EFFECT OF FLY ASH ON ALKALI-SILICA REACTION

M. Berra*, T. Mangialardi**, A.E. Paolini**, R. Turriziani**
*ENEL-CRIS, Milano, Italy
**Faculty of Engineering, University of Roma "La Sapienza", Italy

The effectiveness of different fly ashes in preventing deleterious expansion caused by alkali-silica reaction was investigated using mortar specimens containing fused silica as a reactive aggregate. Expansion tests (ASTM C227 method) and pore solution analyses were performed on bars 25x25x280 mm or 40x40x160 mm. The latter bars always expanded to a greater extent than ASTM prisms and this was not due to a different alkali leaching effect. All fly ashes were effective at a cement replacement level of 35% by weight while, at 25% replacement level, they behaved as expansion delayers. No correlation between the effectiveness of fly ashes and their physical properties was found. The threshold concentrations of OH⁻ ions for alkali-silica expansion were lower than the postulated value of 250 mmoles/l. There was a good agreement between the ash effectiveness predictions based on pore solution analysis and italian pozzolanic activity test.

INTRODUCTION

The use of fly ash in concrete is of increasing importance in many countries both in order to produce concrete more economically and in order to obtain advantageous properties such as lower heat of hydration, increased resistance to aggressive chemicals and reduced susceptibility to alkali-silica reaction (ASR).

There is, however, not universal agreement about the actual effectiveness of fly ash in preventing deleterious expansion due to ASR (Hobbs (1)), principally because the beneficial influence of this admixture, as well as of other mineral additions, is related to many factors such as chemical composition and physical properties of admixture, type and amount of cement being replaced and its alkali content, mix proportioning (reactive silica content, water/cement ratio, cement content) (Swamy and Al Asali (2)).

The concept that the alkali-silica reaction is initiated and sustained above a threshold concentration of hydroxyl ions in the pore solution of the cementitious matrix is generally accepted (Nixon and Page (3)). Thus, the study of the pore solution composition and, particularly, the effect of the partial replacement of cement with fly ash on the OH⁻ ion concentration in the pore solution could be an effective tool to reveal the ability of different fly ashes in counteracting the alkali-silica reaction. However, such an approach is, at present, made problematic by the poor knowledge about the influence of the nature and amount of reactive aggregate on the threshold level of the hydroxyl ion concentration in the pore solution. Furthermore, the composition of the resulting gel and hence its capability to cause expansion would be related to the available amount of sodium, potassium or calcium in the pore fluid.

Another question is whether the ASTM C227 mortar bar expansion test method (4), based on the expansion limit of 0.10% at 6 months, could be used to evaluate the effectiveness of different fly ashes, at least when an alkali-free, highly reactive aggregate is used as working

aggregate.

This paper deals with the above aspects of the alkali-silica reaction in mortar bars made with different fly ash-portland cement combinations, when fused silica was used as highly reactive aggregate and the bars were stored in accordance with the ASTM C227 test procedure.

MATERIALS AND METHODS

Three fly ashes (FA) with different total alkali content (FA_1 = 0.41%, FA_2 = 0.84%, and FA_3 = 1.96% as Na_2O equivalent), a high- alkali (0.80% as Na_2O equivalent) portland cement (PC_1), and a low-alkali (0.43% as Na_2O equivalent) portland cement (PC_2) were used.

These materials were characterized for chemical composition and physical properties according to standard procedures (ASTM C311 (5), ASTM C114 (6)). The available alkali content of the two cements was determined as described in (5), except that no calcium hydroxide was added. The specific surface area of all the materials was measured by the Blaine apparatus. Table 1 gives the chemical and physical characteristics of portland cements and fly ashes.

The crystalline constituents of the three fly ashes were detected by X-ray diffraction (XRD) analysis and the glass content was determined indirectly by a quantitative XRD method (Klug and Alexander (7)). The particle size distribution was determined by a laser analyzer, after an aliquot of each ash was suspended in ethanol and then dispersed for 5 min in an ultrasonic bath equipped with a shaker. The pozzolanic activity index was evaluated by using mortar bars (40 x 40 x 160 mm in size) made with natural quartzitic sand (a non-reactive aggregate having particle size range from 0.15 to 2.5 mm) and $FA-PC_1$ combinations, at cement replacement levels of 0 and 35% by volume (about 25% by weight) and constant water/portland or blended cement (w/c) ratio (w/c = 0.50). Distilled water was used as mixing water. The bars were stored and tested for compressive strength, as described in (5).

Fly ash-portland cement blends with cement replacement levels of 0, 15, 25 and 35% by weight were prepared. These blends were firstly tested for pozzolanic activity according to the italian test procedure (8). This test consisted of storing cement suspensions (water/solid (w/s) ratio = 5.0) at 40°C for 8 days, filtering the suspensions and determining the total alkalinity (expressed as mmoles OH^-/l) and Ca^{+2} ion concentration (expressed as mmoles CaO/l) on the filtrates. These data were then plotted in a graph where the solubility of calcium hydroxide was also reported as a function of total alkalinity.

The mortar specimens for the expansion tests were made with the above portland cements or portland-fly ash blends, natural quartzitic sand and fused silica (FS). Fused silica was virtually free of alkalies (0.01% as Na_2O equivalent) and consisted of 99.2% SiO_2, about 90% as a vitreous phase and the remaining 10% as quartz and cristobalite. This aggregate was used in the 30 to 350 μm fraction, in a proportion of 4 g per 100 g of total aggregate (Berra et al. (9)). The w/c ratio of the mortars was always 0.50, differently from what is established by the ASTM C227 test procedure (variable amount of mixing water to produce a constant flow of mortars).

The expansion tests were performed both on 25 x 25 x 280 mm bars (as established by the original procedure) and on 40 x 40 x 160 mm specimens, and the specimens were stored at 38°C and relative humidity (R.H.) > 95% up to 180 days and, in several cases, up to 365 or 540 days. The lengths of the bars were periodically measured using a comparator with a sensitivity of 0.001 mm.

TABLE 1 - Chemical and physical characteristics of Portland cements and fly ashes.

	Cement			Fly ash	
Chemical Analysis(%)	PC_1	PC_2	FA_1	FA_2	FA_3
SiO_2	22.27	22.11	49.16	51.94	56.02
Fe_2O_3	2.63	4.76	3.57	3.96	7.45
Al_2O_3	4.76	3.62	34.40	30.25	29.14
CaO	63.98	65.93	1.85	2.75	0.04
Free CaO	0.55	0.14			
MgO	1.21	0.57	1.72	1.76	0.40
SO_3	2.40	1.51	0.66	0.61	0.40
Mn_2O_3	0.06	0.04	0.09	0.06	0.07
TiO_2	0.16	0.08	0.85	0.59	0.30
P_2O_5	0.10	0.11	0.13	0.11	0.15
SrO	0.08	0.04			
Na_2O					
Total	0.21	0.11	0.08	0.29	0.73
Available	0.16	0.08	0.07	0.05	0.14
Water-soluble	0.04	0.02	0.016	0.006	0.03
K_2O					
Total	0.90	0.49	0.50	0.83	1.87
Available	0.53	0.32	0.19	0.19	0.44
Water-soluble	0.38	0.16	0.003	0.004	0.003
Na_2O equivalent					
Total	0.80	0.43	0.41	0.84	1.96
Available	0.51	0.29	0.20	0.18	0.43
Water-soluble	0.29	0.13	0.018	0.009	0.03
L.O.I.	1.80	0.72	6.30	6.35	3.03
Density, kg/m^3	3150	3160	2245	2225	2331
Specific surface area Blaine, m^2/kg	493	366	521	462	446
Bogue compounds					
C_3S	46.46	64.67			
C_2S	28.97	14.67			
C_3A	8.16	1.56			
C_4AF	8.00	14.47			

At fixed time intervals, the bars were also analyzed for the chemical composition of the pore solution. Pore solution was expressed from each bar by using high pressure apparatus (550 MPa) and the extracted liquid was analyzed for the OH^- ion concentration by titration against hydrochloric acid to the phenolphtalein end point. The K^+ and Na^+ ion concentrations were measured by atomic absorption spectroscopy. In some cases, the pore liquid composition was also determined on mortar bars made with or without fly ash but not containing the reactive aggregate.

In this paper, the composition of the pore liquid was principally discussed in terms of OH^- ion concentrations and, in several cases, the actual OH^- values were presented. In other cases, the OH^- ion concentrations were adjusted to take into account the changes in free water content of mortar specimens. The proportions of evaporable and non-evaporable water in the specimens were estimated by heating to constant weight at 105°C.

RESULTS AND DISCUSSION

Figures 1 to 3 show the time depending expansion curves for mortar bars made with high-alkali cement (PC_1) or FA-PC_1 combinations. Figure 4 shows the results obtained in the case of using low-alkali cement (PC_2) or its combination with fly ash. In these figures, the percent linear expansions (E%) were the average values of three specimens.

In the absence of fly ash, the bars made with cement PC_1 exhibited significant expansion (Figure 1), thus revealing the high alkali-reactivity of fused silica. The expansion limit of 0.10% was reached after only 25 days for both sizes of bars, and the expansion level at 6 months was 0.80% and 1.10% for 25 x 25 x 280 mm and 40 x 40 x 160 mm specimens, respectively. No further expansion was observed at longer exposure times. As expected, the use of a low-alkali cement greatly reduced the expansion of the bars of both sizes (Figure 4). The E% values always remained below 0.10% even when the exposure time was prolonged up to 365 days.

If an expansion level less than 0.10% at 6 months was adopted as a criterion to evaluate the effectiveness of the fly ashes in preventing deleterious expansion, then all fly ashes tested were found to be effective only when the replacement level of cement PC_1 was at least 25% by weight (Figures 2 and 3).

If the expansion tests were prolonged above six months and an expansion level less than 0.10% was again used as judgement criterion, all fly ashes resulted to be effective, at least up to the ultimate time of the present investigation (540 days), only when they were used at a cement replacement level of 35% by weight (Figure 3). An exception was represented by 40 x 40 x 160 mm bars with 35% FA_3-65%PC_1, which exhibited an expansion level slightly higher than 0.10% after about 365 days of exposure. The high delayed expansions registered for 25%FA-75%PC_1 blends (Figure 2) indicated that, at this replacement level, the fly ashes tested only acted as expansion delayers.

The addition of fly ash to cement PC_2 (Figure 4) did not modify or further reduced the ultimate expansion of the bars and this was also observed for FA_3-PC_2 blends, in spite of the fact that the available alkali content of ash FA_3 was higher than that of cement PC_2 (Table 1).

As evidenced by Figures 1-4, at fixed mortar composition and exposure time, bars 40 x 40 x 160 mm in size always exhibited higher linear expansions as compared to ASTM prisms and the percentage difference in expansion varied from about 40 to 400%. In most cases, this difference was more pronounced for FA_1-PC_1 blends and less pronounced for FA_3-PC_1 specimens but no correlation with the bar composition was found. On the other hand, the results in Table 2, showing similar pore solution compositions for specimens with the same composition and different dimensions, would exclude the possibility that the difference in expansion could be related to a more pronounced alkali leaching effect for 25 x 25 x 280 mm bars, characterized by a higher external surface area/volume (A/V) ratio (A/V = 1.68 cm^{-1} against 1.13 cm^{-1} for 40 x 40 x 160 mm specimens). A possible cause could be the different warping tendency of the two types of specimens as a consequence of their different length/width ratio. More work is, however, needed to understand the effect of bar dimensions on expansion level.

As far as the nature of the fly ashes was concerned, ash FA_3 was found to be more effective than ashes FA_1 and FA_2 when the replacement level of cement PC_1 was 15 or 25 % by weight (Figures 1 and 2). At 35% replacement level, the effectiveness of the three ashes did not differ significantly, particularly when the expansion tests were performed on bars 25 x 25 x 280 mm in size (Figure 3).

TABLE 2- Pore liquid compositions and linear expansions at 365 days for mortar bars made with 25%FA-75%PC$_1$ blends.

Fly ash	Bar dimensions (mm)	Linear expansion (E%)	Pore liquid composition		
			OH⁻	Na⁺ (mmoles/l)	K⁺
FA$_1$	25 x 25 x 280	0.15	87	34	55
FA$_1$	40 x 40 x 160	0.66	76	35	42
FA$_2$	25 x 25 x 280	0.23	79	37	44
FA$_2$	40 x 40 x 160	0.59	74	36	39
FA$_3$	25 x 25 x 280	0.17	81	32	46
FA$_3$	40 x 40 x 160	0.25	80	36	47

As shown in Table 1, with respect to ashes FA$_1$ and FA$_2$, ash FA$_3$ had higher SiO$_2$ and SiO$_2$+ Al$_2$O$_3$+ Fe$_2$O$_3$ contents, lower loss on ignition (L.O.I.), and much higher total and available alkali contents. As reported in Table 3, the glass content of ashes FA$_2$ and FA$_3$ was identical (80%) and it was higher than the value found for ash FA$_1$ (76%). XRD analyses showed that the three ashes contained the same principal crystalline phases (quartz, mullite, hematite, periclase, lime, spinel (Mg(Al, Fe)$_2$O$_4$)), except that periclase and lime were not identified in ash FA$_3$ and spinel was not detected in ash FA$_1$. The particle size analysis data (Figure 5) indicated that ashes FA$_1$ and FA$_3$ were respectively the materials with highest and lowest percentages of fine particles. The particle size distributions essentially differed over the size range from 10 to 50 μm and this explained the little difference in fineness of the three ashes, when evaluated in terms of 45 μm sieve residue (Table 3). The specific surface areas calculated from the particle size analysis (Table 3) were qualitatively in good agreement with the Blaine surface areas (Table 1), but obviously lower values were obtained with the former method.

The differences in fineness and glass content of ashes FA$_1$ and FA$_2$ (lower fineness and higher glass content for ash FA$_2$) could explain their similar pozzolanic activity index values (Table 3), while the lower fineness of ash FA$_3$ could explain its reduced pozzolanic activity index (84% against 100% and 103% for ashes FA$_1$ and FA$_2$, respectively), thus confirming the importance of fineness in determining strength development.

No correlation was, however, found between the behaviour of the three fly ashes in the expansion tests and their total and available alkali content and physical properties (fineness,pozzolanic activity index, surface area).

Figure 6 compares the changes in both the OH⁻ ion concentration of the pore solution and the expansion level of the bars as a function of the exposure time, in the case of mortar bars (25x25x280 mm in size) made with cement PC$_1$ or 15% FA$_1$- 85%PC$_1$ blend, in the absence and the presence of the reactive aggregate. In this figure, the adjusted values for the OH⁻ ion

TABLE 3- Additional characteristics of fly ashes.

	FA$_1$	FA$_2$	FA$_3$
Glass, %	76	80	80
Specific surface area, m^2/kg (from particle size analysis)	297	276	264
Fineness (45 μm sieve residue), %	23.0	23.5	24.5
Pozzolanic activity index, %	100	103	84

concentration were presented to discuss the changes in this concentration with exposure time and bar composition. For mortars containing reactive aggregate (reacting mortars), the actual OH⁻ ion concentrations were also reported to correlate this parameter to the expansion level.

In the absence of fly ash, after 7 days of exposure, pore solutions expressed from reacting mortars showed significantly reduced concentrations of hydroxyl ions as compared to companion mortars not containing reactive aggregate (non-reacting mortars). This provided indication that the alkali-silica reactions were taking place in reacting specimens. A steady-state OH⁻ ion concentration in non-reacting mortars was established after about 28 days of exposure while, in reacting mortars, no steady-state concentration was established, at least up to 180 days, although after 90 days the change in concentration reduced to a lower extent with increasing time.

Similar considerations could be made for mortar bars prepared with 15% FA_1-85% PC_1 blend. However, in this case, the differences in the OH⁻ ion concentration between non-reacting and reacting mortars were less pronounced because of the ability of fly ash in counteracting the alkali-silica reaction. Furthermore, at a fixed exposure time, the OH⁻ ion concentrations were always lower than the respective values found in the absence of fly ash, and the reduction in the OH⁻ ion concentration was higher than what could be expected in the case if the portland cement was diluted with an inert material.

It was noteworthy that reacting mortars showed further significant expansion even when the corresponding reduction in the actual OH⁻ ion concentration was not significant (Figure 6). On the other hand, according to Diamond et al. (10), the physico- chemical process of absorption of pore solution by alkali- calcium-silica gels lags behind the chemical process of the alkali-silica reaction.

Although the changes in the actual OH⁻ ion concentration with the exposure time depended on the bar composition, however, these curves (not presented in this paper) showed that, over a period of 6 months, the OH⁻ ion concentration at 90 days could be approximately assumed as a "steady-state" concentration for all the blends examined. Thus, for mortar bars 25x25x280 mm in size, a correlation between the expansion levels at 6 months and the OH⁻ ion concentrations at 90 days was attempted, as shown in Figure 7. In this figure, the expansion levels at 6 months were also correlated with the respective actual OH⁻ ion concentrations at 28 days and, in both cases, the expansion data at 18 months were also considered for the specimens which exhibited delayed expansion.

In both cases (OH⁻ ion concentration at 90 or 28 days), there was poor correlation between the expansion level of the bars and the actual OH⁻ ion concentration of the pore solutions. However, it was possible to individuate a threshold value for the OH⁻ ion concentration, below which the expansion level of the bars did not exceed 0.10% even if the exposure time was prolonged up to 18 months. This threshold value (lower limit) corresponded to about 150 or 195 mmoles/l when the actual OH⁻ ion concentration of the pore solution was determined at 90 or 28 days, respectively. It was also possible to individuate an upper limit for the OH⁻ ion concentration (about 165 or 215 mmoles/l, depending on the reference time considered for the OH⁻ ion concentration), above which the expansion level at 6 months was always higher than 0.10%. Thus, a narrow range of the OH⁻ ion concentration (150-165 or 195-215 mmoles/l) was detected where all fly ashes tested behaved as expansion delayers (25% cement replacement level) (Figures 2 and 7). In other words, when fused silica was used as a reactive aggregate in a proportion of 4 g per 100 g of total aggregate, replacing 25% of cement PC_1 with fly ash was not enough to reduce the OH⁻ ion concentration in the pore solution to a level less than the lower threshold limit.

As shown in Table 2, for 25%FA-75%PC$_1$ blends, the OH⁻ ion concentrations at 365 days were significantly lower than the values found at 90 days (Figure 7). This provided indication that further consumption of hydroxyl ions by fused silica would be taking place over the period from 90 to 365 days and this could explain the delayed expansions of these specimens (Figure 2). However, the reduction in the OH⁻ ion concentration could also be partially due to an alkali leaching phenomenon.

The threshold values for the OH⁻ ion concentration did not vary significantly if the expansion data for 40 x 40 x 160 mm mortar bars (Figures 1-4) were plotted in Figure 7 instead of the values obtained for 25 x 25 x 280 mm specimens.

It was noteworthy that the threshold concentrations of hydroxyl ions found in this study were lower than the conservative value of 250 mmoles/l postulated by Diamond (11).

As shown in Figure 8, the same considerations could be made if the expansion data were plotted against the OH⁻ ion concentrations determined on cement suspensions (w/s = 5.0) after their storage at 40°C for 8 days, according to the italian test procedure adopted for the evaluation of pozzolanic cements. In this case, the threshold concentrations of OH⁻ ions were obviously much lower than the respective values found for the pore solution of the mortar bars. The data in Figure 8 showed that, in the presence of fused silica, portland cements or portland-fly ash blends did not cause deleterious expansion only when the OH⁻ ion concentration, measured on cement suspensions without reactive aggregate, was below about 49 mmoles/l (cement PC$_2$, 35%FA-65%PC$_1$ blends). Else, delayed or fast deleterious expansions were observed if the OH⁻ ion concentration was within the range from about 49 to 52 mmoles/l or above 52 mmoles/l, respectively. Thus, these preliminary data revealed the potential for the italian pozzolanic activity test as simple and rapid method to gauge the ability of different mineral admixtures in preventing deleterious expansion due to ASR, once the threshold level for the OH⁻ ion concentration was known.

According to the results of the italian pozzolanic activity test (not presented here), all portland-fly ash blends examined, except 15%FA-85%PC$_2$, could be classified as pozzolanic cements.

CONCLUSIONS

The ASTM C227 test method, based on the expansion limit of 0.10% at 6 months, was not able to detect the retarding effect of all fly ashes tested on the expansion development, when these admixtures were used in combination with a high-alkali portland cement at a replacement level of 25% by weight and an alkali-free, highly-reactive aggregate such as fused silica (4% by weight of total aggregate) was used.

All fly ashes were effective, at least up to the ultimate time of the present investigation (540 days), only when used at 35% cement replacement level.

There was no correlation between the effectiveness of fly ashes and their total and available alkali content and physical properties (fineness, pozzolanic activity index, surface area).

Mortar bars 40x40x160 mm in size always expanded to a greater extent than 25x25x280 mm specimens (ASTM prisms) with the same composition and the different expansion level was not due to a different alkali leaching effect.

There was poor correlation between the expansion level of the mortar bars at 6 or 18 months and the OH⁻ ion concentration of the pore solutions at 28 or 90 days. However, a narrow range

of the OH⁻ ion concentration (150-165 or 195-215 mmoles/l, depending on the reference time considered for this concentration) was detected where all fly ashes behaved as expansion delayers. These admixtures were effective or uneffective if the OH⁻ ion concentration was respectively below the lower limit or above the upper limit of the concentration range. Both the limits were lower than the postulated threshold concentration of 250 mmoles/l.

There was a good agreement between the ash performance predictions based on the pore liquid analysis and italian pozzolanic activity test.

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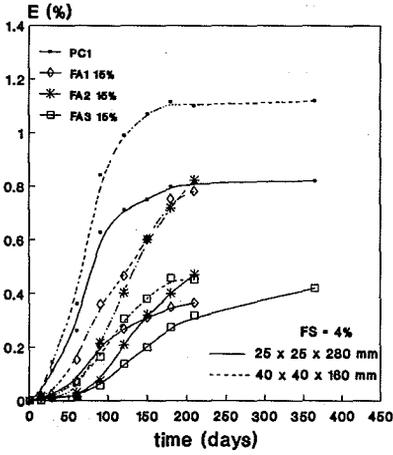
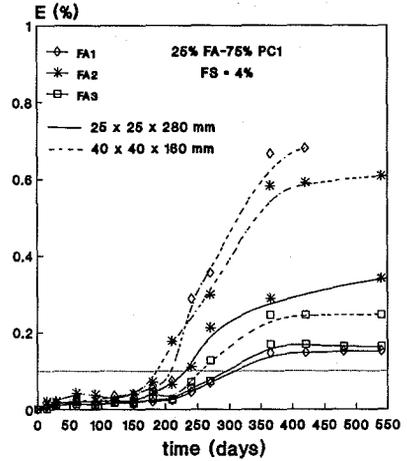Figure 1 Expansion of mortar bars made with cement PC_1 or 15%FA-85%PC_1 blends
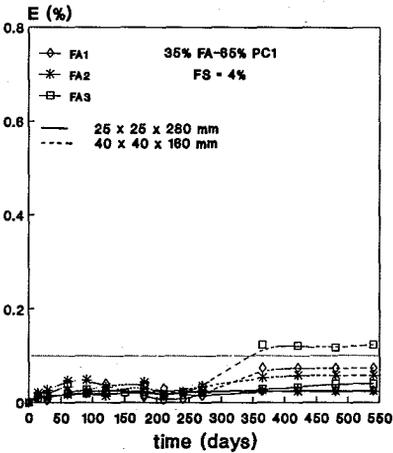
Figure 2 Expansion of mortar bars made with 25%FA-75%PC_1 blends
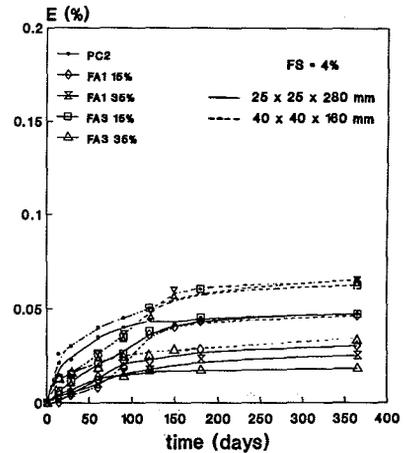

Figure 3 Expansion of mortar bars made with 35%FA-65%PC_1 blends

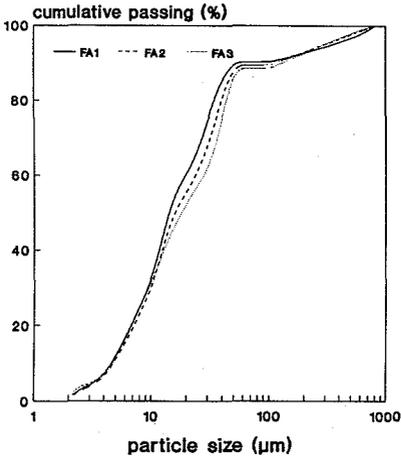Figure 4 Expansion of mortar bars made with cement PC_2 or FA-PC_2 blends
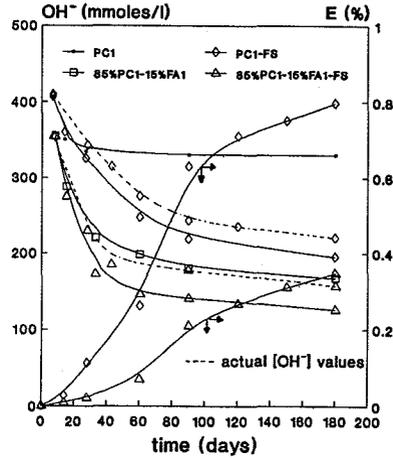
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Figure 5 Particle size analysis of fly ashes

Figure 6 Time-dependent changes in expansion and OH⁻ ion concentration
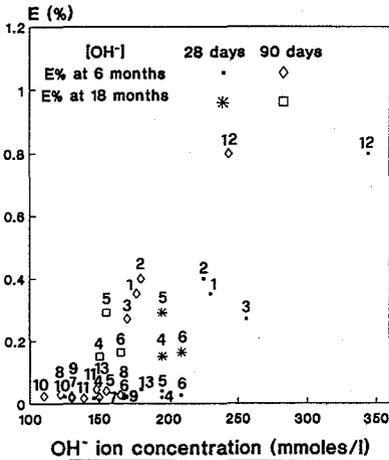
1) 15%FA$_1$-85%PC$_1$; 2) 15%FA$_2$-85%PC$_1$; 3) 15%FA$_3$-85%PC$_1$; 4) 25%FA$_1$-75%PC$_1$; 5) 25%FA$_2$-75%PC$_1$; 6) 25%FA$_3$-75%PC$_1$; 7) 35%FA$_1$-65%PC$_1$; 8) 35%FA$_2$-65%PC$_1$; 9) 35%FA$_3$-65%PC$_1$;10) 35%FA$_1$-65%PC$_2$;11) 35%FA$_3$-65%PC$_2$; 12) PC$_1$; 13) PC$_2$

Figure 7 Correlation between mortar bar expansion and OH⁻ ion concentration of pore solutions
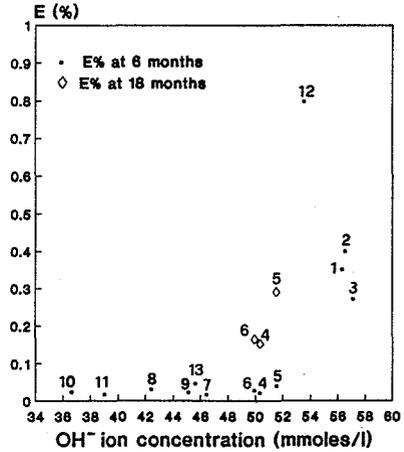
Figure 8 Correlation between mortar bar expansion and OH⁻ ion concentration of cement suspensions

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