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Research Article

EFFECT OF SULPHURIC ACID (H_2SO_4) ON BLENDED CEMENT (FLY ASH BASED) AND IT'S CONCRETE

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ABSTRACT

This paper presents the effect of sulphuric acid (H_2SO_4) on Blended Cement (fly ash based(BC)) and its concrete. The BC and its concrete BCC produced with H_2SO_4 dosage of 100, 150, 300, 500 and 900 mg/l added in deionised water. In addition to this control specimens were prepared with deionised water (without H_2SO_4) for comparison. The setting times and compressive strength were evaluated for 28 and 90 days apart from studying rapid chloride ion permeability. The results show that, as H_2SO_4 concentration increases, there is retardation in initial and final setting of cement (BC). The compressive strength of BCC has come down with an increase in the concentration of H_2SO_4 at both 28 and 90 days. Compressive strengths of BCC have decreased in the range of 2 to 23%, with an increase in H_2SO_4 concentration, when compared with the control specimens. It was also observed that chloride ion permeability has increased with an increase in the concentration of the acid. X-ray diffraction analysis has been carried out for BCC specimens at H_2SO_4 concentration of 300 mg/l in deionised water.

Key Words: *H_2SO_4, Setting Time, Compression, Chloride Ion Permeability, X-ray diffraction*

INTRODUCTION

Water is an important ingredient of concrete, which is not only actively participates in the hydration of cement but also contributes to the workability of fresh concrete. Cement is a mixture of complex compounds, the reaction of cement with water leads to setting and hardening. All the compounds present in the cement are anhydrous, but when brought in contact with water, they get hydrolyzed, forming hydrated compounds. Since water helps to form the strength giving cement gel, the quality of water is to be critically monitored and controlled during the process of concrete making as the water universally the most abundant and natural available solvent, can contain large no of impurities ranging from less to very high concentration of them. In practice, very often, great control on properties of cement and aggregate is exercised but the control on the quality of water is often neglected.

A popular yardstick to the suitability of water for mixing concrete is that, if it is fit for drinking, it is fit for making concrete. This doesn't appear to be a true statement for all condition. Sometimes, water contain a small amount of sugar would be suitable for drinking, but not for making concrete and conversely water suitable for making concrete may not be necessarily be fit for drinking, especially if the water contains pathogenic microbial contaminants. In connection research work has been carried out on effect of polluted/chemical water on hardened concrete strength and durability. The damage impact of various deicing chemicals and exposure conditions on concrete materials were studied by Kejin *et al.,* (2006), and results indicated that the various deicing chemicals penetrated at different rates in to a given paste and concrete resulting in different degree of damages. Gorniniski *et al.,* (2007), presented an assessment of the chemical resistance of eight different compositions of a polymeric mortars. Adnan et.al (2009), reported the effects of environmental factors on the addition and durability characters of epoxy bonded concrete prisms. Fikret *et al.,* (1997), investigated the resistance of mortars to magnesium sulphate attack and results reported that there is a significant change in compressive strength properties. Venkateswara Reddy *et al.,* (2006), studied the influence of strong alkaline substances (Na_2CO_3 and

$NaHCO_3$) in mixing water on strength and setting properties of concrete. In many places ground water and surface water contains the impurities, more than that of limits specified by the IS-456-2000. As there is scarcity of potable water in many places, this impure water is being used for mixing as well as curing of concrete in the civil engineering constructions. Hence an attempt is made to study the effect of water containing H_2SO_4 at various concentrations in cements and their concretes.

MATERIALS AND METHODS

Cement

Portland Pozzolana Cement containing 30% of fly ash was used in this investigation. The compositions of major compounds present in the cements are presented in Table 1 and 2.

Fine Aggregate

Locally available River sand was used.

Coarse Aggregate

Machine Crushed granite stone of max size 20mm confirming to IS 383 -1970 was used.

Water

Deionised water spiked with H_2SO_4 at different concentrations i.e. 100, 150, 300, 500 and 900 mg/l.

Experimental programme

The influence of H_2SO_4 at different concentrations was studied when the H_2SO_4 is spiked with deionised water. Test samples are compared with the control samples. This comparison may not be possible in case of control samples made with locally available potable water since it varies in chemical composition from place to place. With the above reason, H_2SO_4 was mixed with deionised water as per the dosage mentioned above. This water is used for preparation of samples for setting times (initial & final) of BC and its concrete namely BCC. The IS mix design is adopted for concrete mix.

Table 1: Chemical composition of blended cement (Fly ash based)

Sl. No.	Parameter	Result
1.	Insoluble Material (% by mass)	18.90
2.	Magnesia (% by mass)	0.99
3.	Sulphuric Anhydride (% by mass)	2.67
4.	Loss on Ignition (% by mass)	2.04
5.	Total Chlorides (% by mass)	0.001

Table 2: Characteristics of Silica Fume

S. No.	Constituent	Percent content
1	SiO_2	95.65
2	$Al_2 O_3$	0.23
3	Fe_2O_3	0.07
4	CaO	0.31
5	MgO	0.04
6	$S O_3$	0.17
7	Na_2O	0.15
8	K_2O	0.56
9	LOI	2.27
10	Specific surface area m^2/g	16.7

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For determining the initial and final setting times of cement, Vicat apparatus is used and to assess the compressive strength, 30 cubes of size 150x150x150mm for concrete were cast and tested. To determine the chloride ion permeability, RCPT was used, for which15 specimens were cast and tested.

RESULTS AND DISCUSSION

Effect on setting times of Blended Cement (BC)

The effect of H_2SO_4 on initial and final setting times is shown in Table 3, from which it is observed that both initial and final setting times got retarded with increase in H_2SO_4 concentration in deionised water. IS 456-2000 (Clause 5.4.1.3) stipulates that, when the difference in setting time(s) is less than 30 minutes, the change is considered to be negligible or insignificant and if it is more than 30 minutes, the change is considered to be significant. From the experimentation work it was observed that, when the H_2SO_4 concentration exceeded 300 mg/l, the retardation for initial and final setting times was significant (i.e., more than 30 minutes),..

Table 3: Variation of setting times of BC corresponding to H_2SO_4 levels

Sl. No.	Water sample	Setting time in minutes & Percentage change			
		Initial	% change	Final	% change
1	Deionised water (Control)	133	--	361	--
2	100 mg/l	142	6.42	374	3.53
3	150 mg/l	156	16.93	383	5.97
4	**300 mg/l**	**167***	**25.61**	**394***	**9.01**
5	500 mg/l	176	32.13	412	14.1
6	900 mg/l	188	41.39	444	23.06

***- *Significant*

Table 4: Compressive strength of Blended Cement Concrete corresponding to H_2SO_4 concentrations

Sl. No	Water Sample	Blended Cement Concrete			
		Compressive Strength		% variation	
		28 days	90 days	28 days	90 days
1	Deionised Water (Control)	23.89	27.47	--	--
2	100 mg/l	23.34	26.46	-2.29	-3.69
3	150 mg/l	23.03	26.06	-3.58	-5.12
4	300 mg/l	21.40	24.36	-10.42	-11.33
5	500 mg/l	20.52	23.26	-14.1	-15.31
6	900 mg/l	18.71	21.30	-21.67	-22.45

When sulphuric acid content is 900 mg/l (Maximum), initial setting time was 188 minutes which is 55 minutes more than that of control mix. Similarly a difference of 83 minutes was observed in the case of final setting time

Effect on Compressive Strength of Blended Cement Concrete (BCC)

The effect of H_2SO_4 concentration on the compressive strength Blended Cement Concrete (BCC) is presented in Table 4. Decrease in compressive strength of specimens prepared with H_2SO_4 solution is observed as the sulphuric acid concentration increases, the maximum concentration being 900 mg/l. Although there is decrease in the compressive strength of concrete cubes of all age samples, significant decrease is observed at a concentration of 300 mg/l. The rate of decrease in compressive strength also gradually increases with increase in the concentration of the H_2SO_4. When H_2SO_4 concentration is maximum, i.e., 900 mg/l, the decrease in compressive strength is 21.67% for 28 day concrete and 22.41% for 90 day concrete respectively, when compared with that of cubes prepared with the deionised water (control test sample).

Pacheco-Torgal and Jalali (2009) studied the effect of sulphuric acid resistance on plain, polymer modified and fly ash cement concretes. Samples prepared by them were exposed to sulphuric acid after 28 days of curing. The results revealed that the use of polymers impregnation enhances chemical resistance of concrete considerably. It was also observed that the fly ash cement concrete shown a noteworthy resistance vis-a-vis the plain concrete. Similar observations were also noticed in the present experimental investigation.

Effect of H_2SO_4 on Chloride ion Permeability

The rapid chloride permeability levels in terms of coulombs passed through BCC observed are tabulated and listed in the Table 5. A glance at the said results establishes that the chloride ion permeability of the concrete studied has increased with the increase in the concentration of H_2SO_4 up to 900 mg/l which is the maximum experimented concentration. Quantum of variation in coulombs passed is 20.21% at 28 days for BCC when compared with the control sample i.e has increased from 2036 to 2447 coulombs.

Parande *et al.,* (2011) reported same behavior with respect to chloride permeability on cement mortars. Based on their studies, it was noticed that the chloride permeability (electric charge (coulombs)) was more in OPC than PPC. The permeability results reflect the interconnected pores network of concrete in which ions migrate.

Table 5: Chloride ion permeability in terms of coulombs passed in BCC corresponding to H_2SO_4 concentrations

Sl. No.	**Water sample**	**Coulombs passed**			
		28 days	% change	90 days	% change
1	Deionised water (Control)	2036	--	1187	--
2	100 mg/l	2085	2.41	1231	3.71
3	150 mg/l	2103	3.27	1252	5.49
4	300 mg/l	2265	11.24	1321	11.27
5	500 mg/l	2357	15.79	1371	15.47
6	900 mg/l	2447	20.21	1445	21.71

The use of porous concrete would not lead to lower permeability of concrete. If the interfacial between aggregate and cement is intact then the lesser pores are formed which will lead minimal migration of ions, ultimately resulting in lower permeability. This mechanism may be one of the reasons to get lower chloride permeability in BCC. Pengfei *et al.,* (2005) also observed similar trend of increased porosities i.e

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increase in chloride ion permeability for different grades of concretes. The permeability of concrete is mainly attributed to the diffusivities of aggressive ions in concrete, which depends on material microstructure and fluid properties. However, in general, chemical reactions are much faster than the diffusion rate, and thus, the overall rate of the degradation processes will be governed by the slower diffusion of one of the species (reactant or product).

Powder X-ray Diffraction Analysis

Powder X-Ray Diffraction pattern shown in Fig 1 is for BCC specimen prepared with H_2SO_4 (300 mg/l) in deionised water. Upper portion of the said graph indicates the XRD pattern of the control sample prepared with deionised water. A look at the said graph establishes that the compounds such as Ettringite, C_2S, C_3S, Calcium Hydroxie (CH), C-H-S, Calcium Sulphate and M-S-H are found at 41^0, 16^0, 17^0, 21^0, 31.8^0, 22.3^0 and 55^0 respectively. Comparing with control sample, the sample of $MgSO_4$ additionally produced Ettringite, M-S-H and Calcium Sulphate.

Setting times of the BC were observed to get retarded with the increase in H_2SO_4 concentration in the mixing water. Chemical equations when H_2SO_4 is added in mixing water with cement are given below.

$$2H_2SO_4 + 2Ca(OH)_2 \rightarrow 2CaSO_4 + 4H_2O$$

$$3CaSO_4 + Al_2O_3 + 32\ H_2O \rightarrow 3CaSO_4.Al_2O_3.32H_2O$$

Compressive strength has decreased with an increase in the concentration of H_2SO_4. The XRD patterns indicate that the formation of ettringite and M-S-H takes place which decreases the compressive strength of the concrete. When H_2SO_4 is added in the mixing water, it reacts with $Ca(OH)_2$ resulting in the formation of $CaSO_4$. Further, $CaSO_4$ reacts with C_3A resulting in ettringite production. Ettringite has expansive in nature, hence compressive strength is decreased. Apart from this, with the action of said chemical porosity increases which reduces compressive strength.

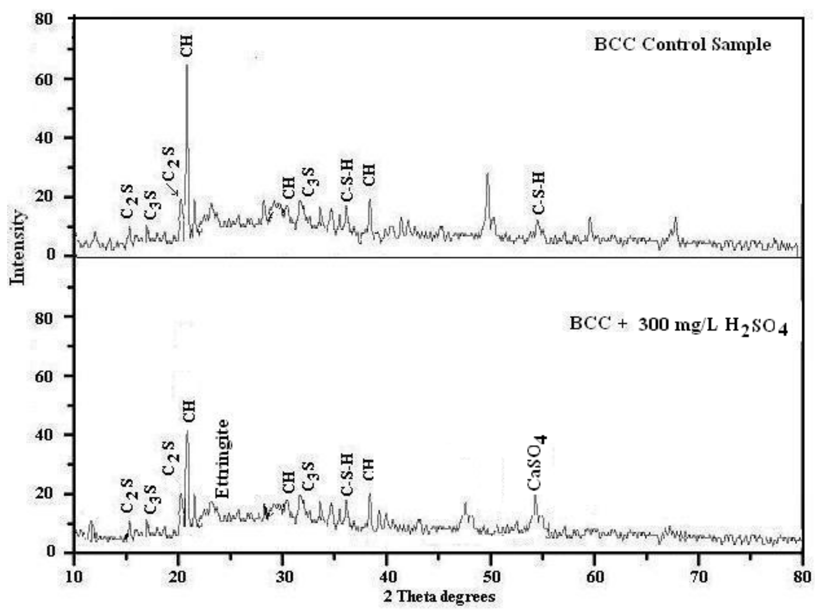

Figure 1: X-Ray diffraction pattern of powdered BCC sample prepared with H_2SO_4 (300 mg/l) in deionised water

Conclusions

The following observations were made from the experimental work.

- Both initial and final setting times of BC got retarded with an increase in sulphuric acid concentration in deionised water.

- The retardation for initial and final setting times is significant (i.e., more than 30 minutes), when the acid content exceeds 300 mg/l.
- Continuous decrease in compressive strength BCC specimens prepared with H_2SO_4 acid solution is observed as the acid concentration increases till the maximum concentration (900 mg/l) tested.
- Although there is decrease in the compressive strength of concrete cubes of all samples, significant decrease is observed only from concentration of 300 mg/l. The rate of decrease in compressive strength also gradually increases with the increase in the concentration of the H_2SO_4.
- Results indicated that the chloride ion permeability of BCC has increased with the increase in the concentration of H_2SO_4 up to 900 mg/l which is the maximum concentration.

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