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Influence of Strong Acids (Hydrochloric and Sulphuric) In Water on Properties of Natural Admixture Cements

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ABSTRACT: *Admixtures and the water have an important role on application of concrete. The study is aimed at investigating the effect of strong acids as mixing water on setting times, soundness and compressive strength of admixture cements. This paper therefore presents the result and findings of an experimental research on the influence of strong acids on setting and strength development of admixture cements. In the research PPC plus 10% silica fume was added by weight and cubes were casted with deionised water and deionised water containing strong acids of HCl and* H_2SO_4*. The results shows the HCl and* H_2SO_4 *in deionised water accelerate the intial and final setting times in all concentrations. Both HCl and* H_2SO_4 *in deionised water decrease the compressive strength from early age (3-days) to the maximum age (2years). In the present work analysis the hydration characteristics of the admixture cements using the techniques of X-ray diffraction analysis and useful conclusions are obtained regarding the influence of strong acids.*

Key words: *PPC, Silica fume, setting times, strength development, X-ray diffraction*

The presence of acids in water changes the properties of concrete in setting times as well as strength. Water is an essential ingredient of concrete as it effectively participates in the chemical reactions with natural admixture cements like natural pozzolana and other supplementary cementitious materials (Silica fume). Pozzolans are commonly used as an addition to Portland cement to increase the long-term strength and other material properties. Concrete is not only material that is risk to physical and chemical process of deterioration associated with water. Therefore, it will be desirable to review, in general, the characteristics of water that make it the principal agent of destruction of material. The I.S.Code 456-2000 also specifies the minimum pH-value as 6.0 and also permissible limits for solids in the water to fit for construction purposes. The code has not specified the limits to the individual components like acidic substances. The use of natural and economical materials seems to be one of the possible solutions for the future. The development of an economical cement concrete with interesting properties in the fresh and hardened state will certainly help and encourage the use of this material in the construction industry. Hence, in the present investigation to find the effects and quality of water on setting and strength properties of admixture cement. The effect of strong acidic substances on setting, hardening and strength development of admixture cement are not known much. Hence, an investigation is carried out on setting time, soundness and strength of admixture cements.

I. Materials and Methods

Materials: The details of various materials used in the experimental investigation are presented below.

Cement: The cement used in the present investigation is of 43 grade Pozzolana Portland Cement manufactured by ACC Ltd.

Fine aggregate: The fine aggregate used in this investigation is the river sand obtained from Swarnamukhi River near Tirupati, Chittoor district in Andhra Pradesh.

Silica fume: Silica fume used in the present study was obtained from Elkem India Pvt. Ltd., Mumbai.

Water: Deionised water spiked with strong acidic substances (H_2SO_4 and HCl) with different concentration is used in mixing water.

Experimental System: The following equipment is used for casting and testing of specimens: (i)Cube moulds, (ii) 200T U.T.M(Universal Testing Machine) for cube compressive strength determination,(iii)Vicat's apparatus including moulds conforming to IS4031(part-5)-1988 for setting times, (iv)Le-Chatelier's equipment to determine the soundness of cement and (v) cement cubes prepared with water containing, H_2SO_4 in the concentration of 50 mg/L,150 mg/L,200 mg/L,500 mg/L and 800mg/L and HCl, in the concentration of 50 mg/L,150 mg/L,200 mg/L,500 mg/L and 800mg/L, in mixing water.

Setting time: Vicat's apparatus confirming IS4031(part-5) 1988 consist of a frame to which a movable rod having an indicator is attached which gives the penetration, weighing 100g and having diameter and length of 10mm and 50mm respectively. Vicat's apparatus included three attachments- square needle for intial setting time, plunger for determining normal consistency and needle with annular collar for final setting time.

Compressive Strength: The test specimens for determination of compressive strength of admixture cement prepared using standard metallic cube moulds adopting IS procedure for the compactions. The cubes were demoulded after 24hours of casting and cured in water having similar quality as used in preparation of mix. The cubes are tested for compressive strength for short term and long term. The compressive strength is computed as the average value of the three samples.

II. Results and Discussion

The results of the present investigation are presented both in tabular and graphical forms. In order to facilitate the analysis, interpretation of the results is carried out at each phase of the experimental work. This interpretation of the results obtained is based on the current knowledge available in the literature as well as on the nature of result obtained. The significance of the result is assessed with reference to the standards specified by the relevant I S codes;

1. The averages of both the initial and final setting times of three cement samples prepared with mixing water containing typical chemical or biological component of varying concentrations under consideration is compared with those of the cement specimens prepared with deionised water. If the difference 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.
2. The average compressive strength of at least three cubes prepared with water under consideration is compared with that of three similar cubes prepared with deionised water. If the difference in the strength is less than 10%, it is considered to be insignificant and if it is greater than 10%, it is considered to be significant.

Setting time:

Hydrochloric Acid (HCl):

The effect of hydrochloric acid on the initial and final setting times is shown in Fig 1. Both initial and final setting got retarded with increase in hydrochloric acid concentration in deionised water. The retardation for initial and final setting times is significant (i.e., more than 30 minutes), when the hydrochloric acid content exceeds 150mg/l and 200 mg/l respectively. When the acid content is 800 mg/l, the initial setting time is about 192 minutes, which is 64 minutes more than that of the control mix. Similarly a difference of 44 minutes is observed in the case of final setting time.

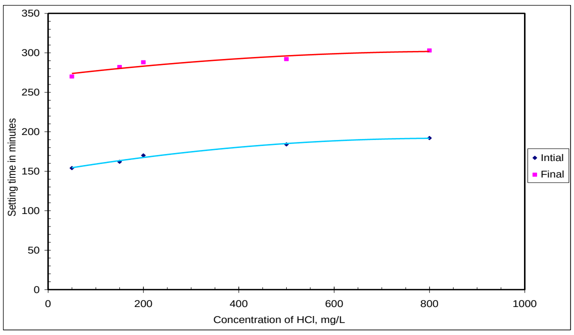


Fig.1 Variation of Setting times of admixture cement (PPC cement + 10% Silica fume) corresponding to various concentrations of HCl in deionised water.

Sulphuric Acid (H_2SO_4):

The effect of sulphuric acid on the initial and final setting times is shown in Fig 2. Both initial and final setting got retarded with an increase in sulphuric acid concentration in the deionised water. The retardation for initial setting time is significant, when the sulphuric acid concentration exceeds 150 mg/L respectively. When sulphuric acid content is 800 mg/L, initial setting time is 202 minutes which is 74 minutes more than that of control mix; similarly difference of 53 minutes is observed in the case of final setting time.

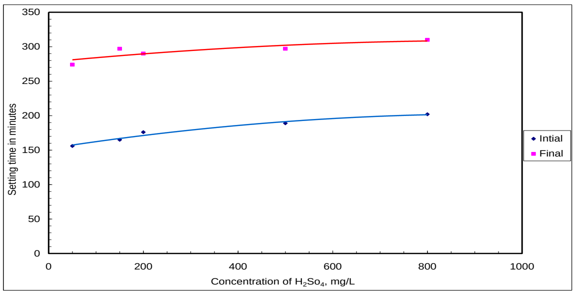


Fig.2. Variation of Setting times of admixture cement (PPC cement + 10% Silica fume) corresponding to various concentrations of H_2So_4in deionised water.

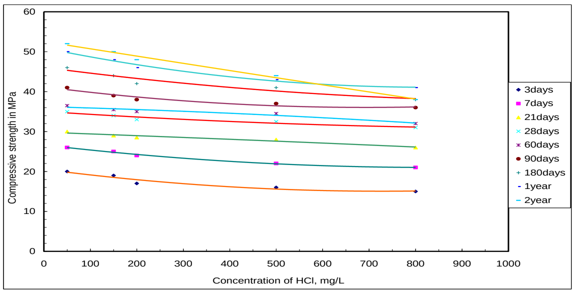


Fig.3. Variation of compressive strength of admixture cement (PPC cement + 10% Silica fume) mortar cubes at different ages corresponding to various concentrations of HCl in deionised water.

The effect of HCl on the compressive strength of admixture cement mortar is presented in Fig 3. Continuous decrease in compressive strength of the cement mortar cubes prepared with HCl acid solution is observed as the acid concentration increases till the maximum concentration (800 mg/L) tested.

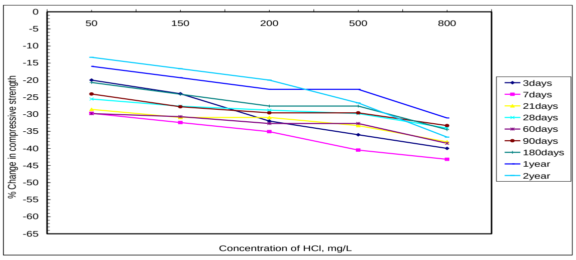


Fig.4. Percentage variation of compressive strength of admixture cement (PPC cement + 10% Silica fume) mortar cubes at different ages corresponding to various concentrations of HCl in deionised water.

The percent change in compressive strength is presented in Fig 4. Although there is decrease in the compressive strength of cement mortar cube of all age samples, significant decrease is observed in all age samples. The rate of decrease in compressive strength also gradually increases with the increase in the concentration of the HCl as well as age. For the 3-day sample, significant decrease in strength starts at 200 mg/L. Similarly, for 28-day,60-day, 90-day, 180-day,1-year and 2- year samples, significant decrease in strength starts at 200 mg/L, 500 mg/L concentrations respectively and the trend continues up to the maximum concentration. 2-year sample shows the maximum decrease in compressive strength with increase in concentration of HCl. When HCl concentration is 800 mg/L, the decrease in compressive strength is 36.70 % than that of the control mix.

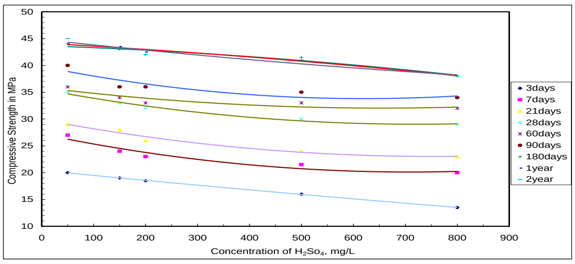


Fig.5. Variation of compressive strength of admixture cement (PPC cement + 10% Silica fume) mortar cubes at different ages corresponding to various concentrations of H_2So_4 in deionised water.

The effect of H_2SO_4 on the compressive strength of cement mortar cubes is presented in Fig.5. Decrease in compressive strength of the cement mortar cubes prepared with H_2SO_4 solution is observed as the sulphuric acid concentration increases, the maximum concentration being 800 mg/l.

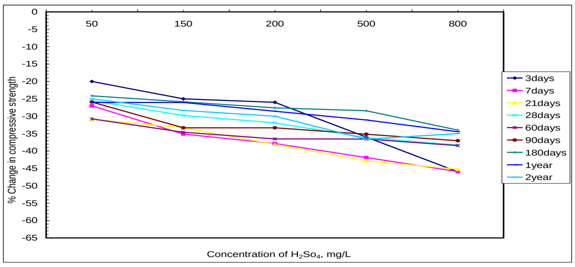
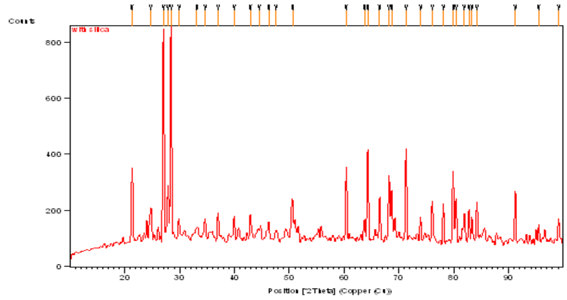


Fig.6 Percent variation of compressive strength of admixture cement (PPC cement + 10% Silica fume) mortar cubes at different ages corresponding to various concentrations of H_2So_4in deionised water.

The percent change in compressive strength of mortar cubes prepared with various concentration of H_2SO_4 in deionised water is shown in Fig 6. Although there is decrease in the compressive strength of cement mortar cube of all age samples, significant decrease is observed in all age samples. The rate of decrease in compressive strength is normally varies with the increase in the concentration of the H_2SO_4 as well as age. And the trend continues up to the maximum concentration. 2-year sample shows the maximum decrease in compressive strength with increase in concentration of H_2SO_4. When H_2SO_4 concentration is 800 mg/L, the decrease in compressive strength is 35 % when compared with that of the control mix.

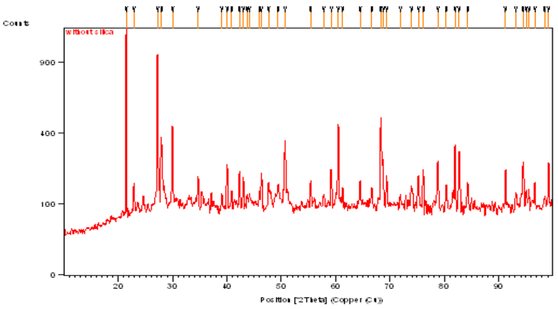
III. X-RAY DIFFRACTION



Peak List

Pos.[°2Th	Height[ct	FWHM[°2Th	d-	Rel.Int.[
21.2999	284.22	0.2854	4.1681	34.84
24.6774	108.28	0.4955	3.60474	13.27
27.044	808.37	0.2522	3.29443	99.08
27.8776	207.59	0.2099	3.19779	25.45
28.4259	815.83	0.2414	3.13734	100
29.8629	61.46	0.017	2.98956	7.53
33.171	21.87	1.773	2.69859	2.68
34.6229	69.14	0.3235	2.58867	8.48
36.999	95.41	0.3082	2.42769	11.69
39.9368	86.14	0.2457	2.25562	10.56
42.9052	94.01	0.2153	2.10619	11.52
44.5205	26.66	1.9653	2.03345	3.27
46.209	62.09	0.3433	1.963	7.61
47.5966	1.82	0.0013	1.90896	0.22
50.5851	112.53	0.5774	1.80296	13.79
60.3873	271.37	0.2481	1.53164	33.26
63.6841	71.39	0.2107	1.46006	8.75
64.2899	349.65	0.2524	1.44776	42.86
66.4734	167.42	0.2302	1.4054	20.52
68.16	278.63	0.1668	1.37467	34.15
68.6174	127.28	0.8095	1.36662	15.6
71.3241	356.41	0.2348	1.32126	43.69
73.9238	51.08	0.6812	1.28109	6.26
76.0972	158.41	0.2823	1.24982	19.42
78.0615	135.48	0.2351	1.22321	16.61
79.882	266.63	0.2495	1.19985	32.68
80.4209	154.97	0.2624	1.19316	18.99
81.8855	95.91	0.3066	1.17548	11.76
82.7426	103.41	0.2265	1.16547	12.68
83.3019	89.55	0.202	1.15906	10.98
84.2383	135.62	0.2587	1.14854	16.62
91.1915	195.95	0.2402	1.07822	24.02
95.4305	30.71	0.952	1.04121	3.76
99.0816	87.25	0.2877	1.0124	10.69

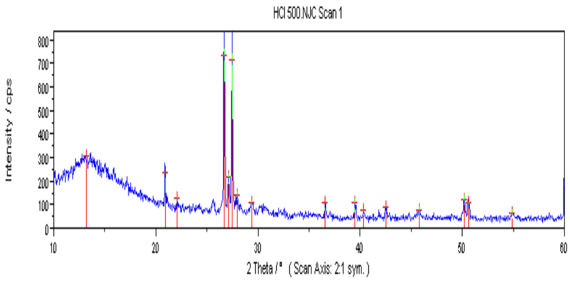
Fig.a. X-ray diffraction pattern of powdered admixture cement (PPC cement +10% silica fume) mortar cube prepared with deionised water.



Peak List

Pos.[°2Th	Height[ct	FWHM[°2Th	d-	Rel.Int.[
21.437	1171.43	0.3008	4.1418	100.00
22.888	86.60	0.1805	3.8824	7.39
27.210	897.90	0.2406	3.2747	76.65
27.914	284.63	0.2406	3.1937	24.30
29.965	345.90	0.3008	2.9796	29.53
34.701	91.95	0.2406	2.583	7.85
39.052	45.30	0.361	2.3047	3.87
40.027	156.15	0.2406	2.2508	13.33
40.831	93.60	0.122	2.2082	7.99
42.307	120.72	0.3008	2.1346	10.31
42.999	92.95	0.1805	2.1018	7.93
43.745	64.71	0.1498	2.0677	5.52
44.137	62.00	0.1389	2.0502	5.29
46.003	89.30	0.1348	1.9713	7.62
46.352	116.21	0.1805	1.9573	9.92
47.667	76.26	0.361	1.9063	6.51
49.402	88.41	0.3519	1.8433	7.55
50.675	254.95	0.2406	1.8	21.76
55.387	84.72	0.2406	1.6575	7.23
57.805	70.69	0.1562	1.5938	6.03
59.194	128.95	0.2406	1.5596	11.01
60.475	358.90	0.2406	1.5296	30.64
61.274	103.90	0.101	1.5116	8.87
64.471	86.67	0.2406	1.4441	7.40
66.641	56.12	0.361	1.4023	4.79
68.282	396.76	0.2406	1.3725	33.87
68.742	241.70	1.6638	1.3645	20.63
69.355	100.93	0.2406	1.3539	8.62
71.922	61.25	0.1721	1.3118	5.23
73.960	57.10	0.4813	1.2806	4.87
75.247	106.14	0.3008	1.2618	9.06
76.135	132.14	0.1805	1.2493	11.28
78.772	171.85	0.2406	1.214	14.67
80.324	75.96	0.3008	1.1944	6.48
81.939	251.31	0.2406	1.1749	21.45
82.719	221.84	0.3008	1.1657	18.94
84.281	85.40	0.2406	1.1481	7.29
91.240	137.60	0.2406	1.0778	11.75
93.175	50.58	0.361	1.0604	4.32
94.473	171.35	0.3008	1.0492	14.63
95.057	91.55	0.1539	1.0443	7.82
95.516	106.10	0.1072	1.0405	9.06
96.651	87.40	0.2406	1.0313	7.46
98.430	77.50	0.1145	1.0173	6.62
99.141	168.76	0.2406	1.012	14.41

Fig.b. X-ray diffraction pattern of powdered PPC mortar cube prepared with deionised water.



No	d_Fit (A1)	Ang-parab	Ang-COG	Low Limit	Upp. Limit	I-net	I-bgr	FWHM	2-Theta
1	6.5094	13.5922	13.2283	10.0500	20.8000	308.41	0.00	7.1856	13.5922
2	4.2462	20.9038	20.9076	17.2500	22.0000	238.14	0.00	0.1787	20.9038
3	4.0188	22.1007	22.0881	21.0500	25.4500	125.72	0.00	18.7403	22.1007
4	3.3367	26.6954	26.6991	26.2500	26.9000	736.76	0.00	0.1412	26.6954
5	3.2835	27.1361	27.1222	26.9000	27.3000	219.26	0.00	0.2423	27.1361
6	3.2452	27.4626	27.4595	27.2500	27.6500	712.83	0.00	0.1377	27.4626
7	3.1857	27.9857	27.9803	27.6500	29.2500	143.40	0.00	1.0316	27.9857
8	3.0315	29.4403	29.3970	28.3500	30.1000	108.48	0.00	25.9794	29.4403
9	2.4530	36.6038	36.5903	34.2500	39.4000	107.85	0.00	0.2121	36.6038
10	2.2781	39.5262	39.4987	37.1500	40.2500	108.91	0.00	0.2735	39.5262
11	2.2334	40.3514	40.3364	39.7000	41.7500	73.72	0.00	0.8347	40.3514
12	2.1240	42.5273	42.5229	41.9000	45.7500	88.66	0.00	0.2558	42.5273
13	1.9802	45.7854	45.7995	42.6000	49.9500	76.19	0.00	1.1762	45.7854
14	1.8162	50.1912	50.2025	45.9000	50.4500	118.21	0.00	0.3999	50.1912
15	1.8025	50.6000	50.5988	50.3000	54.8000	106.00	0.00	0.3325	50.6000
16	1.6709	54.9054	54.9069	50.8000	59.8500	65.67	0.00	1.8828	54.9054

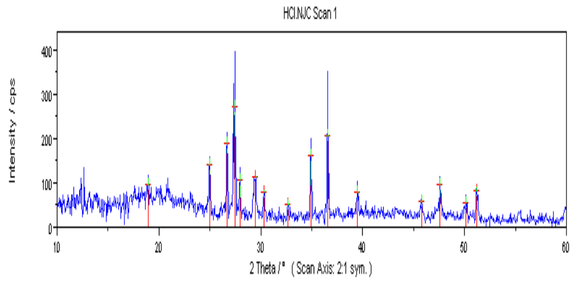


Fig.7. Powdered X- Ray diffraction pattern for the cement mortar cubes prepared with HCl (500 mg/L and 800 mg/L) in deionised water.

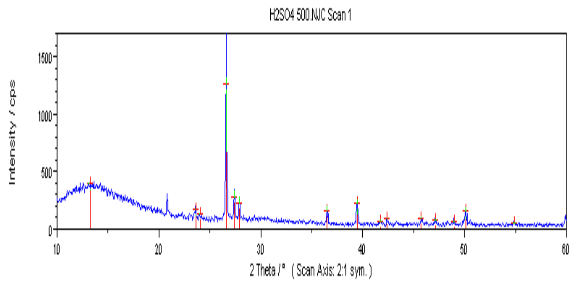
Powdered X- Ray diffraction pattern for the admixture cement (PPC+10% Silica fume) mortar cubes prepared with HCl (500 mg/L and 800 mg/L) in deionised water is shown in fig.7.

The comparison of this pattern with that for deionised water indicates the formation compounds α-C2S hydrate ($Ca_2(HSiO_4)(OH)$),$CaCl_2$ and $AlCl_3$ which are evident from the presence of sets of d-spacing's 1.670Å for ($Ca_2(HSiO_4)(OH)$,6.509Å, 4.246Å and 3.245Å for Cacl2 and 3.336Å for $AlCl_3$ respectively, which are absent in the matter for the control mix(Fig a and Fig b).

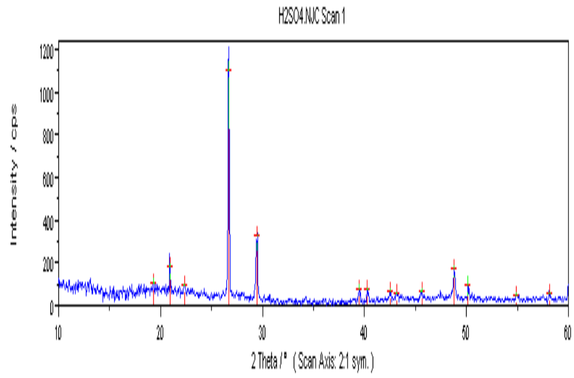
The possible chemical reaction upon the hydration of cement with mixing water containing HCl is

3Cao + Al_2O_3 + SiO_2 + 8 HCl→$Ca_2(HSiO_4)(OH)$ + $CaCl_2$ + $2AlCl_3$ + $2H_2O$

The reason for the retardation of setting time of cement could be attributed to the formation of α-C_2S hydrate (Ca_2 ($HSiO_4$) (OH)). The reaction of cement with water containing HCl leads to the formation of chlorides of calcium and aluminum in addition to α-C_2S Hydrate ($Ca_2(HSiO_4)(OH)$). Among these, chlorides of calcium and aluminum are soluble. Even silica forms a colloidal solution. Significant decrease in compressive strength at longer duration, i.e., 21-day onwards, could be due to the formation of soluble chlorides.



N	d_Fit	Ang-	Ang-	Low	Upp.	I-net	I-bgr	FWH	2-
1	6.752	13.10	13.27	10.10	20.70	397.6	0.00	7.977	13.10
2	3.769	23.58	23.60	23.05	25.45	173.8	0.00	0.542	23.58
3	3.709	23.96	24.03	23.70	26.25	126.6	0.00	17.15	23.96
4	3.347	26.60	26.60	26.25	27.15	1256.	0.00	0.134	26.60
5	3.250	27.41	27.39	26.80	27.75	282.6	0.00	0.212	27.41
6	3.195	27.89	27.90	27.60	34.75	225.7	0.00	0.243	27.89
7	2.457	36.53	36.50	34.25	39.30	159.6	0.00	0.183	36.53
8	2.281	39.46	39.44	39.00	39.85	223.3	0.00	0.213	39.46
9	2.161	41.76	41.75	39.65	42.25	72.00	0.00	0.665	41.76
10	2.129	42.41	42.38	41.90	43.20	88.80	0.00	0.291	42.41
11	1.981	45.75	45.75	42.80	47.00	96.80	0.00	0.336	45.75
12	1.927	47.10	47.11	45.95	48.20	81.67	0.00	0.424	47.10
13	1.859	48.94	48.95	47.35	49.75	72.34	0.00	0.749	48.94
14	1.819	50.09	50.10	49.65	51.35	154.0	0.00	0.332	50.09
15	1.672	54.85	54.90	53.80	58.30	57.20	0.00	1.877	54.85



N	d_Fit	Ang-	Ang-	Low	Upp.	I-net	I-bgr	FWH	2-
1	4.5955	19.298	19.314	16.850	20.800	107.32	69.6	0.7462	19.298
2	4.2453	20.908	20.909	16.850	22.300	182.13	36.3	0.1398	20.908
3	3.9720	22.364	22.363	21.050	24.800	95.87	25.4	0.6246	22.364
4	3.3403	26.665	26.683	26.500	26.850	1104.1	32.4	0.1423	26.665
5	3.0306	29.449	29.457	29.150	29.650	327.98	34.9	0.1813	29.449
6	2.2798	39.495	39.504	39.300	39.700	79.13	16.7	0.2497	39.495
7	2.2356	40.310	40.284	40.150	40.500	73.18	14.1	0.2290	40.310
8	2.1244	42.518	42.512	41.850	43.100	63.51	6.62	0.3539	42.518
9	2.0944	43.159	43.202	42.650	45.400	53.93	4.13	0.6775	43.159
10	1.9870	45.618	45.653	45.250	48.600	67.13	0.00	0.5764	45.618
11	1.8644	48.806	48.778	48.150	49.300	175.24	0.00	0.2658	48.806
12	1.8171	50.165	50.156	49.700	50.900	95.75	0.00	0.2424	50.165
13	1.6707	54.910	54.912	53.650	58.000	53.12	0.00	0.5028	54.910
14	1.5852	58.148	58.140	57.950	59.850	60.55	0.00	0.3299	58.148

Fig.8. Powdered X- Ray diffraction pattern for the admixture cement (PPC+10% Silica fume) mortar cubes prepared with H_2SO_4 (500 mg/L and 800 mg/L) in deionised water.

Powdered X- Ray diffraction pattern for the admixture cement (PPC+10% Silica fume) mortar cubes prepared with H_2SO_4 (500 mg/L and 800 mg/L) in deionised water is shown in *fig.8*.

The comparison of this pattern with that cubes prepared with deionised water indicates the formation of α-C_2S(α-Ca_2SiO_4),$CaSo_42H_20$ and $Ca(OH)_2$ compounds which are evident from the presence of sets of d-spacing's 3.195Å,1.981Å and 1.672Å for α-Ca_2SiO_4 , 4.245Å and 1.817Å for $CaSo_42H_20$ and 3.195Å and 2.457Å for $Ca(OH)_2$ respectively, which are absent in the matter for the control mix (Fig a and Fig b).

The possible chemical reaction upon the hydration of cement with mixing water containing H_2SO_4 is

4Cao + SiO_2 + H_2SO_4 + $2H_2O$→Ca_2SiO_4 + $CaSO_4$ $2H_2O$ + Ca $(OH)_2$

The possible reason for the retardation of setting of cement is the formation of $CaSO_4,2H_2O$. if the cement is mixed with H_2SO_4, it leads to formation of gypsum and calcium hydroxide, in addition to α–Dicalcium silicate (α-Ca_2 SiO_4). Among these, sulphate of calcium is soluble. Even silica forms a colloidal solution. Significant decrease in compressive strength at shorter duration may be due to the formation of soluble sulphates.

The sulphate-bearing water penetrates the hardened cement paste and the final damage to the hardened cement paste is due to the formation of voluminous water-rich calcium compounds. Calcium sulphate formation goes through a process leading to the reduction of stiffness and strength; this is followed by expansion and cracking, and eventual transformation of the material into a mushy or non cohesive mass at longer periods.

IV. Effect of Strong Acids

The strong acidic compounds generally present in water are HCl and H_2SO_4. The effects of these at various concentrations in deionised water on the initial and final setting times of cement and on the compressive strength of cement mortar cubes were already discussed in the above sub sections. The behavior of strong acidic compounds is elucidated in a comprehensive manner as follows:

Both HCl and H_2SO_4 in deionised water retard the initial as well as final setting times phenomena at all concentrations (Fig 1. and Fig 2.).

HCl in deionised water decreases the compressive strength of mortar cubes, the decrease being significant only at ages varying from 3–day to 2- year samples. H_2SO_4 also decreases compressive strength at all ages, is significant only at ages varying from 3-day to 2- year samples (Figs 3 to 6).

By comparing all these results of strong acidic compounds with those of the control mix, it is evident that both HCl and H_2SO_4 affect the compressive strength negatively. This negative effect increases with respect to increase inn concentration as well as duration. Hence, both HCl and H_2SO_4 are the most critical compounds against which a lot of care should be taken if they exist in the mixing water and their concentrations should not be more than 50 mg/L.

It is evident that between these two, the effect of H_2SO_4 is the more critical, against which great care is mandatory. Reaction with HCl leads to the formation of chlorides of calcium and aluminum, which are soluble. But the reaction with H_2SO_4 leads to formation of calcium sulphate and calcium hydroxide. Sulphate-bearing water penetrates the hardened cement paste and forms voluminous calcium compounds. Reduction in stiffness and strength along with expansion and cracking finally transforms the materials into a non cohesive mass at long duration. It is observed form the results that the strength of 2-year sample made with HCl is 36.67 %, which is comparatively higher than that of H_2SO_4 which is 35%.

V. CONCLUSIONS

1. Presence of HCl in water retards significantly the initial and final setting in concentrations more than 200mg/L. Its concentration higher than 200mg/L results in significant decrease in compressive strength.
2. Presence of H_2SO_4 in water retards significantly the initial and final setting in concentrations more than 200mg/L. Its concentration higher than 200mg/L results in significant decrease in compressive strength.
3. Strong acidic substances like HCl and H_2SO_4 in water reduce the compressive strength significantly with respect to increasing concentration as well as age. Thus, care to be taken while using such water for cement mortar and concrete.

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