

# Performance of plain and blended cements exposed to high sulphate concentrations

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**Abstract:** The sulphate resistance of plain (ASTM C150 Type I and Type V) cements and cements blended with silica fume, fly ash, blast furnace slag or Superpozz, a new generation of supplementary cementing materials, exposed to sodium sulphate solutions was evaluated in this study. Cement mortar specimens were exposed to sulphate concentrations of up to 25000 ppm. The sulphate resistance of the selected cements was evaluated by visual examination and measuring expansion and reduction in the compressive strength. Morphological changes in cements, due to sulphate exposure, were evaluated by scanning electron microscopy. The mineralogical changes in cements exposed to a solution with 15 000 ppm sulphate were evaluated. Cracks were noted in Type I and silica fume cement mortar specimens exposed to a sulphate concentration of 15 000 ppm or more. In type V cement, the sulphate tolerance was 25000 ppm. Cracks were not noted in the blast furnace slag, fly ash, and Superpozz cement mortar specimens exposed to 25000 ppm sulphate solution. It is suggested to use Type V cement or Type I cement blended with fly ash, blast furnace slag or Superpozz in sulphate-bearing environments.

## Introduction

It is well known that sulphate ions in soil, groundwater and seawater can cause deterioration of reinforced concrete structures. They react with the cement hydration products, particularly calcium hydroxide (CH) to produce gypsum and calcium alumino-sulphate hydrate, or monosulphate ( $C_4ASH_{12}$ ), to form ettringite ( $C_6AS_3H_{32}$ ) [Note the use of cement nomenclature, i.e., C = CaO; A =  $Al_2O_3$ ; S =  $SO_3$ ; etc]. Both reactions are expansive and they contribute to strength loss and in extreme cases, lead to disruptive expansion and degradation of concrete into a non-cohesive granular mass. Ettringite formation is considered to be the cause of most of the expansion and disruption of concrete structures involved in external sulphate attack (ESA). ESA may be expected when

- (a) cement is susceptible to chemical reactions with sulphate
- (b) concrete is highly permeable
- (c) the environment is sulphate-rich
- (d) there is an ample supply of moisture.'

Two main chemical approaches are taken to control sulphate attack. The first is to limit the potential for formation of calcium alumino-sulphate hydrates by the use of a sulphate-resistant Portland cement (e.g., ASTM C 150 Type V). This cement has a low  $C_3A$  content and therefore, has a reduced potential for the formation of calcium alumino-sulphate hydrates compared with normal Portland cement (i.e., ASTM C 150 Type I). The second approach is to reduce the quantity of cement (and therefore the  $C_3A$  content) with supplementary cementing materials (SCMs). These materials react with CH derived from cement hydration to provide further pore-filling with calcium silicate hydrates and calcium aluminosilicate hydrates. This provides a further protection against aggressive sulphate attack by reducing interconnected porosity, thereby limiting the internal surface for reaction. SCMs conventionally used in concrete for enhancing the sulphate-resistance of cements are silica fume (SF), fly ash (FA), and blast furnace slag (BFS). BFS is not strictly a pozzolan

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because it has its own calcium content and does not require additional CH to react in the presence of water.<sup>2-5</sup>

There have been numerous studies highlighting the beneficial use of SCMs in enhancing the sulphate-resistance of concrete. Al-Amoudi *et al.*<sup>6</sup> reported a lower reduction in strength and reduced expansion in blended (FA, SF, and BFS) cement mortar specimens exposed to Na<sub>2</sub>SO<sub>4</sub> solution compared with plain cement mortar specimens. A good correlation between the degree of deterioration and ettringite content was observed by Wee *et al.*<sup>7</sup> Al-Dulaijan *et al.*<sup>8</sup> showed superior durability of a range of blended systems exposed to solutions representing highly saline soils compared with ordinary Portland cement. Similar observations were reported by Nehdi and Hayek<sup>9</sup> and Lee *et al.*<sup>10</sup>

Moon *et al.*<sup>11</sup> studied the influence of C<sub>3</sub>A content and silicate ratio (C<sub>3</sub>S/C<sub>2</sub>S) in Portland cements as well as the addition of SCMs on sulphate attack and concluded that the improved performance of blended cements was due to a reduction in the quantities of portlandite, gypsum, and ettringite, as well as the higher denseness of the cement matrix due to pozzolanic reaction and latent hydraulic property of mineral admixtures.

Goni *et al.*<sup>12</sup> investigated mixtures of two fly ashes (ASTM C618 class F) with 0, 15 and 35% replacement of Portland cement immersed in Na<sub>2</sub>SO<sub>4</sub> solutions for 90 days. Despite the high Al<sub>2</sub>O<sub>3</sub> content of the FA, the results showed that all the mixtures were sulphate-resistant; indicating that alumina derived from the FA cannot be considered as detrimental as alumina from the cement. Further, the study suggested that diffusion of SO<sub>4</sub><sup>2-</sup> and Na<sup>+</sup> ions through the pore solution activated the pozzolanic reactivity of the fly ashes, causing microstructural changes beneficial for improving their sulphate-resistance.

Kim *et al.*<sup>13</sup> reported improved performance of SF blended cement mortars relative to OPC in mixed sulphate solutions for 270 days. Lagerblad<sup>14</sup> confirmed superior performance of up to 5 years although he observed that expansion reactions continued. Largely, these conclusions were consistent with the studies of Lee *et al.*,<sup>10</sup> although the aggressiveness of magnesium sulphate solutions was highlighted.

In a study of FA and SF-substituted binders, Hassan *et al.*<sup>15</sup> observed that whereas SF contributed to both short- and long-term properties of concrete, FA required a relatively longer time to demonstrate its beneficial effect. This is consistent with the relative rates of hydration expected for these materials in cement systems.

It is evident from the cited literature that SCMs can contribute significantly to enhancing binder durability against sulphate attack by consuming deleterious CH via the pozzolanic reaction and by producing further C-S-H or C-A-S-H gel in the pore structure. Thus,

cements blended with SCMs give pastes with lower potential for expansive gypsum and ettringite formation, and they have typically lower levels of connected porosity than those derived from Portland cement alone, so that reduced levels of sulphate ion transport into the internal microstructure and reduced reactive surface can be expected.

The main factor that distinguishes these blending agents from each other is their reactivity in the cement system. Among the factors that control this are composition (they are all glassy silicate or aluminosilicate materials), glass content and particle size. Recently, a new pozzolanic material, known as Superpozz (SP), has been introduced in the market. SP is an ultra-fine fly ash with a mean particle size of 5 μm. This is effectively a fly ash subjected to additional grinding such that 90% of it passes an 11 μm sieve and 99% passes a 25 μm sieve. It is a highly reactive aluminosilicate pozzolan that adds strength to cementitious mixes and its fineness and spherical particle shape improves the workability of concrete.<sup>16,17</sup> In the context of FA fineness, Chindaprasit *et al.*<sup>18</sup> studied the performance of hardened cement mortars containing 40% FA. Significant improvement of resistance to sulphate expansion was obtained for all fineness values except for the coarse fly ash where greater expansion was observed.

In this paper, the performance of SP, as a cement replacement, is reported and compared with the other SCMs, with respect to their sulphate-resistance.

Santhanam *et al.*<sup>19,20</sup> suggest that the expansion of mortars in sodium sulphate solution follows a two-stage process. During stage 1, there is little expansion but this is followed by a sudden and rapid increase in the expansion in stage 2. Microstructural studies suggest that the onset of expansion in stage 2 corresponds to the appearance of cracks in the chemically unaltered interior of the mortar. Beyond this point, the expansion proceeds at an almost constant rate to complete deterioration of the mortar specimen. These ideas would also be examined in the light of the data developed by the authors.

## Experimental programme

### Materials

Six cement formulations were studied, namely Type I, Type V, Type I with 10% SP replacement, Type I with 8% SF replacement, Type I with 20% FA replacement, and Type I with 70% BFS replacement. Table 1 summarises the characteristics of the cementitious components. Deionised water and dune sand were used in the preparation of the mortar specimens.

### Specimen preparation

Cement mortar specimens, 50 mm × 50 mm × 50 mm, were prepared from each of the selected cementi-

Table 1. Chemical and physical characteristics of cements and SCMs used

Constituent	Portland cement		Superpozz	Fly ash	Silica fume	Blast furnace slag
	Type I	Type V				
SiO <sub>2</sub> (S)	20.52	22.00	53.5	52.8	93.7	33.32
Al <sub>2</sub> O <sub>3</sub> (A)	5.64	4.08	34.3	34.3	0.16	14.09
Fe <sub>2</sub> O <sub>3</sub> (F)	3.80	4.24	3.6	3.6	0.12	0.73
CaO (C)	64.35	64.07	4.4	4.4	0.65	42.4
MgO (M)	2.11	2.21	1.0	1.1	0.39	6.98
SO <sub>3</sub>	2.10	1.96		0.1	0.23	0.22
Loss on ignition	0.70	0.80		0.8	5.14	0.6
K <sub>2</sub> O	0.36	0.31	0.8	0.5	0.20	
Na <sub>2</sub> O	0.19	0.21		0.4	0.20	0.14
Na <sub>2</sub> O equiv	0.43	0.41				
MnO <sub>2</sub>				0.1		0.33
TiO <sub>2</sub>			1.7	1.6		
Chloride					0.07	0.012
C <sub>3</sub> S	56.70	54.57				
C <sub>2</sub> S	16.05	21.91				
C <sub>3</sub> A	8.52	3.64				
C <sub>4</sub> AF	11.56	12.90				
Σ(C + M + S)						82.7
(C + M)/S						1.48
C/S						1.27
Relative density			2.2	2.25		
Surface area: cm <sup>2</sup> /g			13 000	3000	23 000	4580
pH in water			11–12	11–12		
Moisture: %			< 0.2		0.98	0.24
Retained on 45 μm: %					3.25	
APAI: %					87	
IODS @ 28 days: %					0.001	

APAI, accelerated pozzolanic activity index (percent relative to control); IODS, increase over control of drying shrinkage of mortar bars.

tious materials to evaluate the reduction in compressive strength due to sulphate attack. Cement paste specimens 25 mm × 25 mm × 25 mm were prepared to assess the mineralogical changes in the cement due to sulphate exposure utilizing scanning electron microscopy and X-ray diffraction techniques. Prismatic mortar specimens measuring 25 mm × 25 mm × 285 mm were utilised to measure the change in length due to sulphate exposure. Details of the mixture types and the number of specimens cast are shown in Table 2.

Deionised water was used in the preparation of ce-

ment and mortar specimens. For the mortar specimens, the water/binder ratio was 0.5, and the sand to cementitious materials ratio was 2.5. The constituents were mixed in a laboratory mortar mixer until uniform consistency was obtained. In blended cements, the SCMs were first mixed thoroughly with the cement and then deionised water was added. The mortar was poured into the moulds and vibrated using a vibrating table to remove the entrapped air. After casting and finishing, the moulds were covered with plastic sheets and kept under laboratory conditions for 24 h and then de-

Table 2. Details of the mixture types and the number of specimens cast

Cement	Number of specimens		
	Mortar cubes (50 mm × 50 mm × 50 mm)	Paste cubes (25 mm × 25 mm × 25 mm)	Mortar prisms (25 mm × 25 mm × 285 mm)
Type I	84	10	15
Type V	84	10	15
Type I + 10% superpozz	84	10	15
Type I + 20% fly ash	84	10	15
Type I + 8% silica fume	84	10	15
Type I + 70% blast furnace slag	84	10	15

moulded. After demoulding, the specimens were cured in saturated calcium hydroxide solution maintained at  $23 \pm 2^\circ\text{C}$  for further 27 days.

### Exposure

After 28 days of curing, the specimens were placed in tanks containing sodium sulphate solution. The sulphate concentration in the solutions, shown below, was selected to correspond to exposure regimes defined in ACI 318.<sup>21</sup>

- (a) Moderate exposure, 0.15%  $\text{SO}_4^{2-}$  (1500 mg/l)
- (b) Severe exposure, 0.5%  $\text{SO}_4^{2-}$  (5000 mg/l)
- (c) Very severe exposure, 1.5%  $\text{SO}_4^{2-}$  (15 000 mg/l)
- (d) Extremely severe exposure, 2.5%  $\text{SO}_4^{2-}$  (25 000 mg/l).

The above exposures cover the range from moderate to extremely severe categories noted in ACI 318.<sup>22</sup> Very severe to extremely severe exposures represent the sulphate concentrations noted in saline soils, denoted as *sabkha salinas* that are widely prevalent in many parts of the world.

The concentration of the exposure solutions was checked periodically and adjusted as necessary. Further, the solutions were changed every 4 months. The temperature of the exposure solution was maintained at  $23 \pm 2^\circ\text{C}$ . The effect of sulphate concentration on the performance of plain and blended cements was evaluated by visual examination and by monitoring the reduction in compressive strength. Three mortar specimens representing similar composition were retrieved from the exposure solutions after 4, 8, 12, 16, and 20 months of exposure for assessment. The specimens were visually inspected to classify the extent of deterioration. Then the degree of deterioration was quantitatively evaluated by measuring the reduction in compressive strength.

In addition, the sulphate attack was also evaluated by measuring the expansion of mortar prisms (on the linear face). The expansion readings were taken each month for up to 15 months.

**X-ray diffraction.** X-ray diffraction (XRD) was conducted on powdered samples obtained from the central 20 mm  $\times$  20 mm section of cement paste specimens exposed to 15 000 ppm sulphate solution for 20 months. The samples were ground to pass a No. 325 sieve and placed on a sample holder of the diffractometer. XRD patterns were obtained using  $\text{Cu-K}\alpha_1$  radiation across the  $2\theta$  range of 5 to  $80^\circ$ .

**Scanning electron microscopy.** Scanning electron microscopy (SEM) images were obtained using a Jeol electron microscope operating at an accelerating potential of 20 kV. Fresh fractured surfaces were obtained from the top portion of the central 20 mm  $\times$  20 mm section and immediately carbon

coated before being placed in the sample stage of the microscope.

## Results and discussion

### Visual observations for prism mortar specimens

Cracking was evident in the Type I cement mortar specimens exposed to solutions with sulphate concentrations of 15 000 and 25 000 ppm but not in any of the Type V cement mortar specimens. Excessive bending and cracks were noted in the SF cement mortar specimens exposed to solutions with a sulphate concentration of 25 000 ppm but deterioration was not evident in the FA, SP, and BFS cement mortar specimens.

### Visual observations for mortar cube specimens

Figure 1 shows Type I, Type V, and SF cement mortar cube specimens exposed to  $\text{Na}_2\text{SO}_4$  solutions with varying sulphate concentration. Cracks were noted in all the Type I cement mortar specimens, the intensity of cracks increasing with the sulphate concentration; they were wide in the specimens exposed to solutions with sulphate concentration of 15 000 and 25 000 ppm. Cracks were noted in the Type V cement mortar cube specimens exposed to sulphate concentration of 5000 ppm and more. The intensity of cracks was not as high as that in the Type I cement mortar specimens. Cracks were noted only in SF cement mortar specimens exposed to solution with a sulphate concentration of 25 000 ppm. Cracks were not evident in the FA, SP, and BFS cement mortar specimens.

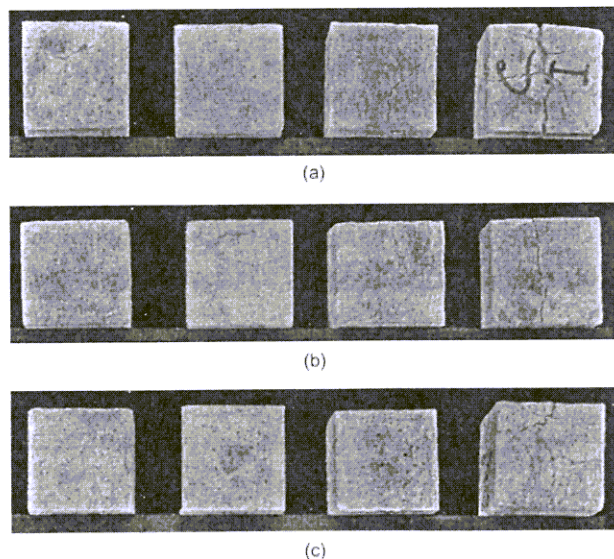


Fig. 1. Cement mortar specimens exposed to  $\text{Na}_2\text{SO}_4$  solution with varying sulphate concentration for 12 months (Sulphate concentration from left to right: 1500, 5000, 15 000 and 25 000 ppm). (a) Type I cement; (b) Type V cement; (c) silica fume cement

Expansion

The expansion in Type I cement mortar specimens exposed to various sulphate solutions for 15 months is depicted in Fig. 2. The change in length was insignificant up to about 3 months of exposure; thereafter, it increased almost linearly and expansion in all the specimens exceeded the threshold value of 0.1%. It can be noted that the rate of expansion increased with the sulphate concentration in the solution.

All the other specimens were considerably more

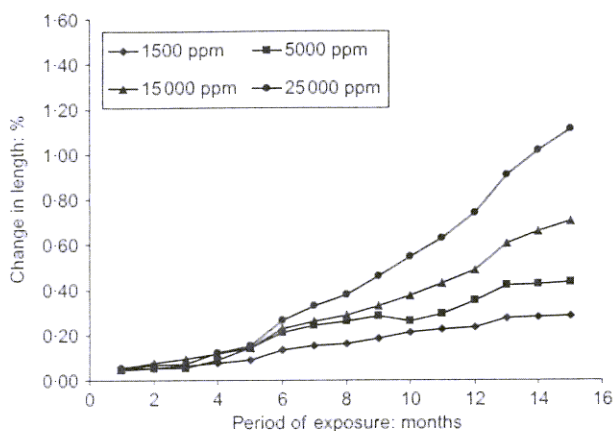


Fig. 2. Expansion Type I cement mortar specimens exposed to varying concentrations of sodium sulphate solution for 15 months

stable during exposure to sulphate solutions as indicated by the data in Table 3.

The expansion in Type V cement mortar specimens, due to exposure to varying sulphate concentrations, exceeded the threshold value of 0.1% after about 6 months of exposure. The expansion in SP10 cement mortar specimens remained low (<0.1%) and essentially constant for 11 months; although, for the higher sulphate concentrations the expansion increased after 12 months. Similar behaviour was observed in the SF cement mortar specimens although the increase was observed after 7 months of exposure. The FA and BFS cement mortar specimens did not expand significantly within the period of exposure.

Another aspect of the expansion data summarised in Table 3 is that the expansion in Type V, BFS, and FA specimens exposed to a sulphate concentration of 15 000 ppm was more than that in similar specimens exposed to solution with a sulphate concentration of 25 000 ppm. This may be attributed to the non-availability of C<sub>3</sub>A in these cements to react with the excess sulphate ions. In the Type I, SF, and SP cements sufficient C<sub>3</sub>A is available for the excess sulphate ions to react leading to further expansion.

The expansion characteristics of all specimens exposed to the maximum sulphate concentration are summarised in Fig. 3 from which the two-stage expansion process as observed by Santhanam *et al.*<sup>19,20</sup> is evident. The figure permits a comparison of different cement

Table 3. Expansion in plain and blended cement mortar specimens exposed to sulphate solutions

Cement	Sulphate concentration: ppm	Expansion: %				
		3 Months	6 Months	9 Months	12 Months	15 Months
Type I	1500	0.0646	0.1341	0.1847	0.2332	0.2832
	5000	0.0556	0.2111	0.2855	0.3533	0.4325
	15000	0.0929	0.2316	0.3282	0.4867	0.7048
	25000	0.0735	0.2674	0.4600	0.7427	1.1124
Type V	1500	0.0624	0.0768	0.0864	0.0955	0.1021
	5000	0.0510	0.0648	0.0734	0.0999	0.1199
	15000	0.0777	0.0928	0.1031	0.1259	0.1913
	25000	0.0396	0.0557	0.0655	0.0851	0.1073
Type I + 10% superpozz	1500	0.0551	0.0624	0.0600	0.0499	0.1104
	5000	0.0658	0.0693	0.0705	0.0802	0.0904
	15000	0.0548	0.0551	0.0655	0.1182	0.1343
	25000	0.0381	0.0603	0.0629	0.1620	0.1882
Type I + 20% fly ash	1500	0.0468	0.0491	0.0530	0.0628	0.0735
	5000	0.0534	0.0464	0.0494	0.0648	0.0699
	15000	0.0535	0.0540	0.0654	0.0747	0.0796
	25000	0.0337	0.0542	0.0493	0.0402	0.0776
Type I + 8% silica fume	1500	0.0502	0.0574	0.0642	0.0735	0.0879
	5000	0.0618	0.0665	0.0814	0.1001	0.1150
	15000	0.0624	0.0805	0.1143	0.1759	0.2283
	25000	0.0587	0.0985	0.1464	0.2515	0.4211
Type I + 70% blast furnace slag	1500	0.0562	0.0597	0.0630	0.0676	0.0781
	5000	0.0677	0.0645	0.0657	0.0833	0.0929
	15000	0.0863	0.1045	0.1069	0.1209	0.1317
	25000	0.0610	0.0759	0.0833	0.0867	0.1074

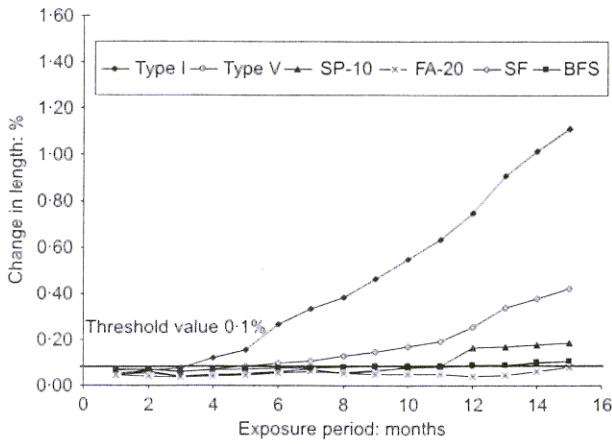


Fig. 3. Expansion in plain and blended cement mortar specimens exposed to 25 000 ppm sulphate solution for 15 months

types to be made and if it is assumed that the mechanism of the two-stage expansion is attributed to the initiation of cracking (and exposure of fresh surface for enhanced rate of reaction to initiate stage 2), then it can be proposed that the order of crack initiation due to sulphate attack is Type I, SF, followed by SP.

*Reduction in compressive strength*

The reduction in compressive strength due to sulphate attack is presented in Figs 4 to 9 for Type I, Type V, SP, FA, BFS, and SF cements. The compressive strength tends to decrease with increasing period of exposure and the sulphate concentration in the exposure solution. The increased reduction in the compressive strength with increasing sulphate concentration may be attributed to the formation of expansive sulphate phases.

The reduction in compressive strength was more than the failure criterion of 30% in Type I and SF cement mortar specimens exposed to solutions with high sulphate concentrations. However, it must be noted that,

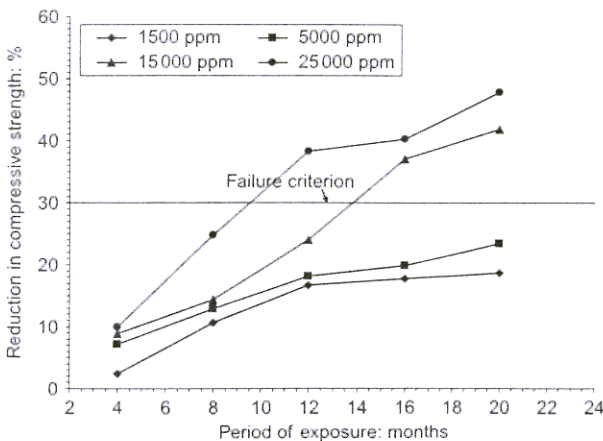


Fig. 4. Strength reduction in Type I cement mortar specimens

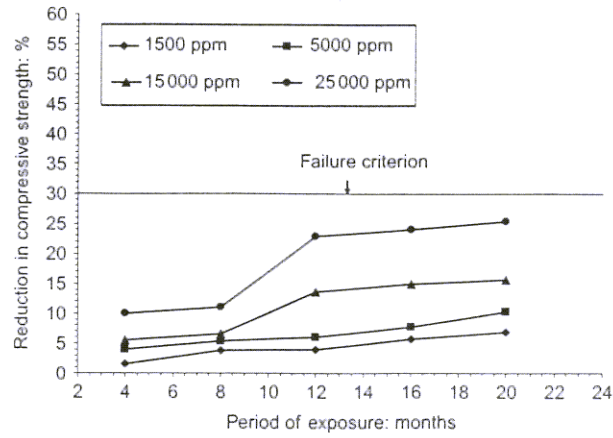


Fig. 5. Strength reduction in Type V cement mortar specimens

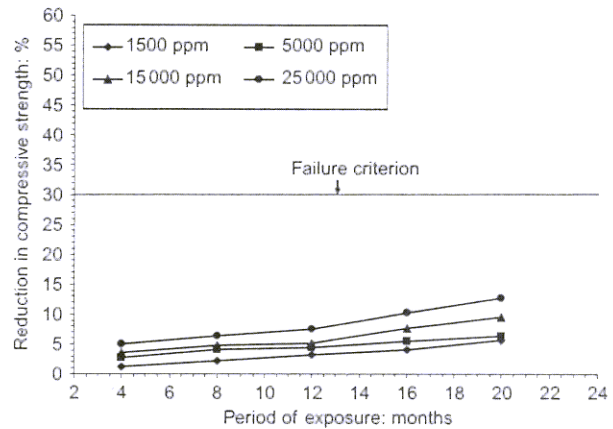


Fig. 6. Strength reduction in FA cement mortar specimens

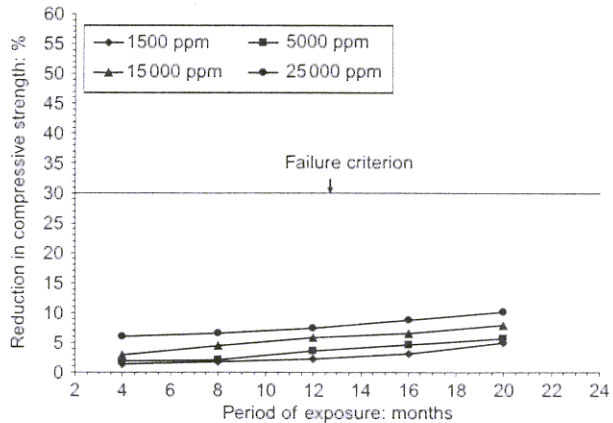


Fig. 7. Strength reduction in SP cement mortar specimens

again, the best performance against sulphate attack was exhibited by SP, FA, and BFS cement mortars followed by Type V cement. In these cements the strength reduction was not more than 30% even in the most extreme exposure conditions.

The data in Figs 4 to 9 indicate a decrease in the

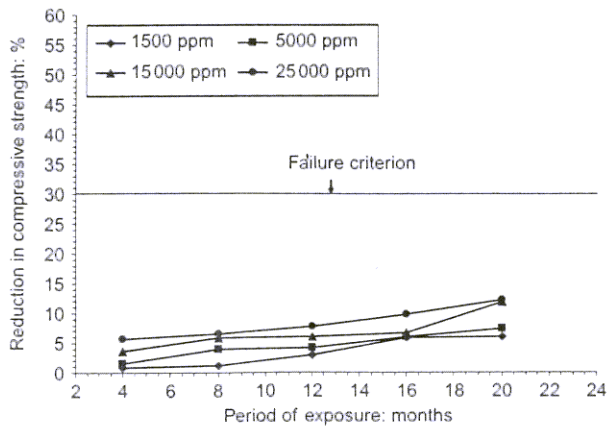


Fig. 8. Strength reduction in BFS cement mortar specimens

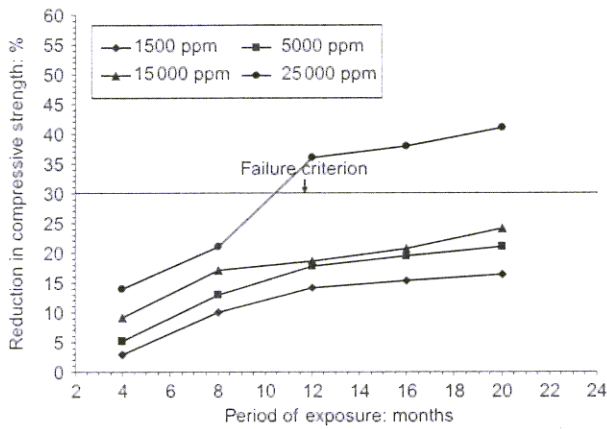


Fig. 9. Strength reduction in SF cement mortar specimens

compressive strength with increasing sulphate concentration in all the cements. However, an increase in the sulphate concentration in the exposure solutions did not cause a proportionate increase in the expansion in Type V, BFS, and FA cements (Table 3). The probable cause for such a differential behaviour has already been explained earlier. The other probability could be that the expansive sulphate phases formed due to the sulphate attack tend to decrease the compressive strength by breaking the bond between the hardened cement paste and aggregates; however they may not proportionately generate expansion.

*X-ray diffraction and scanning electron microscopy*

Figures 10 to 15 show selected X-ray diffraction patterns derived from the top portion of the central 20 mm × 20 mm section cut from cement paste cubes exposed to sulphate (15 000 ppm) solution for 20 months. These results can be correlated with the strength and expansion data reported above.

The significance of ettringite as a disruptive expansive phase is evident from the XRD data that correlates with its presence in mortars demonstrating the highest levels of expansion and reduction in strength loss. It may be

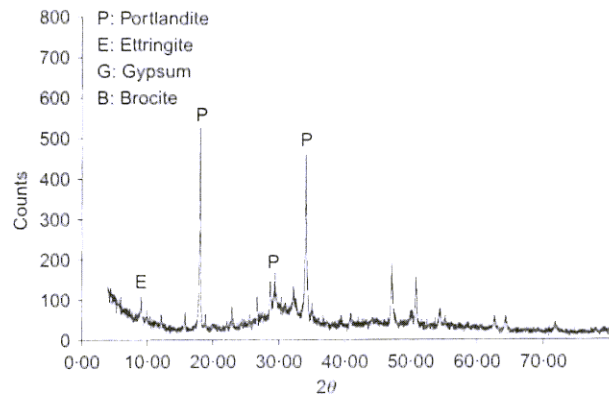


Fig. 10. X-ray diffraction for Type I cement

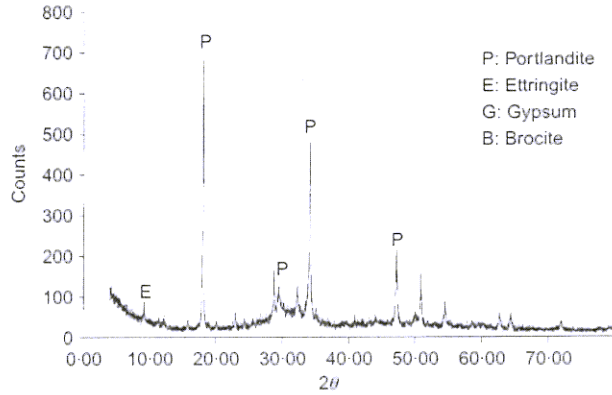


Fig. 11. X-ray diffraction for Type V cement

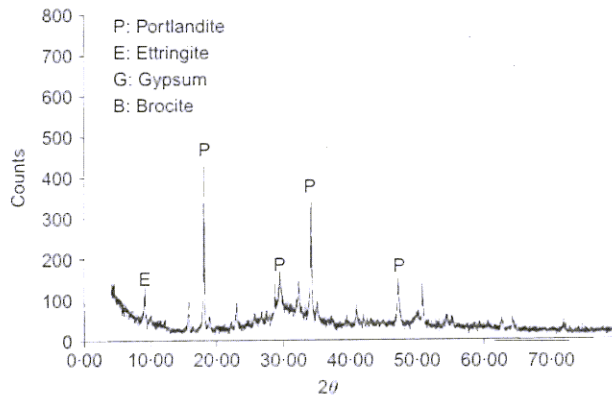


Fig. 12. X-ray diffraction for SP cement

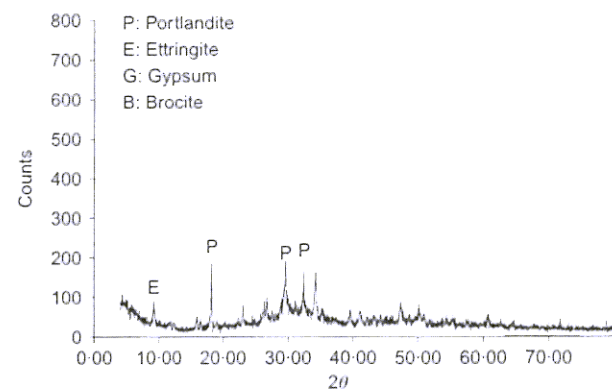


Fig. 13. X-ray diffraction for FA cement

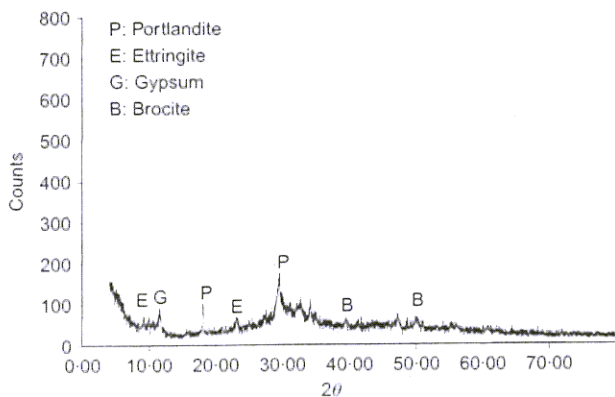


Fig. 14. X-ray diffraction for BFS cement

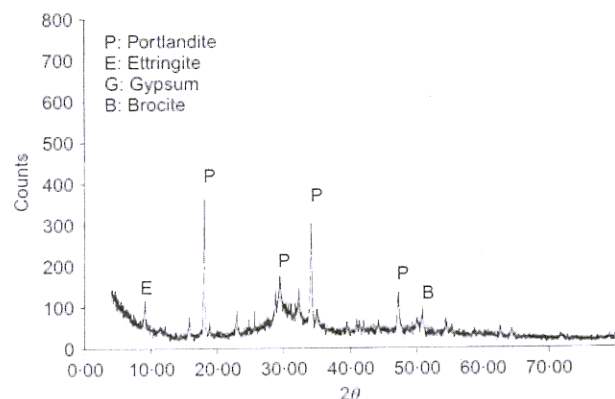


Fig. 15. X-ray diffraction for SF cement

proposed that the superior durability of BFS cement is due to the absence of sufficient  $C_3A$  that is necessary to convert the sulphate to ettringite. It must be recalled that the increased level of pore filling to be expected from the pozzolanic reaction will limit the available surface for reactivity so that while there may be a potential for expansive ettringite formation, this is limited by mass transport factors. All other specimens contain ettringite and portlandite and, in fact, the broad band peak centred around  $30^\circ 2\theta$  confirms the presence of the poorly crystallised C-S-H or C-A-S-H binder. The relative amounts of the various phases have not been quantified but relative peak heights of the sulphate compounds suggest the relative performance of the selected cements.

The strength reduction was more than the threshold value of 30% in Type I and SF mortar specimens for sulphate concentration of more than 15 000 ppm. The strength reduction is attributed to the cracking of the specimens due to the formation of expansive phases as noted in the scanning electron micrographs for Type I and SF cements in Figs 16 and 17, respectively.

## Conclusions

There follows a summary of the findings of this study.

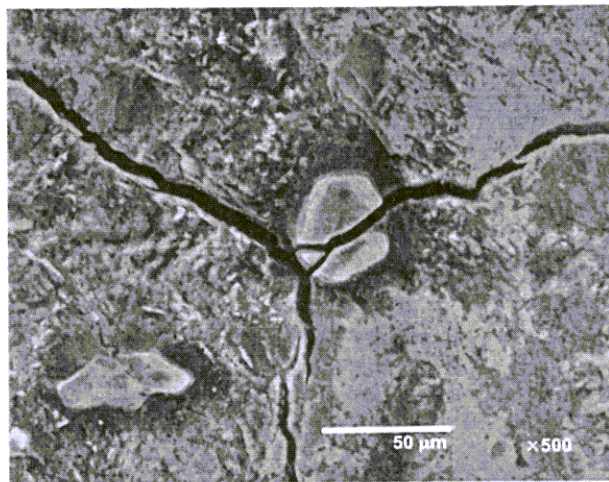


Fig. 16. SEM of Type I cement paste specimen exposed to sodium sulphate solution

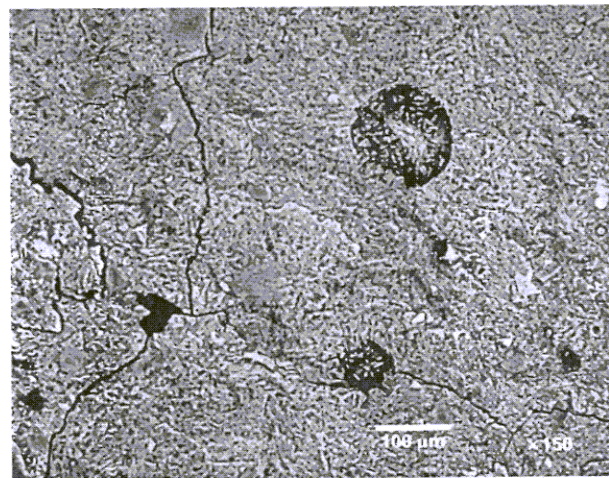


Fig. 17. SEM of SF cement paste specimen exposed to sodium sulphate solution

- Type I and SF (Type I plus 7% SF) cement mortar specimens have shown good resistance to sulphate attack up to a sulphate concentration of 15 000 ppm, as indicated by cracking of specimens, and Type V cement was able to tolerate a sulphate concentration of 25 000 ppm. Blast furnace slag, fly ash, and Superpozz cement mortar specimens have shown good resistance to sulphate attack even when the sulphate concentration was as high as 25 000 ppm.
- The expansion of mortar specimens exposed to sodium sulphate solution followed a two-stage process. In the initial stage, stage 1, there was little expansion. This was followed by a sudden and rapid increase in the expansion in stage 2. However, the length of stage 1 depended on the type of cement and the concentration of the sulphate solution.
- SEM and XRD indicated the formation of ettrin-



gite in almost all the cements leading to cracking that has deteriorated the internal structure of the cement resulting in a reduction in the compressive strength. While the cracks were uniformly distributed in Type I and SF cement paste specimens, they were isolated in Type V, FA, BFS and SP cements.

(d) The data suggest that Type V cement or Type I cement blended with FA, BFS or SP is preferable for sulphate exposures. Blending of Type I cement with the aforesaid SCMs has the advantage that it decreases the cost of the cementitious material and also the resulting cement provides both corrosion and sulphate resistance.

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