A RAPID TEST FOR DETECTING THE REACTIVITY OF AGGREGATES : THE MICROBAR METHOD

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The procedure is a modified version of the autoclave method proposed by Tang *et al* (1). A reactivity threshold is proposed, of 0.11 % expansion obtained for about 100 aggregates. The influence of several key parameters (nature of cement preparation of aggregates, operator or equipment switch) is reported. The chemistry of AAR in the conditions of the test is discussed. The Microbar method delivers a reliable verdict regarding alkali-silica or alkali-silicate reactivity. It has been adopted in France as a draft method under the number P 18-588.

INTRODUCTION

Because of its light implementation with respect to the other methods described in the literature (2), (3), we have carried out extensive work on the Chinese autoclave method proposed by Tang (1). The main peculiarities of the procedure, such as the size of the small bars and the alkaline curing at 150°C, were fully respected (Criaud *et al* (4)). This paper describes the investigations undertaken to design the specific equipment, improve the confidence of the method and establish its reliability.

MATERIALS AND METHOD

Procedure

The aggregate is reduced by crushing, grinding and sieving to the fraction 160-630 µm. Here we have adapted the original mesh size (150-750 µm) to the closest standard Afnor sieves.

Ordinary Portland cement is used to prepare three mixes, with different cement to aggregate weight proportions equal to 2, 5 and 10. The water/cement ratio is 0.30, and the alkali content of the cement is boosted to 1.5 % with NaOH. For each mix, four small bars (1*1*4 cm) are casted. The curing takes place in a humid chamber (> 65 % RH) at 20° C. The bars are demoulded after 24 hours, and their initial length measured. The bars then undergo a vapor curing at 100° C during 4 hours. The next day, the bars are soaked in a 10 % KOH solution for 6 hours at 150° C. After cooling and rincing, the lengths are measured again and the expansion calculated. The aggregate is thus

characterized by three values of expansions, one (the mean of the values obtained for the 4 bars) for each of the three c/a ratios. The highest expansion is retained.

The whole procedure, including preparation of the sample of aggregate takes less than 4 days.

Apparatus

The equipment was designed and optimized in order to take advantage of the small amounts of materials needed to fill the moulds. The paddle and bowl of a commercial Perrier mixer were modified to enable mixes of about 150 g to be prepared. The 4-bar moulds made out of a high density polymer, enable an easy demoulding and can be dismantled. They comprise stainless steel studs for precise measurements to be achieved.

The vapor curing is completed by placing the bars in a basket located at the neck of a boiling flask fitted with a reflux condenser. The condensed water droplets are canalized in a funnel in order to avoid alkali leaching from the mortar bars.

Alkaline curing are performed in small stainless containers, similar to those used for the ASTM C289 chemical test. The four specimens of a particular mix (one value of c/a) are treated together with 120 g of KOH 10 % solution.

Measurements are completed at room temperature. The measuring tool and the studs ensure a precision of 1 to 2 µm. All bars are measured against an Invar 4 cm reference bar.

Variations concerning the abovementionned equipment are possible and are mentionned in the standard method P 18-588 (5).

RESULTS

Influence of the mix

The expansions obtained versus the cement/aggregate ratio are represented for 4 aggregates on figure 1. Most of the aggregates, including limestones, sandstones, siliceous sands or volcanic rocks show a greater expansion at c/a equal to 2. At the opposite, aggregates of the chert type behave differently, producing the highest expansions either at c/a's of 5 or 10. The latter materials include aggregates which contain opaline minerals displaying a pessimum effect type of behaviour. As a consequence, the reactivity of any aggregate is better estimated if the highest of the three expansion values at c/a = 2, 5 or 10 is retained. The slowly expanding materials (such as Sudbury) are especially better detected at lower c/a ratios.

Reproducibility

The reproducibility of the method was assessed in three different ways within the laboratory of TSA (Criaud *et al* (4)) :

(i) Standard deviation for the four bars of a single batch : the

variation coefficients are respectively 15 %, 8 % and 6 % for typical expansions of 0.05, 0.10 and 0.20 %.

(ii) Standard deviation for different mixes run on the same composition by two different operators at random : Variation coefficients are respectively equal to 13, 15 and 30 % for expansions of 0.30, 0.10 and 0.02 %.

(iii) Influence of the operator : The mean value of the expansions, obtained by three operators for three aggregates always lies in the 2 σ range of the individual expansion results.

Interlaboratory comparison

For these preliminary tests, five laboratories were involved.

TABLE 1 - Preliminary interlaboratory tests. Expansions (%) measured at c/a of 2, 5 and 10, respectively.

Lab No	1	2	3	4	5	AVG	STD
Cement	Mx	Ca	Or	Gu	Ca	%	%
Afnor	0.130	0.303	0.177	0.311	0.212	0.227	0.079
Sand	0.089	0.129	0.112		0.092	0.106	0.019
	0.066	0.076	0.064		0.069	0.069	0.005
Meuse	0.045				0.060	0.053	0.011
Sand	0.045				0.030	0.038	0.011
	0.046				0.015	0.031	0.022
Spratt		0.343	0.386	0.345	0.341	0.354	0.022
Gravel		0.201	0.270		0.197	0.223	0.041
		0.144	0.110		0.097	0.117	0.024
Chert		0.147	0.153		0.137	0.146	0.008
Gravel		0.318	0.189		0.412	0.306	0.112
		0.353	0.302		0.470	0.375	0.086
Limestone		0.036	0.028	0.020	0.020	0.026	0.008
Gravel		0.045	0.028		0.020	0.031	0.013
		0.042	0.032		0.018	0.031	0.012
Pure paste		0.018	0.031	0.000	0.020	0.020	

Each participant used his own cement and two or three aggregates supplied as ready to mix 160-630 fractions, with the exception of the Afnor standard sand, which was prepared by each laboratory on his own stock. The results are given in table 1.

Most of the results compare reasonably well. However, large variations are observed for the Afnor sand at c/a = 2 and for the chert aggregate at c/a = 5 and 10. For the former, they probably reflect quality variation in the supply and/or some inexperience in the aggregate preparation. As to the latter, a number of replicate tests run in our laboratory repeatedly

showed larger variations than for other aggregate types, probably as a consequence of the presence of opaline materials. In general, the variation coefficients are less than 20 % for expansions greater than 0.10 %.

A larger multi-laboratory campaign will be conducted under the auspices of the AFREM in 1992.

Influence of the cement

Ten different portland cements were used to test the Spratt limestone, an unreactive siliceous sand from Canada and a sound limestone from France. The alkali content of the cements expressed as Na_2O eq, and their C_3A content, range from 0.2 to 1.2 %, and 2 to 11 %, respectively. In addition, pure pastes of each of them were subjected to a whole cycle of curing. Under these conditions, the cements reacted distinctively to the Microbar treatment, producing expansions from 0 up to 0.05 %. For comparison, pure pastes subjected to the South African test at 80° C (6), (7), lead to expansions ranging from 0.02 to 0.10 %. For both NBRI and Microbar methods, there is neither evidence of relationship between paste expansion and initial alkali or C_3A contents. As a conclusion, the nature of the cements used has little influence provided its paste expansion does not exceed 0.03 %.

Methods for preparating the aggregates

The reduction of the aggregate to the final 160-630 fraction µm was investigated in order to assess the influence of any mineralogical segregation. Figure 2 shows the influence of several procedures on the expansions obtained for the Spratt gravel and for a calcareous sand containing silica under various forms. The expansions measured are little dependent on the preparation method, provided its recovery yield (i.e. mass of 160-630 fraction/initial mass of sample) exceeds 50 to 85 %, depending on the aggregate mineralogy.

CALIBRATION OF THE METHOD

As indicated before, the highest of the expansions obtained for the three different c/a ratios is retained to establish a diagnosis. We had provisionally suggested (4) two limits for the expansion, thus defining three zones of different reactivity. These thresholds (respectively 0.08 and 0.10 %) have proven too severe as more aggregates were tested. The revised limit of expansion proposed thereafter is based on extensive testings on 94 different aggregates with various composition and sources. A selection of data is presented in Table 2.

Comparison with field record services

Some 19 aggregates with proven field record were tested. All of them but one (Pittsburgh) yield expansions greater than 0.11 %. This class of materials includes the well-known aggregates of Spratt, Sudbury, Malmesbury hornfels, Postdam sandstone or Trent Valley aggregates.

TABLE 2 - Microbar expansions and diagnostics for a selection of aggregates. Comparison with field records or previous testings. a = ASTM C 227 b = ACNOR c = NBRI d = danish MBT e = P18-585 (8) f = P18-587 (9) S = sound PR = potentially reactive.

Aggregate	Microbar			Reactivity		
	Diagn	Exp.%	c/a	Field	Other method	
Canada						
Spratt	PR	0.333	2	PR	PR	a,b,c
Nelson	S	0.021	2	S	S	a,b,c
Oxford	S	0.015	2	S		
Postdam	PR	0.129	2	PR	PR	b
					S	a
CBM sand	S	0.054	2	S	S	a,c
Malay Falls	PR	0.241	2	PR	PR	a,c
Sudbury	PR	0.185	2	PR	PR	b,c
					S	a
Pittsburgh	S	0.050	2	PR	S	a,c
Dolomite					PR	b
USA						
Ottawa quartz	S	0.029	2		S	a,c
France						
Chambon	PR	0.332	2	PR	PR	e,f
Brix	PR	0.252	2	PR	PR	a
Limestone 1	S	0.038	2		S	f
Granite 1	S	0.100	2		S	e,f
Chert F	PR	0.390	10		PR	f
Belgium						
Tournaisis	PR	0.250	2	PR	PR	a,c,f
Porphyry	PR	0.208	2	PR	PR	c
					S	a
Meuse gravel	PR	0.202	10	PR	PR	a,d
Visean limest.	S	0.027	2	S		
Quartzite	PR	0.277	2	PR	PR	c
United Kingdom						
Thames Valley	PR	0.227	5	PR	PR	b
Trent Valley	PR	0.155	2	PR	PR	b
Norway						
Ardal sand	S	0.043	2		S	a,b,c
Metarhyolite	PR	0.254	2	PR	PR	a,b,c
Australia						
Phyllite	PR	0.170	5	PR	PR	c
Dacite	PR	0.308	2	PR		
New Zealand						
Tuakau	PR	0.429	2	PR	PR	a
Andesite	PR	0.357	2	PR		

The Pittsburgh dolomitic limestone, an alkali-carbonate reactive aggregate, yields very low expansions whatever the c/a ratio. Three sound aggregates (two limestones and a quartz sand) produce an expansion generally lower than 0.05 %.

Comparison with other expansion test results

The tests used for testing these 72 aggregates are very different in nature and include ASTM C227, Acnor concrete tests and their modifications as well as accelerated tests such as the NBRI and danish mortar bar tests. Aggregates for which the sole information was based on the chemical ASTM C289 test were intentionally avoided for correlation purposes.

43 out of 72 aggregates are potentially reactive according to previous results and yield Microbar expansions greater than 0.11 %. 24 aggregates of no or negligible reactivity bring about Microbar expansions smaller than 0.11 %.

At last five aggregates yields substantial expansion (i.e. > 0.11 %) with the Microbar test while the other methods diagnose then sound. Yet, the petrographic examinations indicate the presence of silica bearing minerals, which is consistent with the possible development of AAR. Further investigations would be necessary to settle these discrepancies.

Threshold and field of application

A unique limit of expansion at 0.11 % best fits the numerous data available so far. The slowly and marginally reactive aggregates tends to yield expansions in the range 0.11-0.14 %.

It should be emphasized that this reevaluated expansion limit of 0.11 % is the same as the one suggested by Tang *et al* (1). However, the Microbar expansion refers to the highest value obtained at three different c/a, instead of one.

The Microbar method provides a reliable evaluation of aggregates with regard to their alkali-silica and alkali-silicate reactivity. The alkali-carbonate reactivity is not unveiled with this test.

CHEMISTRY OF AAR AT 150° C

Cause of expansion and associated reactions

Experiments were carried out with two synthetic cements containing only C_3S or C_3S + C_3A + gypsum. An unreactive limestone aggregate and a chert type material were used. The Microbar procedure was applied at one single cement/aggregate ratio, leading to maximum expansions. As shown in table 3, only the siliceous aggregate yields substantial expansions, emphasizing the effect of the siliceous phases in the swelling process.

TABLE 3 - Microbar tests conducted with synthetic cements

Mix	c/a	Expansion (%)
C_3S		
Paste		0.012
Limestone	2	0.004
Chert	5	0.209
C_3S + 8 % C_3A + 2 % gypsum		
Paste		0.033
Limestone	2	0.012
Chert	5	0.380

The chemical composition of the alkaline solution after treatment was monitored. Neither variations of the concentration of K, nor the alkalinity (as titrated with HCl) are significant with respect to the original values. However, the concentration of dissolved silica generally increases with the expansion and can reach 20 g/l, indicating that the reaction product goes into the solution during curing.

Microstructure of the mortars after treatment

Cracks, crazing pattern and sweaty patches can be seen on the external faces of mortars made with the reactive aggregates. The examination of thin sections of such mortars shows the presence of empty cracks penetrating into the paste and aggregates. Some particles react only superficially while some others such as cherts may be completely dissolved. No gel can be seen within the mortar. By contrast, the unreactive aggregates appear normally bound to the paste and the paste itself is not modified. The porosity of the paste, as observed by SEM, is increased after treatment.

Nature of hydration and AAR products at 150° C

The CSH are not cristallized within the short period of treatment at 150° C, so that amorphous, massive or fibrous morphologies are encountered. Occasionally, tobermorite is detected by XRD. Portlandite and anhydrous components are well evidenced. C_3AH_6 is rarely noticed. AFm and AFt phases are not detected. The degree of hydration at the cement phases was estimated to 70 to 80 % by semi-quantitative XRD analysis. The SEM-EDS showed that the paste was markedly enriched in potassium, especially on the edges of cracks and around some aggregates. It is thought that the reaction product is intimately formed and distributed in the paste until cracks open and enable this product to be dissolved in the solution.

CONCLUSIONS

The Microbar test (French draft method P 18-588) provides a quick and reliable mean to evaluate the reactivity of aggregates with respect to alkali-silica-silicate reactions in concrete. Despite the fact that the chemistry of these reactions at 150° C

slightly differ from those at ordinary temperatures, our studies demonstrate that the measured expansions are actually due to the interaction of reactive silica bearing minerals with the alkaline fluid. The method does not detect alkali-carbonate reactivity. Microbar data obtained for about 100 aggregates with various nature, origin and kinetics of reaction were correlated with field records or other test results and lead to set the expansion threshold at 0.11 %.

The method is easily performed and space saving. Provided that the sampling is properly done, little amount of aggregates (3 to 5 kg) is needed. Detailed quarry studies and quality controls of a production are therefore possible at low costs.

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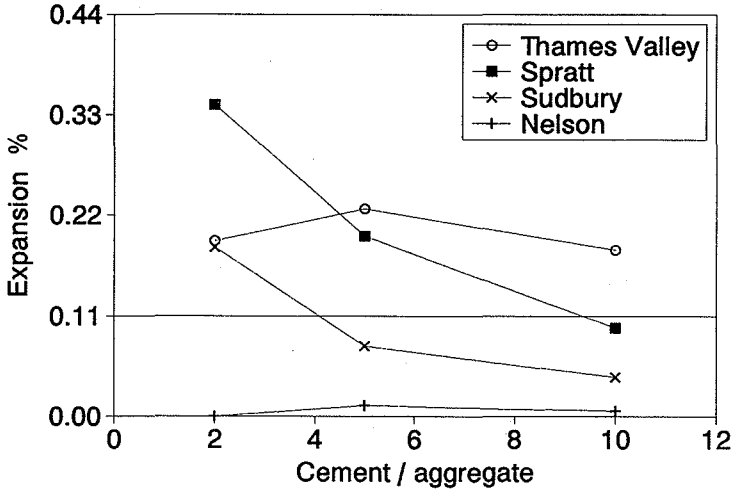

Figure 1 Relationship between Microbar expansions versus c/a ratios in the mix for typical aggregates.

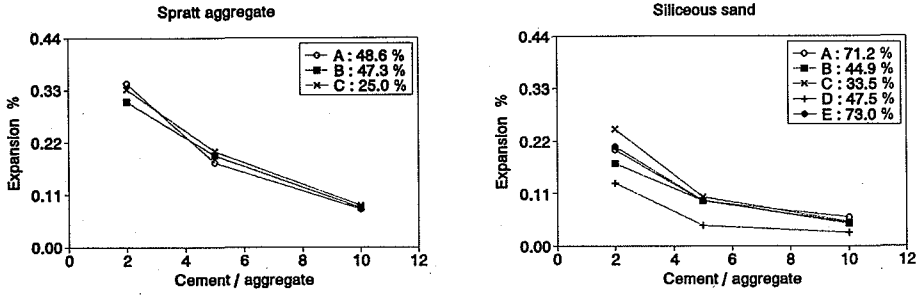

Figure 2 The methods A, B, C, D and E involve different steps and equipment for crushing, grinding and sieving the aggregate. The recovery yield is given in %