste available to give lubricating effect is less per unit surface area on the aggregate [42]. The dosage level of the super plasticizer was kept at 1.5 % of the weight of the cement to keep the concrete with optimal workability. Combined with MK due to its smooth surface texture and low absorption properties, copper slag improves the workability of the concrete as shown in the Figure 1.

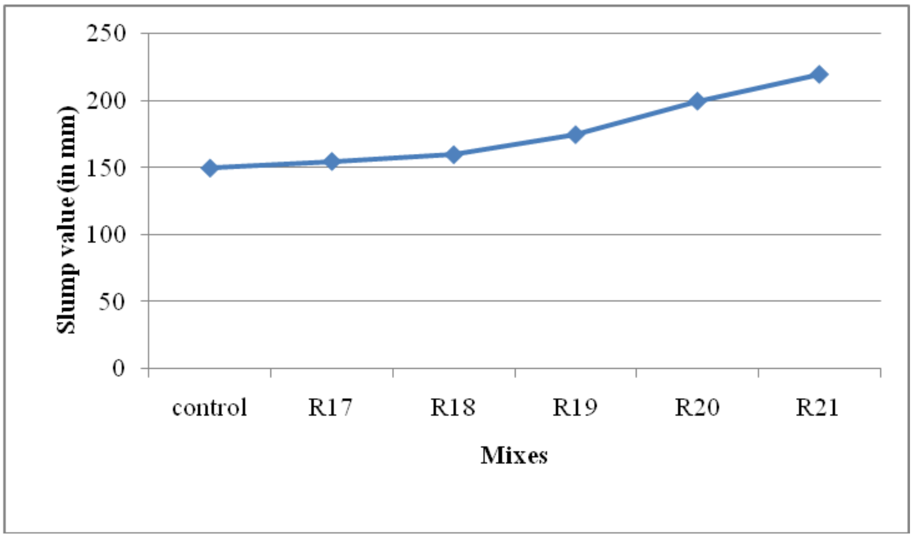

Fig. 1- Comparison of slump values of different mixes with varying copper slag %

6.1.1 Properties of hardened concrete: Compressive strength & Splitting tensile strength

Figure 2 shows the average compressive strength of the concrete specimens for control concrete mix and cube specimens with different copper slag contents as a replacement of fine aggregate at 7, 28, 90 and 120 days of curing periods. Compressive strength improved with the rise in the quantity of the copper slag up to 50 %. CS is better than sand in compressibility and hence relieve the stress concentration partially. CS improves the cohesiveness of concrete due to its angular sharp edges giving good interlocking between cement and coarse aggregates [43]. An improvement of 75% in the strength is observed at 50% substitution level of copper slag as compared to the control concrete. When sand is replaced with copper slag beyond 50% the strength of concrete starts reducing but still comparable to control concrete even after 100% replacement due to low w/b ratio counteracting the low water absorption properties of copper slag which otherwise causes excessive bleeding at higher copper slag content. The compressive strength at 100% copper slag substitution declined by 7% to 15% at all curing ages. The reduction in the compressive strength of the concrete with more than 50% copper slag substitution can be due to increased porosity of the concrete specimen having free excess water remaining in the concrete blend.

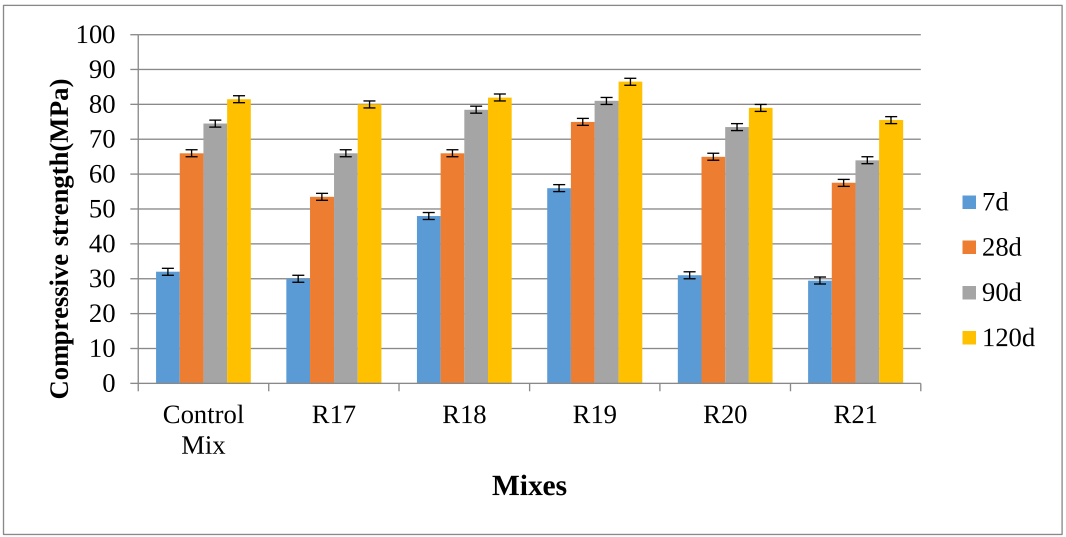

Fig. 2- Compressive strength values of different mixes with varying copper slag%

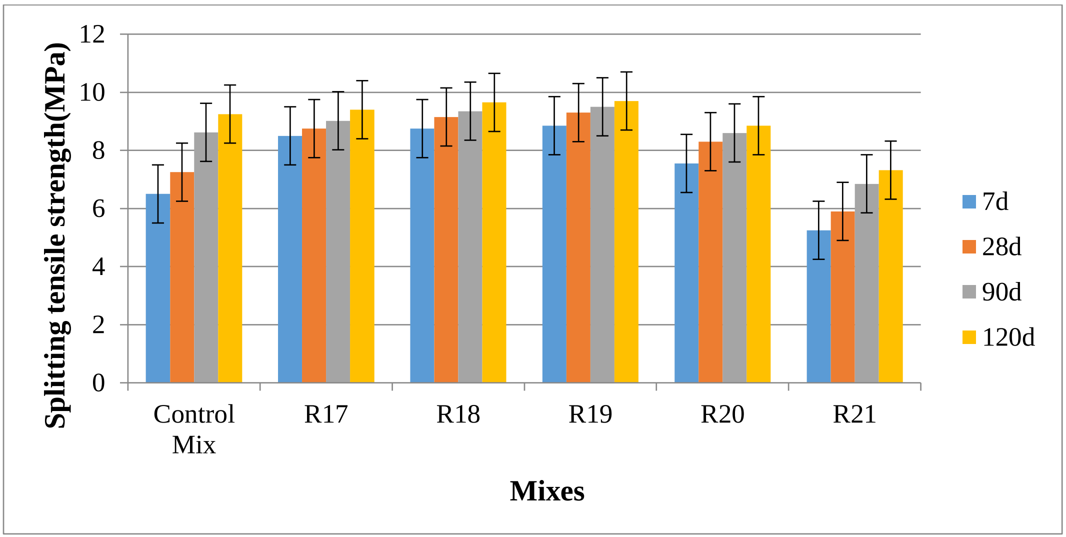

Fig. 3- Splitting tensile strength values of different mixes with varying copper slag %

Fig 3 shows the Splitting tensile strength values of different HPC mixes with varying proportions of CS. Splitting tensile strength of various concrete mixes shows the same pattern as that of compressive strength with peak strength for 50% CS substitution due to high toughness of CS. Splitting tensile strength of R19 shows rise in the strength between 5% to 36% for all curing periods as compared to the control mix. CS substitution of 100% for sand shows decline in 18-20% of tensile strength values. This drop is due to the increasing porosity of the concrete leading to tensile cracking in the weaker parts of the concrete matrix

Fig. 4-(a) Compressive strength test

Fig. 4-(b) Splitting tensile strength test

6.2 Durability properties

6.2.1 Sorptivity

The value of sorptivity decreased with the incorporation of the copper slag in comparison to the control concrete. The inclusion of the MK in the HPC mix improved the resistance against capillary absorption by filling the microspores and densifying the microstructure by the reaction of the MK with CH to form C-S-H gel resulted in the decline of the sorptivity of the concrete. The results of sorptivity with different proportions of copper slag at 28, 90, 120 days of curing period are presented in the Fig.5.

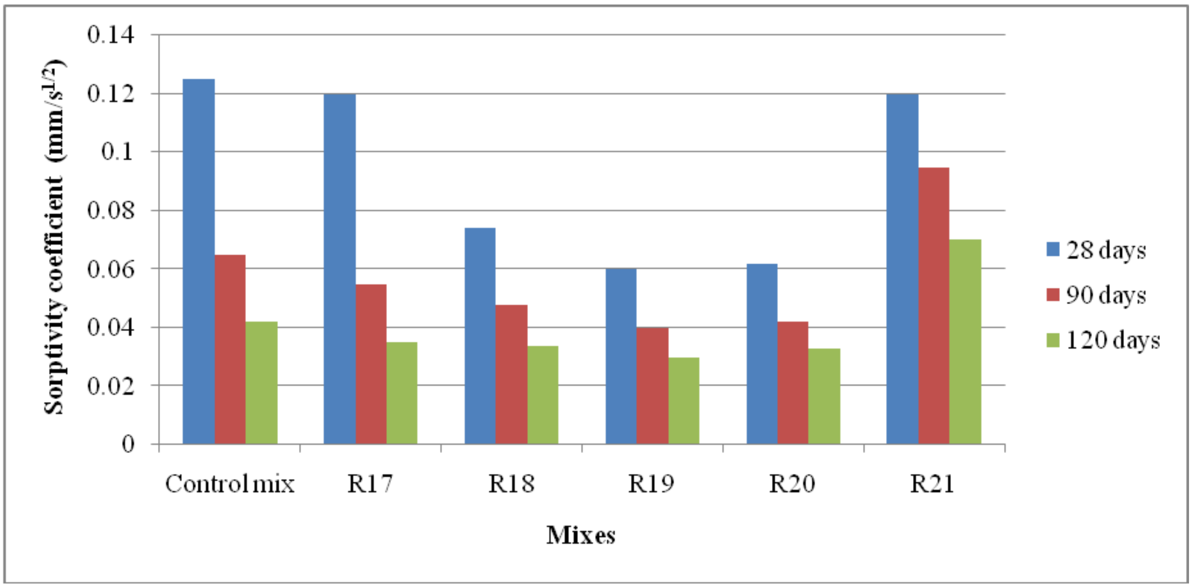

Fig.5- Sorptivity coefficients of various mixes

The lowest sorptivity is noted for the mix containing 50% copper slag at each curing age. For the HPC mix with copper slag replacement of 50%, sorptivity decline by 52%, 38% and 28.5% with respect to control concrete for the curing periods of 28,90 and 120 days.

6.2.2 Rapid chloride penetration test (RCPT)

Chloride permeability of the high performance concrete for the control mix concrete and all other respective mixes shown in table 4 exhibited encouraging results at 25% and 50% replacement of copper slag after 28 days of curing period passing "very low" charge of 740 C & 850 C respectively. The ingression of chloride at 50%, 75% and 100% replacement levels of copper slag increased showing the charge passed as 1002 C, 1205 C & 1487 C falling in the category of "Low". The decreased value of RCPT has a great effect due to low water binder ratio increasing the resistance to chloride penetration [44]. The inclusion of the Metakaolin having finer particles makes the pore structure of the high performance concrete denser resulting in low chloride permeability in addition to the good strengthening results. Metakaolin has an excellent potential to be used as a supplementary cementitious material in structures made of HPC since it minimises the deleterious expansion due to alkali silica reaction in concrete mix and consequently reduces the ingression of the chloride by improving the microstructure [45].

Table 4: Chloride Penetration of concrete mixes after 28, 90 & 120 days of curing period

Mix	Charge (coulombs)	Cl^- penetration after 28 days curing	Charge (coulombs)	Cl^- penetration after 90 days curing	Charge (coulombs)	Cl^- penetration after 120 days curing
Control	4286	High	2985	Moderate	1950	Low
R 17	740	Very Low	612	Very Low	483	Very Low
R18	850	Very Low	605	Very Low	503	Very Low
R19	1002	Low	840	Very Low	626	Very Low
R20	1205	Low	950	Very Low	814	Very Low
R21	1487	Low	1104	Low	987	Very Low

6.2.3 Accelerated Carbonation

Influence of copper slag on the carbonation resistance of high performance concrete for control mix and different mixes with varying proportions of copper slag as 0%, 25%, 50%,75% and 100% after curing periods of 28,90 and 120 days with their respective carbonation periods of 4,8,12 and 16 weeks is illustrated in the fig. 6 to fig.11 respectively. Fig.6 illustrates the carbonation depths well within limits was not observed for the control mix at the end of all curing periods with their respective carbonation periods. Figs.7, 8, 9,10 and 11 describes carbonation depths of HPC with 15% MK showing a delay in the advance of the carbonation front due to decrease in capillary pores. The incorporation of MK in the mix containing 0% CS reduces the carbonation depth by 40-45% as compared to control mix. Mix R19 shows the best results for carbonation resistance with the decline in carbonation depths from 53-59% for all carbonation periods. This drop may due to the pozzolanic behavior of MK which reacts with C-H obtained from the hydration of OPC to form dense C-S-H gel. This leads to the formation of compact microstructure of concrete with reduced porosity resulting in less diffusion of CO2 into it [46]. However, overall scenario of carbonation depths is very encouraging as the depth is well below 20mm, which is much lesser than the usual concrete cover over the reinforcements as observed in other studies also [47]. This deduced that concrete with copper slag has a minor effect of CO2 due to its closely compact hardened structure as compared to the control mix [48].

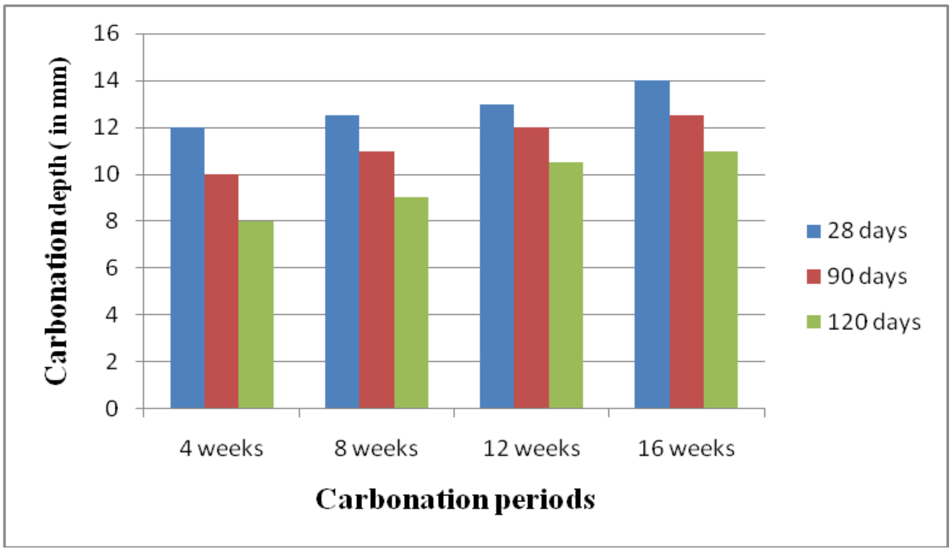

Fig.6- Carbonation depths for HPC Control mix

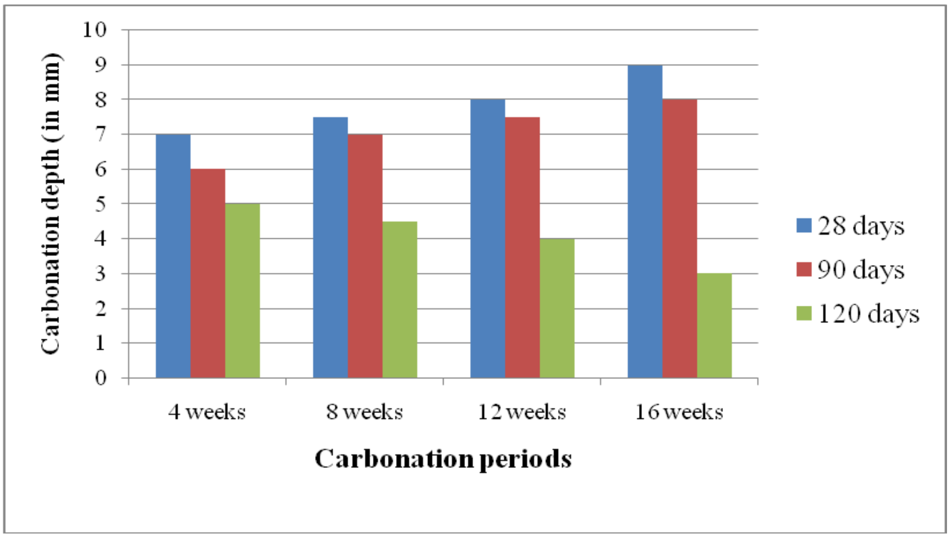

Fig.7- Carbonation depths for HPC mix with 85%cement+15%MK+0%CS

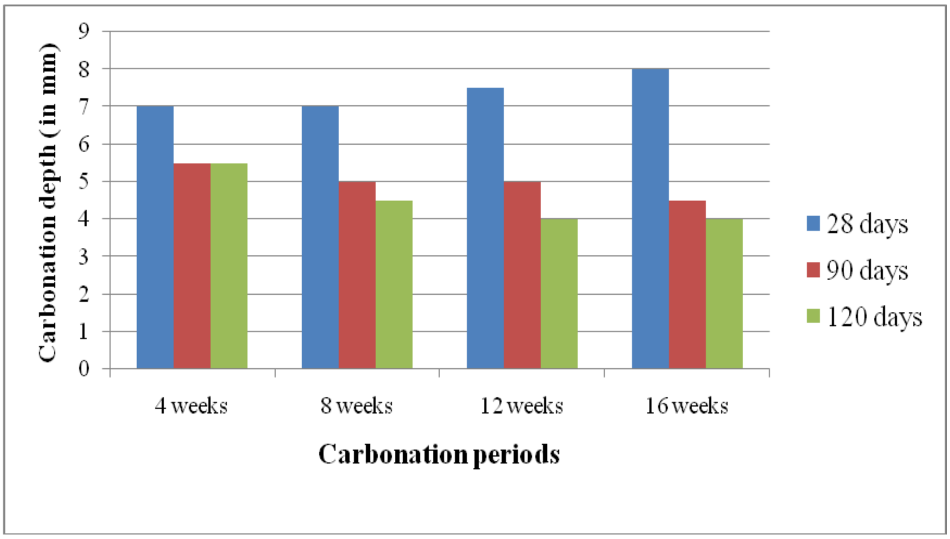

Fig.8- Carbonation depths for HPC mix with 85%cement+15%MK+25%CS

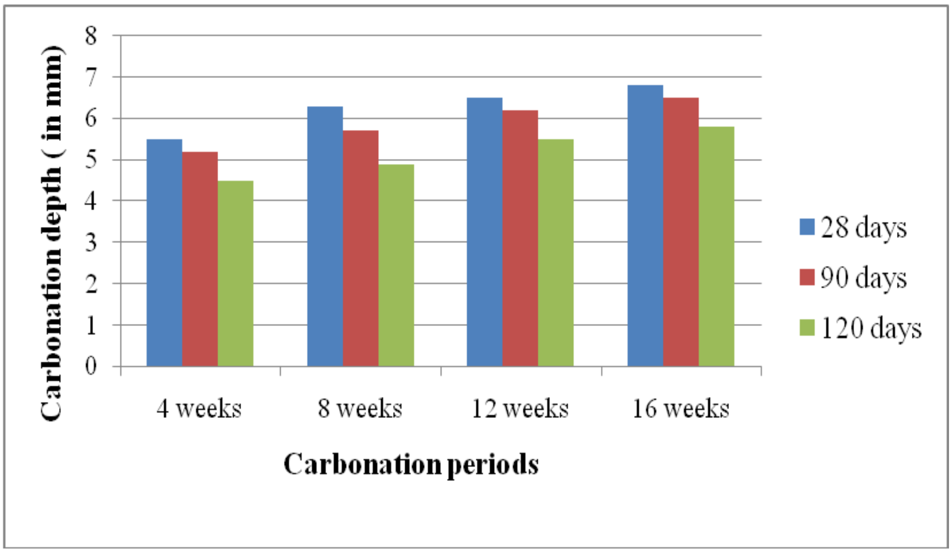

Fig. 9- Carbonation depths for HPC mix with 85%cement+15%MK+50%CS

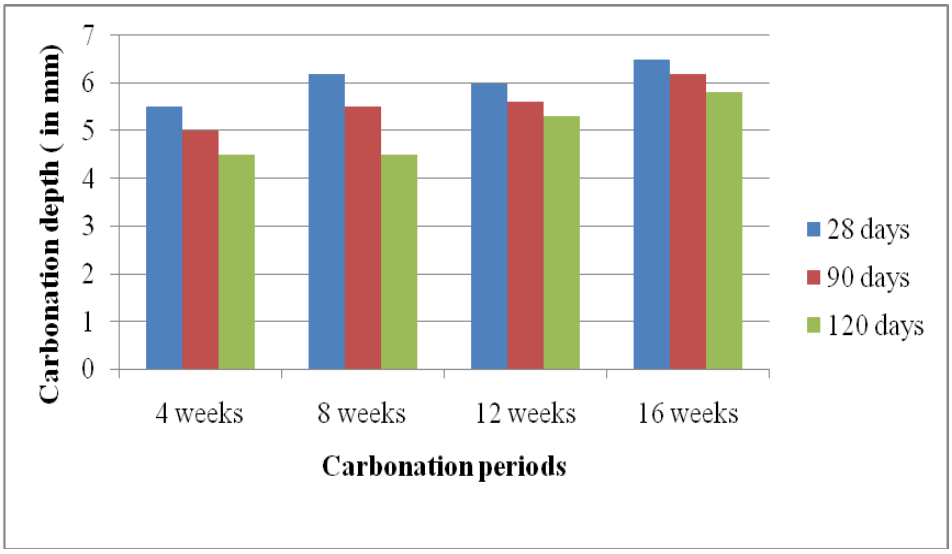

Fig. 10- Carbonation depths for HPC mix with 85%cement+15%MK+75%CS

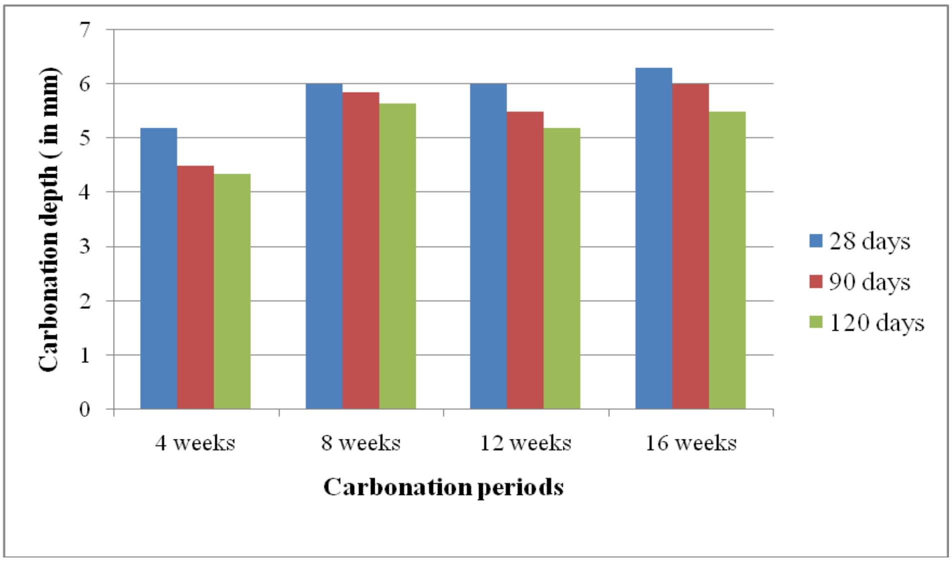

Fig.11- Carbonation depths for HPC mix with 85%cement+15%MK+100%CS

6.2.4 Scanning Electron Microscopy (SEM) Analysis

The SEM micrograph of control concrete mix in Fig.12(a) illustrates presence of some ettringite with porous microstructure whereas substitution of 15% metakaolin as replacement to cement improved the microstructure by acting as filler and reducing the porosity of the mix as shown in Fig.12(b). The SEM micrograph of Fig.12(c) reveals dense microstructure with huge amount of C-S-H gel which is the main reason for maximum compressive strength among all mixes. It can be observed from SEM micrograph in Fig.12(d) that almost similar microstructure is also observed in Fig.12(c). The SEM micrograph in Fig.12(e) shows less formation of C-S-H gel with porous microstructure. Moreover, few small size needle shape crystals can also be observed which depicts the presence of ettringite. The SEM micrograph in Fig.12(f) clearly shows large number of voids, porous microstructure and occurrence of ettringite which are the mains reasons for decrease in compressive strength on replacement of 100% sand by CS.

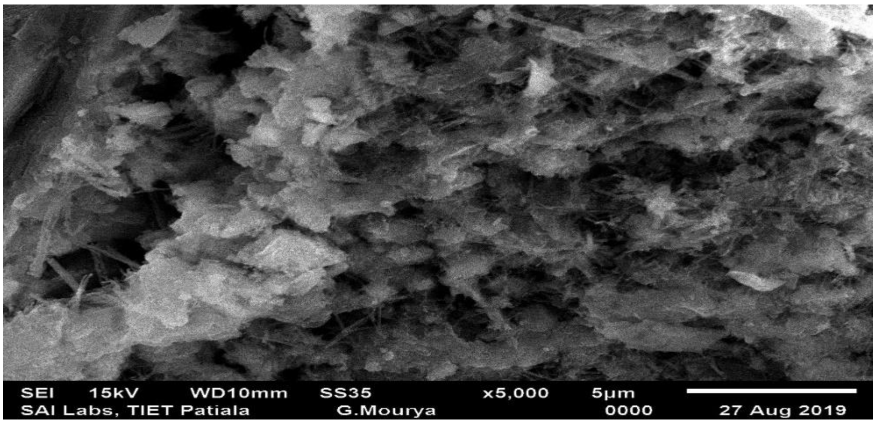

Fig. 12(a). SEM analysis after 120 days of HPC mixes: (a) R1

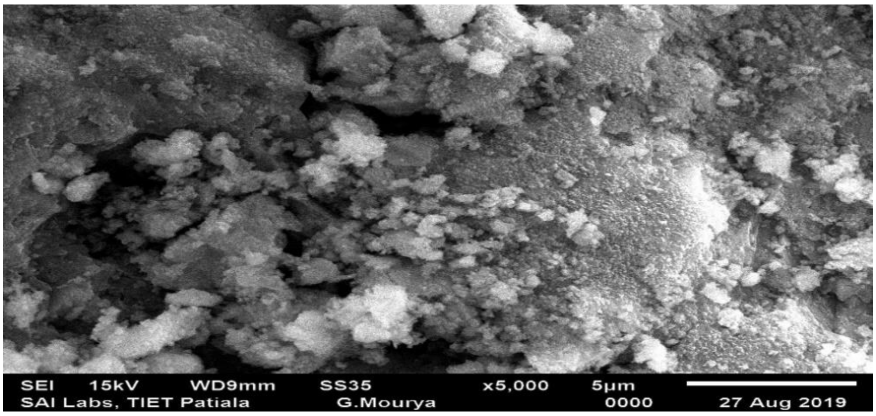

Fig. 12(b). SEM analysis after 120 days of HPC mixes: (b) R17

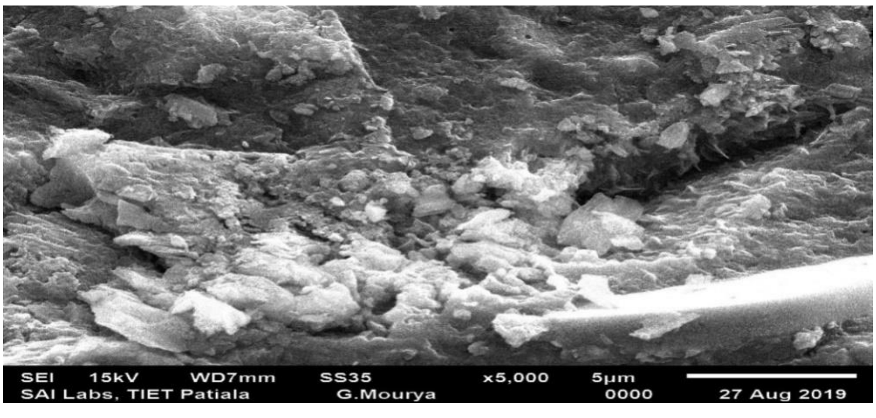

Fig. 12(c). SEM analysis after 120 days of HPC mixes: (c) R18

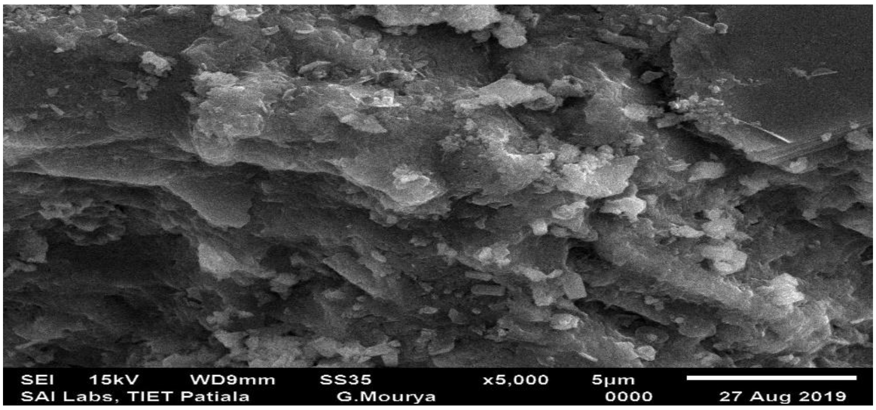

Fig. 12(d). SEM analysis after 120 days of HPC mixes: (d) R19

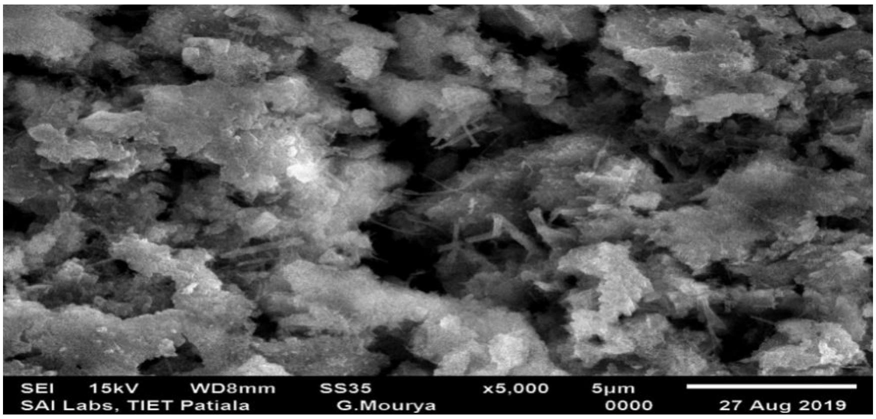

Fig. 12(e). SEM analysis after 120 days of HPC mixes: (e) R20

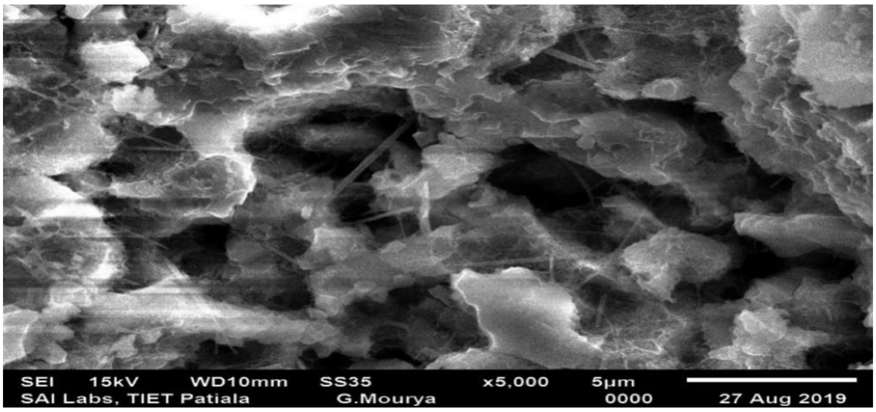

Fig. 12(f). SEM analysis after 120 days of HPC mixes: (f) R21

7 Conclusion

The present study was aimed to evaluate the fresh, mechanical and durability properties of HPC containing CS as fine aggregate and MK as partial replacement of OPC. The following conclusions were drawn from the experiments done:

The introduction of MK as a partial replacement of OPC resulted in the decrease of flow ability for R17 and R18 mixes which is compensated by using superplasticizer to maintain optimal workability. However, slump values escalated for R19, R20 and R21 mixes with the addition of more CS due to its glossy surface texture and low water absorption properties.

The compressive strength was found to be maximum for R19 mix with 50% CS amongst all other mixes at each curing period. CS due to its angular sharp edges provides good interlocking between cement and coarse aggregates ultimately leads to the improved cohesiveness of concrete. The SEM micrograph of R19 supports the same showing dense microstructure with CSH gel due to the incorporation of MK resulting in the maximum compressive strength.

The Splitting tensile strength showed the same pattern as that of compressive strength giving increase of 36% for R19 mix as compared to control mix. On the other hand, Mix R21 showed decline in Splitting tensile strength due to tensile cracking in concrete due to increased porosity in the weakest parts of concrete mix.

Sorptivity decline is observed by 52%, 38% and 28.5% with respect to control concrete for the curing periods of 28,90 and 120 days for R19 mix. The optimum sorptivity for R19 is due to filling of the micro pores and densification of the microstructure by the reaction of the MK with CH to form CSH gel.

The results of chloride ingress are expressed as the total charge passed in Coulombs. The charge passed for R17 & R18 HPC mixes were found to be very low for all curing periods. Other mixes also showed the same results for 90d and 120d of curing periods. It may be due to the MK minimising the deleterious expansion due to alkali silica reaction in concrete mix. Increase in percentage of copper slag gave favourable results for chloride penetration due to decrease in the depth of capillary suction ensuring only little amount of gel pores available inside the concrete matrix.

The overall scenario of carbonation depths for control mix and other mixes are very encouraging as the depth is well below 20mm, which is much lesser than the usual concrete cover over the reinforcements. Mix R19 shows the best results for carbonation resistance with 53-59% decline in carbonation depth comparing it with control mix for all carbonation periods. The MK admixed concrete mixes resulted in the drop of carbonation depth due to the pozzolanic behaviour of MK which reacts with CH obtained from the hydration of OPC to form dense CSH gel.

This study suggests the mechanical and durability properties of HPC were higher than that of control concrete when CS was substituted more than 50% in combination with 15% MK as partial replacement of cement. HPC developed with CS and MK can be effectively used for bridges, high rise buildings and dams. HPC with other combinations of materials can be developed in the future.

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