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METHODOLOGY OF AN INDUSTRIAL RESEARCH LABORATORY TO ASSESS THE REACTIVITY OF AGGREGATES. FOCUS ON REPRODUCTIBILITY PROBLEMS

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1. INTRODUCTION

The main purpose of this paper is to present the way in which the research laboratory of Lafarge Coppée (cement manufacturer, aggregate and concrete producer), is approaching the field of alkali aggregate reactions.

Investigations on the subject have already been conducted at the Technical Center of Lafarge Corporation in Montréal [1], however, as the situation looked quiet in France, the subject was little developed at the Central Research Laboratory, until 3 years ago.

This privileged position is somewhat changing : a few cases of alkali aggregate reactions are now officially recognized and the probability of its occurence in new constructions is greater.

These facts explain why, decision has been taken to develop our own competence in this field. The present paper is intended to deal with aggregates.

The methodology, we are setting up, is based on the simple - and unfortunate - finding that several tests exist, but no one test is good enough to be used by itself. We should add that, in this preliminary stage, available tests will be used rather than new ones developed.

In such conditions the first problem to be solved is : which tests shall we select in order to have the surest answer concerning the potential reactivity of the aggregates ?

2. METHODOLOGY and MATERIALS

2.1 Aggregates : selection and characterization

In order to check the validity of a possible decisional flow chart, we have chosen to use natural aggregates, identified as reactive on existing structures : thus, three Canadian aggregates representative of the three types of reactions and considered as standards, are now under tests.

A minimum information will be given about these aggregates because, as they are those used in the Interlaboratory tests programs conducted by C. Rogers (MT, Ontario), the basic data are contained in a periodically up - dated booklet (General information on standard alkali - reactive aggregates from Ontario ; last issue 1988).

Note : We are participating in the current program (determination of the best of 4, storage conditions) on CSA. A 23.2-14A with the Pittsburgh dolomitic limestone ; these results will therefore not appear here.

- The Spratt aggregate is from a limestone quarry, in the vicinity of Ottawa, however damage are typical of an alkali - silica reaction. In fact, the rock consists of calcite and some areas of dolomite (see FIG. 1), but chemical analysis gives an insoluble residue of 9.45%. XRD shows that it is partly made of quartz, illite and pyrites ; also, EDS analysis on the small dark grey spots reveal they consist of fine size grains of silica and argillaceous matter, mainly illite (Si - Al - K).

TABLE 1 - Chemical analysis of the Spratt and Sudbury aggregates and of the high alkali content cement

	SiO2	Al2O3	Fe2O3	CaO	MgO	SO3	Na2O	K2O	L.O.I	TOTAL	Ins. res
SPRATT	8.9	0.6	0.3	48.85	1.45	0.30	0.03	0.09	39.7	100.2	9.45
SPRATT Ins.Res	86.5	4.2	1.45	1.50	0	3.15	0.10	0.56	5.00	99.3	
SUDBURY	69.2	13.0	4.4	1.75	3.10	0.15	3.18	1.95	1.80	99.2	87.1
CEMENT	19.3	5.4	2.65	63.0	1.30	3.90	0.31 Tot 0.20 Sol	0.87 Tot 0.76 Sol	2.25	99.6	0.60

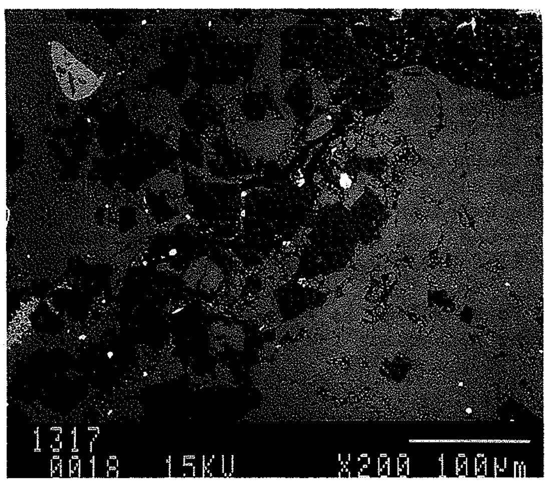

FIG. 1 - Polished section of a Spratt aggregate (BEI). A rich dolomitic area, (dark rhomboedral crystals) inside the limestone matrix (light grey, background). The small dark grey spots are silica and shale ; the white ones are pyrites

In every case a thorough petrographic study is done. The preparation of thin sections receive particular attention. We follow the method developed at the Danish Technological Institute, as advised by Idorn Consult : the thickness is 20 μm, and observation is made easier by adding a fluorescent dye to the epoxy used for impregnation.

The chemical standard ASTM C-289, has been first applied in its original version and, as others, we found the aggregate non reactive. We now use the modified version, i.e, the alkaline attack is made on the insoluble residue containing the reactive part of the aggregate. But more work needs to be done on this method.

- The Sudbury aggregate, is a partly crushed gravel from a pit near Sudbury airport. Typical of the alkali silicate reaction, it has been investigated by several Canadian researchers, like P.G. Bellew [2,3], or C.A. Rogers [4,5] who noticed several peculiarities : expansion develops slowly with the mortar bar test and only in severe conditions with the Canadian concrete prism test (38°C, high alkali level). The quantity of visible alkali-silica gel in damaged concrete, is rather small. The gravel contains about 75% reactive materials, among which argillite is the most reactive one, but the important feature seems to be that all of them include illite and microcrystalline quartz ; they are undoubtedly active in the deterioration process [2].

Once these investigations are done on aggregates, the following step is testing their behaviour in the presence of cement, as mortars or concretes. The most developed mortar bar test is the ASTM C 227 ; the canadian CSA.A 23.2-14A concrete prisms test, seems also to be under increasing development. However, from various sources of information, we are not very confident with the validity of such tests, especially the reproducibility. We have therefore decided to start a series of experiments in order to form our own opinion.

3. MORTAR BAR TEST

3.1 General

The procedure of the ASTM C 227-87 mortar bar test has been followed for the preparation and grading of the aggregates, the mixing and placing of the mortars, as well as for the basic curing conditions (38°C, 100% RH), but after some experience, we have voluntarily introduced changes.

As we are in a program of testing aggregates and not a particular aggregate/cement couple, a high 1.25% Na2O eqv. has been maintained - following the recommendation in the concrete prism Canadian sdandard - ; this is realized by boosting with NaOH, a high alkali-cement (0,9% Na2O eqv. ; Table 1).

The newly adopted container (1987 version) is far from being trustworthy [6], so we have used the 25 bar metal container, firstly at is was, and then modified. The 30 bar container, made of stainless steel, has a suitably designed cover in order to avoid alkali leaching.

It quickly became clear to us that the relative humidity was playing a prominent role ; consequently a great deal of attention has ben paid to the maintenance and homogeneous repartition along a given bar and from one bar to another, of the 100% R.H. This is achieved with a double regulation system of T° and R.H., by placing the container with water in the bottom, in a large box, thermoregulated at 38°C and also containing some water.

The container is thus in a saturated vapour atmosphere, making easier the thermal exchanges. All the following results (mortars and concrete) have been obtained in such conditions.

3.2 Reproducibility with the Spratt aggregate

Three storage modes have been experienced (sections a, b and c, table 2). For each of them, three series of 4 bars have been prepared successively (at one month intervals, by different operators) : lines 1,2 and 3 section a and corresponding FIG. 2 ; in sections b and c, only the mean value is given, but the reproducibility is as good. The tests are still in progress, oldest specimens are now 10 months.

TABLE 2 - Expansion (%) of mortar bars with the Spratt aggregate

		1 m	2 m	3 m	4 m	6 m	8 m	10 m
Container (a)	1	0.023	0.078	0.137	0.204	0.284	0.300	0.310
	2	0.025	0.084	0.165	0.245	0.306	0.330	
	3	0.020	0.109	0.182	0.224	0.263	0.298	
	$\bar{x}$	0.023	0.090	0.160	0.244	0.284	0.308	
Container+wick (b)	$\bar{x}$	0.022	0.087	0.152	0.201	0.250	0.260	0.25
Plastic bag (c)	$\bar{x}$	0.023	0.095	0.162	0.231	0.290	0.308	0.325

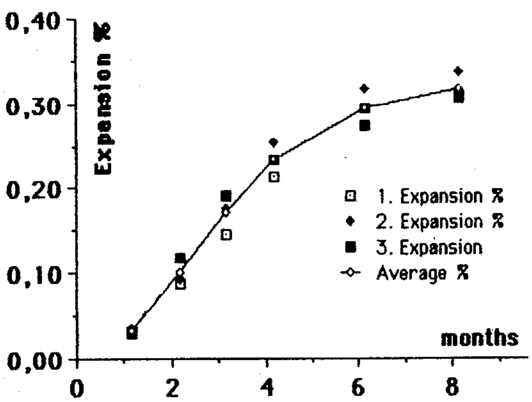

FIG. 2 - Results of part a, table 2. Scattered points are relative to the series 1, 2, 3, while the line is the average of the three series

Whatever storage condition is used, expansion of the bars develops well, rating this aggregate as reactive. It appears, that the best method is also the simpliest one, i.e., the container alone a ; no wicks, and no plastic bag. The results between a and c look equivalent while those with b, are somewhat smaller. This has been observed by D. Hooton but with a much larger difference [7].

We have decided to utilize method a for further tests.

3.3 Preliminary results with the Sudbury aggregate

We are aware of the difficulty of expansion of mortar bars with this particular aggregate (see Ch. Rogers document) ; nevertheless we consider that we should test it, as a part of our educational program. Three series of 4 bars have been prepared at one month intervals. After 3 months, the average expansion is about 0.02% ; the low value confirms previous findings.

This family of aggregate raises the problem of the limit of the test validity : it may work, but the delay becomes too long. Its accelerated version, developed at NBRI, looks very attractive [8].

4. ACCELERATED MORTAR BAR TEST

Another important reason why we tried this quick test is that all the problems encountered with the containers and curing conditions are avoided.

Expansions of 2 sets of 4 bars containing the Spratt or the Sudbury aggregate, recorded from 3 to 28 days appear in Table 3 and FIG. 3.

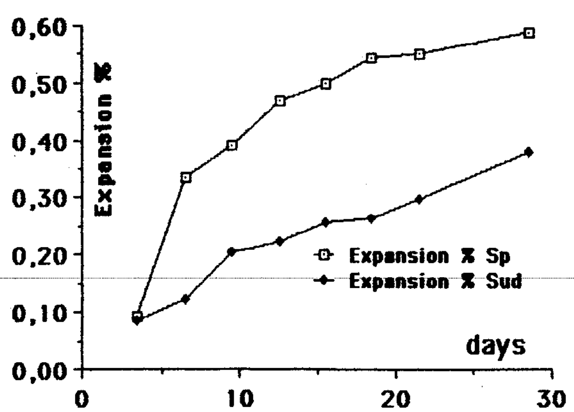

FIG. 3 - Expansions measured for the Spratt and Sudbury aggregates

In both cases, and even though Sudbury is a late expanding aggregate, length changes are above the accepted limit (0.11% at 12 days).

Although the results are fast and correct in these cases, research is currently being conducted in several places (Laval University) to determine its limits ; it seems to severe with some unreactive aggregate in particular those containing chlorite [9].

TABLE 3 - Expansion (%) of bars with the NBRI accelerated test

Aggregate	3 d	6 d	9 d	12 d	15 d	18 d	21 d	28 d
Spratt	0.077	0.320	0.380	0.456	0.483	0.528	0.535	0.573
Sudbury	0.069	0.108	0.190	0.210	0.240	0.250	0.283	0.366

5. CANADIAN CONCRETE PRISM TEST

We have already mentioned that we are taking part in the Interlaboratory program on the alkali carbonate reaction (Pittsburgh aggregate). Aside from this, and because the test is recommanded for all the types of reactions, we are also exploring this test for alkali silica and silicate reactions. First of all, using the same procedure as for the mortar bar test, we have conducted:

5.1 Reproducibility tests with the Spratt aggregate

The 1988 version of the test is being used : 1.25% Na20 equivalent. All the specimens are cured at 38°C, 100% R.H. The non reactivity of the sand has been verified with the ASTM C 227 (0.013% from 1 to 10 months).

Like in table 2, the full set of values is given in one case only ; mean values are given for the other ones.

TABLE 4 - Expansion (%) of concrete prisms cured in different conditions

		1 m	2 m	3 m	4 m	6 m	8 m	10 m
Container (a)	1	0.038	0.073	0.099	0.122	0.148	0.159	0.170
	2	0.009	0.032	0.060	0.085	0.108	0.123	
	3	0.008	0.040	0.070	0.090	0.117	-	
	$\overline{x}$	0.018	0.045	0.076	0.099	0.124	0.141	
Container+wick (b)	$\overline{x}$	0.016	0.043	0.076	0.099	0.119	0.138	0.156
Plastic bag (c)	$\overline{x}$	0.090	0.022	0.043	0.061	0.076	0.090	0.113

Generally speaking, expansion develops well any time, but results obtained, when using a plastic bag are systematically below the others. Once more, mode a seems the most appropriate one. We are now using it for the following study of the:

5.2 Influence of the cement content with Spratt and Sudbury aggregates

Cement contents are 310 and 410 kg/m3. Preliminary results (3 months) show a higher expansion (about twice) with 410 kg/cm3 cement for both aggregates.

5.3 Restrained concrete

Concrete prisms, made with the Spratt aggregate and two cement contents (310 and 410 kg/m3), are restrained with stainless steel rods, (0.25 and 0.66%). Results will be presented orally.

6. MICROSTRUCTURE

The studies undertaken have several objectives : First, take the advantage of the availability of such a large number of standard samples to collect maximum information on their microstructure and chemical composition. It must be underlined that very few microstructure studies have been done on samples at early ages and that they deal usually with deteriorated field concrete. Second, try to correlate such results with the other ones, mainly length changes. Third, attribuate a fair position to this type of investigations.

7. CONCLUSIONS

From the results of the large experimental program, based on the use of standard natural reactive aggregates, several points can be stressed out :

- The chemical C-289 ASTM standard is not satisfactory, even if using the insoluble resisue [9].
- Concerning the tests ASTM C-227 mortar bar and Canadian concrete prism, we are now convinced that trustworthy and reproducible results can be obtained, provided that basic rules and some changes are adopted : 1.25% Na2O eqv. should be used any time (high alkali content cement+NaOH) ; 38°C (rather than 23°C) and a high cement content (410 kg/m3) promote expansion, especially in the case of late expanding silicate aggregates.
- The quality of thermal exchanges is essential, whereas the container by itself is not.
- The NBRI method is very attractive but requests complementary work.
- Microstructure studies, deserve a full space in the methodology, if associated with serious petrographic examinations.

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