

Assessment of Aggregate Reactivity Using Slurry Tests

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Abstract

The testing and screening of aggregates for their alkali-silica reactivity (ASR) is generally carried out initially by petrographic analysis. If reactive aggregates are identified by petrographic analysis then a rapid screening of the aggregate's potential to cause expansion using the accelerated mortar bar test (AMBT, AS-1141.60.1) is carried out to determine further reactivity potential. Aggregates that are found to be reactive in the AMBT method may be further screened using the concrete prism test (CPT, AS-1141.60.2). Both AMBT and CPT methods are a compromise between introducing accelerated and reactive conditions and monitoring the expansion over short and long periods of time but with conditions that are more closely aligned with field conditions.

Given that these tests are empirical estimates of reactivity potential, alternate testing may be developed for the screening of aggregates. Alternate laboratory tests are rapidly carried out using slurry tests on small samples of ground aggregate (e.g. ASTM C289). Simulating storage temperatures used in the AMBT (80°C) and CPT (38°C) in 1 M NaOH (1.25% Na₂O_e) is an alternate approach to the development of new rapid screening tests. To assess the degree of aggregate reactivity a co-reactant, calcium hydroxide (CH), may be added to the reaction mixture aiding reactivity assessment through the consumption of CH. The results of a laboratory trial into the reactivity of aggregates using a ground aggregate slurry test of this nature are reported in this paper. The results are correlated with standard test method data using AMBT and CPT (AS-1141.60.1 and 2) with a view to assessing this method (or methods of this type) as an alternative rapid screening approach in the identification of aggregate reactivity for ASR potential.

Keywords: ASR, ground aggregate, slurry tests, calcium hydroxide, AMBT, CPT

Introduction

The alkali silica reaction (ASR) is a reaction that occurs in concrete for aggregates that contain reactive silica. Expansion accompanying ASR formation can result in deleterious cracking (c.f. [1]). ASR occurs through the reaction of hydroxyl ions at elevated pH with reactive silica derived from the aggregate to form an amorphous silica gel (ASR gel) [2-3]. The ASR gel then absorbs water and expansion occurs resulting in hydrostatic stresses exerted on the surrounding matrix. If sufficient hydrostatic stress is present the formation of cracks will arise within the concrete matrix [2]. The mechanism of ASR is therefore a two-step process involving the formation of the gel through chemical means followed by the physical action of the gel on the concrete resulting in cracking.

Test methods screening aggregates for reactivity potential can be applied to the identification of reactive phases (i.e. potential of the ASR gel to form) or through methods that investigate the action of the ASR gel on the mortar and concrete such as in expansion tests. Typically, aggregates are screened for reactivity initially through petrographic analysis for the identification of reactive silica phases. Once reactive silica has been identified in the aggregate, expansion testing using the accelerated mortar bar test (AMBT) and the concrete prism test (CPT) is carried out following the procedures laid out in AS-1141.60.1 and AS-1141.60.2, respectively [4-6]. The assessment protocol for aggregate reactivity is outlined in Section 2 of Standards Australia Handbook HB79-2015 [1].

Alternate testing has also been applied to determine the level of reactivity of aggregates such as withdrawn test method ASTM-C289-07 [7] (withdrawn due to unreliability with reference to field performance), but has generally investigated silica dissolution in alkali solution at elevated temperature although some effort has been applied to the insoluble silica content of the reaction

Table 1 Particle size distribution analysis of 3 aggregates, the numbers are percentages which passes the size.

Sizes in μm	M (%)	G (%)	B (%)
100	100	96	99
75	99	90	97
45	92	77	88
10	64	51	60

media [8]. The ASR gel formed, however, not only contains alkali, but also contains calcium ions in the silica network in an atom ratio of approximately 70 Si : 20 (Na + K) : 10 Ca [9, 10]. The presence of calcium in the ASR gel suggests that calcium ions are also relevant to the ASR process. This is especially true as ASR occurs in the pore solution saturated with calcium ions due to the formation of calcium hydroxide (CH) as a by-product of cement hydration [11]. Given that CH is a consistent component of all mortars and concretes, and that calcium ions are consumed in the ASR process, CH content has the potential to be used as a screening tool in laboratory test methods investigating aggregate reactivity. This study reports on the investigation of CH consumption in ground aggregate slurry tests containing CH and 1M sodium hydroxide (NaOH) solution carried out at 38°C over a period of 1 year to complement the findings of the 80°C study reported in Concrete 2017 [12].

Materials and Methods

Materials:

The coarse aggregates used in this study, micro-diorite (M), greywacke (G) and basalt (B), were supplied by Cement, Concrete and Aggregates Australia (CCAA). These aggregates were used as part of an inter-laboratory study to evaluate the reactivity potential using AS-1141.60.1 and AS-1141.60.2 test methods. The aggregates were classified according to AS-1141.60.1 (AMBT) as: non-reactive (Aggregate B), slowly reactive (Aggregate M) and reactive (Aggregate G), and according to AS-1141.60.2 (CPT): non-reactive (Aggregates M and B) and reactive (Aggregate G).

The as-received aggregates were ground in a RockLab ring grinder for 5 min and stored in resealable plastic bags in a desiccator until required. The particle size distribution for each aggregate was determined using a Malvern Mastersizer 2000 laser diffraction particle size analyser and is listed in Table 1.

Chem-Supply sodium hydroxide (98%) containing 1.0% sodium carbonate, 0.5% chloride and 0.01% iron oxides as impurities was used to prepare the 1.0 M sodium hydroxide solution. Chem-Supply calcium hydroxide (98%) containing 0.60% magnesium oxide, 0.12% silicon dioxide, 0.08% aluminium oxide, 0.05% iron oxides and 0.50% carbon dioxide as impurities was used as the co-reactant in the slurry test.

Slurry Test:

Slurry tests were carried out by mixing 1.00 ± 0.01 g of ground aggregate with 1.00 ± 0.01 g of calcium hydroxide in HDPE centrifuge tubes with 10 mL of 1.0 M of sodium hydroxide added. The tubes were sealed and shaken to produce homogenous slurries and placed in a 38°C oven for 1, 7, 14, 21 and 28 days and thereafter at 28 day intervals up to 364 days. The solids recovered using a Buchner funnel, were rinsed with 10 mL of methanol and transferred to a watch glass and dried in a 50°C vacuum oven for 72 hours. The solids were then lightly ground in a mortar and pestle, transferred to a new sealed tube and stored in the 50°C vacuum oven until required for analysis.

Thermogravimetric Analysis (TG):

Mass loss data was collected using a Netzsch Jupiter F5 STA thermogravimetric analyser. The dried, ground samples (25 ± 1 mg) were weighed in an alumina crucible, placed into the instrument and stabilised at 40°C for 10 min prior to heating at a rate of 10°C/min to 1000°C in a helium atmosphere at a flow rate of 60 mL/min. The mass fraction of calcium hydroxide (CH) remaining in each sample was determined from the mass loss between 390 and 470°C normalised to the mass loss at 900°C. The mass loss resulting between 390 and 470°C is based on the dehydroxylation reaction:



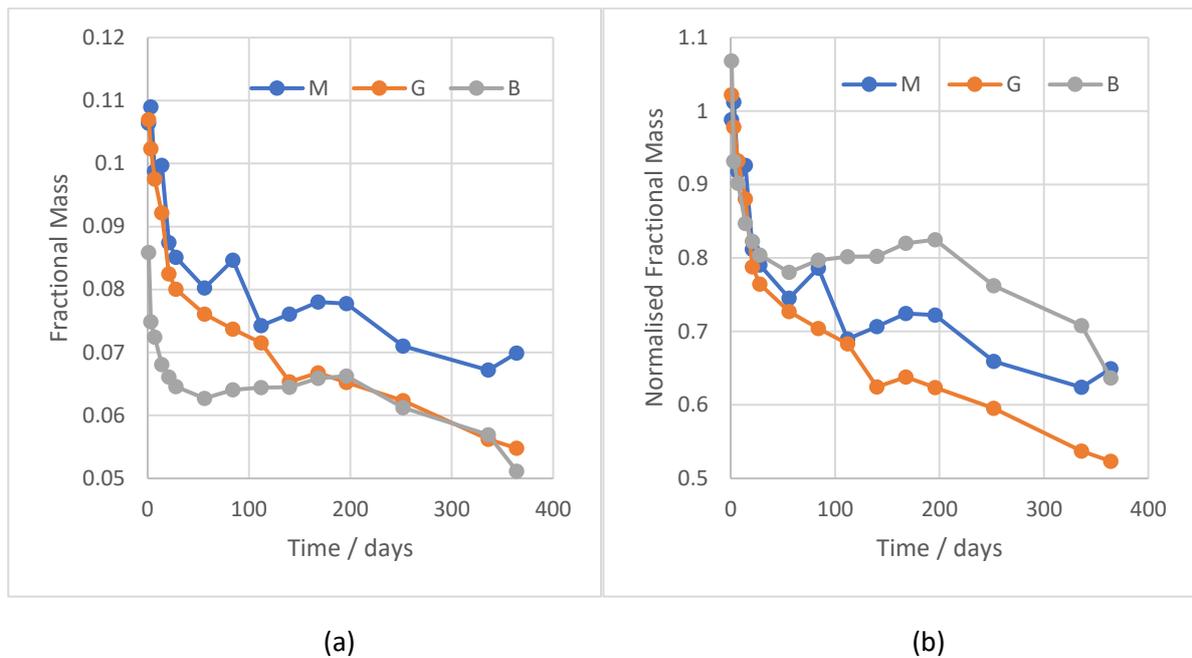


Figure 1 TG data of CH (a) fractional mass and (b) normalised fractional mass as a function of aging time.

X-ray Diffraction (XRD):

XRD data was collected using a Bruker Discovery D8 XRD using Cu K α radiation with a wavelength of 1.5148 Å. Diffraction patterns of hand ground powder samples were collected in the range 3° to 75° with a step increment of 0.01949° at 0.630 s for each point. Phase identification was determined using the International Centre for Diffraction Data (ICDD) ICDD Products - PDF-4+ database.

Results and Discussion

Three aggregates, micro-diorite (M – AMBT slowly reactive; CPT non-reactive), greywacke (G – AMBT reactive; CPT reactive) and basalt (B – AMBT non-reactive; CPT non-reactive), were investigated in this study, which have known reactivity based on standard methods testing (AMBT and CPT; AS 1141.60.1 and 2 [4, 5]). The aggregates were ground to sub 100 microns and used to investigate reactivity in slurry tests using CH as a co-reactant in 1M NaOH solution as the reaction medium with the reactions carried out at 38°C. Data for these slurry mixes has already been reported elsewhere for a reaction temperature of 80°C [12]. In the current study, the CPT condition temperature of 38°C has been chosen to investigate reactivity over a CPT time scale (up to 1 year). The 1M NaOH solution was selected as it is consistent with AMBT test conditions and considered to be 1.25% Na₂O_e, as used in the CPT method. The particle size reduction was designed to increase the reactivity potential of the aggregates. In the AMBT and CPT test methods, the aggregate is graded and the fines are discarded. For the slurry tests carried out in the current study, the complete sample was used in order to reduce the potential for selectively discarding reactive components. CH was used to provide a saturated solution in calcium ions as well as being available for reaction in ASR gel formation; the principle being that the calcium ions in solution aid the process of precipitation of the silica gel and therefore become a reactant in the process.

As the CH takes part in the reaction in the process of precipitating the ASR gel, the progress of the reaction can be followed by determining the proportion of CH present in the sample as a function of time. TG analysis of the decomposition of the CH according to Equation (1) allows the quantification of the proportion of CH remaining in the sample. The data is represented as fractional mass with respect to the total mass of solids recovered by filtration (Figure 1(a)). It is interesting to note that the non-reactive Aggregate B shows a significant degree of consumption of CH in the very early stages of the reaction (mass fraction of CH for an ideally unreacted slurry would be 0.16). Data reported for this aggregate for the 80°C slurry test also showed rapidly consumed CH before 1 day of storage in the 28-day test period. Reactivity tests on Aggregate B (to be published elsewhere) have been carried out and have found that zeolitic phases are formed within 1 day of immersion in 1M NaOH. This type of reactivity is not consistent with ASR and therefore rapid consumption of CH is not indicative of reactivity in an ASR context. In order to correlate reactivity between aggregates over longer ages, the

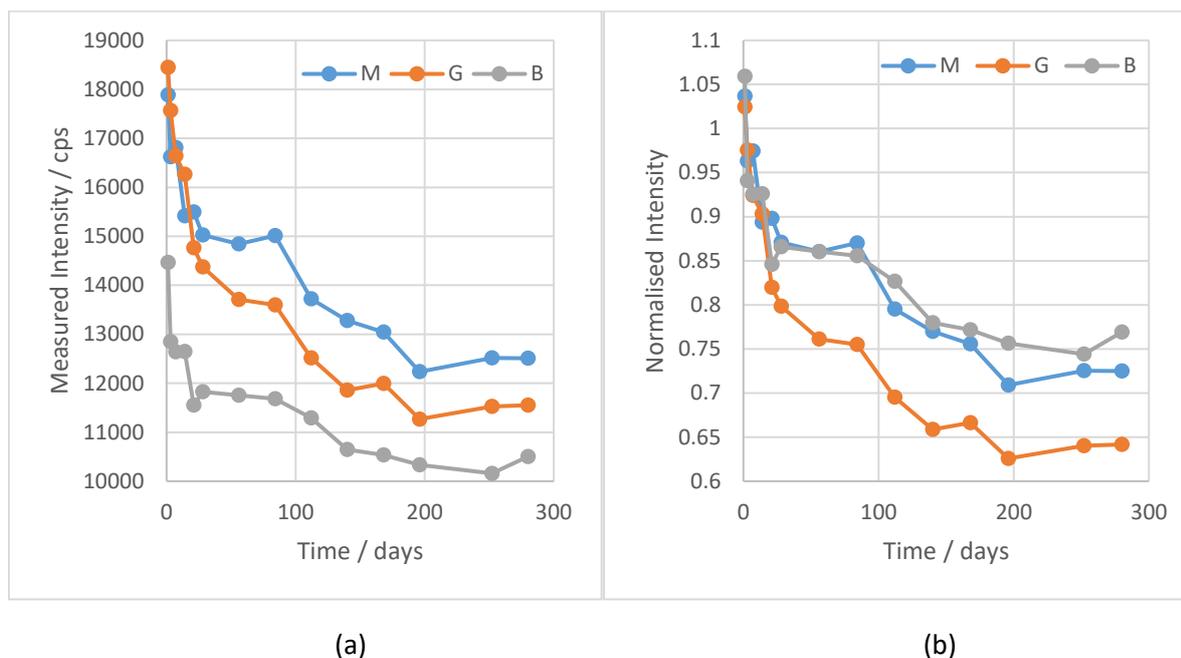


Figure 2 XRD data of CH (a) absolute intensity of the (101) CH peak and (b) normalised intensity.

data was normalised to 7 days (Figure 1(b)). The relative reactivity of these samples based on the normalised data correlates well with the reactivity determined from standard test methods; that is, Aggregate G shows greater reactivity than Aggregate M and Aggregate B shows the least reactivity. This correlation is consistent with the data presented at Concrete 2017 for AMBT mirrored conditions (80°C). It is also interesting to note that Aggregate B reactivity based on this screening method appears to increase beyond 200 days (reduced CH content is observed). There is some discussion that aggregates containing feldspars such as the basalt used here may be subject to delayed ASR through the dissolution of the feldspars as a source of dissolved alkali and silica [13].

To confirm the relative consumption of CH in these slurry tests, XRD was also carried out and the proportion of CH remaining was determined semi-quantitatively using the absolute intensity of the (101) CH diffraction peak at $34.1^{\circ}2\theta$ (Figure 2(a)). Comparison of the intensity dependence of the (101) peak with the TG data shown in Figure 1(a) shows significant similarities especially with respect to the relative proportion of CH remaining in Aggregates M and G with respect to Aggregate B. The data was also normalised to the 7 day intensity and plotted in Figure 2(b). A similar correlation between the reactivity is observed with Aggregate G being significantly more reactive than Aggregates M and B.

The correlation of reactivity between the slurry test carried out at the CPT temperature (38°C) with the slurry tests carried out and presented at Concrete 2017 under conditions aligned with the AMBT (80°C) suggest that there is significant potential in using CH as a marker for aggregate reactivity. Further testing on a range of aggregates with known reactivity from field and laboratory expansion test data, however, is required to validate and demonstrate the viability of using methods of this type as alternative screening methods. The key to the advancement in this method is based on the CH present and is not dependent on the aggregate. Aggregates vary significantly in their mineralogy, such as the aggregates used in this study, which also vary in their morphology as well as their mineral composition. The mortar and concrete environment, however, is consistent in that the hydration of cement produces CH as a by-product, which is then involved in the precipitation of the ASR gel. Monitoring the consumption of CH in the reaction therefore has significant potential as a simplified laboratory test method for aggregate screening for determining the reactivity potential of an aggregate to ASR.

Summary

Aggregate reactivity has been investigated by monitoring the CH content of the solid phase in ground aggregate-CH-alkali slurries for aggregates with known reactivity based on expansion test data. The

correlation between reactivity determined from standard expansion tests and CH content in the slurry tests carried out in this study and the study reported in Concrete 2017, particularly when the CH content is normalised to early age specimens, demonstrates that CH content determination has the potential to be developed into a new screening test for aggregate reactivity. At this stage limited data has been collected, but plans are in place to continue the investigation on a range of aggregates with known reactivity based on laboratory expansion testing and field performance. An extended range of aggregate reactivity data will allow further assessment of the efficacy of testing based on CH content rather than the more traditional chemical test methods which rely solely on silica dissolution.

Acknowledgements

The authors would like to acknowledge the Australian Research Council Research Hub for Nanoscience Based Construction Materials Manufacturing (NANOCOMM) and Cement Concrete and Aggregates Australia (CCAA) for their financial support.

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