A SIMPLE CHEMICAL TEST METHOD FOR THE DETECTION
OF ALKALI-SILICA REACTIVITY OF AGGREGATES

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1. ABSTRACT

In this paper a simple and quick chemical test method is
proposed for the detection of alkali-silica reactivity of aggre-
gates. The proposed method consists of suspending a mixture of
CaO and the aggregates to be tested in a saturated solution of
KCl maintained at an elevated temperature, eg 70°C. After 24
hours the suspension is cooled to 20°C, filtered and the OH ion
concentration of the filtrate determined. The measured OH ion
concentration is then compared to that of a control suspension
of a mixture of pure quartz sand and CaO treated in the same way.
Any lowering of OH ion concentration in the test solution, com-
pared to the control, indicates a potential alkali-silica reac-
tivity of the test aggregate. The difference in the OH ion con-
centrations between the control and the test is a measure of the
alkali-silica reactivity of the aggregate under test. Repeated
measurements on a number of sand samples showed that the standard
deviation of measured OH ion concentrations of the test solutions
is about 2% of the mean. So far three different laboratories have
tried this method and obtained similar standard deviations.

This simple and quick method could be carried out in the
field and the observed low standard deviation indicates that
it could be used for the quality control of the aggregates.

2. INTRODUCTION

From the reports of the recent alkali-silica conferences
it will appear that the incidence of alkali-silica reaction in
concrete is increasingly occurring in countries which were pre-
viously assumed to be free of reactive aggregates. In those
countries it is not possible to rely on the service records for
the selection of the aggregates. This calls for a quick and
simple method for the identification of reactive aggregates.
Furthermore, at present there is no suitable test method for al-
kali-silica reactivity which could be used in the quarries for
the quality control of the aggregates especially for the sand
fractions. This paper addresses the above two points.

On the basis of an extensive series of research work on al-
kali-silica reaction and associated expansion we have recently
proposed a new mechanism for alkali-silica reaction and the as-
sociated expansion (1). According to this mechanism any soluble
alkali compound eg NaX present in the cement environment takes
part in the alkali-silica reaction. During the reaction the al-
kali ions from the alkali compound and OH ion from $Ca(OH)_2$ enter
the reactive grains forming solid alkali-silica complexes leav-
ing behind Ca and X ions in the liquid phase. The solid $Ca(OH)_2$
which is present in the cement paste will then tend to dissolve
to maintain the liquid phase saturated with $Ca(OH)_2$. However Ca
ions which are already in the liquid phase will decrease the so-
lubility of $Ca(OH)_2$ in the liquid phase by the common ion effect
i.e. the concentration of OH ion in the liquid phase will be de-
pressed compared to the case where no such alkali-silica reac-
tion had occurred.

Basing upon this observation it has been suggested that the
potential alkali-silica reactivity could be detected by storing
the aggregate in a suspension of $Ca(OH)_2$ in a saturated KCl so-
lution at an elevated temperature eg $70^{o}C$ and measuring the
OH ion concentration of the solution and comparing it to that
of a control suspension of quartz sand. This paper reports work
done to develop the above suggestions into a suitable test me-
thod for the detection of the potential alkali-silica reactivity
of aggregates.

3. MATERIALS AND EXPERIMENTAL TECHNIQUE

In this investigation one pure quartzic sand and four reac-
tive sand types of varying expansivity have been used. A freshly
burnt lot of CaO and a saturated solution of KCl (at $20^{o}C$) were
used to make suspensions.

Detailed step by step description of the proposed method is
given in the appendix. In the investigation two temperatures,
i.e. $50^{o}C$ and $70^{o}C$ were used as reaction temperature. Experiments
were repeated on two different days in order to evaluate the re-
producibility of the proposed method.

4. RESULTS AND DISCUSSION

The results of this investigation are shown in Table 1;
note that two of the experiments, marked with *) are obviously
unusable.

The OH ion concentrations of the filtrates from suspensions
containing sand No. 1, a pure quartz sand, should be independent
of the reaction temperature and should represent the solubility
of $Ca(OH)_2$ in the saturated KCl solution at $20^{o}C$. The mean of
the four valid experiments is 597 mg OH/litre with a standard
deviation of 6 i.e. 1% of the mean value. Subsidiary experiments
showed that the solubility of $Ca(OH)_2$ in a saturated KCl solu-
tion corresponds to 600 mg OH/litre, i.e. as expected a quartz

Table 1

The OH ion concentration of the filtrates in mg/l solution

Sand No.	Expansivity	Set 1 50°C	Set 1 70°C	Set 2 50°C	Set 2 70°C	Set 3 50°C
1	Nil	590	603	601	580 *)	593
2	Very low	550 *)	565	567	561	
3	Low	541	527	549	522	
4	High	499	463	510	457	500
5	High	486	458	488	457	

*) have some difficulties during filtration

Table 2

The mean OH ion concentrations of the filtrates in mg/l

Sand No.	50°C	70°C	Diff. 50°	Diff. 70°
1	597	597	–	–
2	567	563	30	34
3	545	525	52	73
4	503	460	94	137
5	487	457	110	140

sand shows no alkali-silica reactivity. The three experiments
with sand No. 4 at 50°C gave a mean OH ion concentration of
503 mg/l solution with a standard deviation of 6 i.e. 1.2% of
the mean. The filtrates from suspensions containing sands Nos. 3
and 5 also showed very low spread of OH ion concentrations at
each temperature. This low spread of results at each temperature
indicates a high degree of reproducibility and also allows for
the use of the mean OH ion concentration at any one temperature
for further consideration. The mean OH ion concentrations are
tabulated in Table 2.

The difference in the OH ion concentrations between the filtrate from sand No. 1 and any other filtrate represents the amount of alkali-silica reaction that had occurred at a given temperature. These differences are also tabulated in Table 2 as Diff. 50° and Diff. 70°. It is also possible to calculate the individual Diff. 50° or Diff. 70° from Table 1 and the maximum uncertainty, so far obtained, is about 4% of the individual mean.

The results from the industrial laboratories showed that a reproducibility of about 2% of the mean can easily be obtained. This low spread of results indicates that this technique could be used for the quality control of a chosen sand. No work has yet been done to evaluate the inter-laboratory reproducibility of the method especially the Diff. values.

Though the results so far obtained is very encouraging a note of caution is in order. This method like all other chemical methods eg ASTM Quick Chemical Method, does not have any inbuilt limit for acceptance. To select an acceptance criterion for the selection of an aggregate the method has to be calibrated against observed expansions as has been done in the case of ASTM Quick Chemical Method. However, the sand No. 3 has such expansivity that its Diff. values may be taken as the preliminary acceptance criterion. The advantages of this method over ASTM Quick Chemical Method are (i) no special instrument is necessary, (ii) its sim-plicity, (iii) sand samples could be treated as received and gravels could be crushed down to follw the ASTM grading, (iv) a larger sample could be tested, (v) the OH ion concentration never exceeds that of a cement paste thereby avoidding some complica-tions inherent in the ASTM Quick Chemical Method, eg consumption of OH ions by carbonate minerals.

ACKNOWLEDGEMENTS

REFERENCES

[1] S. Chatterji et al - Studies of Alkali-Silica Reaction. Part 3, Cement & Concrete Research, 16, 246-254, 1986.

APPENDIX

The detailed description of the test method

1) Prepare representative duplicate dry samples of ca. 100 g each. Note the exact weights of each of the samples. Place both the samples in an air oven maintained at the desired reaction temperature, i.e. 70°C.

2) Prepare a saturated solution of KCl in distilled water. Store the saturated solution for two days at 20°C before use

3) In two numbered conical flasks, with ground glass joints, weigh out the saturated KCl solution such that solution/sand ratio is 2.

4) Weigh out two lots of CaO, 3.5 g each. Add one lot of CaO in each of the conical flasks containing saturated KCl solution and stopper them.

5) Store both the conical flasks in the air oven maintained at the desired reaction temperature for 16 hours.

6) Add the heated sand samples to the corresponding conical flasks and stopper them. Swirll the flasks carefully, but thoroughly. Maintain at the desired temperature for 24 hours. For the first 8 hours resuspend sand samples every 2 hours.

7) Cool the suspensions in a water bath maintained at 20°C for at least 3 hours. During the cooling period resuspend the mix every 20 minutes; this will ensure resaturation of the solution with $Ca(OH)_2$ at 20°C.

8) Filter the suspensions through 0.4 μm size membrane filters under suction. Avoid carbonation after the solutions have been filtered.

9) Determine the OH ion concentrations of the filtrates acidimetrically using 0.025 N HCl and phenolphthalein as the indicator.

10) Treat a quartz sand as above and use its OH ion concentration as the control.

11) A reaction temperature of 70°C appears to be desirable.