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# Protocols for investigating the reactivity of aggregates and alkali thresholds for ASR prevention

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#### Abstract

Alkali in concrete pore solution and reactive silica in aggregate are integral features required for alkalisilica reaction (ASR). When high amounts of alkali are present, expansive ASR gel forms that cause cracking of concrete. Thus, limits have been imposed, restricting allowable alkali contents for use in concrete. However, these limits are known to be generalised with a single limit specified for all aggregate types. This study investigates the reactivity potential and critical alkali threshold for individual aggregates and aggregate combinations, by increasing alkali content (0.60-1.25% Na<sub>2</sub>O<sub>e</sub>) in concrete, varying exposure temperature (38-80°C) and extending test duration. A combination of RILEM recommended methods and modified versions of the standard accelerated mortar bar test (AMBT) and concrete prism test (CPT) expansion test methods have been used. The key findings of this study suggest that the potential exists for specifying a determined alkali threshold in concrete based on the reactivity classification of aggregates used, thus, allowing a relaxation of the current alkali limit for concrete. This approach permits greater flexibility in the potential safe use of reactive aggregates in concrete. Furthermore, this study shows that the determination of an aggregate's reactivity and potential to ASR is not only highly dependent on its chemical composition and the alkali content in the concrete but also the test method, exposure temperature and test storage age used to assess changes in expansion.

Keywords: aggregate reactivity; alkali limit; alkali-silica reaction; alkali threshold; expansion tests

## 1. INTRODUCTION

Alkali-silica reaction (ASR) initiates with the dissolution of metastable silica in the presence of high alkali content in pore solution [1]. Most of the alkalis available for reaction in concrete are known to originate from Portland cement. However, other potential sources of alkalis can also include those originating from within the aggregates used in the production of concrete. Although, these alkalis are released over much longer periods of time when compared with those alkalis released from Portland cement hydration. The initial hydration of Portland cement releases calcium (Ca<sup>2+</sup>), potassium (K<sup>+</sup>), sodium (Na<sup>+</sup>), hydroxyl (OH<sup>-</sup>) and sulphate (SO<sub>4</sub><sup>2-</sup>) ions into pore solution. The concentration of alkali cations (Na<sup>+</sup> and K<sup>+</sup>) present at any given age primarily determine the alkalinity of the pore solution.

To reduce the risk of ASR, aggregates that demonstrate the potential to be reactive are often restricted from use in concrete. Preferential use of non-reactive aggregates in the production of concrete places a significant burden on the demand and availability of quarry suppliers to provide these aggregate sources. With the rise in construction activity, non-reactive aggregate sources are becoming increasingly scarce. Additionally, guidelines have been imposed on the alkali limit allowed in concrete to reduce the amount of alkali available to react with reactive silica in aggregates. This limit is conservative and has been generalised particularly in Australia and in New Zealand as 2.8 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> and 2.5 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> respectively for all aggregates used in concrete [2]. To achieve the set alkali limit in concrete, low alkali cements are employed in the production of concrete. Low alkali cements are those with alkali content not exceeding 0.6% Na<sub>2</sub>O<sub>e</sub> [3, 4]. Achieving low alkali contents in cement manufacture is both an environmental and economic challenge. There are however instances reported in literature where concretes containing low alkali cement still experience ASR [5]. Therefore, the alkali content that will induce ASR in concrete is primarily dependent on the mineralogical composition of the aggregate used

in concrete [4]. Consequently, it is important to identify appropriate alkali limits for distinct aggregates and aggregate combinations to ascertain their ASR reactivity in the presence of alkali.

The objective of this study is to investigate the potential reactivity of some commercially available aggregates using standard laboratory expansion tests to determine the behaviour of these aggregates with variations in alkali content. The purpose of introducing a variable alkali content is to establish the alkali threshold- the alkali content at which initiation of expansion due to ASR becomes evident [6]. Consequently, the study also hopes to propose the likelihood of using reactive aggregates in concretes, as well as the potential of introducing flexible alkali limits that exceed the current alkali limits in place thus conserving natural resources and reducing cement manufacturing costs.

# 2. EXPERIMENTAL PROCEDURE

## 2.1 Materials

For this study, two New Zealand aggregates classified by petrographic examination as slowly reactive and reactive have been selected. These aggregates were used in combination with two Australian non-reactive aggregates to determine their effectiveness in reducing expansion due to ASR, and their respective alkali threshold for safe use in concretes.

A cement with alkali content of 0.58% Na<sub>2</sub>O<sub>e</sub> conforming to the current alkali limit for cement in New Zealand was selected as the source of the binder for use in this work. A description of the aggregates used, and their reactivity classification is given in Table 2.1.

Aggregate	Symbol	Reactive Minerals	Reactivity Classification
New Zealand Aggregate A	NZ-A <sub>f</sub>	Glassy volcanic clasts, quartz, cherts and feldspar	Reactive natural sand
New Zealand Aggregate B	NZ-B <sub>f</sub>	Hornblende, pyroxene, feldspar, quartz	Slowly reactive Manufactured sand
	NZ-Bc	and epidote	Slowly reactive
Peats Ridge	PR₀	Not Applicable	Non reactive
	PR <sub>f</sub>	Νοι Αμμισαρίε	NOT-LEACTIVE
Maroota sand	Mf	Not Applicable	Non-reactive

Table 2.1: Selected	aggregates	under	investigation.

\*c = Coarse Aggregate; f = Fine Aggregate

## 2.2 Methods

Throughout this study, AS 1141.60.1 accelerated mortar bar tests (AMBT) and AS 1141.60.2 concrete prism tests (CPT) were conducted to assess the amount of expansion and classify the reactivity status of aggregates and aggregate blends. CPT tests have been conducted at 38°C and 60°C.

#### 2.2.1 Petrographic Assessments

Before undertaking any AMBT and CPT studies, RILEM AAR-0 recommends that petrographic examination be undertaken to determine the potential of aggregates to alkali reactivity. In this study, petrographic examinations were conducted following the requirements of the AS 1141.65 test method and CCANZ Technical Report TR 3 guidelines.

At the conclusion of undertaking AMBT studies, mortar bars were subjected to petrographic examination to establish the presence of any ASR and whether any other cement-aggregate reaction might have taken place. The extent of the reaction, the nature of the reaction products and the effects of the reaction

produced with exposure to a chemical aggressive environment were also evaluated. Test methods covered under the requirements of ASTM C856 (Clauses 5.5.1, 5.5.3, 5.5.4, 5.5.6 and 5.5.8) were used to undertake these petrographic analyses.

#### 2.2.2 Accelerated Mortar Bar Test

The AMBT method is primarily intended for use in the screening of aggregates and aggregate blends for ASR reactivity potential [4]. In this study, three alkali environments of 0.6M, 0.8M and 1.0M NaOH were selected to vary the alkali concentration for an extended test period of 56 days. Expansion results were compared at the standard ages of 10 and 21 days; however, the expansion of aggregates and aggregate blends at the end of the 56 days has also been studied to determine the reactivity status of aggregates changing with age. The standard expansion limit of 0.1% at 10 days and 0.3% at 21 days [7] were applied for all AMBT mixes including those AMBT mixes evaluated under the modified AMBT conditions of 0.6 M and 0.8 M NaOH. As per the Australian AMBT standard, an expansion limit of 0.15% at 10 days was used for mortars bars containing natural sand.

#### 2.2.3 Concrete Prism Tests

Expansion tests on CPT mixes for alkali contents exceeding the current alkali limit of 2.5 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> in New Zealand [2] were conducted for a test duration of 12 months. Five alkali contents of 2.5, 3.0, 3.5, 4.0 and 5.25 kg/m<sup>3</sup> have been used. The alkali threshold was determined in accordance to RILEM AAR-3.2 [8] with an additional alkali content of 5.25 kg/m<sup>3</sup> incorporated into the test programme to mimic the 1.25% alkali content used in the Australian CPT standard AS 1141.60.2. These alkali contents were achieved by adding 98% high purity NaOH to the concrete mix water.

Due to the longer timeframe required to undertake CPT at 38°C (12 months), this test method is more likely to identify aggregates that exhibit a change in reactivity status with time. However, to obtain some early indication of an aggregates' reactivity potential, CPT carried out at 60°C (ACPT) was also evaluated. In ACPT, results on ASR assessment are typically obtained at the end of a 6 month test period [4]. However, for this study, the test method was extended to 8 months to further observe any change in reactivity status resulting with time. ACPT follows a similar procedure to CPT for classifying the reactivity potential of aggregates. A review of available test data shows that applying the CPT expansion limit of 0.03% at 3 months for ACPT gives the same indication of an aggregate's reactivity classification as that obtained at 12 months for CPT [4]. Consequently, ACPT was also carried out at the five alkali contents chosen in the CPT study at 38°C.

## 2.3 Aggregate Combinations

Mortar and concrete mixes consisting of combinations of the aggregates under investigation were tested by the designated test methods. Table 2.2 describes the mix composition and test programme that were adopted in this study. In the CPT method, coarse and fine aggregates were tested together in a standard mix combination. In general, one component of the standard mix combination, coarse or fine aggregate, has known reactivity such that at any point in time the reactivity status of the unknown aggregate can be determined. However, certain variations of RILEM AAR-3.2 suggest that aggregates of unknown reactivity can also be evaluated as an aggregate blend [8].

In this study, fine aggregate blends that were identified as reactive in AMBT were selected for further assessment using CPT and ACPT. Non-reactive coarse aggregate sourced from Australia and slowly reactive coarse aggregate NZ-B sourced from New Zealand were used to determine the long term alkali reactivity potential of the fine aggregate blends. A fine aggregate blend comprising of 3 parts reactive aggregate to 2 parts of non-reactive/slowly reactive aggregate was used as the basis of the mortar and concrete mixes to determine the alkali reactivity potential of the proposed fine aggregate blends.

Standardised Tests		Aggregate and Aggregate Blends
AS 1141.60.1 at 80°C 3 alkali solutions: 0.6, 0.8 and 1.0 M NaOH	1a	NZ-A <sub>f</sub>
	1b	NZ-B <sub>f</sub>
	1c	NZ-A <sub>f</sub> + NZ-B <sub>f</sub>
		NZ-A <sub>f</sub> + PR <sub>f</sub>
AS 1141.60.2 at 38 and 60°C		NZ-A <sub>f</sub> +NZ-B <sub>f</sub> + NZ-B <sub>c</sub>
5 alkali contents: 2.5, 3.0, 3.5, 4.0 and 5.25 kg/m <sup>3</sup> Na <sub>2</sub> O <sub>e</sub>	2d	NZ-A <sub>f</sub> + M <sub>f</sub> +PR <sub>c</sub>

#### Table 2.2: Experimental Test Programme

# 3. RESULTS AND DISCUSSION

## 3.1 Accelerated Mortar Bar Test at 80°C (AMBT)

The expansion data of the AMBT mixes as a function of age up to 56 days for the three alkali solutions assessed are shown in Figure 3.1. By applying the standard AMBT expansion limit of 0.1% (0.15% for natural sand) and 0.3% at 10 and 21 days, respectively, for classifying aggregate reactivity (AS 1141.60.1), Mixes 1a, 1c and 1d showed expansion exceeding these 2 limits. Therefore, in accordance with these expansion limits, aggregate NZ-A<sub>f</sub> and its blends can be classified as reactive for all the alkali concentrations tested. Under these test conditions, it is observed that the reactivity of the fine aggregate blend is dominated by the reactivity of the aggregate NZ-A<sub>f</sub>. For Mix 1b, mortar bars in 1.0 M NaOH showed expansions of 0.03% at 10 days and 0.20% at 21 days. In accordance with AS 1141.60.1 expansion limits, this behaviour is indicative of a slowly reactive aggregate.

In general, an increase in expansion with increasing alkali concentration was observed for all AMBT mixes. At 10 days, the mortar mix composed of reactive aggregate NZ-A<sub>f</sub> (Mix 1a) showed a lower expansion (0.13%) in 0.8 M NaOH compared to the 0.30% expansion noted in 1.0 M NaOH. For all the alkali concentrations assessed, the blends containing reactive aggregate (Mixes 1c and 1d) showed similar expansions. This finding suggests that as a function of increasing alkali concentration, although able to differentiate reactive from non-reactive aggregates or blends, the AMBT method appears to be limited in appropriately identifying the reactivity level of the aggregates and aggregate blends. Particularly, at the standard condition of 1M NaOH and 0.6M NaOH, the reactive specimens are seen to expand similarly.

As the AMBT study was extended to 56 days, an increase in expansion was noted for all aggregates assessed including the slowly reactive aggregate NZ-B<sub>f</sub> (Mix 1b). What is of interest is the expansion noted for aggregate NZ-B<sub>f</sub> (Mix 1b) in 0.8 M and 1.0 M NaOH at 56 days. Based on the 0.3% expansion limit used, this aggregate may also be classified as reactive over time with expansions of 0.44% and 0.56% noted, respectively. Overall, the fine aggregate blends comprising reactive aggregate NZ-A<sub>f</sub> showed the highest expansion in 0.6 M NaOH, which again suggests the limitation and inconclusive indication of an aggregate's reactivity level obtained when AMBT is used alone.

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Figure 3.1: AMBT expansion results in (a) 0.6 M, (b) 0.8 M and (c) 1.0 M NaOH storage solution for tested aggregates

## 3.2 Petrographic Examination of AMBT Mixes

Following the AMBT studies, petrographic examination was conducted on mortar bars to determine if any ASR gel was present and if this gel had contributed to the expansion observed. Assessments conducted on thinly sliced representative samples of mortar bars classified as reactive under AMBT conditions showed the presence of hydrated gel containing scattered regions of calcium hydroxide. Remnants of isotropic ASR gel were also observed in the micro-fractured space of the hardened cement regions. Cracks filled with ASR gel were found to emanate through and around the periphery of aggregate components identified as glassy shards, acidic volcanics and quartz grains; components with perceived potential for ASR. Grains affected by ASR were also found to be porous from the depletion of silica. Although further microscopic studies and phase analyses are recommended, these observations are indicative of expressions of alkali-silica reactivity.

## 3.3 Concrete Prism Tests

At present, the criteria for ascertaining aggregate classification for CPT involves the use of prescribed expansion limits taken from 3 standard test methods, as described in Table 3.1. For this study, however, the expansion limits set out in AS 1141.60.2 were adopted for determining the reactivity classification of the fine aggregate blends assessed.

Interpretation	1-year Expansion Limit (%)			
Interpretation	AS 1141.60.2	ASTM C1293	RILEM AAR-3	
Non-reactive	< 0.03	< 0.04	< 0.05	
Reactive/Potentially Reactive	≥ 0.03	≥ 0.04	≥ 0.05	

Table 3.1: Assessment of aggregate reactivity based on current CPT standard methods

#### 3.3.1 Concrete prism test at 38°C (CPT)

Expansion measurements of CPT mixes containing varying alkali contents were taken at monthly intervals after 7 and 28 days until 12 months test duration. The expansion data captured for the fine aggregate blends assessed at varying alkali contents are shown in Figure 3.2. It is evident that the CPT mixes assessed for expansion show signs of shrinkage. As the alkali content was increased to  $5.25 \text{ kg/m}^3 \text{ Na}_2\text{O}_e$ , the expansion of CPT mixes was seen to increase. However, the 0.03% expansion limit for classifying reactivity in accordance with AS 1141.60.2 was not reached. Therefore, from these results, the fine aggregate blends were inferred to be non-reactive. Consequently, this finding suggests that these New Zealand aggregate blends may perform well at alkali contents lifted from the current stipulated limit of 2.5 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>. Overall, the CPT mixes containing NZ-B<sub>f</sub> aggregate showed no expansion. This behaviour is consistent with the petrographic and AMBT classification of slowly reactive assigned to NZ-B<sub>f</sub> aggregate. At 12 months, no discernible expansion was observed in CPT mixes for these fine aggregate blends containing up to and including 4.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> alkali content. However, a the notable increase in expansion was observed for the fine aggregate blend containing non-reactive aggregate (2d) at 5.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>.

RILEM AAR-3.2 (comparable to AS 1141.60.2) proposes an effective method for determining the alkali threshold of an aggregate or aggregate combination for use in concrete. Used in conjunction with RILEM AAR-7.1, a safety margin (usually -1.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>) is applied to the alkali threshold of the aggregate or aggregate blend under evaluation to establish a safe alkali limit at which potentially reactive aggregates can be used [9]. This safety margin accounts for differences between laboratory and field tests as well as other sources of alkali that cannot be accounted for at the time of testing. The alkali threshold is defined as the highest alkali content that is found to not induce significant expansion in an aggregate or aggregate blend. As discussed, the results in this study show no evidence of expansion for the fine aggregate blends assessed at alkali contents up to and including 4.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>. Although further testing is recommended, this finding suggests that with the implementation of a safety margin the current alkali limit in New Zealand (2.5 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>) may be lifted to 3.0-3.5 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> to incorporate the New Zealand fine aggregate blends evaluated in this study. A similar approach can thus be adapted to determine the alkali thresholds that apply to other aggregates or aggregate combinations intended for use in concrete.

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Figure 3.2: 12-month CPT expansion behaviour for aggregate blends of varying alkali content.

The effect of age on the rate of expansion of CPT mixes was also considered for the highest alkali content assessed in this study ( $5.25 \text{ kg/m}^3 \text{ Na}_2 O_e$ ). This data is presented in Figure 3.3. Mix 2c showed initial shrinkage before expansion. Generally, the rate of expansion of both fine aggregate blends evaluated is noted to increase with age. Although the 0.03% expansion limit was not reached, a continuous increase in expansion with age was observed at the end of the 12 months testing period. This finding suggests that monitoring expansion up to 12 months, and beyond, is beneficial in establishing the rate of expansion that applies for classifying the reactivity status of aggregates as a function of age.





#### 3.3.2 Concrete prism test at 60°C (ACPT)

The reactivity classification of fine aggregate blends assessed under ACPT conditions has also been interpreted using the CPT expansion limit of 0.03% applied at 3 months [4]. A comprehensive study carried out in Europe suggests that for aggregates classified as slowly reactive in initial tests, the expansion limit of 0.03% at 20 weeks of ACPT provides a more reliable assessment of the ASR potential of the aggregate [10]. Similarly, the results of ACPT at 3 months and 5 months of mix 2c and 2d are presented in Figure 3.4. At age 3 months for both fine aggregate blends evaluated, neither was found to reach the 0.03% expansion limit. Therefore, following the reactivity classification discussed in Table 3.1 both fine aggregate blends are inferred to be non-reactive. It is interesting to note that like in the CPT studies, the concrete prisms containing slowly reactive aggregate (Mix 2c) were again observed in ACPT to exhibit shrinkage for all the alkali contents evaluated up to 4.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>. In terms of expansion, the fine aggregate blend containing the non-reactive aggregate (2d) showed higher expansion than prisms containing the slowly reactive aggregate (2c). For instance at age 3 months for 5.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>, mix 2d showed an expansion of 0.014% where mix 2c expanded 0.006%. A similar observation was noted for this aggregate blend in the CPT studies conducted at 38°C for 12 months. At age 5 months, the fine aggregate blend containing non-reactive sand showed expansion that exceeded the 0.03% limit. This observation in a way supports the CPT behaviour of this sand blend in Figure 3.3, in that the rate and nature of expansion is seen to exhibit a change in positive slope at age 11 months suggesting that the 0.03% CPT limit will be extended when the test duration is extended. On the other hand, specimens containing slowly reactive aggregate showed expansion that is similar to 3 months ACPT and did not exceed the limit. This suggests that the aggregate blend is potentially non-reactive towards ASR.



Figure 3.4: 3-month and 5-month ACPT expansion data for aggregate blends at varying alkali content.

The rate of expansion with age of the fine aggregate blends evaluated in this study was also considered at the maximum alkali content ( $5.25 \text{ kg/m}^3 \text{ Na}_2\text{O}_e$ ) under ACPT testing. This data is reflected in Figure 3.5. In general, an increase in the rate of expansion was observed with age for both fine aggregate blends assessed. However, the rates of expansion differed significantly between both blends. For Mix 2c, the steady increase in reactivity with age was attributed to the slowly reactive nature of the aggregate NZ-B<sub>f</sub>. In comparison, a steep increase in the rate of expansion was observed for Mix 2d with distinct changes noted in the slope at both 3 and 7 months.

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Figure 3.5: ACPT expansion behaviour of aggregate blends at 60°C and 5.25 kg/m<sup>3</sup> alkali content

From the CPT and ACPT studies that have been undertaken, the trends observed in expansion for changes in the alkali content and age appear to be similar in behaviour with the exception that ACPT data shows significantly higher expansion. Elevating the temperature in the ACPT method to 60°C is the most probable cause for the observed increase in expansion. However, a good correlation can still be established between the expansion data obtained from ACPT studies at 3 months and the expansion data obtained from CPT studies at 12 months. When ACPT studies are extended as recommended by Lindgard et al [10], the results obtained are collaborative to field performance, particularly for slowly reactive aggregates.

## 4. CONCLUSIONS

In this investigation, AMBT, CPT, ACPT and petrographic examination have been utilised as screening tools in establishing the alkali thresholds that apply in the safe use of potentially reactive aggregates in concrete. The alkali thresholds determined for the fine aggregate blends used in this investigation have been derived following the guidelines specified under RILEM AAR-3.2 and AAR-7.1.

From the results obtained, the following conclusions can be drawn:

- AMBT is limited in its ability in screening the effectiveness of fine aggregate blends in reducing the potential to expansion by ASR.
- Although the fine aggregate blends assessed have shown expansion below the CPT expansion limit of 0.03%, an increase in expansion with increasing alkali content, age and elevated temperature is evident. As age is, therefore, a critical factor in determining the reactivity potential of aggregates including those that are non-reactive after standard testing periods, it is recommended that CPT should be extended up to 2 years or more if the reaction rate is still observed to increase. This will allow for investigations to be undertaken in the long-term to establish changes in the reactivity potential of aggregates. Subsequent petrographic examination on CPT mixes is also encouraged to ascertain the presence of ASR.
- The maximum alkali content that will not induce ASR is dependent on the reactivity level of the aggregate used in the concrete. It can, therefore, be concluded that the potential exists for the revision of the current alkali limits set for concretes; upon a comprehensive study including additional aggregates, to accommodate the safe use of potentially reactive aggregates.
- A good correlation in expansion data between CPT and ACPT exists. The reactivity status of the aggregates determined from CPT expansion data at 12 months corroborates to ACPT expansion data at 3 months and 5 months for slowly reactive aggregates.

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