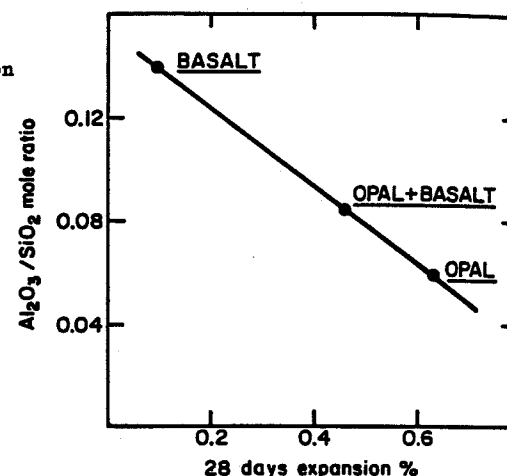


Fig. 9. Cement + Basalt (180 days). (a) Fibrous structure of thaumasite/woodfordite. (b) Higher magnification of (a).

Fig. 10. (Right) Plot of 28 days expansion vs. Al_2O_3/SiO_2 mole ratio.



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L Boswell and E C Robert*
Department of Civil Engineering
The City University
Northampton Square
London, England

and F G Buttler and S R Morgan
Department of Chemistry
Teeside Polytechnic
Middlesbrough
Cleveland, England

ABSTRACT

The influence of pulverised fuel ash on the expansion due to the alkali silica reaction has been studied at 38°C. The expansions of mortar bars made with a high alkali Portland cement and containing a pulverised fuel ash of BS 3892 Part 1 : 1982 quality were monitored and both pyrex glass and Beltane opal were used as expansive aggregates. The expansions were measured at regular intervals and the bars were analysed at the end of six months in order to determine the amount of alkali removed from the bars.

The results show that the expansion caused by the alkali silica reaction was reduced when pulverised fuel ash was used to replace some of the Portland cement despite the fact that such replacements led to a considerable increase in the dilute acid soluble alkali content of the bars. It was also found that the amount of alkali leached from the bars was reduced when pulverised fuel ash was incorporated.

The results are explained in terms of changes in permeability with time due to the pozzolanic or acid-base reaction of the pulverised fuel ash. (Keywords : expansion, mortar bars, alkali content, pfa).

1. INTRODUCTION

The results described in this paper have been obtained as part of a detailed study of the alkali silica reaction (ASR) in which both physical and chemical aspects of the reaction have been investigated. In the full programme the influence of pulverised fuel ash (pfa) at different replacement levels, the type and amount of reactive aggregate and the water content have been studied with respect to Portland cements of low (0.46% Na_2O_{eq}), medium (0.72% Na_2O_{eq}) and, high (1.17% Na_2O_{eq}) alkali contents. There is only room in this paper to describe the results obtained with the Portland cement of highest alkali content but the results obtained with the other Portland cements show similar trends.

Whenever possible, existing standard test methods were used. In particular, all expansion measurements were conducted on 25 x 25 x 250mm mortar bars produced and conditioned in accordance with ASTM C 227-71 and ASTM C 441-69. The latter standard was suitably modified to allow for the use of pulverised fuel ash at more than one replacement level. The chemical analyses for sodium and potassium content were carried out according to the method described in BS 4550.

2. MATERIALS

2.1 Portland Cement

A high alkali cement from a particular works of a major UK producer was used. The relevant chemical and physical properties of this cement were: relative density = 3.03, % Na_2O = 0.40, % K_2O = 1.17, % Na_2O_{eq} = 1.17.

* Now of Harry Stanger Ltd., Materials Consultants, Elstree, England.

2.2. Pulverised Fuel Ash

A commercially available pulverised fuel ash was used of BS 3892 Part 1 quality and to Agreement Board Certificate No. 81/841. The relevant physical and chemical properties of the pulverised fuel ash were: relative density = 2.33, water soluble alkalis, $\text{Na}_2\text{O} = 0.13\%$, $\text{K}_2\text{O} = 0.07\%$, $\text{Na}_2\text{O}_{\text{eq}} = 0.18\%$; total alkalis, $\text{Na}_2\text{O} = 1.32\%$, $\text{K}_2\text{O} = 2.36\%$, $\text{Na}_2\text{O}_{\text{eq}} = 2.87\%$.

2.3 Aggregates

Two reactive and one non-reactive aggregates were used, these being pyrex glass, Beltane opal and crushed sand respectively. All three materials were crushed and graded to the requirements of ASTM C 441-69. Their properties are listed in Table 1.

Table 1 - Properties of Aggregates

	Relative Density		Water Absorption
	Oven Dry	Saturated Surface Dry	
Sand	2.56	2.60	1.4%
Pyrex Glass	2.23	2.23	Negligible
Beltane Opal	2.02	2.08	3.2%

2.4 Mix Designs

A four digit number has been used to describe each of the mixes examined in the full study. The digits in the mix reference numbers are in the order cement - pfa - aggregate - water. Thus, for this paper, the first digit denotes the use of the high alkali cement, the second the percentage replacement of the cement by pfa, the third the proportions of sand and reactive aggregate relative to the total aggregate and the fourth digit denotes the water content of the mix. The letter following the four digit number shows the type of reactive aggregate used, P = pyrex glass, B = Beltane opal. The full details of each mix are given in Table 2 where, for example, mix reference number 2121P contained a high alkali cement with 15% cement replacement by pfa (relative to mix 2021P which contained no pfa), the aggregate consisted of 33.3% sand and 66.6% pyrex glass and a high water content was used. The corresponding mix with a low water content was 2120P.

For those mixes which did not contain pfa, free water/cement ratios of 0.40 and 0.55 by mass were used for the low and high water contents respectively. However, for mixes which did contain pfa, the free water/cementitious ratios by mass varied slightly from the above figures due to the differences in the relative density of cement and pfa and the manner in which replacement was conducted. The free water contents when quantified as percentages of the total mass of each mix varied from 10.9 to 11.3% (low water content) and 14.4 to 15.0% (high water content).

The volume of cementitious material was kept constant and equal to the volume of Portland cement in the mixes containing no pfa. Thus, for a 15% replacement of cement by pfa the cementitious mixture was 15% pfa and 85% Portland cement by mass. It therefore followed that, because of the difference in the relative densities of pfa and Portland cement, the total mass of cementitious material in a blend was less than the mass of cement in a mix containing no pfa.

Table 2 : Mix Design of Bars

Level Ref. No.	Parameter			
	Cement	pfa replacement of cement	Aggregate	Free Water Content
0		0%	100%S	Low
1	High Alkali	15%	66%S/33%P or B	High
2		30%	33%S/66%P or B	
3		45%	100%P or B	
4		96%S/4%P or B		
5		92%S/8%P or B		
6		88%S/12%P or B		
7		84%S/16%P or B		
N.B. S = sand : P = Pyrex glass : B = Beltane opal				

3. EXPERIMENTAL

Three bars of each mix were made and stored according to the methods of ASTM C277-71 and ASTM C 441-69 and measurements of expansion were taken at ages of 14 days, 2 months, 6 months, and 12 months. After 6 months one of the three bars was removed for chemical analysis leaving two bars for the determination of expansion at 12 months. The liquid at the bottom of the expansion tanks was removed for chemical analysis and replaced with fresh water. The alkali analyses were carried out according to the method given in BS 4550.

The averages of the expansions observed for the bars of each mix are given in Tables 3 and 4. The alkali analyses of the bars and of the solutions from the bottom of the expansion tanks are given in Tables 5 and 6. For ease of comparison the alkali contents of the solutions are quoted with respect to the mass of the bars from which they were derived. The relative densities quoted in Tables 5 and 6 were those determined at the time of the chemical analyses and were obtained by measuring the linear dimensions and masses of the bars. There was a close correspondence between the densities determined this way and those calculated from the densities of the ingredients and the mix design of each bar. The pH of the solutions at the bottom of the expansion tanks ranged from 10.1 to 11.2.

4. RESULTS

4.1 Bars Containing Pyrex Glass

1. The most rapid expansion occurred during the first two months and, in general, there was little further expansion up to twelve months (vid. 2000 to 2030P and 2001 to 2071P).
2. An increase in the expansion was observed as the pyrex glass content of the bars was increased (vid. 2000 to 2030P and 2001 to 2071P).
3. The expansion of the bars increased as the water content of the mix was increased (cf. 2000 to 2030P with 2001 to 2031P).
4. There was a trend towards an increase in the amount of acid soluble alkali present in the bars as measured at six months and a decrease in the amount of alkali leached from the bars as the amount of pyrex glass in the bars was increased (vid. 2000 to 2030P and 2001 to 2071P).
5. Only a very slight indication of a pessimum content for pyrex glass was observed, possibly in the sequence 2000 to 2030P.

4.2 Bars containing Pyrex Glass and Pulverised Fuel Ash

6. There was a marked reduction in the measured expansions when pfa was used as a partial replacement for some of the high alkali Portland cement (vid. 2010P to 2310P, 2020P to 2320P, 2030P to 2330P, 2011P to 2311P, 2021P to 2321P and 2031P to 2331P).

7. Relative to the amount of pfa used the most marked reduction in expansion occurred when 15% of the Portland cement was replaced by pfa, but further reductions in expansion were observed at 30% and 45% replacement levels (vid. the mixes listed under 6 above).

8. Relative to the expansions observed when pfa was absent there was a greater reduction in expansion for those mixes with the higher water content (cf. 2010P to 2310P with 2011P to 2311P, 2020P to 2320P with 2021P to 2321P, 2030P to 2330P with 2031P to 2331P).

9. There was a considerable increase in the acid soluble alkali contents of all the bars containing pfa compared with the bars in which Portland cement was the only cementitious component and, using the same comparison, there was a decrease in the amount alkali leached from the bars (vid. the mixes listed under 6 above).

4.3 Bars Containing Beltane Opal

10. The most rapid expansions occurred during the first two months and there was little change between six and twelve months (vid. 2011B to 2071B).

11. There was considerably less expansion with the mixes containing Beltane opal than with their pyrex glass counterparts (cf. 2011P to 2071P with 2011B to 2071B).

12. A pronounced pessimum was observed when 4% of the aggregate was Beltane opal (vid. 2011B to 2071B).

13. There was a marked reduction in the amount of alkali leached from the bars when Beltane opal was used as expansive aggregate (vid. 2011B to 2071B).

4.4 Bars containing Beltane Opal and Pulverised Fuel Ash

14. At the pessimum concentration of Beltane opal there was a marked reduction in the amount of expansion observed (cf. 2041B with 2341B). At all other Beltane opal concentrations the use of pulverised fuel ash at high cement replacement levels and in bars with a high water content increased the amount of expansion to a small extent (vid. 2301B to 2371B). This increase in the expansion was very small compared with the decreased observed with comparable mixes containing pyrex glass (cf. 2011P-2311P, 2021P-2321P and 2031P-2331P with 2011B-2311B, 2021B-2321B and 2031B-2331B).

15. There was a considerable increase in the amount of dilute acid soluble alkali present in the bars when pfa was used as a partial replacement of some of the Portland cement. Despite this there was very little difference in the amount of alkali leached from the bars (cf. 2011B to 2071B with 2311B to 2371B).

4.5 Bars Containing No Expansive Aggregate

16. The substitution of Portland cement by pfa caused an increase in the acid soluble alkali content of the bars and a decrease in the amount of alkali leached from the bars (vid. 2000 to 2300 and 2001 to 2301).

17. There were regular but non-linear relationships between the amount of alkali in the bars, the amount of alkali leached from the bars and the level of pfa substitution (vid. 2000 to 2300 and 2001 to 2301).

5. DISCUSSION

The results show that when pyrex glass is used as a reactive aggregate the partial replacement of a high alkali Portland cement by a pfa of BS 3892 Part 1 quality produces a significant reduction in the expansion of mortar bars. This is true for all of the pfa and pyrex glass levels studied. Other workers have found similar reductions in expansion (1).

Following the work of Stanton (2) it has been suggested than an upper limit be placed on the alkali content of a Portland cement (expressed as $\text{Na}_2\text{O}_{\text{eq}}$) when used with potentially reactive aggregates. Subsequently it was recommended that an upper limit be placed on the acid soluble alkali content of concrete containing reactive aggregates (3). However, such an approach should only be used for those alkalis which are rapidly released and this, in most cases, implies a calculated figure based on the mix design and the alkali content of the Portland cement. The results in this paper demonstrate that the alkali content of mortars containing pfa and which show a marked reduction in expansion compared to their Portland cement controls can be very much in excess of that calculated from the Portland cement. These "extra" alkalis have been liberated by the acid-base or pozzolanic reaction of the pfa and should not be directly related to an equivalent hydroxyl ion concentration.

The reduction in the amount of alkali leached from the bars which contained pfa, despite the fact that more soluble alkali was present, can be explained in terms of the lower permeability of these mixes to the migration of alkali metal ions and this has arisen because of the pozzolanic reaction of the pfa. The expansions observed for those mixes which contained Beltane opal as reactive aggregate were a factor of 10 smaller than those observed with comparable mixes containing pyrex glass. In addition, there was an extremely prominent pessimum content, evident from the results shown in Table 5, in the region of 4% Beltane opal by weight of aggregate. This is in agreement with earlier published work (4,5).

It has also been suggested that the expansions tended to zero when the amount of Beltane opal was increased above the pessimum content. However, the results from this study show that this was not the case since, following the minimum in expansion with mixes containing 8 and 12% Beltane opal, there was a steady increase in expansion as the proportions of this expansive aggregate were increased further.

The differences in behaviour of the bars containing pyrex glass and Beltane opal maybe due to the fact that although they both act as hydroxyl ion "sinks" the former can also act as a source of its own positive counter-ions whereas the latter must accept these from the pore fluids. Thus the Beltane opal is a stronger "acid" than pyrex glass and could, in principle, act as a pozzolan in competition to the pfa so that the full benefit of the latter is not observed in the test method at high concentrations of Beltane opal. The pessimum position is, in effect, an example of the Law of Mass Action as stated by Guldberg & Waage (6) in which the most "reactive combination" depends on the concentration of reactive sites within the aggregate and the "availability" of hydroxyl ions to react with them.

Previous work has shown (7) that in pastes of pfa and calcium hydroxide there is a close correspondence between the rates of dissolution of the pfa and liberation of alkali metal ions to solution. If the same behaviour is true in mortars the alkali metal ion analyses can also be used to estimate the amount of reaction of the pfa. On this basis, and using the results from series 2000 to 2300, 91% of the pfa had reacted at a 15% replacement level, 87% had reacted at a 30% replacement level and 80% had reacted at a 45% replacement level.

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Table 3 Expansion of Bars containing Pyrex Glass

Mix Ref. No.	Expansion in Microstrain			
	14 Days	2 Months	6 Months	12 Months
2000	96	101	125	92
2100	71	71	104	96
2200	61	87	137	156
2300	91	113	133	140
2010P	1317	1953	1993	1952
2110P	539	597	597	584
2210P	301	335	371	372
2310P	253	299	331	328
2020P	3447	4065	4099	4112
2120P	1307	1441	1479	1464
2220P	855	916	964	964
2320P	501	532	568	572
2030P	3643	3843	3887	3920
2130P	1743	1873	1924	1936
2230P	1413	1505	1556	1552
2330P	985	1047	1077	1084
2001	92	95	105	88
2101	125	124	156	156
2201	96	129	177	176
2301	96	131	163	152
2011P	1309	2931	3048	3028
2111P	508	969	1732	1864
2211P	301	407	491	520
2311P	187	237	309	300
2021P	3432	6217	6467	6612
2121P	1768	2911	3557	3676
2221P	707	904	1164	1448
2321P	375	465	543	600
2031P	4680	6777	6948	6848
2131P	2163	2919	3201	3288
2231P	1125	1461	1872	2104
2331P	593	683	600	664
2041P	117	168	181	192
2051P	153	228	245	232
2061P	232	419	489	472
2071P	372	705	771	772

Table 5 Alkali Contents of Bars containing Pyrex Glass

Mix Ref. No.	Density kg/m ³ *	Na ₂ O eq kg/m ³ **	*** Analysis of bar			*** Analysis of liquid below each bar		
			%Na ₂ O	%K ₂ O	Na ₂ O _{3q} kg/m	%Na ₂ O	%K ₂ O	Na ₂ O _{3q} kg/m
			2000	2230	7.09	0.094	0.229	5.46
2100	2250	5.85	0.115	0.308	7.16	0.015	0.046	1.01
2200	2210	4.57	0.124	0.357	7.93	0.013	0.035	0.80
2300	2240	3.52	0.148	0.349	8.47	0.013	0.027	0.69
2010P	2220	7.08	0.137	0.266	6.93	0.018	0.036	0.93
2110P	2210	5.77	0.102	0.349	7.34	0.015	0.033	0.82
2210P	2140	4.45	0.143	0.343	7.90	0.015	0.034	0.82
2310P	2170	3.41	0.146	0.311	8.11	0.008	0.019	0.46
2020P	2140	6.83	0.105	0.301	6.12	0.017	0.022	0.66
2120P	2120	5.53	0.191	0.343	8.84	0.014	0.023	0.61
2220P	2120	4.41	0.167	0.344	8.72	0.016	0.028	0.72
2320P	2110	3.33	0.175	0.361	8.71	0.013	0.022	0.57
2030P	2110	6.77	0.102	0.313	6.29	0.013	0.017	0.51
2130P	2060	5.40	0.202	0.337	8.73	liquid lost		
2230P	2030	4.22	0.190	0.332	8.28	0.018	0.024	0.69
2330P	2040	3.22	0.183	0.316	7.98	0.011	0.019	0.49
2001	2070	6.31	0.081	0.205	4.47	0.026	0.081	1.64
2101	2150	5.38	0.097	0.294	6.24	0.020	0.054	1.20
2201	2130	4.24	0.111	0.335	7.05	0.021	0.051	1.17
2301	2110	3.17	0.135	0.349	7.70	0.014	0.028	0.68
2011P	2060	6.30	0.148	0.253	6.47	0.022	0.042	1.03
2111P	2140	5.37	0.148	0.301	7.40	0.023	0.049	1.18
2211P	2120	4.22	0.143	0.349	7.91	0.019	0.040	0.95
2311P	2060	3.11	0.124	0.319	6.88	0.015	0.031	0.72
2021P	2090	6.42	0.179	0.284	7.65	0.016	0.023	0.65
2121P	2070	5.20	0.177	0.329	8.14	0.019	0.034	0.85
2221P	2060	4.12	0.152	0.329	7.58	0.016	0.031	0.74
2321P	2050	3.10	0.156	0.336	7.73	0.015	0.028	0.68
2031P	2000	6.16	0.088	0.301	5.72	0.011	0.014	0.40
2131P	2020	5.09	0.193	0.307	7.98	0.016	0.026	0.67
2231P	2010	4.02	0.186	0.319	7.96	0.016	0.024	0.64
2331P	1990	3.02	0.175	0.361	8.20	0.017	0.031	0.74
2041P	2180	6.65	0.071	0.196	4.36	0.023	0.066	1.44
2051P	2190	6.68	0.074	0.194	4.42	0.023	0.064	1.42
2061P	2170	6.62	0.089	0.217	5.03	0.028	0.068	1.58
2071P	2150	6.56	0.084	0.206	4.73	0.024	0.058	1.33

* As determined at time of chemical analysis
 ** Calculated using the alkali content of the Portland cement
 *** Expressed with respect to the mass of each bar

Table 4 : Expansion of Bars containing Beltane Opal

Mix Ref No:	Expansion in Microstrain			
	14 Days	2 Months	6 Months	12 Months
2011B	88	139	164	172
2021B	84	172	216	236
2031B	120	235	295	304
2041B	276	493	529	528
2051B	144	189	200	200
2061B	112	152	159	152
2071B	109	140	153	156
2311B	112	238	262	264
2321B	127	293	337	324
2331B	160	368	416	396
2341B	129	219	251	268
2351B	121	223	247	248
2361B	109	224	239	244
2371B	92	223	252	224

Table 6 : Alkali Contents of Bars containing Beltane Opal

Mix Ref No.	Density kg/m ³ *	Na ₂ O _{eq} kg/m ³ **	*** Analysis of Bar			*** Analysis of liquid below each bar		
			%Na ₂ O	%K ₂ O	Na ₂ O _{eq} kg/m ³	%Na ₂ O	%K ₂ O	Na ₂ O _{eq} kg/m ³
			2011B	2070	6.30	0.113	0.314	6.62
2021B	1980	6.00	0.109	0.295	6.00	0.001(5)	0.004(3)	0.086
2031B	1910	5.77	0.098	0.286	5.47	0.001(7)	0.006(7)	0.117
2041B	2180	6.65	0.089	0.251	5.54	0.009(1)	0.025(3)	0.561
2051B	2180	6.65	0.098	0.280	6.15	0.005(0)	0.014(1)	0.311
2061B	2170	6.62	0.102	0.275	6.14	0.003(2)	0.008(5)	0.191
2071B	2160	6.59	0.105	0.292	6.42	0.003(4)	0.008(6)	0.196
2311B	2070	3.10	0.147	0.386	8.30	0.003(9)	0.009(8)	0.214
2321B	1970	2.94	0.140	0.372	7.58	0.002(6)	0.006(4)	0.134
2331B	1890	2.81	0.148	0.395	7.71	0.001(7)	0.006(1)	0.108
2341B	2140	3.21	0.128	0.337	7.48	0.008(9)	0.020(2)	0.475
2351B	2120	3.18	0.140	0.352	7.88	0.005(4)	0.013(7)	0.306
2361B	2120	3.18	0.138	0.353	7.85	0.005(6)	0.012(8)	0.297
2371B	2130	3.20	0.139	0.359	7.99	0.004(6)	0.010(7)	0.248

* As determined at time of chemical analysis
 ** Calculated using the alkali content of the Portland cement
 *** Expressed with respect to the mass of each bar

EFFECT OF SILICA, PFA AND SLAG ADDITIVES ON THE COMPOSITION OF CEMENT PORE FLUIDS

J. Marr and F.P. Glasser

Department of Chemistry, University of Aberdeen
 Meston Walk, Old Aberdeen AB9 2UE, Scotland UK.

The pore fluid of a moderately low alkali cement with and without additives has been analyzed after 30 and 90 d cure. During this time, typical PFA's are not sufficiently reactive at ~18°C to affect significantly the aqueous phase composition and pH, but slag and a chemically-produced SiO₂ do exhibit significant reactivity. These data are assessed with a view to designing long-term experiments relevant to elucidating the chemical evolution of the aqueous phase in real cement-based systems.

Pore fluid, PFA, Slag.

1. INTRODUCTION

Slag, PFA and other pozzolanic addition appear to be helpful in controlling the extent to which alkali-aggregate reaction occurs in set cements. These materials might act by effectively removing alkali from the aqueous pore fluid, thereby lowering the alkalinity of the system. Once setting has occurred, it is difficult to sample directly the pore fluid composition, although this has been partly surmounted by the development of powerful squeezers which can extract a sample of the pore fluid even from set cements. Using squeezer designs developed elsewhere /1,2/ we report on exploratory studies on the role of pozzolans in controlling the pore fluid composition.

2. MATERIALS

A cement having a moderately low alkali content was selected. Additives used include (i) a chemically reactive, high surface area silica produced by flame hydrolysis of SiO₂ and sold commercially by Degussa (FK 320) (ii) ground iron blast-furnace slag, containing > 98% glass, ex Ravenscraig (iii) a suite of five British PFA materials consisting of a siliceous glass and having a variable content of crystalline materials (e.g., mullite, spinel, quartz, feldspar) and (iv) a natural Italian pozzolan, consisting largely of crystalline phases. Each sample was analyzed for Na and K contents by flame emission, following LiBO₂ fusion with the results shown in Table 1.

Table I: Alkali Contents.

Material	(wt %) Na ₂ O	K ₂ O
OPC	0.19	0.48
Degussa SiO ₂	0.88	< 0.02
Slag	0.37	0.64
PFA		
Barony	0.33	3.04
Eggborough	1.14	3.73
Fiddler's Ferry	0.81	3.43
Ironbridge	0.84	2.83
Longannet	0.28	1.39
Natural Pozzolan	0.59	2.67

The cement has a moderate-to-low alkali content with K₂O > Na₂O. The flame hydrolysis silica also has a substantial alkali content, especially of Na, relative to the cement. Slag, while normally considered to be low in alkali also has a comparatively high alkali content while the PFA materials, as well as the natural pozzolan, all have a high alkali content such that K₂O > Na₂O. Thus the total alkali content of the reactive components of composite materials made with these additives is likely to be raised or at least maintained, relative to neat cement. In the examples studied the additives, if reactive, do not dilute the alkali content of the system.