

## AUTOCLAVE TESTING OF CONCRETE WITH RESPECT TO AAR

W. J. French

Geomaterials Research Services Limited, 1 Falcon Park, Crompton Close, Basildon,  
Essex, U.K. SS14 3AL

Caroline Tye,

Department of Civil Engineering, Imperial College, Exhibition Road, London, U.K.

### ABSTRACT

Concrete prisms and mortar bars have been subjected to periods of from 4 to 16 hours at 120°C and 0.1 MPa in an autoclave. The aggregate used was crushed granodiorite and quartz sand to which 15% of fused silica had been added. Alkali concentrations were made up to 4% by weight of cement using sodium hydroxide. In addition, some sodium and potassium silicates and silica gel were added to sets of bars. The purpose of this was to see if these reactants and reaction products influenced the rate and magnitude of reaction. The results showed that gel formation and cracking was generated by the autoclave procedure at a level comparable with two or more decades of field exposure. The added compounds took part in the reaction and where potassium silicate was added the gels became potassium-rich despite the presence of abundant sodium in the pore fluid. The technique could allow concrete designed for structural use to be tested directly at lower alkali concentrations than used here. The potential for further expansion in previously damaged concrete could also be evaluated and tests are in hand to evaluate this possibility, the first of which showed no further expansion.

*Keywords: Autoclave, concrete, fused silica, sodium silicate, potassium silicate.*

### INTRODUCTION

This paper describes some tests carried out on concrete containing reactive aggregate (fused silica) to which various substances were added. The purpose of the work was to evaluate the effects of the additions on the magnitude of expansion and cracking. The concrete was made with the alkalis enhanced to 4% by weight of cement using NaOH solution and the concrete was heated in an autoclave to accelerate the reaction some days after curing. The period of heating varied from four to sixteen hours. The added materials were chosen on the basis that they were either adding to the reactants or to the potential products. They are listed in Table 1. Two suppositions were to be tested. Firstly it was postulated that the addition of reaction product or allied substances would reduce reaction by preventing further product formation. Alternatively, it was supposed that the added reaction product would break down by reaction with the cement paste to liberate additional alkalis so that reaction is enhanced.

*Table 1 Compounds added to the concrete prisms  
(the admixtures)*

A	Sodium silicate solution	$\text{Na}_2\text{O}/\text{SiO}_2 \sim 0.5$
B	Sodium silicate solution	$\text{Na}_2\text{O}/\text{SiO}_2 \sim 0.3$
C	Zeolite powder	$\text{Na}_2\text{OAl}_2\text{O}_3\cdot 2\text{SiO}_2$
D	Potassium silicate solution	$\text{K}_2\text{O}/\text{SiO}_2 \sim 0.25$
E	Sodium silicate powder	$\text{Na}_2\text{O}/\text{SiO}_2 \sim 0.4$
F	Sodium silicate powder	$\text{Na}_2\text{O}/\text{SiO}_2 \sim 0.5$
G	Silica gel powder	$\text{SiO}_2$
H	Sodium silicate powder	$\text{Na}_2\text{O}/\text{SiO}_2 \sim 1$

## CONCRETE

The concrete was made with the following recipe.

Water	Approximately 180 kg/m <sup>3</sup>
Cement	400 kg/m <sup>3</sup>
Sand (0.073 to 3.18 mm)	450 kg/m <sup>3</sup>
Fused silica (mostly 3 to 5 mm)	270 kg/m <sup>3</sup>
Coarse aggregate (5 to 20 mm)	1080 kg/m <sup>3</sup>

Cube tests of the control concrete gave a strength at 28 days of 48 MPa and a bulk density of 2380 kg/m<sup>3</sup>. The coarse aggregate was a crushed granodiorite which was carefully washed and thoroughly dried. Similarly, the fine aggregate was a fine quartz sand which was washed and oven dried. The reactive part of the aggregate, the pure silica, was sieved to separate the fraction from 1 to 5 mm and this was used in the test. In practice, most of the material was in the size 3 to 5 mm. This was added to the sand to make a total of 15% of the total aggregate. The cement employed was an ordinary portland cement to BS12. Sodium hydroxide was added to the water to produce a final concentration in the concrete of 4% Na<sub>2</sub>O equivalent by mass of cement (Criaud et al 1992, Fournier et al 1991, Saloman and Gallias, 1992, Tang and Han 1983a and b, and Tang et al 1987). It was necessary to carry out some trials with this in order to establish the correct recipe for the water content because the addition of the NaOH greatly stiffened the mixture. Having added the alkaline solution to obtain the necessary alkali to cement ratio, further water was added until the required workability was achieved. The mixes had to be blended and cast rapidly because it was found that the added materials acted variously as accelerators. The added ingredients were mainly added as solutions with the concentrations being used to make effectively 2 and 5% by volume (20 and 50 ml/L) of the total mixture added as part of the aqueous phase. The solutions of admixture used contained 14% by mass of Na<sub>2</sub>O equivalent. The cement and solution or suspension was first blended and then the aggregates were added.

Prisms measuring 75 x 75 x 250 mm were cast with vibration and these were cured for between 14 and 19 days in a moist atmosphere. They were then transferred to an autoclave operating at working conditions of 120°C and 0.10 MPa. The apparatus is a Swing Clave autoclave measuring 65 x 75 x 100 cm. This is normally used for

biological sterilisation. It requires an hour to reach the steady state condition and an hour to cool. Times referred to in Table 2 relate to the time at 120°C and 0.1 MPa. The tests were run overnight and samples were measured and examined the following day. All the prisms were studied using thin sections and with the electron microscope and with an attached energy dispersive analytical system.

Much gel and numerous microcracks were produced and the concrete after four hours resembled field concrete more than twenty years old and containing a highly reactive aggregate. There was some increase after 16 hours but the shorter time would have been sufficient to identify the potential for adverse reaction and to identify the reactive materials. It was found that after 16 hours a little of the siliceous sand and a few grains of the granodiorite also exhibited some reaction. Gel issued onto some of the prism surfaces and was removed and analysed using the energy dispersive system attached to the electron microscope. The prism lengths were measured using a Mitutoyo Digimatic Indicator type 1DF-130E at room temperature and the prisms were weighed at the time of measurement.

### EXPANSION TEST RESULTS

The results obtained are given in Table 2. The controls gave an average expansion of 0.37% in four hours. This compares closely with the expansion created in the ASTM C1260-94 test (ASTM 1994) which gave about 0.40% for the same aggregate. Mortar bars were also made of the same materials with alkalis enhanced to the same extent as those in the concrete and these, after being treated in the autoclave for four hours, gave a mean expansion of 0.39% for the control.

The use of sodium silicate with an  $\text{SiO}_2/\text{Na}_2\text{O}$  ratio of unity gave an effectively identical expansion at 2% addition, but the expansion was reduced substantially with 5% addition. All the other added compounds showed a slight increase in expansion relative to the control. However, the expansion at 5% addition was in six out of seven cases less than that at 2% addition. Further, while the controls reached the cited expansion after four hours, others showed a little less expansion after four hours, but matched or exceeded the controls after a further twelve hours in the autoclave. This suggests that the admixtures influence the structure of the binder and retard the early stages of the reaction, but that ultimately the generation of microcracking leads to an acceleration of the reaction and rate of expansion.

### COMPOSITION OF GELS

The petrographic examination of the concrete samples and analyses made with the electron microprobe showed a structure identical with those found in examples of concrete taken from the field. Cracks radiate from within the aggregate particles and often link nearby reactive grains. Gel occurs within the aggregate, in cracks in the paste, and in voids, and was found to issue on to the formed surfaces of the prisms in some instances. These gels have been analysed with the results given in Table 3. The analyses show that the gel compositions are similar to those found in field concrete exhibiting adverse reactions. The gels in the aggregate have high alkalis and low calcium, while those in the cracks in the paste have high calcium (Figure 1)(French

1995). Gel exudations, which have not had access to the cement paste, also have high alkalis and low calcium. Where the added alkali-silicate was potassic the gels produced were also potassium-rich so that clearly the added silicates are taking part in the reactions. It seems that the entry of the potassium ions may take precedence over the sodium ions since sodium was always in excess.

Table 2. Expansion of concrete prisms after a period in the autoclave

Samples	admixture (see table 1)	time in autoclave (hours)	expansion %
1	none	4	0.343
2	none	4	0.387
3	H 2%	4	0.366
4	H 5%	4	0.237
5	E 2%	4	0.418
6	E 5%	4	0.368
7	F 2%	4	0.392
8	F 5%	4	0.391
9	C 2%	16	0.465
10	C 5%	16	0.430
11	A 2%	16	0.510
12	A 5%	16	0.447
13	D 2%	14	0.528
14	D 5%	14	0.459
15	G 2%	14	0.457
16	G 5%	14	0.360
17	B 2%	14	0.567
18	B 5%	14	0.551

## DISCUSSION

No substantial differences were found between the controls and the prisms to which compounds had been added. Observations of cracks generated in the aggregate, cracks produced in the paste, dissolution of the aggregate, gel infilling voids, and gel total quantity, show very little difference from one sample to another. The qualitative observations were scaled from 1 to 5 with 5 indicating the greatest degree of reaction. If these values are summed, then the samples were found to range from 18 to 25, indicating that the most damage occurred where the additional material was dispersed as silica gel. However, this was only marginally less reactive than where potassium silicate ( $K_2O/SiO_2=1$ ) was employed.

Since the addition of potassium silicate to the mixture led to enhanced potassium in the gel it is evident that the admixtures have taken part in the reaction. Gel in the paste has a higher calcium content than that in the aggregate and this is interpreted as a result of reaction between gel and paste leading to release of alkali into the pore fluid. It seems likely therefore that the admixtures may also have reacted with the paste to

liberate alkali metal ions into the pore fluid and hence have enhanced the potential for reaction. The conditions employed in the tests (120° and 0.1MPa) for between four and sixteen hours are very severe and led to some reaction with the granodiorite and quartz sand. These materials are regarded as very stable when used in field concrete. It therefore seems likely that designed concrete mixtures could be tested by this procedure but that lower alkali concentrations should be employed. Experiments are in hand to test these possibilities. It is also possible that the potential for further expansion for concrete already exhibiting some reaction could be evaluated rapidly. Four cores from structures exhibiting reaction were tested in this way all of which showed no further expansion.

Table 3. Chemical analyses of gel in various locations and some areas of paste.

Sample	2	2	2	2	2	6	6	6	6	6	6
Admixture	none	none	none	none	none	E 5%					
(see table 1)											
Location	B	B	B	C	B	D	D	D	C	B	B
SiO <sub>2</sub>	84.4	73.8	83.6	79.2	78.9	45.4	47.2	66.7	68.9	76.9	83.7
Al <sub>2</sub> O <sub>3</sub>	0.0	0.0	0.2	0.3	0.2	0.3	0.0	0.2	0.0	0.0	0.1
Fe <sub>2</sub> O <sub>3</sub>	0.0	0.3	0.1	0.4	0.0	0.1	0.0	0.3	0.2	0.1	0.1
MgO	0.3	0.4	0.4	0.1	0.4	0.1	0.0	0.0	0.5	0.4	0.2
CaO	3.4	13.2	3.8	12.3	8.9	48.0	48.0	28.8	20.0	11.2	7.0
Na <sub>2</sub> O	6.6	6.3	7.2	3.2	6.0	2.4	0.2	1.7	6.1	7.8	6.5
K <sub>2</sub> O	5.4	5.7	4.6	4.4	5.2	0.7	0.3	1.0	4.2	3.4	2.2
SO <sub>3</sub>	0.1	0.3	0.1	0.1	0.1	3.0	3.9	0.2	0.2	0.1	0.2
Sample	6	6	6	6	6	6	10	10	10	10	10
Admixture	E 5%	C 5%									
(see table 1)											
Location	D	C	B	B	B	A	E	E	E	B	C
SiO <sub>2</sub>	68.0	77.7	60.8	85.3	85.9	87.6	37.0	23.2	25.1	83.8	83.8
Al <sub>2</sub> O <sub>3</sub>	0.4	0.0	1.8	0.0	0.1	0.0	6.0	7.2	7.5	0.1	0.0
Fe <sub>2</sub> O <sub>3</sub>	0.1	0.2	0.3	0.0	0.0	0.0	2.3	3.0	3.0	0.3	0.2
MgO	0.9	0.5	0.6	0.4	0.2	0.5	1.8	2.4	2.4	0.5	0.5
CaO	21.7	9.6	23.0	1.8	4.0	0.5	47.1	29.7	57.7	0.2	0.5
Na <sub>2</sub> O	7.1	8.7	10.8	10.1	7.6	8.8	1.9	0.8	0.8	11.6	9.9
K <sub>2</sub> O	1.3	3.3	1.2	2.0	2.2	2.2	0.7	0.3	0.3	3.3	4.9
SO <sub>3</sub>	0.3	0.1	1.2	0.2	0.1	0.2	2.9	2.8	2.8	0.2	0.4
Sample	10	10	10				14	14	14	14	14
Admixture	C 5%	C 5%	C 5%				D 5%				
(see table 1)											
Location	B	B	D				B	A	B	D	C
SiO <sub>2</sub>	81.3	76.3	83.6				81.5	87.0	81.6	78.7	65.2
Al <sub>2</sub> O <sub>3</sub>	0.1	0.2	0.2				0.1	0.2	0.2	0.1	0.7
Fe <sub>2</sub> O <sub>3</sub>	0.0	0.1	0.1				0.1	0.0	0.1	0.0	0.9
MgO	0.9	0.6	0.5				0.3	0.1	0.1	0.4	0.3
CaO	0.1	7.3	1.0				0.2	4.6	1.8	1.7	19.1
Na <sub>2</sub> O	14.0	11.3	7.4				6.8	3.1	3.4	8.0	2.8
K <sub>2</sub> O	3.2	3.9	4.4				10.6	4.9	12.8	11.0	10.9
SO <sub>3</sub>	0.4	0.0	0.2				0.3	0.0	0.0	0.1	0.2

Sample	16	16	16	16	16	16	16	16	16	16
Admixture (see table 1)	G 5%									
Location	A	B	B	A	F	C	A	A	D	B
SiO <sub>2</sub>	49.3	78.4	69.1	44.6	34.2	66.1	34.1	73.4	68.2	38.9
Al <sub>2</sub> O <sub>3</sub>	2.9	0.0	0.0	4.4	4.7	0.4	3.3	0.8	0.0	3.4
Fe <sub>2</sub> O <sub>3</sub>	0.5	0.0	0.3	0.0	2.8	0.0	0.2	0.0	0.2	0.1
MgO	0.0	0.1	0.2	0.3	2.0	0.4	0.2	0.3	0.4	0.0
CaO	43.5	11.3	18.8	16.8	52.3	16.8	55.5	11.6	17.8	53.5
Na <sub>2</sub> O	2.0	5.2	6.0	0.9	2.2	10.7	1.3	5.7	7.8	1.4
K <sub>2</sub> O	0.7	4.7	5.5	0.3	0.6	4.8	0.4	7.3	6.0	0.5
SO <sub>3</sub>	0.7	0.2	0.2	1.9	1.0	0.8	4.9	0.6	0.3	2.0
Sample	18	18	18	18	18		13	13	17	17
Admixture (see table 1)	B 5%		D 2%	D 2%	B 2%	B 2%				
Location	B	B	C	B	C		F	F	F	F
SiO <sub>2</sub>	77.4	73.7	87.0	85.1	72.7		71.8	72.0	74.0	74.9
Al <sub>2</sub> O <sub>3</sub>	0.0	0.0	0.0	0.0	0.0		0.0	0.0	0.0	0.0
Fe <sub>2</sub> O <sub>3</sub>	0.2	0.1	0.1	0.2	0.1		0.0	0.0	0.0	0.0
MgO	0.2	0.2	0.2	0.6	0.5		1.0	1.0	1.1	1.1
CaO	10.7	17.4	0.9	4.8	15.5		0.5	0.1	1.4	0.2
Na <sub>2</sub> O	6.8	4.4	8.3	5.3	6.5		18.8	18.2	17.9	17.8
K <sub>2</sub> O	4.7	4.1	3.5	3.9	4.7		8.5	8.6	5.3	5.9
SO <sub>3</sub>	0.0	0.0	0.0	0.0	0.0		0.0	0.0	0.0	0.0

Note: locations are as follows: A within aggregate, B aggregate surface, C in crack in paste, D in void, E paste analyses, F surface exudations.

## CONCLUSIONS

Concrete containing extra alkalis and a reactive aggregate, treated from 4 to 16 hours in an autoclave at 120°C and 0.1 MPa, shows significant gel formation, cracking and expansion. The expansion created is equivalent to two or more decades of reaction in the field. The addition of dispersed silica gel and soluble sodium and potassium silicates did not suppress the reaction, but if anything the rate of reaction slightly increased. This is interpreted as being due to the release of extra alkalis by reaction with the portland cement paste. The extent to which the autoclave generated expansion, cracking and gel formation in the concrete suggests that this may be a very simple way of detecting whether field concrete is likely to show further expansion or whether a designed concrete may show a potential for reaction. It was observed that some of the quartz sand and some parts of the granodiorite also showed traces of reaction after being held in the autoclave for 16 hours. These aggregates are from field experience known to be thoroughly stable. It seems likely therefore that a reliable test could be established at lower alkali levels than were employed here by means of autoclaving concrete.

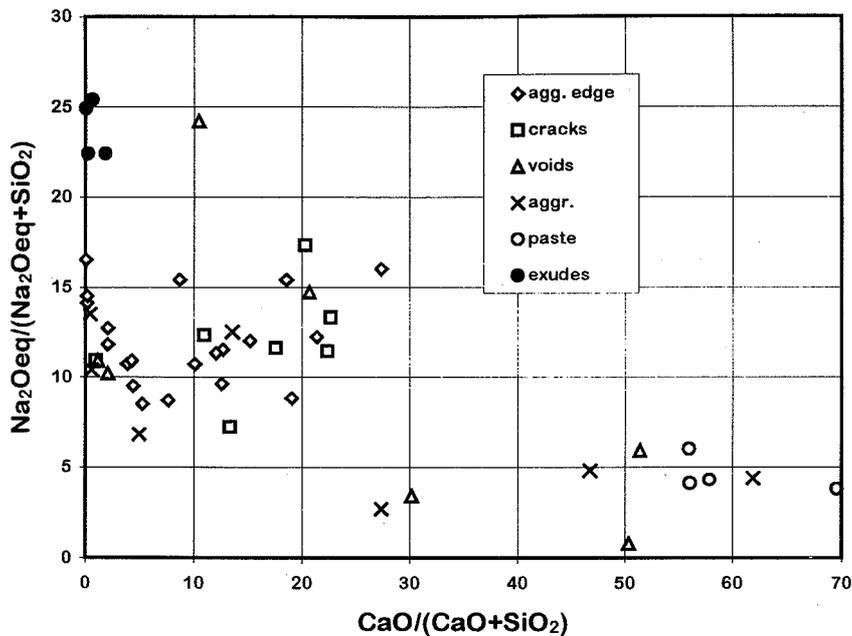


Figure 1. Variation of gel composition with location in the concrete. Note that the values labelled aggregate but with high Ca are altered silica gel phases.

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