

EVALUATION OF RAPID TEST METHODS
FOR DETECTING ALKALI-REACTIVE AGGREGATES

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ABSTRACT

Using 12 aggregates, mostly from Ontario, with known petrographic characteristics and field performances, a series of rapid test methods was evaluated along with standard ASTM C 227 mortar bar tests.

The rapid test methods involving mortar bars included those of Oberholster and Davies (14 days in NaOH at 80°C), Chatterji (8 to 20 weeks in NaCl at 50°C), Duncan (100% rh at 64°C), Nishibayashi (added NaOH in bars, steamed at 125°C for 4 hours), and Tang (steam cured, then autoclaved in KOH at 150°C for 6 hours).

While some tests are still in progress, the NBRI 14-day test published by Oberholster and Davies appears to be the most promising in terms of distinguishing between non-reactive, marginal and reactive aggregates. However, the expansion limits proposed by Oberholster and Davies may only be suitable for distinguishing non-reactive and very reactive aggregates. Other testing may still be required to distinguish slowly expansive aggregates from petrographically marginal aggregates with good field performance. As well, it was found that washing of the aggregate normally done in ASTM C 227 to remove dust, may not be required with this test.

While some of the other mortar bar methods may have been unfavourably influenced by the authors' decision to modify them by using ASTM standard mortar bars (25 mm by 25 mm cross-section) and C 227 aggregate gradations, many of the methods could not distinguish all of the reactive aggregates.

Based on these and other findings, the CSA Standards Committee is presently investigating the possible adoption of the 14-day NBRI procedure.

1. INTRODUCTION

There is an urgent need to develop and standardize the use of reliable rapid test methods for detecting deleteriously alkali-reactive aggregates. The widely used ASTM C 227 mortar bar method involves waiting at least 6 months and is not reliable in a reasonable time frame for detecting slowly-reactive aggregates. Numerous rapid methods have been proposed but there was a need to evaluate the more promising ones with respect to existing standard methods and known field performance within the Province of Ontario.

2. EXPERIMENTAL

2.1 Materials

A single, high alkali ($\text{Na}_2\text{O} = 0.39\%$, $\text{K}_2\text{O} = 1.18\%$, $\text{Na}_2\text{O} = 1.17\%$) portland cement was used. The 12 aggregates, mostly from Ontario, are described by their petrographic and field performance in Table 1. Included are known non-reactive, deleteriously reactive, and petrographically marginal aggregates.

2.2 Test Methods

2.2.1 ASTM C 227 The bars were stored upright at 100% rh and 38°C in wicked, rectangular, stainless steel containers capable of holding 18 bars. Inside the containers, three of the six bars in each set were placed in heat-sealed, polyethylene bags with approximately 10 ml of water. Previous testing had demonstrated that the bars in the bags would expand more rapidly if the aggregates were reactive. ASTM C 33 suggests expansion limits of 0.05% at 3 months and 0.10% at 6 months but many agencies including Ontario Hydro impose more stringent limits of 0.05% at 6 months and 0.10% at 12 months. However, with late-expanding, alkali-silicate reactive aggregate [1], 0.1% may not be exceeded for 18 to 24 months.

2.2.2 NBRI Method This method described by Oberholster and Davies [2,3] subjects ASTM C 227 mortar bars to a 1N NaOH solution at 80°C for 14 days. The bars are measured hot. Oberholster and Davies have suggested expansion limits of 0.10% for innocuous aggregates, 0.10 to 0.25% for slowly expansive aggregates, and greater than 0.25% for rapidly expansive aggregates [3].

2.2.3 Duncan Method In this method, the expansion of four ASTM C 227 bars was accelerated in an 100% rh, 64°C environment [4]. The 4 bars were sealed in polyethylene bags and then placed horizontally above water in a sealed plastic container. A limit of 0.05% at 16 weeks was suggested [4]. Four bars were cast in each set.

2.2.4 Danish Salt Method ASTM mortar bars were cast (instead of ISO mortar bars) and there were subjected to a saturated NaCl solution at 50°C while three more were subjected to deionized water at 50°C [5]. Deleterious expansions (> 0.10%) in the salt solution, corrected for the expansions in water were reported within 8 weeks for some reactive aggregates and in 20 weeks for others [6].

2.2.4 Japanese Rapid Test In this method, proposed by Nishibayashi et al [7], mortar bars are made with added NaOH to raise the Na_2O eq to 3.0%, then, at 24 hours they are subjected to saturated steam at 125°C (0.15 MPa pressure) for 4 h. In this study, the test was modified using four ASTM C 227 mortar bars and the Na_2O eq. was raised to 4.0% based on preliminary tests with one of the reactive aggregates.

2.2.6 Chinese Autoclave Test Mortar bars are cured at 125°C in steam prior to autoclaving in a 10% KOH solution for 6h at 150°C [8]. Since it was decided to modify all tests to use ASTM 25 mm cross-section mortar bars and ASTM proportions (graded aggregate to cement ratio of 2.25 to 1), this modified method was not successful (ie. None of the aggregates expanded to 0.10%) and it was abandoned. However, with the originally proposed mix proportions and bars, it was found to work well elsewhere with Canadian aggregates [9].

3. RESULTS

The expansions of mortar bars in the various tests are summarized in Tables 2 and 3.

In the ASTM C 227 test (Table 2), only the bars that were sealed in bags expanded at all. The lack of expansion in the wicked containers is consistent with the problem of alkali leaching [10]. For the bagged bars, only the Spratt aggregate (#12) exceeded 0.10% at 12 months. However, several of the other

reactive aggregates would be classified as the late-expansive, alkali-silicate type [1] and would be only expected to exceed 0.10% at later ages (Aggregates #8, 10, 11). Bars made with these three aggregates are the only others to have expanded more than 0.05% at 12 months and are approaching 0.10% at 18 months as shown in Table 3.

Of the rapidly methods, NBRI method (Table 2) gave the best results with respect to known field performance. It classified three aggregates as rapidly reactive ($> 0.25\%$ at 14 days: #10, 11, 12), five as slowly reactive, marginal or unclear (0.10 to 0.25% at 14 days: #5, 6, 7, 8, 9) and four as innocuous ($< 0.10\%$ at 14 days: # 1, 2, 3, 4). Of the unclear NBRI test results, from field performance, #5 and #7 are marginal (poor petrographically but good field performance) and #6, #8 and #9 would be considered slowly reactive.

As well, for the 3 rapidly reactive aggregates (#10, 11, 12), both the 14 day NBRI expansions and the 12 or 18 month ASTM C 227 expansions would rank them in the same order. From the 28 and 56 day expansions included in Table 2, it can be observed that all of the aggregates continue to expand in the hot NaOH solution but the relative ranking of the aggregates remains the same at all ages.

The Duncan test expansion data is given in Table 3 for periods of 1.5, 3 and 6 months. The results are not very encouraging and it appears that sealing ASTM C 227 bars in bags at 38°C is more effective in accelerating reactive expansions than raising the storage temperature to 64°C. However, both reactive aggregates #10 and #12 did expand $>0.05\%$ at 16 weeks.

The Danish salt test (Table 3) did not result in any expansions exceeding 0.10% and when the expansions of companion bars in water is subtracted from the expansions in salt solution, the corrected expansions became negligible.

Even with the equivalent alkali content raised to 4.0%, the modified Japanese Rapid method (Table 3) only gave an expansion exceeding 0.10% for the Spratt aggregate (#12). It appears that the original aggregate to cement ratio or the aggregate gradation proposed by Nishibayashi [7] is critical to the success of this method and should not have been altered. The same is likely true for the Chinese Autoclave test.

4. ADDITIONAL TESTS

Based on the success of the NBRI test method in detecting reactive aggregates, it was decided to conduct further tests with some of the aggregates to study the influence of: a) using a fixed water to cement ratio (w/c) of 0.50 rather than varying w/c to obtain a constant flow, and b) not washing the dust off of the crushed aggregate as required in ASTM C 227. The dust reacts pozzolanically and reduces expansion in the ASTM C 227 test but it was thought that the excess NaOH in the NBRI test would be sufficient to continue expansion even if the dust reacted. Using a fixed w/c and not having to wash the aggregate fractions would simplify the test procedure.

The results are given in Table 4. It can be observed that higher expansions were obtained in some cases when a fixed w/c was used. The lack of washing of the aggregate fraction also did not reduce expansions significantly and in the case of the Spratt aggregate, expansion was increased.

5. CONCLUSIONS AND RECOMMENDATIONS

Based on these results, ASTM C 227 takes more than 18 months to indicate potentially deleterious, late-expanding alkali-silicate aggregates (ie: to obtain $> 0.10\%$ expansion).

Sealing the C 227 bars in polyethylene bags accelerates the expansion of reactive aggregates, which has been found elsewhere to be due to reduced alkali leaching.

Of the five rapid mortar bar test methods evaluated, only the NBRI 14 day test method, developed by Oberholster and Davies, was able to distinguish all of the reactive aggregates. The failure of the other rapid test methods may have been due to modifications made to them or the different types of reactive aggregates used in this study.

While test limits need to be developed for the NBRI test method based on a much larger series of tests, as an interim measure it is suggested that the limits suggested by Oberholster and Davies [3] are appropriate. i.e. If less than 0.10% expansion is observed after 14 days in NaOH, then the aggregate is innocuous. Also, if more than 0.25% expansion is obtained, then the aggregate is likely reactive. However, expansion results between 0.10 and 0.25% are less clear. It appears that aggregates with good fold performance but which are petrographically marginal, expanded up to 0.175% at 14 days (#5 and #7). As well, some slowly reactive aggregates also expand approximately the same (#6 and #8). Therefore, for expansions between 0.10% and 0.25%, further testing and evaluation is required. In all cases, it is cautioned that petrographic examination is required to ensure that high expansions are in fact due to alkali-aggregate reactivity.

Based on this work and unpublished work by other Canadian researchers, draft CSA and ASTM test procedures based on the NBRI method have been written by the authors and an interlaboratory test program is progress to establish precision data.

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TABLE 1
Aggregate Description and Field Performance

Aggregate Name	Description	Reactive Component	Field Performance
Nelson stone	quarried dolostone	none	Excellent - > 30 years
Ottawa sand (Illinois)	pure quartz sand	none	None
Fleming stone	quarried granite	strained quartz	None ⁴
Guelph sand	carbonate sand	0.2% chert	Excellent - > 30 years
Paris sand	carbonate sand	0.3% chert	Excellent - > 30 years
Neyes sand	siliceous sand	2.5% chert	Reactive ²
Paris stone	carbonate gravel	1.6% chert	Excellent - > 30 years
Grant stone	siliceous gravel	27% argillite, greywacke, sandstone	Reactive ¹
Natural sand (Michigan)	siliceous sand	7.4% chert	Reactive ¹
Sudbury stone	siliceous gravel	75% argillite, greywacke, sandstone	Reactive ¹
Lower Notch stone	quarried meta- sediments	98% argillite, greywacke, sandstone	Reactive ³
Spratt stone	quarried limestone	< 5% chalcedony	Reactive ¹

- Footnotes: 1. Alkali reaction identified in many structures.
 2. Alkali reaction identified in part of one 30-year old structure.
 3. Alkali reaction identified in small part of one 20-year old dam
 (but the use of fly ash has prevented reaction in most of this structure).
 4. Petrographically similar to alkali-silica reactive granite in 70-year structure.

TABLE 2
ASTM C 227 and NBRI Test Expansions (%)

#	AGGREGATE NAME	ASTM C 227						NBRI METHOD			
		MTO CONTAINER			BAGGED			12d	14d	28d	56d
		6m	12m	18m	6m	12m	18m				
1	Nelson	.019	.034	.036	.021	.036	.040	.009	.012	.020	.039
2	Ottawa	.023	-	-	.019	-	-	.038	.038	.053	.139
3	Fleming	.005	.021	.019	.021	.039	.040	.038	.046	.086	.205
4	Guelph	.029	.039	.041	.029	.042	.049	.077	.093	.177	.297
5	Paris Sand	.023	.039	.049	.029	.047	.050	.122	.153	.306	.399
6	Neyes	.024	-	-	.020	-	-	.118	.164	.369	.513
7	Paris	.012	.025	.020	.025	.045	.044	.144	.175	.293	.336
8	Grant	.011	.024	.014	.030	.054	.057	.143	.185	.423	.533
9	Natural	.023	-	-	.041	-	-	.172	.213	.494	.525
10	Sudbury	.022	.031	.032	.036	.062	.080	.221	.266	.466	.736
11	Lower Notch	.017	.032	.035	.036	.077	.094	.282	.326	.540	.746
12	Spratt	.046	.055	.058	.166	.222	.234	.309	.356	.626	.956

TABLE 3
Duncan, Danish Salt and Japanese Rapid Test Expansions (%)

#	AGGREGATE NAME	DUNCAN TEST			DANISH SALT TEST			JAPANESE RAPID TEST 4% NaOH
		12w	16w	26w	in Sat. NaCl		CORRECTED	
					8w	20w		
1	Nelson	.033	.023	.025	.003	.013	.001	.034
2	Ottawa	.027	.028	.019	-	-	-	.033
3	Fleming	.040	.044	.050	.022	.054	.008	.047
4	Guelph	.032	.034	.036	.003	.007	.007	.060
5	Paris Sand	.036	.035	.037	.008	.019	.005	-
6	Neyes	.035	.042	.061	.022	.027	.003	.057
7	Paris	.035	.034	.037	.017	.026	.007	.060
8	Grant	.029	.033	.041	-	-	-	.043
9	Natural	.025	.026	.025	-	-	-	.054
10	Sudbury	.057	.055	.063	.016	.049	.002	.044
11	Lower Notch	-	-	-	.016	.023	.008	.069
12	Spratt	.068	.071	.081	.021	.054	.013	.225

TABLE 4
Influence of Test Variations on NBRI Expansions

#	AGGREGATE NAME	14 DAY EXPANSIONS (%)			W/C FOR CONSTANT FLOW
		CONSTANT FLOW	W/C = 0.50	UNWASHED AGGREGATE CONSTANT FLOW	
5	Paris Sand	.153	.197	.147	.46
7	Paris	.175	.163	.151	.54
10	Sudbury	.266	.255	.275	.53
11	Lower Notch	.326	.403	-	.54
12	Spratts	.356	.375	.441	.54