

Strength and durability of ultra fine slag based high strength concrete

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Abstract. The use of ground granulated blast furnace slag (GGBFS) from steel industries waste is showing perspective application in civil engineering as partial substitute to cement. Use of such waste conserves natural resources and minimizes the space required for landfill. The GGBFS used in the present work is of ultra fine size and hence serves as micro filler. In this paper strength and durability characteristics of ultra fine slag based high strength concrete (HSC) (with a characteristic compressive strength of 50 MPa) were studied. Cement was replaced with ultra fine slag in different percentages of 5, 10, and 15% to study the compressive strength, porosity, resistances against sulfate attack, sorptivity and chloride ion penetration. The experiments to study compressive strength were conducted for different ages of concrete such as 7, 28, 56, and 90 days. From the detailed investigations with 16 mix combinations, 10% ultra fine slag give better results in terms of strength and durability characteristics.

Keywords: ultra fine slag; compressive strength; sorptivity; porosity; sulfate attack; chloride ion penetration

1. Introduction

Concrete, a homogeneous material made with number of heterogeneous components and used in all spheres of civil engineering construction. According to Basheer *et al.* (2001), ingress of any ionic and molecular species from the prevailing environment causes deterioration and affects the service life of concrete structures. Chloride, sulfate and carbonation attacks are well known in this category. Microstructure of concrete was responsible to resist such attacks and could be modified using finer particles in concrete. Several works were done using ultra fine particles in concrete as mineral admixtures to improve physical, mechanical and durability properties. Many researchers have done their research work with commercially available nano SiO₂, nano Al₂O₃, nano Fe₂O₃ and nano Zinc-iron oxide particles as mineral admixtures in concrete (Li 2004, Qing *et al.* 2007, Jo *et al.* 2007a, b, Li *et al.* 2006, Lin *et al.* 2007, Lin *et al.* 2008). Her and Lim (2010), studied the effect of rapidly chilled and air-cooled nano slag as alternate material to silica fume on physical, chemical, and dynamic properties of the mortar. They found that enhancement of fineness decreased the fluidity and bigger grains surrounded by smaller grains.

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They concluded that SEM study was adequate to assess existence of cohesion between particles and nano slag in mortar gained better strength only in later age. Lim (2004, 2005, 2008) investigated strength properties of slag powder on high strength concrete and found that addition of nano slag modified the microstructure and improved the strength of concrete. Karthikeyan and Dhinakaran (2014) conducted experiments on concrete with ultra fine silica and found that there was a gain in compressive strength. Vijayasathya and Dhinakaran (2014) performed experiments on strength and durability characteristics of high performance concrete with GGBFS and M-Sand and found that there was an improvement in both compressive strength and sorptivity. From the literature available, it was understood that research work on performance of concrete with locally produced ultra fine slag was scanty. Hence it was decided to study the strength and durability characteristics of ultra fine slag based high strength concrete in the present research work. Many early researchers used commercially available ultra fine particles (very high cost) for their research work. In the present study, ultrafine slag was locally produced by grinding the normal slag in a planetary ball mill for a designated period of time.

2. Experimental investigations

2.1 Material properties

Ordinary Portland cement of 53 grade was used in this paper. GGBFS obtained from near by steel industries was used as substitute to cement after grinding. River sand was used as fine aggregate. Broken stone used as coarse aggregate. The specific gravity was 3.15 for cement, 2.40 for GGBFS and 2.65 for fine aggregate and coarse aggregate. The chemical composition of cement and GGBFS was obtained by X-ray fluorescence using an XRF spectrometer, which is given in Table 1.

Table 1 Chemical composition of cement and GGBFS

Formula	Concentration (%)	
	Cement	GGBFS
CaO	68.05	34.85
SiO ₂	25.91	34.01
Al ₂ O ₃	5.85	16.62
MgO	0.07	9.11
Fe ₂ O ₃	0.12	1.71
SO ₃	-	1.55
TiO ₂	-	0.69
Na ₂ O	-	0.48
K ₂ O	-	0.46
MnO	-	0.27
BaO	-	0.10
P ₂ O ₅	-	0.04
SrO	-	0.04
Cl	-	0.03
ZrO ₂	-	0.03
As ₂ O ₃	-	37 ppm

Table 2 Details specimen cast for strength and durability tests

Curing medium	Age of Concrete in days	Test	Size in mm	Codal Provision	No. of specimens
Normal	7, 28, 56 & 90	Compression	100×100×100	BS 1881	36
		Porosity	100 mm (dia)× 50 mm height	ASTM C642	08
		Sorptivity	100 mm (dia)× 50 mm height	ASTM C1585	08
		Chloride ion Penetration	100 mm (dia)× 50 mm height	ASTM C1202	08
HCl	56 days	Compression	100×100×100	BS 1881	12
H ₂ SO ₄	56 days	Compression	100×100×100	BS 1881	12

2.2 Specimen details

The concrete mix design was performed according to ACI 211.1-91 method for concrete with a characteristic compressive strength of 50 MPa and the mix proportion arrived at was 1:1.04:2.13 (cement: fine aggregate: coarse aggregate) with a w/b ratio of 0.33. The addition of super plasticizer (high range water reducer) reduces the w/c ratio from 0.36 to 0.33. The workability of fresh concrete was verified with slump cone test according to ASTM C143. Preparation of fresh concrete followed this sequence: High range water reducer was added with water and stirred for a minute to have homogeneity. Then ultra fine slag was added with water and stirred at a high speed for the duration of 5 minutes to ensure uniform dispersion. The other ingredients namely cement, fine aggregate and coarse aggregate were mixed together in a concrete blender with a little bit low speed for the duration of 2 minutes. Finally, prepared water (with high range water reducer and ultra fine slag) and defoamer was poured into the dry concrete mix and stirred further for duration 2 minutes to get the desired workability. Cube specimens were used to test compressive strength. The specimens of size 100 mm×100 mm×100 mm were cast according to BS 1881. Cylindrical specimens of size 100 mm diameter and 50 mm height were cast to study porosity (according to ASTM C642), sorptivity (according to ASTM C1585) and chloride ion penetration (according to ASTM C1202). Details of specimens cast for different tests are given in Table 2.

3. Results and discussion

The results of compressive strength, porosity, resistance against corrosion tested in this paper are discussed in the following sections. The combinations chosen are as follows: control concrete (concrete with no ultra fine slag); concrete with 5, 10, and 15% ultra fine slag. The above parameters were considered to understand the effect of ultra fine slag on strength and durability characteristics.

3.1 Characterization of ultra fine slag

3.1.1 XRF and particle size analysis

The chemical composition was obtained by X-Ray Fluorescence test using XRF spectrometer

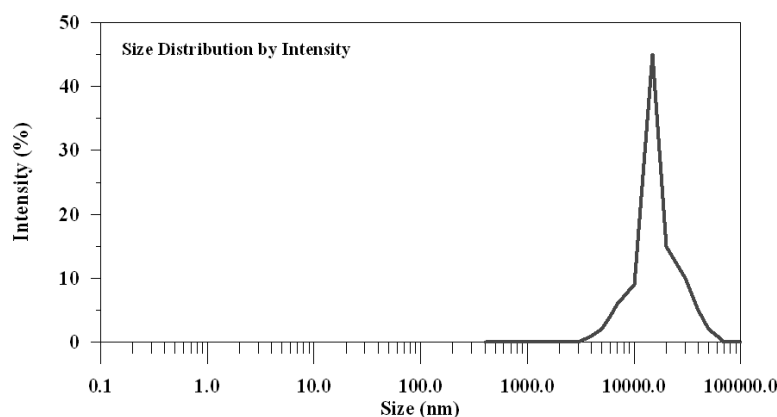


Fig. 1(a) Particle size of slag before grinding

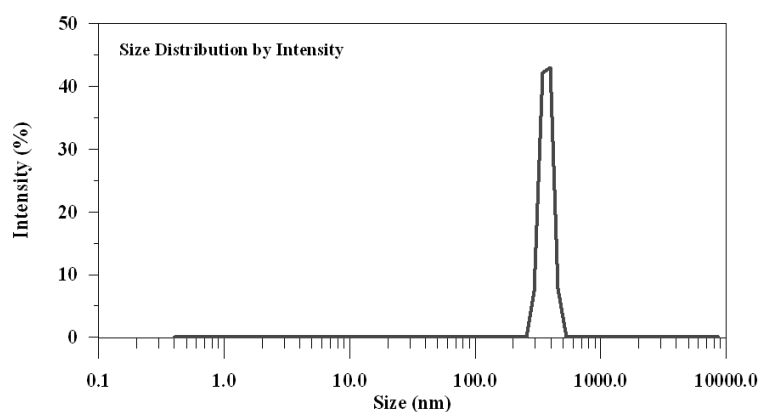


Fig. 1(b) Particle size of slag after grinding

of model Tiger 88. Table 1 shows the major and trace elements in solids (expressed as oxides). Cao and Silica (SiO_2) were the major components in the slag and constituted 68.86 percentages. The other major elements were Al_2O_3 and MgO with 16.62 and 9.91% respectively. All the other components constituted only 4.61%. Ultra fine slag was obtained by grinding precursor slag obtained from steel industry in dry ball mill. Precursor slag was fed into the dry ball mill with 25 ceramic balls weighing 10 g each in a tungsten carbide jar of diameter 90 mm with a constant speed of 324.5 rpm. The precursor slag was subjected to grinding for different grinding periods such as 60 minutes, 120 minutes and 150 minutes. The samples of precursor slag ground at different durations were subjected to Particle size analysis and sample subjected to grinding for 150 minutes was found to be finer and the same was used for the research work. The average particle size of precursor slag was $17.15 \mu\text{m}$ (see Fig. 1(a)) and ground slag (Ultra fine slag) was $0.37 \mu\text{m}$ (see Fig. 1(b)). The size was reduced about 46 times smaller than the original size.

3.1.2 X-Ray Diffraction (XRD)

XRD is one of the best methods to assess the concentration of compounds or minerals in concrete. It is also a direct method to find the crystalline phases present in the concrete. The powder sample for XRD test was prepared by crushing concrete specimens at the age of 28 days.

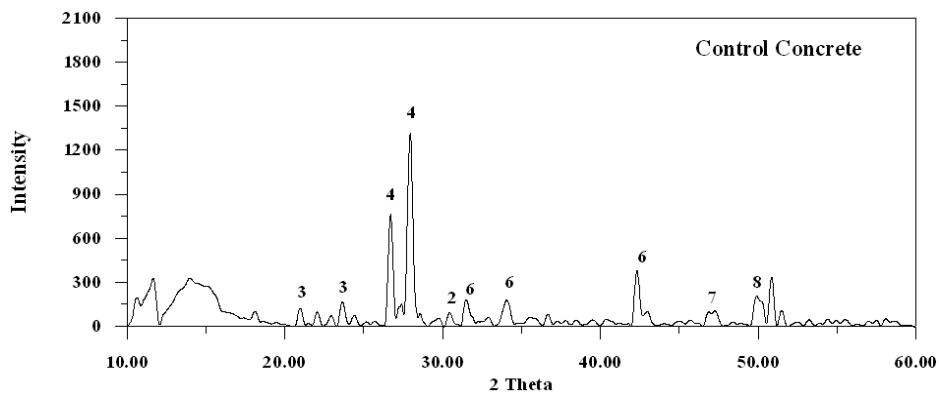


Fig. 1(c) XRD pattern of control concrete

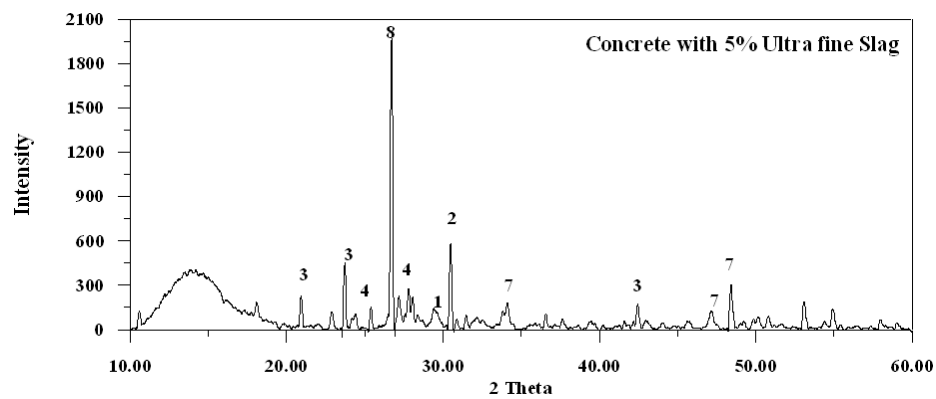


Fig. 1(d) XRD pattern of concrete with 5% ultrafine slag

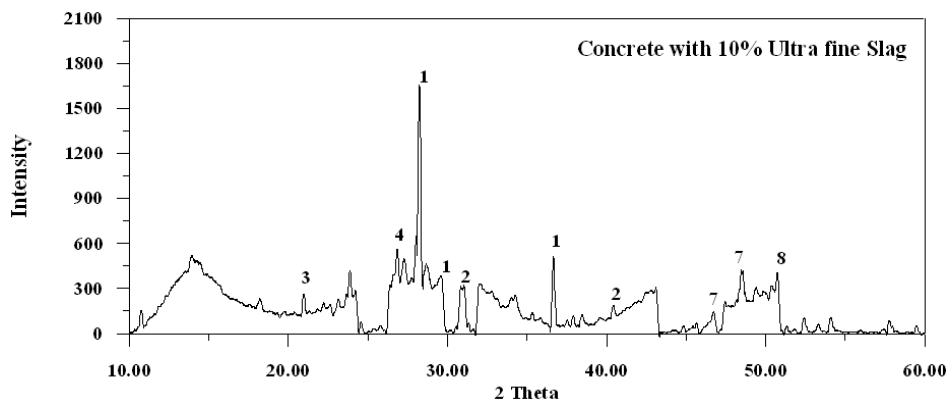


Fig. 1(e) XRD pattern of concrete with 10% ultrafine slag

The powder was sieved through 150μ sieve and scanned for 10° to 60° . The XRD results were shown in the form of peaks for all samples (see Figs. 1(c) to 1(f)). The peaks in the graph indicate the presence of portlandite, ettringite, calcite, gypsum, quartz, feldspar, dolomite and Portland cement. In control concrete specimen, Feldspar, Portland cement, portlandite and ettringite were

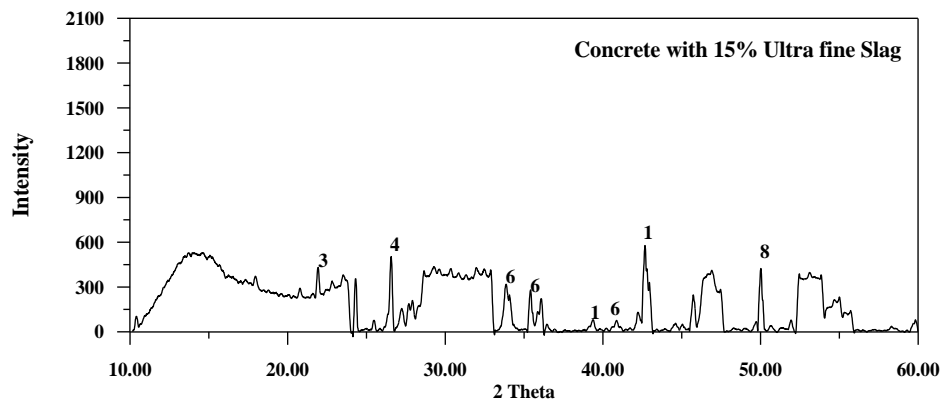


Fig. 1(f) XRD pattern of concrete with 15% ultrafine slag

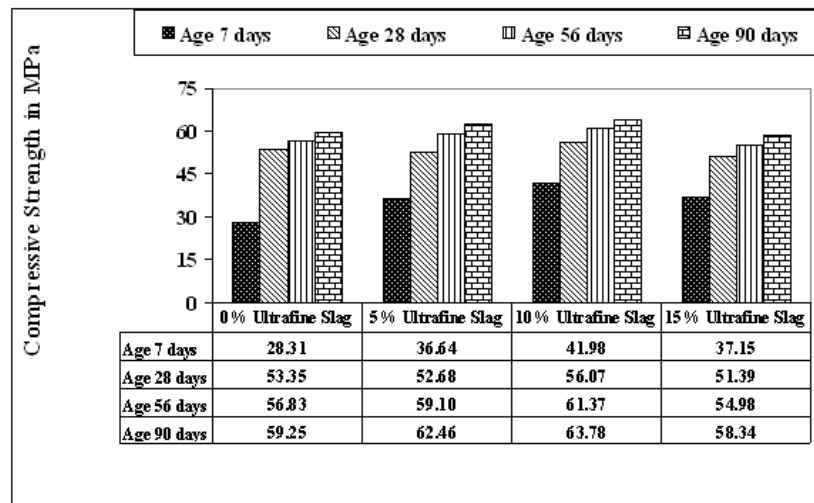


Fig. 2 Effect of age on compressive strength of concrete with ultrafine slag

the major crystalline phases dominated. There was also a broad and diffuse hump that appears between 10° and 20° . In the case of concrete with 5 and 10% ultra fine slag, domination of calcite and quartz was found. The presence of higher amount of calcite and quartz filled the voids between cement and aggregate grains and improved the resistance against permeability characteristics of the concrete. It also influenced the later development strength of the concrete. In the case of concrete with 15% ultra fine slag Portland cement, portlandite, ettringite and feldspar were dominated as seen in control concrete.

3.2 Compressive strength

The variation of compressive strength of concrete with ultra fine slag for different ages of concrete was depicted in Fig. 2. The compressive strength of concrete with ultra fine slag at the early age was higher than control concrete. The rate of increase in compressive strengths of concrete with 5, 10 and 15% of ultrafine slag were 29, 48 and 31% with respect to control

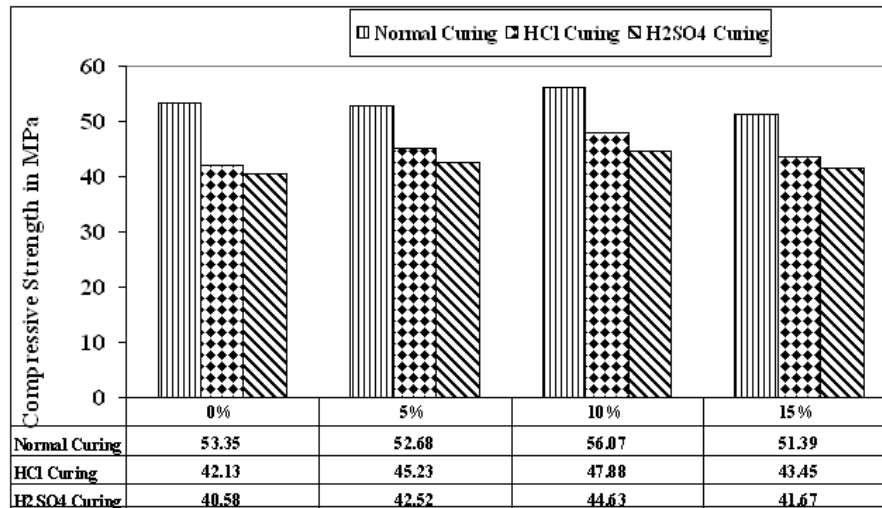


Fig. 3 Compressive strength of concrete with ultrafine slag in normal and acidic curing

concrete. At the age of 28 days, compressive strength of concrete with 5 and 15% ultra fine slag was slightly less than the control concrete. However for concrete with 10% of ultra fine slag yielded 5% higher strength than control concrete. The reason could be contribution of 5% ultra fine slag in gaining strength was insignificant. The reason for reduction in strength for concrete with 15% ultra fine slag was due to aggregation of finer particles together and formation of larger particles. The formation of larger particles did not serve the purpose of modification of microstructure in terms of densification. In contrast it makes concrete with more voids due its increased size of agglomerated ultra fine particles and finally the higher displacement ratio affected the fluidity. Hence there was a reduction in strength. Concrete with 10% ultra fine slag modified the microstructure effectively and hence the strength development was successful. At the ages of 56 days and 90 days, concrete with 15% ultra fine slag concrete gives similar results. For concrete with 5 and 10% ultra fine slag the strength developments at the age of 90 days were found to be more than the control concrete. The rate of increase of compressive strength was in the range of 5 to 10%.

3.3 Resistance against corrosion

All the specimens of control concrete and concrete with 5, 10, and 15% of ultra fine slag were cured in 10% HCl solution and 10% H₂SO₄ separately for 28 days. The compressive strength of these specimens was compared in two aspects. The first aspect of comparison was performance of control concrete and concrete with ultra fine slag in aggressive and normal environment. Specimens tested in aggressive environment were first cured in normal water for 28 days and then cured in aggressive environment (HCl or H₂SO₄ solution) for another 28 days. Hence to have realistic comparison, specimens cured in normal water at the age of 56 days were compared with the specimens cured in aggressive environment at the age of 28 days. The comparison of these results was depicted in Fig. 3. Compressive strength of control concrete was 56.83 MPa in normal curing, 42.13 MPa in HCl solution curing and 40.58 MPa in H₂SO₄ solution curing. Control concrete suffers with 26 and 29% lesser strength in HCl and H₂SO₄ than normal curing. Similar

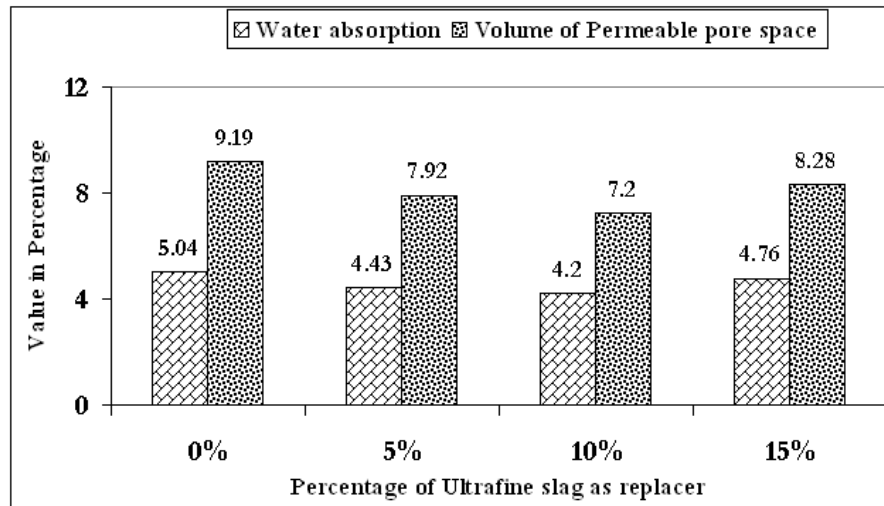


Fig. 4 Effect of ultra fine slag based concrete against water absorption and voids

Table 3 Calculations of Water absorption and Voids (as per ASTM C642)

Description	Formula	Percentage of Nano slag			
		0	5	10	15
Mass of oven-dried sample in air (g)	A	992	1015	1096	1092
Mass of surface-dry sample in air after immersion (g)	B	1028	1055	1132	1136
Mass of surface-dry sample in air after immersion and boiling (g)	C	1042	1060	1142	1144
Apparent mass of sample in water after immersion and boiling (g)	D	498	492	502	516
Absorption after immersion (%)	$[(B-A)/A] \times 100$	3.63	3.94	3.29	4.03
Absorption after immersion and boiling (%)	$[(C-A)/A] \times 100$	5.04	4.43	4.20	4.76
Bulk density, dry (g/cc)	$g_1 = [A/(C-D)] \times \rho^*$	1.82	1.79	1.71	1.74
Bulk density after immersion (g/cc)	$[B/(C-D)] \times \rho$	1.89	1.86	1.77	1.81
Bulk density after immersion and boiling (g/cc)	$[C/(C-D)] \times \rho$	1.92	1.87	1.78	1.82
Apparent density (g/cc)	$g_2 = [A/(A-D)] \times \rho$	2.01	1.94	1.85	1.90
Volume of permeable Pore space (voids) (%)	$[(g_2 - g_1)/g_2] \times 100$ or $[(C-A)/(C-D)] \times 100$	9.19	7.92	7.19	8.28

* ρ =density of water in g/cc

trend was observed in concrete with ultra fine slag also. But the resistance of ultrafine slag admixed concrete to aggressive environment was higher than control concrete. The second aspect of comparison was between control concrete and concrete with ultra fine slag concrete in aggressive environment. Concrete with 5% ultra fine slag gives 5% more strength in HCl solution and 7% more strength in H₂SO₄ solution than the control concrete. In the case of concrete with 10% ultra fine slag the rate of increase in compressive strength was doubled compared to concrete

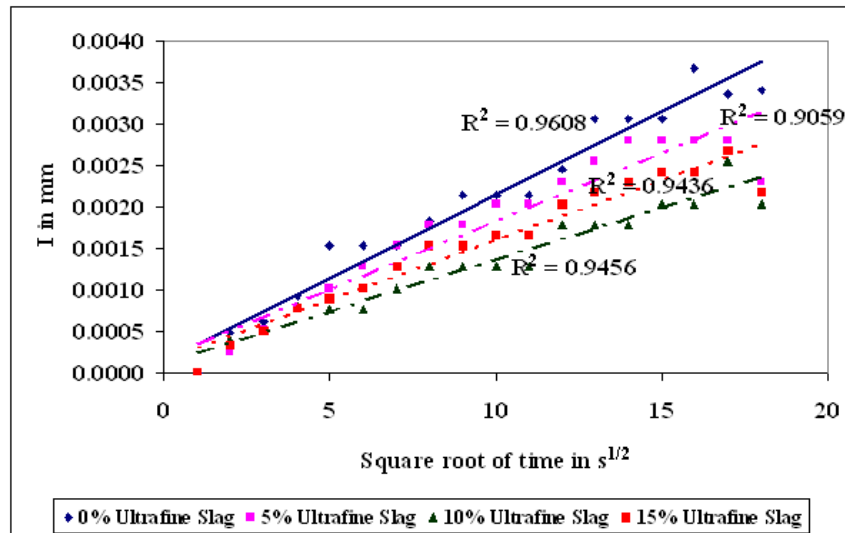


Fig. 5 Sorptivity characteristics of concrete with ultra fine slag

with 5% ultra fine slag. In concrete with 15% ultra fine slag, the rate of increase in compressive strength was insignificant for the same reason of agglomeration.

3.4 Evaluation of water absorption and voids in hardened concrete

The water absorption and voids value were calculated according to ASTM C642 and was given in Table 3. The water absorption values were 5.04% for control concrete, 4.43, 4.20 and 4.76% for concretes with 5, 10, and 15% of ultra fine slag respectively (see Fig. 4). The amount of water absorption was higher for control concrete and come down due to the addition of ultra fine slag in concrete.

Increase in percentage of ultra fine slag from 5 to 10 % reduces water absorption and further increase of ultra fine slag to 15% shown a reverse trend. However water absorption of concrete with 15% ultra fine slag was less than that of control concrete. Concretes with 5, 10 and 15% ultra fine slag absorbs 12, 17 and 6% lesser amount of water than control concrete. Hence it was evident that addition of ultra fine slag served as micro filler in the pores of concrete and increased the density of concrete. The volume of pores was 9.2% in control concrete, 7.9, 7.2 and 8.3% respectively for concrete with 5, 10 and 15% ultra fine slag.

3.5 Evaluation of sorptivity characteristics

The sorptivity values were plotted with respect to time elapsed and was shown in Fig. 5. The value of primary sorption (upto 6 hours) was 0.0016 mm for control concrete, 0.0014 mm, 0.0010 mm and 0.0012 mm respectively for concretes with 5%, 10% and 15% ultra fine slag. The sorption values of concretes with all percentages of ultra fine slag were lesser than control concrete. Minimum sorption value was observed in concrete with 10% ultra fine slag. Similar trend as in porosity was observed in sorptivity also for concrete with 15% ultra fine slag. In a similar way the values of secondary sorption were calculated from 6 hours to 9 days. The secondary sorption value

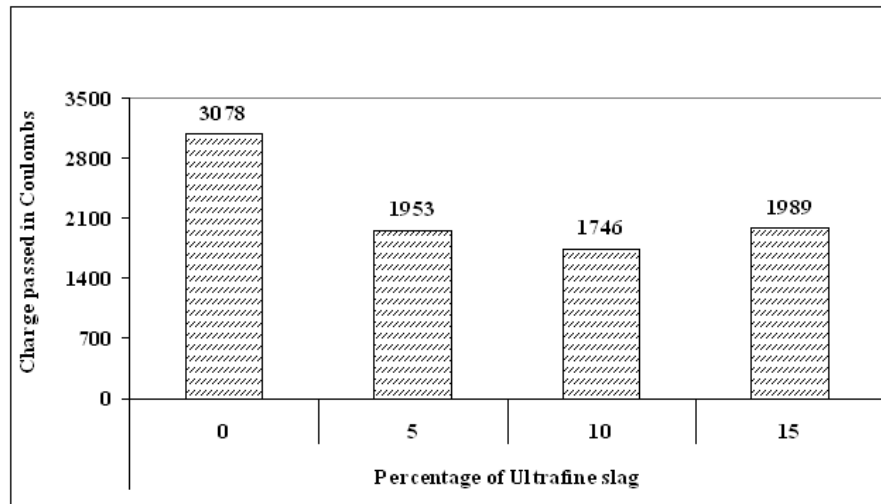


Fig. 6 Effect of ultra fine slag based concrete against chloride ion penetration

was 0.003 mm for control concrete, 0.0027 mm, 0.0020 mm and 0.0024 mm respectively for concretes with 5%, 10% and 15% ultra fine slag. Here again concrete with ultra fine slag exhibited better performance in terms resistance. The reduction of primary sorption values compared to control concrete was 46, 50, and 41% respectively for concretes with 5, 10, and 15% ultra fine slag. In a similar way, the secondary sorption values were 32, 44, and 36% respectively for concretes with 5, 10, and 15% ultrafine slag. Comparing initial sorption and secondary sorption, sorption at the initial stage was found to be more aggressive irrespective of type of concrete. The R^2 values of control concrete and concretes with 5, 10 and 15% ultra fine slag were found to be 0.96, 0.90, 0.94 and 0.95 respectively. Since R^2 value for all the concretes were more than 0.9, the goodness of fit was found to be good.

3.6 Resistance against chloride ion penetration

The result of chloride ion penetration through the concrete specimens was shown in Fig. 6. It is expressed in terms of charge passed. The charge passed was 3078 for control concrete and fell under the category of moderate as per ASTM C1202. Addition of ultra fine slag as partial substitute to cement significantly improved the resistance of concrete to chloride ion penetration. The charges passed were 1953, 1746 and 1989 for concretes with 5, 10, and 15% ultra fine slag respectively. The rate of reduction compared to control concrete was 37, 43, and 35% respectively for concretes with 5, 10 and 15% ultra fine slag. Maximum resistance was observed in concrete with 10% ultra fine slag due to uniform dispersion of ultrafine particles in concrete. Hence it was understood that, ultra fine slag in concrete was very effective in minimizing the chloride ingress into concrete.

4. Conclusions

Detailed experimental investigations were carried out on high strength concrete to study the

effect of ultra fine slag on strength and durability characteristics. From the experiments conducted, the following conclusions are made:

- Addition of ultra fine slag as partial substitute to cement in high strength concrete modified the micro structure and made improvements in compressive strength and durability characteristics.
- The compressive strength increases till 10% replacement of cement with ultra fine slag and further increase of replacement shown a reverse trend.
- Concrete with ultra fine slag exhibited better resistance to corrosion than control concrete. Concrete with ultra fine slag offered better resistance to acid attack than to sulfate attack.
- Performance of concrete with ultra fine slag till 10% was found to be better in terms of resistance against capillary suction, water absorption, porosity and chloride ion penetration. Use of 15% ultrafine slag in concrete lead to aggregation of molecules and affected the performance.
- From the strength and durability criteria, it was concluded that concrete with 10% ultra fine slag could be used without compromising any parameter and was an optimum percentage of replacement of cement from the experiments conducted.

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