

# Investigating the Alkali Threshold of Potentially Reactive Aggregates for Use in ASR Risk-Free Concretes

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## Abstract

The amount of alkali in concrete plays a pivotal role in the formation of alkali-silica reaction (ASR). Typically, concrete containing high amounts of alkali can show deleterious signs of ASR. For this reason recommendations have been imposed on the allowable alkali limits permitted in cement and concrete to mitigate the potential for ASR.

This study investigates the alkali threshold of different types of aggregates using modified RILEM protocols AAR-3.2 and AAR-7.1 to include alkali contents up to 5.25 kg/m<sup>3</sup>. Subsequently, combinations of aggregates have been studied to determine the effect of aggregate blends on ASR potential. Expansion tests at varying alkali levels have also been carried out using the accelerated mortar bar test method at 80°C and concrete prism test method at 38°C and at 60°C for both mortars and concretes containing these aggregates.

The expansion data for mortar bars containing reactive natural fine aggregate showed the greatest expansion at low alkali concentration of 0.6M NaOH at 21 days whereas a 14% decrease was observed for these aggregates at 21 days in 1.0M NaOH solution. Concrete prisms containing this reactive fine aggregate did not show expansion exceeding the standard expansion limit of 0.03% at 12 months (AS 1141.60.2) for both CPT at 38°C and CPT and 60°C, for all alkali contents tested including the alkali content of 5.25 kg/m<sup>3</sup>.

The results obtained in this study will be discussed in terms of the implications for concrete alkali limits specified by both Australian and New Zealand jurisdictions.

**Keyword:** alkali silica reaction, concrete prism tests, alkali limits, New Zealand aggregates.

## 1.0 Introduction

Alkali silica reaction (ASR) occurs between aggregates containing metastable reactive silica and hydroxyl ions found in concrete pore solution. This results in the formation of an alkali-silicate gel complex. The gel formed is semi-permeable and thus possesses the capacity to absorb moisture and cause deleterious expansion that leads to the deterioration of the mechanical properties of the concrete (Hanson 1944; Stanton 1940).

ASR begins with the dissolution of silica in the presence of high alkali content (Broekmans 1999). Most of the alkalis available for reaction in concretes are known to be supplied by the Portland cement used in the concrete mix. At elevated pH such as attained in concrete pore solution, Si(OH)<sub>4</sub> dissolves producing Si-O<sup>-</sup> species which are then balanced by Na<sup>+</sup> and K<sup>+</sup> cations in solution to form alkali silicates. Alkali silicates then further polymerize when the pore solution is saturated with dissolved silica to form ASR gel (Broekmans 1999; Walther & Helgeson 1977). Ca<sup>2+</sup> cations react to substitute alkali ions in ASR gel forming a calcium-alkali-silica hydrate complex (Walther & Helgeson 1977) such that a lower Ca<sup>2+</sup> concentration in the gel results in subsequent expansion (Powers & Steinour 1955). The process of releasing alkali ions allows the regeneration of a high pH that in turn results in further dissolution of the silica network in the aggregate and the continuity of ASR.

In an effort to reduce the risk of ASR in concrete, aggregates that demonstrate the potential to be reactive are often avoided by industry. However, the preferred use of non-reactive aggregates is impractical and uneconomical as these aggregates may not be available locally, for use in concrete. Due to the rejection of so called potentially reactive aggregates, a depletion in the acceptable aggregate sources has become an increasing concern for the concrete industry in Australia and New Zealand. Additional guidelines have also been imposed on the allowable total alkali in concrete, to reduce the amount of alkali made available to react with meta-stable silica in concretes. These limits are conservative and have been generalised for all aggregates as 2.8 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> in Australia and 2.5 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> in New Zealand (CCANZ 2012). To achieve set alkali limits in concrete, low alkali cements have to be employed in concrete production. Low alkali cements are cements whose alkali content would typically fall below an alkali limit of 0.6% Na<sub>2</sub>O<sub>e</sub> (Standards New Zealand 2009; Thomas et al. 2006). Achieving low alkali contents in cement manufacture present both environmental and economic challenges to industry. There are however instances reported in literature where concretes containing low alkali cement have also been found to experience ASR attack (Bérubé et al. 2000). This is due to the fact that the alkali content that will incite ASR is largely dependent on the mineralogical composition of the aggregate used in the concrete (Thomas et al. 2006). It is therefore important to

identify distinct alkali limits: a threshold, for both individual aggregate and aggregate combinations, to ascertain their reactivity potential in the presence of alkali.

The aim of this study therefore investigates the reactivity potential of different aggregate types and combinations using standard laboratory expansion test methods to determine the behaviour of these aggregates with variations in alkali content. The purpose of introducing a variable alkali content in this study is to establish the alkali threshold: the alkali content at which initiation of expansion due to ASR becomes evident (Hester, McNally & Richardson 2005) for particular commercially available aggregates and aggregate combinations in comparison to current fixed limits of 2.8 kg/m<sup>3</sup> and 2.5 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> set out in Australian and New Zealand guidelines, respectively (CCANZ 2012; Standards Australia 2016). Consequently, the study hopes to recommend the potential of using cements with alkali contents exceeding the 0.6% Na<sub>2</sub>O<sub>e</sub> cement alkali limit, thus conserving natural resources and reducing cement manufacture cost all of which will be beneficial to industry.

## 2.0 Experimental Procedure

### 2.1 Materials

Table 1 describes the aggregates used in this study. A type GP cement with alkali content of 0.58% Na<sub>2</sub>O<sub>e</sub>, conforming to the current alkali content of cements in New Zealand and Australia was selected as the binder.

**Table 1. Selected aggregates under investigation**

Aggregate	Symbol	Reactive Minerals	Reactivity Classification**
New Zealand Aggregate A (natural sand)	NZ-A	Glassy volcanic clasts, Quartz, cherts and feldspar	Reactive (fine aggregate)
New Zealand Aggregate B (manufactured aggregate)	NZ-B <sub>f</sub>	Hornblende, pyroxene, feldspar, quartz and epidote	Slowly reactive (fine aggregate)
	NZ-B <sub>c</sub>		Slowly reactive (coarse aggregate)
Peats Ridge (manufactured aggregate)	PR <sub>c</sub>	Not Applicable	Non-reactive (coarse aggregate)
	PR <sub>f</sub>	Not Applicable	Non-reactive (fine aggregate)
Maroota sand (natural sand)	M	Not Applicable	Non-reactive (fine aggregate)

*c = Coarse; f = Fine \*\*NZ aggregates by petrographic analysis to AS 1141.65; NZ-A by in-situ behaviour; Peats Ridge by accelerated mortar and concrete expansion tests; Maroota sand by accelerated concrete expansion tests.*

### 2.2 Methods

Throughout this study, accelerated mortar bar test (AMBT) and concrete prism test (CPT) in accordance to AS1141.60.1 and AS1141.60.2, respectively, were carried out for determining the reactivity status of the aggregates and their combinations. CPT tests have been carried out at both 38°C and 60°C.

#### 2.2.1 Accelerated Mortar Bar Test

The AMBT test method is primarily intended for the screening of aggregates and aggregate blends for ASR reactivity potential (Thomas et al. 2006). In this study, three alkali environments of 0.6M, 0.8M and 1.0M NaOH were selected to determine the aggregates' sensitivity to alkali concentration. Expansion results were compared for all specimens at ages of 10 and 21 days in accordance with AS 1141.60.1; however, the expansion of the aggregates and aggregate combinations at the end of the 56 days has also been studied to determine the reactivity status of the aggregate changing with age. The AS 1141.60.1 expansion limits of 0.1% (0.15% for natural sands) at 10 days and 0.3% at 21 days (Standards Australia 2014) were applied for all specimens including specimens tested at the modified AMBT conditions of 0.6 and 0.8 M NaOH. Upon the completion of the expansion tests, petrographic assessments were carried out to identify the presence of ASR gel as evidence of ASR occurring. Petrographic assessment was also carried out to determine the extent of any damage observed from the presence of expansion.

#### 2.2.2 Concrete Prism Tests

Expansion tests on CPT specimens for alkali contents exceeding the current alkali limits of 2.5 and 2.8 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> (New Zealand and Australian limits, respectively) were carried out for a test duration of 12 months. This investigation was carried out to primarily establish the alkali threshold of aggregate combinations. The alkali thresholds were determined in accordance to the RILEM AAR-3.2 method (Nixon & Sims 2016b). In addition to the 5.25 kg/m<sup>3</sup> alkali content specified by AS 1141.60.2 for determining potential aggregate reactivity, alkali contents of 2.5,

3.0, 3.5 and 4.0 kg/m<sup>3</sup> were used in this study to determine the alkali threshold. Alkali contents were achieved by adding high purity NaOH to the concrete mix water.

CPT carried out at 60°C (ACPT) was also used in this study. Typically in the ACPT method, results on ASR assessment of aggregates are obtained at after 20 weeks of testing (Nixon & Sims 2016c). For this study, the aggregate combinations assessed by this method have been studied for an extended test period of 8 months, to further capture any change in reactivity status resulting with time. ACPT follows a similar procedure to the standard CPT carried out at 38°C. A review of available test data show that applying the CPT at 38°C expansion limit at 3 months of ACPT generally gives the same indication of an aggregate's reactivity as that obtained at 12 months of CPT at 38°C (Fournier et al. 2004; Thomas et al. 2006). Consequently, ACPT was also carried out at the five alkali contents chosen in the CPT study at 38°C.

### 2.2.3 Aggregate Combinations

Concrete and mortar mixes consisting of combinations of the aggregates under investigation have been tested by the designated test methods. Table 2 describes the mix composition and test program that have been adopted. In CPT, coarse and fine aggregates are tested together in a standard mix combination. Generally, one component of the aggregate mix, coarse or fine aggregate, has known reactivity (usually determined by accelerated tests) such that at any point in time, the reactivity status of only one aggregate (or aggregate combination) can be determined. However, certain variations of the CPT test method described by RILEM AAR-3 suggest that two aggregates (or aggregate combinations) of unknown reactivity can also be tested to determine the potential reactivity of aggregate combinations and mix designs proposed for use (Nixon & Sims 2016b).

In this study, fine aggregate combinations (sand blends) that were identified as reactive in AMBT were selected for further assessment using CPT. Non-reactive coarse aggregate sourced from Australia and coarse NZ-B aggregate have been used to determine the long term alkali reactivity potential of the sand blends in concrete. A sand blend of 60 reactive sand NZ-A: 40 non-reactive/slowly reactive sand (by weight) was used for the mortar and concrete aggregate mixes prepared in this study, to determine the alkali reactivity potential of the proposed aggregate combination. Sand NZ-A is used in blends of approximately this proportion by New Zealand industry. Natural sands were used in the grading 'as supplied', and manufactured aggregates were used in standard test grading. Mix 2c represents an aggregate combination used by New Zealand industry.

**Table 2. Laboratory Test Program**

Standardised Tests	sample ID	Aggregate/ Aggregate Combination
<b>AS1141.60.1 at 80°C</b> 3 alkali solutions of 0.6, 0.8 and 1.0 M NaOH	1a	NZ-A
	1b	NZ-B <sub>f</sub>
	1c	NZ-A + NZ-B
	1d	NZ-A + PR <sub>f</sub>
<b>AS1141.60.2 at 38 and 60°C</b> 5 alkali contents of 2.5, 3.0, 3.5, 4.0 and 5.25 kg/m <sup>3</sup> Na <sub>2</sub> O <sub>e</sub>	2c	NZ-A +NZ-B <sub>f</sub> + NZ-B <sub>c</sub>
	2d	NZ-A + M +PR <sub>c</sub>

## 3.0 Results and Discussion

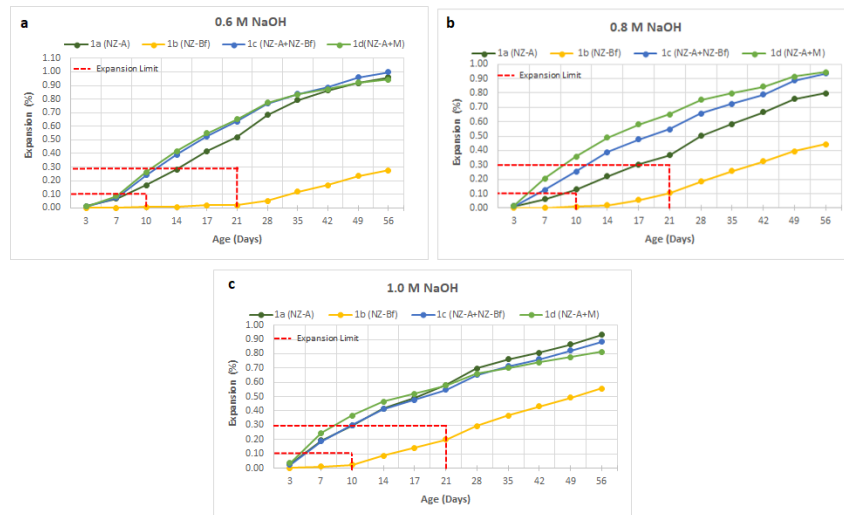
### 3.1 AMBT Expansion Data

The expansion data for mortar bars as a function of age up to 56 days for the 3 storage solutions are shown in Figure 1. By applying the standard AMBT expansion limit of 0.1% (0.15% for natural sand) at 10 days and 0.3% at 21 days, all sand NZ-A combinations 1a, 1c, and 1d showed expansion exceeding both limits at the 3 alkali concentrations assessed, thus classifying sand NZ-A as potentially reactive. The mix specimen from manufactured sand NZ-B (1b), stored in 1.0 M NaOH, solution showed an expansion below 0.1% at 10 days but an expansion of 0.20% at 21 days was observed. In accordance to the classification index described in AS1141.60.1, this behaviour is indicative of a slowly reactive aggregate.

Generally, at an age of 10 days, a consistent increase in expansion with increasing alkali concentration was observed for all specimens. However, mortar bars composed of reactive aggregate NZ-A (mix 1a) showed a lower expansion of 0.13% in 0.8M NaOH whereas a 17% increase in expansion was noted for these specimens in 1.0M NaOH. A similar trend in expansion was also observed at 21 days. Of the 3 alkali concentrations assessed, the data for mortar bars containing reactive aggregate (mixes 1c and

1d) showed the greatest expansion at lowest alkali concentration of 0.6M NaOH at 21 days. Approximately 14% decrease in expansion was observed for these aggregates at 21 days in 1.0M NaOH solution

As the AMBT test was extended to 56 days, a linear expansion was seen for all aggregates including the slowly reactive aggregate NZ-B<sub>r</sub> (mix 1b). At the end of 56 days, the data from the graph in Figure 1 suggests that with the exception of the slowly reactive aggregate, the mortar bars appeared to have expanded more in 0.6M NaOH solution with the highest expansion recorded at 0.96% for mortar bars containing the slowly reactive aggregate combination.



**Figure 1. AMBT expansion results in (a) 0.6M, (b) 0.8M and (c) 1.0M NaOH storage solution for tested aggregates.**

### 3.2 Petrographic Assessment of AMBT Specimens

Petrographic assessment was conducted on AMBT specimens to ascertain whether ASR had occurred and whether other mechanisms may have contributed to the observed expansions observed. Assessments on thinly sliced representative samples of mortar bars identified as reactive showed the presence of hydrated gel containing scattered regions of calcium hydroxide. Remnants of isotropic ASR gel that was presumably removed during the preparation of the thin slices for petrographic examination was also observed in the micro-fracture space of the hardened cement. Cracks filled with ASR gel appear to emanate around and through mineralogical components in the aggregates. These components were identified as glassy shards, acidic volcanics and quartz grains: components with perceived potential for ASR. Additionally, the ASR affected grains appeared to be porous in nature as a result of silica depletion. Although further microscopic studies and phase analyses are recommended, these observations are typically indicative of expressions of alkali silica reactivity.

### 3.3 Concrete prism tests

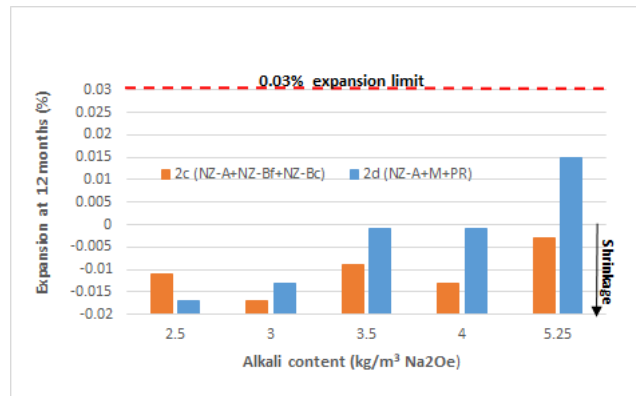
At present, the criteria for assessing potential aggregate reactivity from CPT results vary between jurisdictions, as described in Table 3. For this study, the classification limits set out in AS1141.60.2 were adopted.

**Table 3. Assessment of aggregate reactivity based on current CPT standard methods**

Interpretation	1 year Expansion Limit (%)		
	AS1141.60.2	ASTM C1293	RILEM AAR-3
Non-reactive	< 0.03	< 0.04	< 0.05
Reactive/Potentially Reactive	≥ 0.03	≥ 0.04	≥ 0.05

#### 3.3.1 Concrete prism test at 38°C

Expansion measurements for concrete prisms containing varying alkali content were taken at monthly intervals after 7 and 28 days until 12 months test duration. The expansion results for the tested sand blends at varying alkali content are shown in Figure 2.

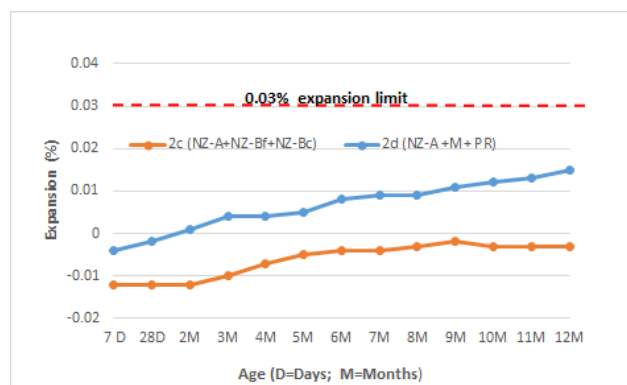


**Figure 2. 12 months CPT expansion data for aggregate blends at varying alkali content.**

Figure 2 shows that the specimens exhibited initial shrinkage after demoulding before starting to expand. As the alkali content was increased to 5.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>, expansion of prisms containing sand NZ-A and non-reactive control aggregates M and PR<sub>c</sub> (2d) was observed to increase. However, the 0.03% expansion limit is not reached by either aggregate combination, therefore, the sand blends can be classified as non-reactive. Consequently, this suggests that these blends may perform well at an alkali content higher than the current stipulated limit of 2.5 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> in New Zealand. Overall, the prisms containing sand NZ-A and NZ-B coarse and fine aggregate (2c) showed no net expansion at the alkali contents assessed. At 12 months, no net expansion was seen for the aggregate combinations containing up to and including 4.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> alkali content. Although leaching of alkali from the concrete specimen during the test may cause expansions lower than expected (Thomas et al. 2006), an initial net expansion is observed for sand NZ-A with non-reactive aggregate (2d) at 5.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>.

The RILEM AAR-3.2 method, similar to the AS 1141.60.2 test method carried out in this study, proposes an effective method for determining the alkali threshold of an aggregate or aggregate combination. Used in conjunction with the RILEM AAR-7.1 method, a safety margin (usually - 1.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>) can be applied to the alkali threshold of the aggregate or aggregate combination to establish a safe amount or range of alkali content (alkali limit) at which the aggregates can be used (Nixon & Sims 2016a). As previously discussed, the results obtained in this study showed no expansion for the sand blends assessed at alkali contents below 5.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>. Whereas further testing is recommended, this suggests that the current alkali limit may yet be revised and/or customised for the current aggregate combination assessed, as 4.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>. A similar approach can thus be adopted to determine the alkali thresholds and limits for other aggregates or aggregate combinations intended for use in concrete.

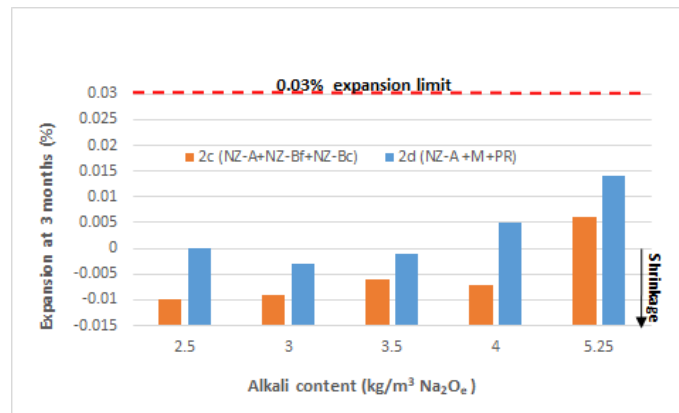
The effect of age on the expansion behaviour of the aggregate combination has also been considered for the highest alkali content studied. This expansion data is shown in Figure 3. At a constant alkali content of 5.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>, expansions associated with both sand blends were seen to increase linearly with age although the expansion limit of 0.03% has not been exceeded. These results suggest that further testing and monitoring is beneficial to establish the rate and degree of expansion of the fine aggregate combinations as a function of age. In accordance to the RILEM recommendation AAR-3.2, the shape of the expansion curve at 12 months should be considered in addition to the magnitude of the 12-month expansion. Such that, if the specimen expansion is continuous but below the expansion limit of 0.03% at 12 months, the test period should be extended.



**Figure 3. CPT expansion data of aggregate blends at 38°C and 5.25 kg/m<sup>3</sup> alkali content**

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

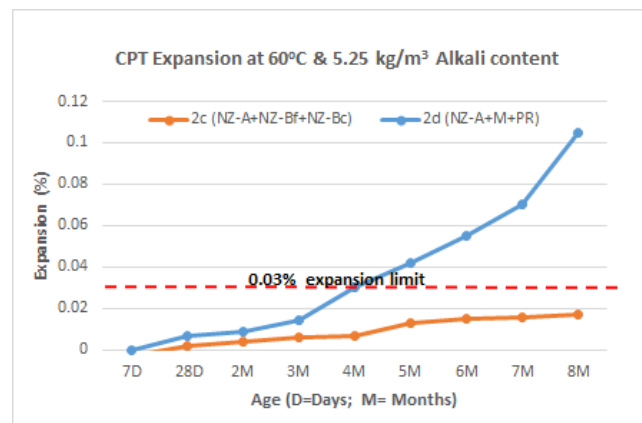
Reactivity classification for concrete prisms assessed under ACPT conditions were interpreted using the CPT limit of 0.03% applied at the 3 months (Thomas et al. 2006). Due to high alkali mass leaching and an increased sulphate content in pore solution is observed for ACPT, the main expansion phase of the test is often completed after 3 months of testing (Folliard et al. 2004). The expansion data of the ACPT study is shown in Figure 4.



**Figure 4. 3 months ACPT (CPT at 60°C) expansion data for aggregate blends at varying alkali content.**

From Figure 4, the sand blends assessed were found to exhibit expansion below the 0.03% limit. Therefore, in accordance with the classification index, both aggregate combinations can again be classified as non-reactive. It is interesting to note that the prisms containing slowly reactive aggregate (mix 2c), exhibited net shrinkage rather than expansion at less than 5.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>. Of the two reactive sand blends assessed, the aggregate combination with non-reactive aggregate was found to expand more. A similar observation was noted for this aggregate combination for CPT undertaken at 38°C at 12 months.

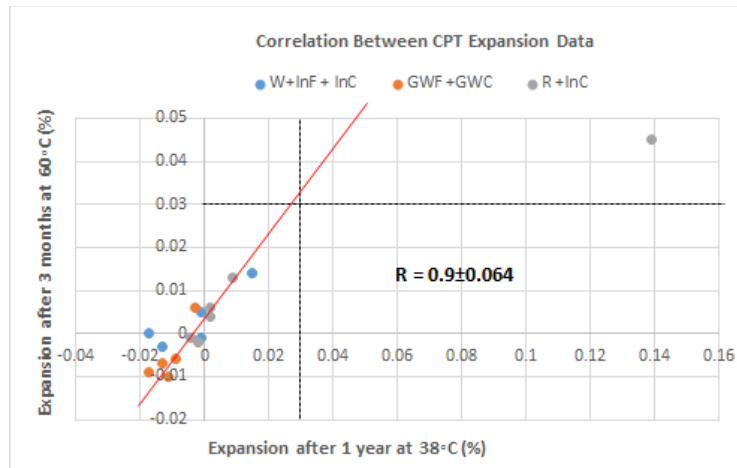
When the expansion of the aggregate combinations are monitored at constant alkali content of 5.25 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub> over time (Figure 5), an increase in expansion is observed. For the aggregate combination used in mix 2c, the steady increase in reactivity with age can be attributed to the slowly reactive nature of aggregate NZ-B.



**Figure 5. CPT expansion of aggregate blends at 60°C and 5.25 kg/m<sup>3</sup> alkali content.**

In contrast, a steep increase in expansion behaviour is observed for the aggregate combination with non-reactive aggregate (mix 2d) at 3 and 7 months. A relatively similar behaviour is observed at 5 and 8 months for CPT undertaken at 38°C. This suggests that this aggregate combination may exhibit a tendency to cause deleterious ASR over time when used in the specified combination. The expansion data observed in CPT at 60°C (ACPT) is similar in trend to the expansion data seen at 38°C with the exception that ACPT data is generally showing significantly higher expansion due to the higher temperature condition. However, a positive correlation can be made from the expansion data between aggregates' classification for ACPT at 3 months and CPT at 38°C at 12 months. This is evident by the strong uphill correlation coefficient (R) of 0.9±0.064 (Figure 6), calculated based on Pearson correlation formula. Thus,

indicating a near exact positive linear relationship between CPT at 38°C and the accelerated CPT at 60°C.



**Figure 6. Comparison of expansions in accelerated (CPT at 60°C) vs. standard concrete prism tests (CPT at 38°C) for New Zealand aggregates under investigation**

#### 4.0 Conclusions

In this study, accelerated mortar bar test at 80°C and concrete prism tests carried out at 38°C and 60°C have been used to investigate the effect of alkali concentration on the alkali reactivity of aggregates and aggregate blends, to determine the alkali threshold and limit at which these aggregates and aggregate combinations can be used safely in concrete. The alkali threshold and limit of the aggregate combinations was determined using RILEM AAR-3.2 and 7.1 protocols modified by using AS 1141.60 test methods.

From the results obtained the following conclusions can be drawn:

- A correlation between CPT at 38°C and at 60°C (ACPT) can be made. Reactivity status of the aggregates determined at 38°C for CPT at 12 months is corroborative to ACPT data at 3 months.
- Whereas the aggregates assessed have shown expansion below the CPT limit of 0.03%, an increase in expansion at constant alkali content with increasing age is evident for aggregate combinations assessed by both AMBT and CPT methods. Therefore, age is a key factor in determining the reactivity behaviour of aggregates and aggregate combinations including those that appear to be non-reactive after standard testing periods. A recommendation can thus be made for the standard CPT test method to be extended up to or exceeding 2 years to investigate the long term reactivity behaviour of aggregates to determine their safe use in concretes. Subsequent petrographic assessment on concrete prisms are also encouraged to ascertain the presence of ASR.
- The maximum alkali content that will not incite 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 to accommodate the safe use of potentially reactive aggregates and cements with increased alkali contents in concrete production. The current alkali limit in New Zealand is conservative and may be reviewed upon a comprehensive study that will also include aggregates sourced from other locations in New Zealand. This recommendation can be further applied to the current alkali limit in Australia.

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