# INFLUENCE OF ALKALINE SUBSTANCES (CARBONATES AND BICARBONATES OF SODIUM) IN WATER ON PROPERTIES OF NATURAL ADMIXTURE CEMENTS

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## Abstract

The impurities in water play a major role in application of concrete. This paper assesses the setting times and compressive strength of natural admixture cements and silica fume is used as an admixture. This study introduces the effect of strong alkalines (carbonates and bicarbonates of sodium) on setting time and compressive strength of natural admixture cements (PPC+SF) is assessed under laboratory conditions. The research programme included tests of soundness, setting times, compressive strength of short and long term and powdered x-ray diffraction analysis. As the experimental results show that  $Na_2CO_3$  in deionised water accelerates the initial and final setting times where as the other compound NaHCO<sub>3</sub> retards the initial and final setting times for all concentrations.  $Na_2CO_3$  and  $NaHCO_3$  in deionised water increase the compressive strength at early age (3-day and 7-day) and decrease significantly from28-day, 60-day, 90-day, 180-day, 1year and 2years. Comparison of the strong alkaline substances with those of control mix levels that both carbonates decrease compressive strength. The rate of decrease is increasing with increase in concentration .The present work analyses the hydration characteristics of admixture cement using X-ray diffraction analysis (XRD) and useful conclusions are obtained regarding the influence of alkaline substances.

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

# **INTRODUCTION**

The impurities in water can impose deleterious effect on concrete. This creates great concern on mixing of water in hydration of cement and the properties of resultant concrete. Most specifications on quality of water concentrate on the added water rather than the moisture in the aggregates. The presence of dissolved inorganic substances in ground water is mostly through mineral origin and would be expected in varied concentrations geographically. Cement used in concrete is a mixture of complex compounds. The reaction of these with water leads to setting and hardening of cement as well as the admixtures that are mixed with cement when it is gauged with water. Most of former specifications for concrete tend to discharge the requirements for mixing water with the stipulation that if the water is fit for drinking, it is acceptable for making concrete. This is hardly the best evaluation of an ingredient which contributes so significantly the hydration process of natural admixture (silica fume) cement. I.S.Code 456-2000 mentioned that the alkalinity of water should not be more than 250mg/L. However, many ground water and surface bodies contain more alkalinity than the limit specified by the I.S.Code. 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 alkaline 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.

# 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 alkaline substances (Na2CO3 and NaHCO3) 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, Na2CO3 in the concentration of 1,2,4,10 and 20g/L and NaHCO3, in the concentration of 1,2,4,10 and 20g/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.

# **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 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.

3. The powdered X-ray diffraction analysis reports prepared to identify the unknown compounds.

**Setting time**: The effect Na2CO3 on initial and final setting times is shown in the Fig 1.0. It is observed that the initial and final setting of cement got accelerated with increase of the sodium carbonate concentration in deionised water. The decrease in the initial and final setting times is significant only when the sodium carbonate concentration exceeds 4 g/L. The decrease in the initial setting time is about 59 minutes and final setting time

is about 69 minutes at the maximum concentration of 20 g/L when compared with that of the test blocks made with deionised water.





The effect of NaHCO3 on initial and final setting times is shown.in.fig.2.0. Both initial and final setting got retarded with an increase in sodium bicarbonate concentration in the deionised water. The retardation of initial and final setting is significant (i.e., more than 30minutes) when the sodium bicarbonate content exceeds 10g/L and 20g/L respectively. When the sodium bicarbonate content is 20g/L, the initial setting time is 85minutes more than that of control mix. The difference observed in the case of final setting time is 66 minutes with respect to the control mix.





## **Compressive strength:**

The effect of Na2CO3 concentration on compressive strength of cement mortar is presented in Fig 3.0. Decrease in the compressive strength is observed as the sodium carbonate concentration increases in deionised water, the maximum concentration studied being 20 g/L.



#### Fig-3. Percent variation of compressive strength of admixture cement (PPC cement + 10% Silica fume) mortar cubes at different ages corresponding to various concentrations of Na2Co3 in deionised water

The percent change in compressive strength of mortar cubes prepared with various concentrations of Na2CO3 solution in deionised water is shown in fig 3.0. There is significant decrease in the compressive strength of all cement mortar cubes at different ages ranging from 60 day to 2 year and the rate of decrease in compressive strength also gradually increases with the increase in the concentration of the Na2CO3. At early ages for the 3 day sample, there is a significant increase in strength, and when the concentration of Na2CO3 is more than 2 g/l. The significant decrease in strength, for 7day, 14 day, 21 day, 28 day,60 day, 90 day, 180 day,1 year and 2 year samples. The trend continues up to the maximum concentration tested. The decrease in the compressive strength is 25% for long duration i.e., for the 2 year sample at the maximum concentration of 20 g/L. The effect of NaHCO3 concentration on the compressive strength of cement mortar is presented in Fig.5.24. Decrease in compressive strength of the cement mortar cubes prepared with NaHCO3 solution is observed as the sodium bicarbonate concentration increases, the maximum concentration considered being 20g/L.



### 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 NaHCo3 in deionised water.

The percent change in compressive strength of mortar cubes prepared with various concentrations of NaHCO3 solution in deionised water is shown in Fig.4.0. There is significant decrease in the compressive strength of all cement mortar cubes of ages varying from 21-day to 2 year and the rate of decrease in compressive strength also gradually increases with the increase in the concentration of NaHCO3. For the 60-day sample, significant decrease in strength starts when concentration of NaHCO3 is more than 20g/L. Similarly for 7-day,21-day,28-day,60-day,90-day,180-day,1 year and 2 year samples, the significant decrease in strength starts at 4g/L,10g/L and 20g/L concentrations respectively. The 2 year sample shows the maximum decrease in compressive strength with the increase in concentration of NaHCO3. When NaHCO3 concentration is 20g/L, the decrease in compressive strength is 20% when compared with that of control mix.

# POWDER X-RAY DIFFRACTION ANALYSIS



Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	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





N	d_Fit	Ang-	Ang-	Low	Upp.	I-net	I-bgr	FWH	2-
1	4.873	18.18	18.12	14.750	19.650	81.0	69.03	2.131	18.189
2	4.246	20.90	20.90	20.750	21.100	370.	28.60	0.121	20.903
3	3.847	23.09	23.13	21.050	23.500	78.8	17.86	0.504	23.099
4	3.766	23.60	23.60	23.250	25.350	87.8	16.16	0.283	23.604
5	3.490	25.49	25.50	23.700	26.150	112.	11.09	0.295	25.499
6	3.335	26.70	26.70	26.500	26.900	733.	8.92	0.139	26.702
7	3.241	27.49	27.47	26.900	27.850	321.	7.88	0.189	27.493
8	3.185	27.98	27.96	27.700	28.550	239.	7.37	0.171	27.986
9	2.952	30.24	30.23	28.350	34.050	84.3	6.34	1.987	30.248
10	2.623	34.14	34.14	30.350	36.100	78.9	8.10	0.629	34.143
11	2.451	36.63	36.60	36.300	36.900	98.4	10.44	0.222	36.633
12	2.279	39.50	39.50	39.350	39.800	55.1	13.31	0.268	39.507
13	2.232	40.36	40.34	40.200	40.550	68.8	14.01	0.227	40.362
14	2.156	41.85	41.84	41.700	42.200	62.7	14.95	0.273	41.856

15	1.977	45.85	45.85	45.700	46.050	66.7	14.50	0.134	45.857
16	1.927	47.11	47.13	46.950	47.450	53.2	13.13	0.237	47.118
17	1.815	50.20	50.20	49.900	51.000	141.	7.10	0.223	50.208



No	d_Fit(A1)	Ang- parab	Ang- COG	Low Limit	Upp. Limit	I-net	I- bgr	FWHM	2-Theta
1	7.1613	12.3499	12.7725	10.1000	20.7500	305.94	0.00	6.9384	12.3499
2	4.2396	20.9365	20.9297	18.2000	23.5000	264.17	0.00	0.2152	20.9365
3	3.7584	23.6535	23.6573	23.5000	23.8000	679.73	0.00	0.1033	23.6535
4	3.6960	24.0588	24.0824	23.8500	25.4500	110.64	0.00	0.9311	24.0588
5	3.3581	26.5219	0.0000	23.8500	26.5000	136.13	0.00	0.3991	26.5219
6	3.3359	26.7018	26.7023	26.5000	26.9000	1528.96	0.00	0.1447	26.7018
7	3.2392	27.5141	27.5083	27.2000	27.8000	383.46	0.00	0.1747	27.5141
8	3.1855	27.9873	27.9724	27.7000	28.5500	499.24	0.00	0.1588	27.9873
9	3.0300	29.4555	29.4611	28.8000	34.0500	93.85	0.00	0.8891	29.4555
10	2.6229	34.1570	34.1618	31.0500	36.4500	78.69	0.00	1.2813	34.1570
11	2.4535	36.5964	36.6061	34.3500	39.4500	110.92	0.00	0.2577	36.5964
12	2.2778	39.5322	39.5052	37.1000	40.2500	80.48	0.00	0.6136	39.5322
13	2.2330	40.3595	40.3389	39.6500	41.6500	78.76	0.00	0.2833	40.3595
14	2.1245	42.5169	42.5022	41.9500	45.7500	88.05	0.00	0.7641	42.5169
15	1.9770	45.8638	45.8579	42.6500	47.1000	91.26	0.00	0.4038	45.8638
16	1.9255	47.1622	47.1652	46.0000	48.1500	65.33	0.00	1.2479	47.1622
17	1.8829	48.2968	48.3045	46.0000	50.0500	87.12	0.00	0.7659	48.2968
18	1.8158	50.2033	50.2051	48.4000	50.5000	143.11	0.00	0.2662	50.2033
19	1.8012	50.6388	50.6493	50.3500	54.8000	108.77	0.00	0.4903	50.6388
20	1.6697	54.9489	54.9537	53.1500	55.7500	156.27	0.00	0.1238	54.9489

Fig-6. Powder X-Ray diffraction pattern for the cement mortar cubes prepared with Na2CO3 in deionised water at the maximum concentration (10g/L and 20 g/L).

The Powder X-Ray diffraction pattern for the cement mortar cubes prepared with Na2CO3 in deionised water at the maximum concentration (10g/L and 20 g/L) is shown in fig.6

The comparison of this present pattern with that of deionised water indicates the formation of Ca4 Si6 O15 (OH)2 3H2O (Gyrolite) compound, which is evident by the d spacing's 3.335 Å, 3.1855Å, 1.669 Å, absent in that of the control mix. Further analysis of compounds formed in the X-Ray Diffraction pattern revealed the formation of Ca5 (Si6 O18 H2) 4H2O (11-Å Tobermorite) compound, which is evident by the presence of d-spacing's 1.925 Å, 1.815 Å and 1.669Å.

The probable chemical reaction upon the hydration of cement with mixing water containing Na2CO3 is

Na2CO3 + 5 CaO + 7 SiO2 + 4 H2O $\rightarrow$ Ca4 Si6 O15 (OH)2 3H2O + Na2SiO3 + CaCO3 ----(1)

The end compound formed i.e., CaCO3 may again participate in the chemical reaction as follows.

4CaO + 6SiO2 + 6H2O + CaCO3 → Ca5(Si6O18H2)4H2O + H2 CO3 -----(2)

The acceleration of setting of cement could be possibly attributed to the formation of Gyrolite. Continuous and significant decrease in the compressive strength for all samples ranging from 60 day to 2 year could be due to the formation of Gyrolite and sodium silicate. Alkali-silicate gel (Gyrolite) of variable chemical composition is formed in the presence of hydroxyl and alkali-metal ions. The mode of attack in the cement mortar involves depolymerization or break down of silica structure of the sand by hydroxyl ions followed by adsorption of the alkali-metal ions (Na2SiO3) on the newly created surface of the reaction product. Gyrolite gel when comes in contact with water; it swells by imbibing a large amount of water through osmotic process. The hydraulic pressure so developed leads to expansion and cracking of the cement paste matrix surrounding the sand, thus leading to the significant decrease in the compressive strength. The byproduct CaCO3 may again involves itself in the reaction with cement constituents in accordance with the equation (2) given to form Tobermorite and carbonic acid (H2CO3). These two byproducts may cause further decrease in the compressive strength of the mortar.



No	d_Fit(A1)	Ang- parab	Ang- COG	Low Limit	Upp. Limit	I-net	I- bgr	FWHM	2-Theta
1	6.4523	13.7130	12.9681	10.1000	20.7000	352.86	0.00	6.2645	13.7130
2	4.2485	20.8921	20.8640	18.1500	23.5500	502.36	0.00	0.1460	20.8921
3	3.7604	23.6410	23.6277	21.2000	26.3500	119.60	0.00	15.5163	23.6410
4	3.3349	26.7099	26.7075	26.4000	26.9000	1751.40	0.00	0.1326	26.7099
5	3.2350	27.5506	27.5487	27.3500	27.7500	1894.57	0.00	0.1374	27.5506
6	3.1797	28.0396	28.0224	27.7500	28.4500	814.40	0.00	0.2156	28.0396
7	2.8971	30.8394	30.7830	28.3500	32.1000	94.84	0.00	9.0009	30.8394
8	2.6235	34.1493	34.1371	32.6500	34.9000	86.69	0.00	0.7801	34.1493
9	2.5617	34.9996	34.9989	34.2500	36.4500	78.31	0.00	2.3988	34.9996
10	2.4522	36.6168	36.6004	36.1000	40.3000	127.60	0.00	0.2198	36.6168
11	2.2748	39.5868	39.5558	36.8000	41.6500	70.14	0.00	0.8710	39.5868
12	2.1574	41.8376	41.8127	40.5000	42.3500	86.17	0.00	0.6011	41.8376
13	2.1225	42.5588	42.5479	41.9500	45.8500	108.08	0.00	0.3689	42.5588
14	1.9751	45.9108	45.9040	42.7000	49.9000	75.80	0.00	0.4399	45.9108
15	1.8157	50.2046	50.2063	49.8000	51.5500	168.49	0.00	0.2302	50.2046
16	1.6696	54.9513	54.9550	51.4000	58.7000	117.24	0.00	0.2784	54.9513
17	1.5700	58.7655	58.7852	55.2000	59.6000	68.44	0.00	1.0727	58.7655



Ν	d_Fit	Ang-	Ang-	Low	Upp.	Inot	Ihan	FWH	2-
0	(A1)	parab	COG	Limit	Limit	1-net	1-ogr	М	Theta
1	6.363	13.90	13.01	10.10	20.75	328.1	108.1	5.485	13.90
1	0	10	51	00	00		7	1	10
2	4.243	20.91	20.93	13.95	23.40	423.0	94.56	0.191	20.91
3	3.758	23.65	23.65	21.10	25.10	281.3	89.22	0.126	23.65
4	3.475	25.61	25.61	23.80	26.40	127.6	85.43	0.604	25.61
5	3.333	26.72	26.70	26.55	26.90	1754.	83.29	0.154	26.72
6	3.239	27.51	27.51	27.30	27.75	1245.	81.74	0.167	27.51
7	3.183	28.00	28.01	27.80	28.20	1043.	80.77	0.158	28.00
8	2.450	36.63	36.60	34.25	38.00	97.43	63.96	0.258	36.63
9	2.277	39.54	39.52	39.35	40.00	93.95	58.32	0.262	39.54
1	2.231	40.38	40.35	40.05	40.60	70.52	56.67	0.462	40.38
1	2.157	41.83	41.83	40.65	42.30	75.83	53.85	0.511	41.83

1	2.125	42.50	42.51	42.25	43.00	166.0	52.59	0.205	42.50
1	1.974	45.91	45.91	43.50	48.50	82.42	45.98	0.316	45.91
1	1.861	48.88	48.83	48.50	50.05	103.7	40.25	0.299	48.88
1	1.815	50.20	50.20	49.90	50.60	180.6	37.63	0.230	50.20
1	1.669	54.95	54.95	54.60	55.95	132.5	28.40	0.099	54.95

**Fig-7**. Powder X-ray Diffraction pattern for the Admixture cement (PPC cement + 10% Silica fume) mortar cubes prepared with NaHCO3 in deionised water at maximum concentration (10g/L and 20g/L).

The Powder X-Ray diffraction pattern for the cement mortar cubes prepared with NaHCO3 in deionised water at the maximum concentration (10g/L and 20 g/L) is shown in fig.7

The comparison of this pattern with that of deionised water indicates the formation of Ca4Al2(OH)12CO36H2O (Tetra calcium aluminate carbonate 12-hydrate) compound which is evident from the presence of d-spacing's 3.448 Å,3.162 Å, 2.862Å, which are absent in the pattern for control mix.

The probable chemical reaction by the hydration of cement with mixing water containing NaHCO3 is

 $5CaO+Al2O3+2NaHCO3+11H2O+SiO2 \rightarrow Ca4Al2(OH)12CO36$ H2O+Na2SiO3+CaCO3 ----(1)

CaCO3 thus formed as byproduct, may again participate in the chemical reaction as follows

4CaO+6SiO2+6H2O+CaCO3→Ca5(Si6O18H2)4H2O+H2CO3 ----- (2)

One of the possible reasons for the retardation of setting of cement could be the formation of tetra calcium aluminate cabonate12-hydrate. Continuous and significant decrease in the compressive strength for all age samples ranging from 28-day to 2-year could be due to the formation of same tetra calcium aluminate carbonate 12-hydrate and sodium silicate. Alkalisilicate gel of variable chemical composition is formed in the presence of hydroxyl and alkali-metal ions. The mode of attack in cement mortar cubes involves depolymerization or break down of silica structure of the sand by hydroxyl ions followed by adsorption of the alkali-metal ions (Na2SiO3) on newly created surface of the reaction product. Tetra calcium aluminate carbonate 12-hydrate gel comes in contact with water and swells by imbibing a large amount of water through osmotic process. The hydraulic pressure so developed may lead to expansion and cracking of the cement paste matrix surrounding the sand and thus leading to the significant decrease in the compressive strength. The byproduct CaCO3 may again participate in the reaction with the cement constituent in accordance with the equation (2) given above to form Tobermorite and carbonic acid (H2CO3). These two byproducts may cause further deterioration of the compressive strength of mortar

## EFFECT OF STRONG ALKALINE SUBSTANCES:

The strong alkaline substances that are generally present in water are Na2CO3 and NaHCO3. The effect of these compounds in various concentrations in deionised water o the initial and final setting times of cement and compressive strength of cement mortar cubes have been already discussed in the above sub sections. The behavior of strong alkaline compounds is elucidated in a comprehensive manner as follows:

Na2CO3 in deionised water accelerates the initial as well as final setting where as other the compound NaHCO3 retards the initial and final setting (Fig 1.0 and Fig 2.0) in all concentrations.

Na2CO3 and NaHCO3 in deionised water decreases the compressive strength of mortar cubes significantly at all ages varying from 28 day to 2- year samples (Figs 3.0 and Fig 4.0). Comparison of the results of strong alkaline compounds with those of the control mix reveals that both Na2CO3 and NaHCO3 affect the compressive strength negatively. The negative effect goes on increasing with the increase in concentration as well as age. Both Na2CO3 and NaHCO3 are most critical compounds; therefore, a lot of care should be taken if they exist in the mixing water and their concentrations should not be more than 5 g/l in either case.

## CONCLUSIONS

Based on the results obtained in the present investigation the following conclusions can be drawn.

• Presence of  $Na_2CO_3$  in water in concentrations more than 4 g/L and 10 g/L accelerates significantly the initial and final setting respectively. Further, a concentration higher than 10 g/L results in significant decrease in compressive strength.

• Presence of NaHCO<sub>3</sub> in concentrations more than 10 g/L retards significantly the initial and final setting respectively. Further, a concentration higher than 10 g/L results in significant decrease in compressive strength.

• Strong alkaline substances under consideration  $(Na_2CO_3 \text{ and } NaHCO_3)$  in water reduce the compressive strength significantly right from the early age, thus requiring caution in the use of wafer containing these substances.

# REFERENCES

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