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RILEM TC 191-ARP: 'Alkali-reactivity and prevention - Assessment, specification and diagnosis of alkali-reactivity'

AAR-5: Rapid preliminary screening test for carbonate aggregates

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The text presented hereunder is a general draft for general consideration. Comments should be sent to the TC Chairman Dr. Philip J. Nixon, Building Research Establishment, Garston WD2 7JR Watford, United Kingdom; Tel: +44 923 664 239; Fax: +44 923 664 786; Email: nixonp@bre.co.uk, by 30 April 2006.

1. INTRODUCTION TO AAR-5

The ultra-accelerated mortar-bar test (AAR-2) has been widely and successfully used as a screening test to identify aggregates that are potentially alkali-reactive. However, there are exceptions; some aggregates that, though alkali-reactive according to both field experience and concrete prism test results, do not show as such in the mortar-bar test.

Trials carried out by members of RILEM TC 191-ARP have established that these aggregates include carbonate materials.

It has been found that any potential reactivity of such carbonate aggregates will be identified if 4/8 mm aggregate is used for making the bars instead of 0/4 mm aggregate. The development of this method (AAR-5), which is sometimes termed the 'concrete microbar' test, and the international trials organised by RILEM TC 191-ARP are described in [1].

The concrete microbar method is particularly beneficial when the aggregate contains both reactive silica and a kind of dolomite that is prone to dedolomitization. In the mortar-bar test most of the (fine) aggregate will dedolomitize throughout, whilst in the concrete microbars only the outer shells of the (coarser) aggregate is dedolomitized [2]. This difference seems to explain why, though alkali-silica gel develops in both the tests, the aggregate develops more pressure and expansion in AAR-5 than in AAR-2.

It is therefore recommended that, if dolomitic carbonate aggregates are being assessed, both the AAR-2 and the AAR-5 tests should be carried out. If the concrete microbars expand

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more than the mortar-bars, the reactivity of the aggregate is not of the normal alkali-silica type and further investigation using the concrete prism test (AAR-3) or the accelerated concrete prism test (AAR-4) will be required.

Draft methods for the AAR-2 and AAR-3 tests (then designated TC 106-2 and TC 106-3 respectively) were published in [3] and are currently being updated by RILEM TC 191-ARP. The AAR-4 accelerated concrete prism test is presently undergoing an international trial, as described in [4], and the draft method will be published in due course by TC 191-ARP.

A draft of the new AAR-5 concrete microbar method follows this introduction. It is based on the Chinese Accelerated Concrete Microbar Method [5] developed by Nanjing University of Chemical Technology, China, and was prepared by the Carbonate Team of TC 191-ARP: H. Sommer, Austria (team leader); I. Sims, UK (TC 191-ARP secretary); B. Fournier, Canada; P. Grattan-Bellew, Canada; S. Guédon-Dubied, France; J. Larbi, The Netherlands; V. Jensen, Norway; T. Katayama, Japan; G. Lorenzi, Belgium; A-M. Marion, Belgium; C. Merz, Switzerland; B. Schouenborg, Sweden; J. Steigenberger, Austria; and M-S. Tang, China.

2. SCOPE

This test method is intended to be used conjunctively with AAR-2 [6] (ultra accelerated mortar bar method) to determine rapidly the potential alkali-reactivity of carbonate aggregates through the evaluation of the expansion of mortar-bars and concrete microbars immersed in NaOH solution at elevated temperature as specified in the methods.

3. SIGNIFICANCE AND USE

Some carbonate aggregates do not show as potentially reactive in the AAR-2 test, though they are alkali-reactive both according to field experience and test results according to AAR-3 [7] (concrete prism method).

AAR-5 provides, when used in conjunction with AAR-2, a means of reliably screening carbonate aggregates for their potential alkali-reactivity, in particular for aggregates that contain dolomitic limestone and/or dolostone.

It is recommended that the test is performed not only with the aggregate to be evaluated but also with a known reactive and a known non-reactive reference aggregate.

4. PRINCIPLE

This method is similar to AAR-2: bars moulded from a mix containing the aggregate to be tested are demoulded after 24 hours, heated up in water to 80°C during another 24 hours, then immersed in 1 M NaOH solution at 80°C and the expansions are measured. However, since some carbonate aggregates produce deleterious expansions only if used in a larger particle size, with AAR-5 4/8 mm aggregate is used instead of 0/4 mm aggregate and 40/40/160 mm bars are used instead of 25/25/285 mm bars.

NOTE 1: *Alternatively, aggregate 5/10 mm (as in the Chinese method) may be used, but expansion may be somewhat higher, see also Annex A3.*

5. APPARATUS

5.1 Sieves

A set of sieves conforming to series A of ISO 6274, having square apertures of 8 mm and 4 mm.

5.2 Balance

A balance capable of weighing 1000 g with an accuracy of 1 g.

5.3 Measuring cylinders

Graduated in ml, with a capacity of 500 ml.

5.4 Mixer, paddle and mixing bowl

Mixer, paddle and mixing bowl, as used in the procedure for testing the strength of cement with plastic mortar, *e.g.* EN 196-1 [11] or ASTM C109 [12].

5.5 Flow table and mould

Flow table consisting essentially of a circular rigid table top, that can be raised vertically by means of a cam, and dropped through a specified height, and a frustum of a cone, both conforming to ASTM C230 [8] or EN 1015-3 [9].

5.6 Vibrating table

Vibrating table as used in the procedure for testing the strength of cement with plastic mortar (not applicable if the specimens are compacted by hand).

5.7 Tamper

Tamper, made of non-absorbent, non-abrasive, non-brittle material and with a cross-section of *e.g.* 20 x 25 mm. A convenient length is 230 mm to 300 mm. The tamping face shall be flat and at right angles to the length of the tamper (not applicable if the specimens are compacted by vibration).

5.8 Moulds

Moulds, providing for bars with a nominal length of 160mm and a cross section of 40mm x 40mm. The end plates of the moulds must have threaded holes in the centres to take stainless steel pins of 6mm diameter and 20 mm length used for length measurements.

5.9 Length comparator

Consisting of:

- an apparatus to measure the length of the specimens conveniently and rapidly,
- a high-grade dial micrometer, or other measuring device,

graduated to read in 2.5µm units, accurate to within 2.5µm in any 25µm range, and within 5µm in any 0.25mm range. The measuring range shall allow for small variations (±10mm) from the nominal gauge length of the specimens,

- an Invar® reference bar (or similar) of the same nominal length as the specimens for checking the measuring device, before and after each set of readings.

5.10 Containers

Rigid containers for the test specimens made of plastic or other material resistant to corrosion by a solution of sodium hydroxide at a temperature of 80°C for a prolonged period of time. Each container must be of such dimension and shape to accommodate at least three specimens and must be provided with lids or other suitable means to prevent loss of moisture by leaking or evaporation. The prisms must be positioned and supported in such a way that the solution has access to the whole of the bar. It should further be ensured that the specimens do not touch each other or the sides of the container. The specimens, if stood upright in the solution, shall not be supported by the steel pins.

5.11 Storage

A cabinet or moist storage room maintained at a temperature and a relative humidity as specified by the standard for testing the strength of cement, *e.g.* EN 196-1 or ASTM C109.

NOTE 2: *EN 196-1 and ASTM C109 specify 20°C ± 1°C and 23°C ± 2°C respectively. However, the different degrees of hydration at the end of the initial curing period will not matter essentially, as the aggregate will be evaluated by comparing the AAR-2 and AAR-5 results, provided both the tests use 40/40/160 mm bars and the moist storage conditions are the same.*

5.12 Oven

An oven or room of suitable size to accommodate the required number of containers maintained at a temperature of 80°C ± 2°C.

5.13 Crusher

A laboratory-type crusher of suitable size and design capable of crushing aggregate to the prescribed size fractions.

6. REAGENTS AND MATERIALS

6.1 Water

Distilled or deionised water.

6.2 Solid Sodium hydroxide

Solid Sodium hydroxide of at least technical grade.

6.3 Sodium hydroxide solution (NaOH)

Each litre of sodium hydroxide solution shall contain 40.0g of NaOH dissolved in 900ml of water and, after cooling to about 20°C, it shall be diluted with additional distilled or deionised water to obtain 1.0 litre of solution. The concentration of the solution shall lie between 0.99 and 1.01M. A new solution shall be prepared for each series of tests.

6.4 Cement

An ordinary Portland cement CEM I or ASTM type I, preferably with a Na_2O equivalent (Na_2O+0.658 K_2O) between 0.9% and 1.1%. The specific surface of the cement, when measured according to the air permeability method (*e.g.* EN 196-6 [13] or ASTM C204 [14]), shall be greater than 450 m^2/kg. The autoclave expansion, determined according to ASTM test method C151 shall be less than 0.20%. Alternatively the MgO soundness can be evaluated through Le Chatelier test (*e.g.* EN 196-3) and the increase in separation of indicator ends shall be 0 mm.

NOTE 3: *The composition of the cement may influence the expansions, even when the alkali content of the mix is kept at the same level according to clause 7.3. Therefore, the same cement should be used both for the AAR-2 and the AAR-5 tests.*

NOTE 4: *High alkali cement as specified above is not available everywhere. In such areas a cement from the market with the alkali content on the high side should be used and enough NaOH added to the mix to obtain the alkali level prescribed in clause 7.3. However, high amounts of added NaOH may make mixing difficult, for recommendations see Note 10.*

7. PROCEDURE

7.1 Preparation of the aggregate sample

The quantity of the sample delivered to the laboratory should be in accordance with the sampling procedures recommended in AAR-1. The material to be tested shall consist of particles < 8 mm and > 4 mm, processed according to 7.1.1 and 7.1.2.

NOTE 5: *For the influence of the size of the aggregate, see Annex A3.*

7.1.1 Natural 4/8 mm aggregate

The natural 4/8 mm aggregate shall be tested in the grading as submitted, except that the aggregate is washed, dried, and sieved and particles retained on an 8 mm screen and particles passing a 4 mm screen are removed.

7.1.2 Aggregate >8 mm

The aggregate > 8 mm shall be crushed, sieved, washed, and dried to produce a 4/8 mm sample without oversize and undersize material as in 7.1.1.

7.1.3 Samples to be tested

When a petrographic examination reveals that the natural 4/8 mm aggregate and the crushed 4/8 mm aggregate from the same source are essentially different, both the samples shall be tested; otherwise only the crushed aggregate needs to be tested.

7.2 Conditioning

The temperatures of the moulding room, apparatus, dry materials, mixing water and of the cabinet or moist storage room and the relative humidities of the moulding room and of the cabinet or moist storage room shall conform to the standards valid in the place of use for testing the strength of cement, *e.g.* EN 196-1 or ASTM C109.

7.3 Proportioning of the concrete

Take 900 g of cement, 900 g of 4/8 mm aggregate, and 290 ml of water (see also NOTE 7) for each batch for three concrete bars.

Before mixing, add enough sodium hydroxide to the water to bring the alkali content of the concrete up to a Na_2O equivalent of 1.5 % by mass of cement. If the mix is not sufficiently workable (*e.g.* when the surface of the aggregate is very rough), add just enough superplasticizer (but not one containing an air-entraining agent) as is necessary. In order to minimize any risk of segregation, flow values in excess of 150 mm (determined according to EN 1015-3) should be avoided. Any alkali content of the plasticizer shall be taken into account when calculating the Na_2O equivalent.

NOTE 6: *In the original Chinese method the water content was 270 ml. In a comparative test conducted in the course of the international trial in 2002, a somewhat higher water content produced the same expansions. The water content of 290 ml was chosen to improve the workability of the mix and to minimize the need for a superplasticizer. In the 2002 trial the superplasticizer did not influence the expansions, but it is thought that a high amount of a superplasticizer in combination with special cements may cause early stiffening.*

NOTE 7: *The water content of 290 ml is the free water available for the workability of the fresh concrete and for the hydration of the cement. The total water to be added to the mix is the free water plus the water absorbed by the aggregate to bring it to a saturated surface dry condition. Water absorption shall be measured according to EN 1097-6 [15].*

NOTE 8: *In the original Chinese method KOH was added to the mix to bring up the alkali level to the required level. In comparative tests the addition of NaOH resulted in somewhat higher expansions; NaOH was chosen for consistency with AAR-2.*

NOTE 9: *If the bars are compacted by hand-tamping, normally no superplasticizer should be necessary. If a vibrating table is used for compacting the mix, the addition of a small amount (e.g. 5 g) of a superplasticizer may be necessary in order to enable proper compaction. A flow of at least 115 mm as determined according to EN 1015-3 [9] has been found to ensure this. With amounts of superplasticizer not exceeding 10 g there is no need to make allowance for its water content.*

NOTE 10: *The clearance between the mixer bowl and the paddle shall be increased in order to avoid blocking and damage to the machine due to the coarse aggregate. Alternatively, especially when coarser aggregate than 4/8 mm is used or when high additions of NaOH make mixing difficult, mix cement and water first, exchange the paddle for a hook type, add the aggregate and remix, or mix in the aggregate by hand.*

7.4 Moulding test specimens

Make at least three 40 x 40 x 160 mm specimens for each aggregate to be tested. The mould shall be filled in two approximately equal layers and the specimens compacted and levelled as specified *e.g.* by EN 196-1 or ASTM C109.

NOTE 11: *Particular care should be taken to attain a consistent compaction of the concrete as the degree of compaction greatly influences the degree of expansion.*

7.5 Initial curing and measurement

Place the moulds in the moist cabinet or in the moist storage room for a period of 24 ± 2 hours. Remove the specimens from the mould and, while they are being protected from loss of moisture, weigh and properly identify each specimen in such a way that they, when subsequently measured, are placed in the measuring equipment in the same manner. Make and record the length (Li) and all subsequent measurements to the nearest 0.002 mm.

Place the specimens made with each aggregate sample in a storage container with sufficient distilled or deionized water, at room temperature, to immerse them totally. The volume proportion of water to the bars shall be the same as in clause 7.6. Seal and place the containers in an oven at 80°C ± 2°C for a period of 24 hours.

Remove the containers from the oven one at a time. Remove other containers only after the bars in the first container have been measured and returned to the oven. Remove the bars one at a time from the water and dry their surfaces with a towel or cloth paying particular attention to the two metal pins. Take the zero measurement of each bar (Lo) immediately after drying and read as soon as possible after the bar is in position. Complete the process of drying and measuring within 15 s of removing the specimens from the water. After measuring is completed the temperature of the water shall not be less than 77°C.

The measuring device should be checked with the reference bar prior to and after measurement of each set of specimens.

7.6 Final storage and measurement

Place the specimens made with each aggregate sample in a container with sufficient 1M NaOH, preheated at 80°C ± 2°C, totally to immerse the specimens. The recommended volume proportion of sodium hydroxide solution to the bars in a storage container shall be 4 ± 0.5 times the volume of the mortar-bars. Seal the container, mark the level of the liquid on the outside, and return it to the oven.

Take subsequent measurements (Ln) of the specimens periodically, with a reading after 24 hours of immersion in the NaOH solution and at least at 2 days (or 3 or 4 days), 7 days, 14 days, 21 days and 28 days. All measurements should be taken at approximately the same time each day. The measuring procedure is identical to that described in clause 7.5 and the specimens are returned to their container after each measurement. If moisture is lost from the container by evaporation replenish with distilled or deionized water.

NOTE 12: *Judgement will normally be made after storage for 14 days in NaOH. Continuation of storage and measurement until 28 days is recommended in order to collect data that may be useful for establishing criteria to predict AAR-3 results.*

7.7 Safety measures

Reliable safety precautions should be taken and suitable personal protective equipment should always be used to avoid the hazards of the hot alkaline solution such as severe burns and injury to unprotected skin and eyes.

8. CALCULATION AND REPORTING OF RESULTS

8.1 Expansion

The linear expansion of each specimen is obtained by calculating the difference between the length of the specimen at each period of measurement (Ln) and the zero measurement (Lo), to the nearest 0.001% of the effective length, as follows:

Expansion, % = 100 x (Ln - Lo)/Gauge length

where:
Ln = reading taken at each period of storage in sodium hydroxide solution,
Lo = measurement of specimen before subjection to sodium hydroxide solution,
Gauge length = distance between inner ends of the metal pins, measured to the nearest 1 mm.

8.2 Expansion recording and further examination

Record and report to the nearest 0.01% the average expansion of the specimens for a given period. If a specimen breaks during the test, the test will be considered valid provided the two remaining specimens do not differ from each more than the values given below. For average values of expansion greater than 0.10%, the repeatability is considered satisfactory if the expansion of each specimen is within 10% of the average value. For average values of expansion less than 0.10%, the repeatability is considered satisfactory if, for each specimen, the deviation from the average value is within 0.01%. If these values are exceeded, deem the test invalid and repeat the test.

After the final measurement, the specimens shall be examined and any relevant features recorded. Warping, if observed, shall be measured on 3 moulded surfaces by placing the specimens on a plane surface, with curved ends facing downwards, and measuring the maximum separation between the specimen and the surface to the nearest 0.2 mm. Location, type and pattern of any cracking should also be recorded.

NOTE 13: *If the bars exhibit an expansion that is judged to be deleterious, a petrographic examination of the interior of the bars that have expanded most should be carried out together with an examination of the internal crack pattern to confirm that the cause of expansion is likely to be an alkali-aggregate reaction. If it has been concluded from the expansion results and supplementary examination of the prisms that a given aggregate should be considered potentially alkali-reactive, additional studies using RILEM Recommendation AAR-3 (Concrete Prism Method) and/or AAR-4 (Ultra-accelerated Concrete Prism Method) may be appropriate to develop further information on its potential alkali-reactivity and to evaluate the effect of coarse aggregate, different aggregate gradings and different alkali contents of the concrete.*

9. TEST REPORT

The following information shall be given in the report:

- identification and source of the aggregate sample and reference to petrographic analysis, if available; date of and state at delivery at the laboratory;
- type and maximum size of the aggregate;
- type of processing undertaken on the aggregate sample in the laboratory (washing, drying, crushing, sieving etc.);
- grading of the aggregate as used in the test;
- identification and source of the Portland cement;
- alkali content of the cement expressed as equivalent sodium oxide (% Na_2O + 0.658 % K_2O);
- autoclave expansion and/or Le Chatelier value of the cement;
- Blaine fineness of the cement;
- workability of the concrete (flow, if measured);
- type and content of superplasticizer (if used), and its contribution to the alkali content of the mortar mix;
- size and mass of the test bar specimens;
- tested reference (reactive and non-reactive) aggregates;
- initial expansion of the bars after 24 h of storage in water at 80°C.
- each single value and average percentage length change after each measurement of the specimens;
- a graph of the percentage length change vs. time from the zero reading to the end of the immersion period in NaOH solution;
- results of any warping measurements of the specimens;
- any significant features revealed by examination of the specimens and the sodium hydroxide solution during and after the test.

ANNEX

A1. Reference aggregates

It is recommended that the test is performed on samples of the aggregate in question, a reference reactive carbonate aggregate (*e.g.* Pittsburg quarry, Kingston, Ontario, Canada; available upon request from: Ontario Ministry of Transportation, Materials Officer, 1201 Wilson Avenue, Downsview, Ontario, Canada, M3M 1J8) and a reference non-reactive aggregate. For the latter, a natural gravel or crushed rock may be used that is known from experience to be non-reactive and, when tested according to AAR-2, exhibits an expansion of not more than 0.02 % (bars 40x40x160 mm).

A2. Interpretation of results

Typically, for alkali-silica reaction, the smaller the aggregate the more reactive it is and expansions in AAR-2 (0/4 mm aggregate) should be greater than in AAR-5 (4/8 mm aggregate), provided the dimensions of the bars and the length of the test period are the same. When, however, the expansions in AAR-5 are as high or even higher than in AAR-2, then something different from normal alkali-silica reaction has probably happened. With concrete, owing to the larger aggregate used, even higher expansions have to be expected.

It has been found that the composition of the cement can influence the expansions; different aggregates (with different amounts and/or kinds of dolomitic material) may require different criteria for maximum admissible expansion.

Therefore, for the time being, expansion limits should be applied only where established by local experience (in China a 28-day criterion of 0.1 % has been used successfully). In this Recommendation no such limit is suggested and the following procedure is recommended instead:

Test the aggregate both in AAR-2 and AAR-5, using the same cement and bars with the same dimensions (40x40x160 mm), and compare the expansions after 14 days in NaOH solution:

-AAR-2 expansion higher than admissible: → further testing by AAR-3 or AAR-4 [10] required

-AAR-2 expansion smaller than admissible:

- ➢ AAR-5 expansion same or higher: → further testing by AAR-3 or AAR-4 required
- ➢ AAR-5 expansion smaller by at least 0.01 %: no further testing required.

A3. Influence of the size of the aggregate

When only 4/7 mm aggregate is available, this will have to be used, but somewhat smaller expansions will have to be expected with aggregates containing dolomitic material.

When 4/11 mm aggregate (or similar) is to be tested instead of 4/8 mm, the amount on the intermediate 8 mm screen should be specified, in order to ensure a well defined grading. With aggregates containing dolomitic material, the expansions will be greater than with 4/8 mm aggregate.

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