

**EXPERIMENTS WITH ACCELERATED TESTS
FOR PREDICTING ALKALI-AGGREGATE REACTIVITY**

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ABSTRACT

The slow rate of reaction between alkali in the pore solution of cement mortar and some aggregates has necessitated the development of rapid accelerated methods for predicting reactivity of aggregate in concrete.

In recent years attempts have been made worldwide to improve the methods for prediction of the potential reactivity of aggregates in concrete. In this study accelerated methods based on (1) storage of concrete prisms or mortar bars in 1M NaOH solution at 80°C, (2) storage of mortar bars made with 1M NaOH solution above water at 80°C in sealed conditions, (3) storage of mortar bars made with 1M NaOH solution in water at 80°C, and (4) storage of mortar bars and concrete prisms in saturated NaCl solution at 50°C have been applied to a number of aggregates with a wide range of rock types, including aggregates known to have reacted in existing concrete structures. Results obtained so far indicate that although very reactive aggregates may be identified by all these methods, method (1) was more suitable for the identification of slowly reactive aggregates. However, further testing is required to establish its general applicability to all rock types.

1. INTRODUCTION

The need for accelerated testing of aggregates for alkali-aggregate reaction has arisen largely because some of the slowly reactive aggregates are not correctly classified by current standard methods. Thus, researchers from various countries have attempted to develop faster and more reliable methods for predicting the alkali-reactivity of aggregates. The rate at which a reactive aggregate reacts with alkali depends largely on the level of alkali available, humidity and temperature, and high levels of these are common features in accelerated methods. Table 1 lists a number of such accelerated methods. Some of these test methods were developed and evaluated on the basis of testing highly reactive materials such as opal and perlite [2] or even synthetic fused silica and opal [8]. Such tests may not be applicable to more complex aggregates. One accelerated test [10] which involves dry heating at 82°C and water immersion cycles might produce expansion due to physical causes totally unrelated to alkali-reactivity. More recently Chatterji and Jensen [11] suggested that OH concentration changes in a mixture of aggregate (sand) and CaO suspended in a saturated KCl solution could be the basis of a simple chemical test, but this needs to be evaluated on a wide range of aggregates. This paper presents the results of a number of modifications of accelerated testing at 80°C and that at 50°C using a wide range of Australian and overseas aggregates, some with a known history of poor performance in concrete structures.

Table 1. Some accelerated alkali-aggregate reactivity tests

Method	Specimen size (mm)	Cement alkali (Na ₂ O equiv.) (%)	Storage condition
Chatterji [1]	40x40x160	n.s.	Saturated NaCl solution, 50°C.
Tang <i>et al.</i> [2]	10x10x40	n.s.	Autoclave 4 hours at 100°C then autoclave in 10% KOH solution for six hours.
Oberholster & Davies [3]	25x25x285	n.s.	1M NaOH solution at 80°C.
Abe <i>et al.</i> [4]	25x25x285	2.5	Autoclave for 3 cycles of 2 hours at 100°C and 100% RH, and 90 minutes at 20°C.
Nishibayashi <i>et al.</i> [5]	40x40x160	1.5	Autoclave for 4.5 hours at 120-130°C.
Tamura [6]	40x40x160	2.5	Autoclave for 2 hours at 110°C.
Yoshioka <i>et al.</i> [7]	25x25x285	n.s.	24 hours saturation in 1M NaOH at 80°C, 24 hours cooling at 20°C and storage for 72 hours at 80°C and 100% R.H.
Swamy & Al Asali [8]	75x75x300	n.s.	Storage in salt water bath at 40°C.
Shayan <i>et al.</i> [9]	25x25x285 75x75x285	n.s.	1M NaOH at 80°C

n.s. = not specified. Alkali level is probably unimportant because the storing solution contains a high level of alkali.

Note: 75x75x285 (or 300) mm size refers to concrete prisms; other sizes refer to mortar bars. See appropriate references for mix design.

2. EXPERIMENTAL

2.1 Equipment

The equipment used for accelerated testing at 80°C was a thermostatically controlled tank of 200 l capacity described by Shayan *et al.* [9]. The tank was either filled with sufficient water or 1M NaOH to cover the specimens, or enough water was added to cover the heating element and to provide steam without water contacting the specimens. The experiment with a saturated NaCl solution at 50°C was carried out in a large plastic bath filled to a depth of 150 mm with the solution, then sealed with plastic sheeting and stored in a 50°C constant temperature room.

2.2 Materials

The aggregates used in the testing program included a river gravel (CG) and a siliceous limestone (CSP) from Canada; a metagreywacke from South Africa (SA); an andesite from Japan (JA); a metadolerite from Perth, Western Australia (GSN); and a phyllite from Victoria, Australia (UY), all of which have reacted in concrete structures in service. Also tested were two known non-reactive basalts (DP and PIO) from Victoria; and two river gravels (TKA and HC) from Western Australia, which were shown experimentally to be reactive (Shayan *et al.* [12]). Other aggregates tested were a suspect sandstone (NBB); three metadolerites (HHM, JDM, TRM); two granites (GSG, LRG); and a river gravel (SPG), all of unknown service record from Western Australia.

A Type A cement containing 0.84% available alkali (Na_2O equivalent), and a known non-reactive sand, when required, were used in the preparation of the specimens. Higher levels of cement alkali were achieved by the addition of NaOH to the mixing water.

2.3 Procedures

All the mortar bars were made according to the Australian Standard AS 1141-38 [13], and concrete prisms (75 x 75 x 285 mm) were of 40 MPa strength grade.

2.3.1 Testing at 80°C. For accelerated testing at 80°C, mortar bars were made either without any added alkali or with 1M NaOH solution instead of mixing water. They were cured and the zero reading at 80°C taken as described by Shayan *et al.* [9]. Specimens were then exposed to each of the following conditions:

- (a) Specimens with no added alkali were placed in a 1M NaOH bath at 80°C, and their length change monitored.
- (b) Replicate specimens with no added alkali were also treated similarly to those in (a) except that the initial fog curing was 14 days instead of 3 days. This was to check the effect of specimen age prior to exposure to the NaOH solution on subsequent expansion.
- (c) Specimens made with 1M NaOH solution were placed over water at 80°C (sealed condition). For each length measurement the specimens were briefly placed in water at 80°C to prevent drying and their length measured as before.
- (d) Replicate specimens made with 1M NaOH solution were also placed in water at 80°C and the length changes monitored.
- (e) Replicate specimens made with 1M NaOH solution were individually placed in a stainless steel tube so that a 2 mm layer of water was available around the specimen, and the tube was placed in the water bath at 80°C. This was to limit leaching of alkali from the specimens.

2.3.2 Testing at 50°C. For testing in saturated NaCl solution at 50°C, mortar bars and concrete prisms were demoulded 24 hours after casting, cured in fog for 28 days, measured for length after 24 hours equilibrium in a water bath at 50°C, and then placed in the saturated NaCl solution at 50°C and their length changes monitored.

3. RESULTS AND DISCUSSION

Figure 1 shows expansion curves for mortar bars subjected to the 1M NaOH solution at 80°C. Mortar bars made with all the known reactive aggregates expand 0.10% or more in 10 days, except those made with the UY aggregate. This is a very slowly reactive aggregate which, in this accelerated test, takes about 20 days to expand by 0.10%. Shayan *et al.* [9] suggested that for a 3-day pre-curing period, mortar bar expansion of 0.10% in 10 days and 0.10% in 22 days be taken as indicating reactive aggregates of short induction period and long induction period, respectively. Figure 1 also shows that mortar bars made with the two non-reactive basalts (DP and PIO) did not expand significantly, and that those made with the sandstone (NBB) appeared to show a pessimum effect.

Six aggregates of unknown reactivity from Western Australia were also subsequently subjected to the 1M NaOH solution at 80°C, and Figure 2 shows their expansion behaviour. Based on these results, the SPG, HHM and GSG aggregates are classed as reactive and LRG, TRM and JDM as non-reactive. Testing mortar bars by the standard method [13] has also shown that SPG and HHM aggregates are reactive and JDM aggregate is non-reactive. Because no service record

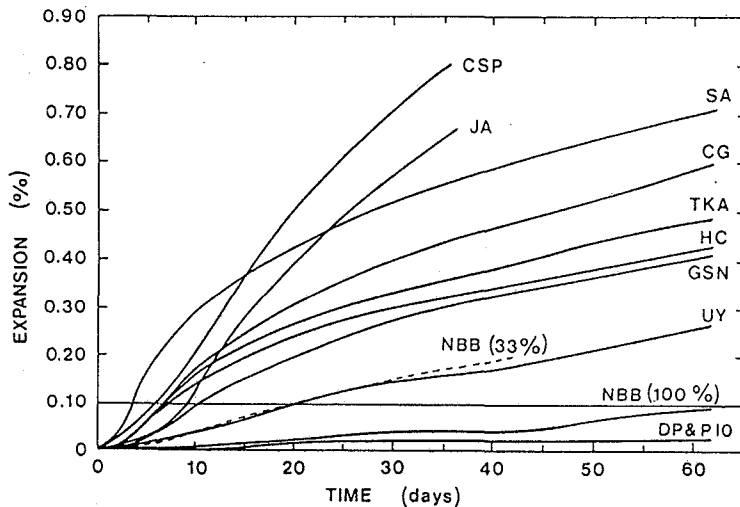


Figure 1. Expansion of mortar bars subjected to 1M NaOH solution at 80°C. Curve designations are those in Section 2.2.

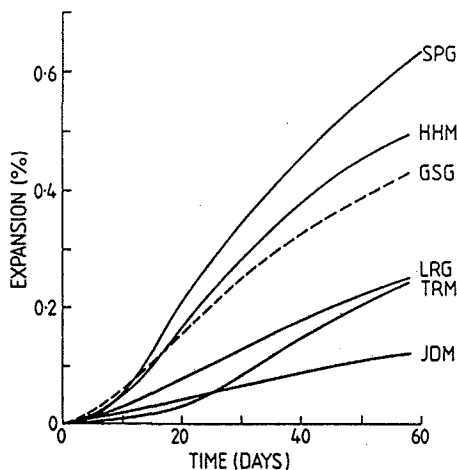


Figure 2. Expansion of mortar bars made with six WA aggregates and subjected to 1M NaOH solution at 80°C. Curve designations are those in Section 2.2

exists for these aggregates, longer testing times are required in order to correlate the results of accelerated and conventional standard testing for the GSG, LRG and TRM aggregates, although more recent work has indicated that the GSG aggregate is potentially reactive.

Table 2 shows the effect of precuring time on subsequent expansion in 1M NaOH (80°C). The longer curing time reduces the initial rate of expansion (except for JA) so that the criterion of 0.10% expansion at 10 days does not apply. However, all the reactive aggregates tested expanded far beyond 0.1% in 20 days regardless of the precuring period.

Concrete prisms were also subjected to 1M NaOH solution at 80°C, but the results were not encouraging for accelerated testing except for the case of a reactive dolomitic limestone (Shayan *et al.* [9]).

The expansion curves of mortar bars made with 1M NaOH solution and held over water at 80°C are shown in Figure 3. Although the rate of reaction was very high for SA, CSP, CG, and particularly JA aggregates, the three Australian reactive aggregates (GSN, TKA and HC) did not expand significantly. The tendency for a pessimum effect shown by NBB aggregate in the earlier method is also observed in this test. The very low expansion of mortar bars made with GSN, TKA and HC aggregates is consistent with their less reactive nature as indicated in Figure 1, but more moisture could probably enhance the expansion.

The expansion of mortar bars that were made with 1M NaOH solution and placed in water at 80°C was generally very low (below 0.03% at 36 days) except for that of mortar bars made with the JA, SA and CSP aggregates which expanded by 0.478, 0.131, and 0.090%, respectively, at 17

Table 2. Expansion at 10 and 20 days of mortar bars subjected to 1M NaOH at 80°C after precuring in fog for 3 or 14 days

Aggregate* designation	3-day precuring		14-day precuring	
	10 day (%)	20 day (%)	10 day (%)	20 day (%)
CS	0.175	0.311	0.070	0.274
GSN	0.097	0.207	0.030	0.149
TKA	0.163	0.270	0.094	0.284
SA	0.294	0.426	0.063	0.431
JA	0.170	0.418	0.238	0.671
BRN†	0.666	0.940	0.358	0.583

* See Section 2.2 for rock type.

† A highly reactive basalt from Australia.

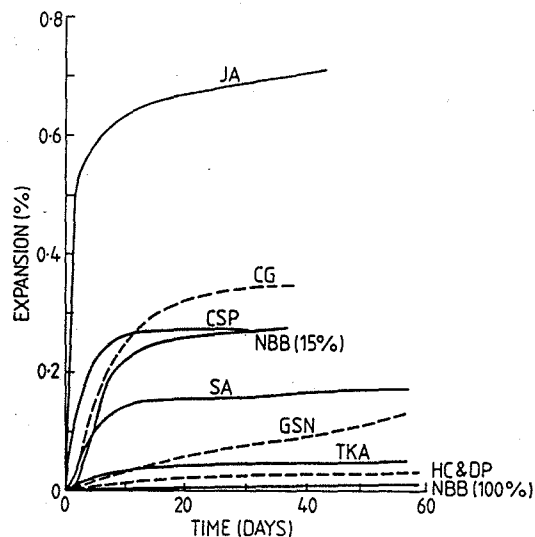


Figure 3. Expansion of mortar bars made with 1M NaOH solution and stored above water at 80°C in sealed conditions. Curve designations are those in Section 2.2.

subjected to the saturated NaCl solution at 50°C were very small even after six months, but at one year the specimens cracked. Additional alkali in the specimens (1.74% Na₂O equivalent) reduced the cracking time, but only to 20 weeks, implying that the method is unsuitable as an accelerated test for the Australian aggregates.

Based on the results obtained so far, it appears that storage of mortar bars in 1M NaOH solution at 80°C is the preferred method for accelerating alkali-aggregate reaction. However, more extensive testing, covering a much wider range of aggregates, is needed in order to verify its general applicability. In another paper in this conference (Shayan and Quick [14]) it has been

days. Although these mortar bars showed initial cracking, the expansion did not increase further probably because a significant proportion of the NaOH was leached out of the mortar bars. Analysis of the water showed that its NaOH concentration was 5.5×10^{-3} M. Considering that 45 mortar bars were immersed in about 190 l of water, it can be calculated that, on average, about 38% of the NaOH was leached out of the mortar bars. The lack of consistency in expansion of mortar bars made with the reactive aggregates makes this modification unsuitable as an accelerated test.

Limiting the amount of water available to each mortar bar to a 2 mm water layer (60 ml) in individual stainless steel tubes did not increase their expansion. After 18 days the concentration of NaOH in this amount of water was about 0.125 M, indicating that about 10% of the NaOH added to each mortar was leached out. Replacing the 60 ml of water with 60 ml 1M NaOH solution caused increased expansion of these mortar bars showing their potential expansivity. Probably the leaching of the reaction products was responsible for the low expansions that occurred in water. For the very reactive JA aggregate that caused a large mortar bar expansion under these conditions, the rate of formation of the reaction product was probably greater than its leaching rate.

Expansion values for mortar bars and concrete prisms made with the reactive Western Australian aggregates and sub-

shown that reaction products formed in this accelerated test and in conventional tests are similar to those found in concrete suffering from alkali-aggregate reaction.

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