

Correlation between Existing Test Methods for Assessing Alkali-Silica Reaction of Aggregates

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Abstract: The most recognised test methods for assessing alkali-silica reaction (ASR) can be categorised as petrography, chemical and expansion tests conducted on mortar bars (AMBT) at 80°C and concrete prisms (CPT) at 38 and 60°C. Individually, these test methods are not considered ideal owing to excessive alkali supply, alkali leaching and the apparent decrease in the accuracy of the results with decreasing test duration. Nonetheless, CPT carried out at 38°C for up to 12 months is considered to give the most reliable indication of an aggregate's reactivity. However, the relatively long test period for CPT of 12-24 months is still an issue for industry, where early results are desired. This work evaluates the correlation between aggregate reactivity predictions based on the different test methods. Furthermore, the relationship between the expansion test methods is studied using Pearson's correlation method to ascertain the appropriate test methods that are most comparable to CPT at 38°C, thus providing the most suitable alternative short-term test method for assessing ASR. Likewise, the influence of test parameters on expansion and alkali leaching during CPT, and consequently, the correlation between CPT and ACPT is investigated using a statistical approach. The findings show a consistency in the reactivity prediction by the different methods. A strong Pearson's coefficient (r) was found between CPT at 38°C and chemical tests ($r=0.85$), ACPT ($r=0.90$) and AMBT ($r=0.67$). The results also demonstrated that alkali and temperature have a strong influence on expansion test methods.

Keywords: alkali-silica reaction, Pearson's correlation, ASR test methods, alkali leaching, AMBT, CPT, ACPT

1. Introduction

Eighty years after its discovery [1], ASR is still considered one of the most detrimental reactions that can occur in concrete that impacts the durability of concrete structures in service [2]. ASR is described as the chemical reaction that occurs between reactive silica from aggregates and alkalis from cement in the pore solution of concrete. The result of ASR is a semi-permeable hygroscopic gel (ASR gel) that expands upon water absorption to cause internal tensile stresses. This leads to cracking in concrete and the eventual premature failure of structures [1]. The most recognised approach to mitigate ASR is the addition of supplementary cementitious materials (SCMs) such as fly ash, ground granulated blast furnace slag and silica fume [3]. However, it can be agreed that the first line of defence against ASR is assessing the reactivity of the aggregates used in the concrete.

A number of test methods exist for identifying the reactivity potential of aggregates and evaluating ASR prevention methods [4]. Usually, field performance and exposure tests are considered the most reliable methods. However, these techniques require long periods (several years) of testing and thus are less favoured in industry where the early indication of aggregates' reactivity is desired for decision making. As such, consensus has been reached on acceptable laboratory test methods for assessing ASR potential and preventive measures. These methods have been standardised and can be categorised as:

- characterisation tests, to identify potentially reactive minerals and free silica content in aggregates;
- chemical tests, to assess the amount and reactivity rate of soluble silica in aggregates, and;
- physical expansion test methods carried out on aggregate-cement combinations intended for use in field structures to evaluate ASR performance or a specific preventive measure employed.

Table 1 presents a list of the common standard test methods that are employed for assessing the ASR potential of aggregates. Individually, these test methods may provide a good indication of the reactivity

potential of the aggregates. However, none satisfy the requirements for an 'ideal' test method as the tests become more conservative with shortened time. Thus, their reliability typically reduces with the severity of the accelerated test duration [5]. Additionally, over the years, there have been emerging concerns and debates surrounding the use of some of these test methods, such as ASTM C 289 and AS 1141.65, which has led to their withdrawal as standardised techniques; even though they are still employed in current research as quick indicators of ASR reactivity [6].

Table 1. Standard laboratory test methods

Category	Test Methods	Standard Designation
Characterization Test	Petrography	ASTM C 295; AS 1141.65; AAR-1
Chemical Test	Chemical Reactivity Test	ASTM C 289
Expansion Tests	Accelerated Mortar Bar Test (AMBT)	ASTM C1260; AS 1141.60.1; CSA A23.2-25A; AAR-2
	Concrete Prism Test (CPT)	ASTM C1293; AS 1141.60.2; CSA A23.1-14A; AAR-3

ASTM (American); AS (Australian) AAR (European: RILEM); CSA (Canadian)

Conversely, the curing conditions of high humidity (>95%) and temperature (38°C) for CPT generates expansion results that are deemed most reliable and comparable to the field performance of aggregates [4, 7]. However, the relatively long test duration of 12 months for evaluating aggregate reactivity and 24 months for assessment of preventive measures poses an inconvenience for concrete producers. Therefore, a modified version of the CPT method carried out at 60°C; accelerated concrete prism test (ACPT) has been adopted as a practical alternative test method to the standard CPT. Owing to the higher curing temperature of ACPT, expansion results can be obtained in a shorter period, usually, after 4 months for aggregate reactivity testing [8] or up to 6 months to evaluate ASR preventive measures [9]. Currently, there is no specific acceptance limit designated for ACPT, as the test method is yet to be accepted as a standardised test method. Nonetheless, ACPT results may correlate with standard CPT [4]. Importing the 0.03% expansion limit of standard CPT after 3 months of ACPT has been reported to provide the same indication of an aggregate's reactivity potential as that obtained after 12 months of CPT [10]. However, other studies suggest that the CPT expansion limit after 5 months of ACPT provides a more reliable assessment of the ASR potential of the aggregate [11]. Evidently, the specific expansion correlation age for ACPT to 12 months CPT is unclear.

Although considered the most reliable test method for ASR assessment, one of the major challenges of CPT is the alkali leaching that occurs during the curing of prism samples. Concrete prisms for CPT are usually cured above a reservoir of water in sealed plastic pails. At high humidity and temperature, condensation of water vapour occurs on the surface of the prisms. This causes a localised reduction of alkali concentration in the surface pore solution of the prisms that induces the transport of alkalis from the inside of the prism towards the surface. As a result, the condensed water enriched with alkali travels down the prisms' surface into the reservoir present at the bottom of the storage pail. Alkali leaching potentially influences the expansion results obtained during CPT. For ACPT, increased alkali leaching is expected due to the higher curing temperature employed [10].

This study aims to investigate the correlation between the three categories of test methods (Table 1) to establish their reliability in predicting the ASR potential of aggregates. Certainly, it is noted that each of these test methods measures a different property, such as silica content and expansion, as an indication of reactivity (or ASR), and the reliability of the various test may vary with aggregates due to differences in their mineralogical composition. Still, it is expected that the results obtained from the individual tests provide some corroborative information on the potential reactivity of aggregates, thus the purpose of this study. Additionally, the relationship between the various expansion test methods, particularly CPT and ACPT, will be explored to establish the correlation between these methods. To achieve this, the extent of alkali leaching in both CPT and ACPT will be considered. Furthermore, the influence of alkali content and temperature, as test parameters, on the validity of CPT will also be investigated. This will provide industry with an alternative short-term test that gives results that are comparable to the preferred long-term test.

2. Materials and Methods

This study forms part of a wider collaborative project between UTS and Concrete NZ. A detailed description of the experimental work has been discussed elsewhere [12, 13], and in an unpublished dissertation.

2.1 Materials

Four aggregates, identified in Table 2, were selected to prepare 10 mortar and 26 concrete mixes investigated in this study. The aggregates used were submitted by industry partners to represent common reactive aggregates in New Zealand. Type GP Portland cement with alkali content of 0.58% Na₂O_e was used in the preparation of mortar and concrete specimens.

Table 2 Selected aggregates used in this study

Aggregate	Symbol	Aggregate Type
Waikato	WT	Natural river sand
Greywacke	GW	Manufactured fine and coarse aggregate
Rangitikei	RT	Natural river sand
Andesite	AS	Manufactured fine aggregate

2.2 Methods

2.2.1 Laboratory Tests

Petrography, AMBT, CPT and ACPT were carried out on aggregates, mortar bars and concrete prisms in accordance with the Australian standards (AS) as described in Table 1. A summary of the test program used has also been reported by Freitag and Mackechnie [13]. Chemical dissolution tests were also carried out on ground aggregate samples as per the ASTM C289 method. The detailed description of the test procedure is reported in respective standards and in several literature sources [4, 14, 15]. The AMBT test was conducted for an extended test duration of 56 days. Three alkali contents of 2.5, 3.5 and 5.25 kg/m³ Na₂O_e were also employed during CPT and ACPT to evaluate the effect of alkali content on the expansion results obtained.

2.2.2 Alkali leaching

During CPT and ACPT, an aliquot (5±0.5 ml) of the storage water was collected at the time of expansion measurements after ages 1, 3, 6 and 12 months for CPT, and each month for ACPT until 6 months of testing. The storage water samples were filtered and diluted with 1% nitric acid. The diluted samples were then analysed using microwave plasma atomic emission spectroscopy (MP-AES) to determine the concentration of Na⁺ and K⁺ ions in the solution. Post MP-AES analysis, the amount of leached alkalis in the storage water was calculated as the total sodium equivalent (Na₂O_e) using Equation (1) [16]:

$$\text{Na}_2\text{O}_e = (\text{Na} \times 1.35) + (0.658 \times \text{K} \times 1.02) \dots \dots \dots$$

Eqn. (1)

2.2.3 Statistical Analysis

The results from the chemical and expansion tests (AMBT, CPT and ACPT) were evaluated using statistical techniques to investigate the reliability and consistency of the ASR test methods. Additionally, the expansion measurements after 12 months of CPT were assessed against ACPT expansion after ages 1 to 6 months to determine the appropriate age at which ACPT is most comparable to the standard CPT method. The correlation studies were achieved using Pearson's correlation coefficient method in the data analysis pack of OriginPro8 software.

3. Results and Discussion

3.1 Reliability of Existing Test Methods

Table 2 presents the prediction on the reactivity of the aggregates as determined by the various test methods. From Table 3, it can be observed that generally, a consistent outcome of reactivity classification of the aggregates was obtained.

Table 2 Comparison of reactivity classification by various test methods

Aggregates	Petrography	Chemical Test ASTM C289	Expansion Tests	
			AMBT at 21 days AS1141.60.1	CPT at 12 months AS1141.60.2
WT	Substantially reactive	Considered deleterious	Reactive	Potentially reactive
RT	Mild - slowly reactive	Considered deleterious	Reactive	Potentially reactive
GW	Mild - slowly reactive	Considered Innocuous	Slowly reactive	Non-reactive
AS	Substantially reactive	Considered deleterious	Reactive	Potentially reactive

Except for aggregate RT, which was identified by petrography to contain reactive minerals in proportions that are likely to form mild to slow ASR yet exhibited significant reactivity in the chemical and expansion test methods, a correlation can be observed between the different test methods. However, it is worth noting that aggregate reactivity classification by petrography depends strongly on the expertise of the petrographer and documented field evidence of the aggregate [5], which could typically vary from one location to another (local or international). This, therefore, affects the precision and reproducibility of the petrographic technique. For the aggregates investigated in this study, the chemical test method showed results that are most comparable to CPT. Further correlation studies on the concentration of released silica from the aggregates as determined by the ASTM C289 chemical method and the CPT expansion of their respective prisms after 12 months indicated a strong correlation of $r=0.85$, as illustrated in Figure 1. The ASTM C289 uses a three-part chart to identify aggregates as innocuous, potentially deleterious or deleterious. Thus, there is no agreed limit on the amount of dissolved silica for aggregate reactivity classification, although an early research suggested a limit of 100mmol/L- above which aggregates can be considered deleterious [17]. However, it can be observed from Figure 1 that by applying the AS1141.60.2 CPT expansion limit of 0.03%, the reactivity potential of the aggregates was correctly identified by both methods.

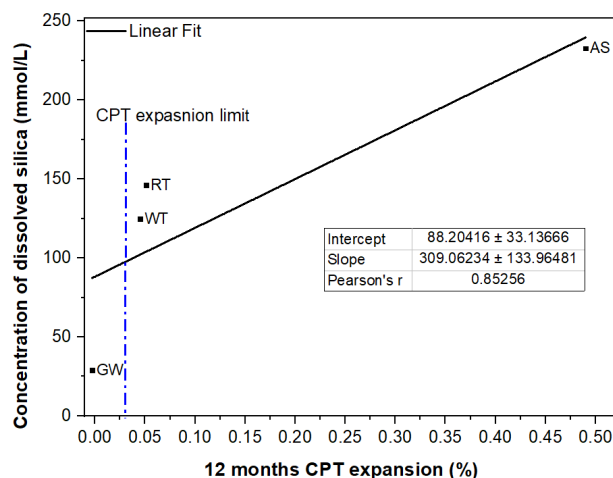


Figure 1 Relationship between ASTM C289 and 12 months AS1141.60.1 CPT expansion test

3.2 Correlation between Expansion Test Methods

3.2.1 AMBT and CPT

The AMBT method is the most widely used accelerated (rapid) screening method for assessing ASR, whereas CPT is recognised as the most reliable test procedure [4]. As such, studies are still ongoing to determine a correlation between AMBT and CPT [4, 18, 19] in the effort to refine AMBT test conditions and establish its validity for assessing ASR globally.

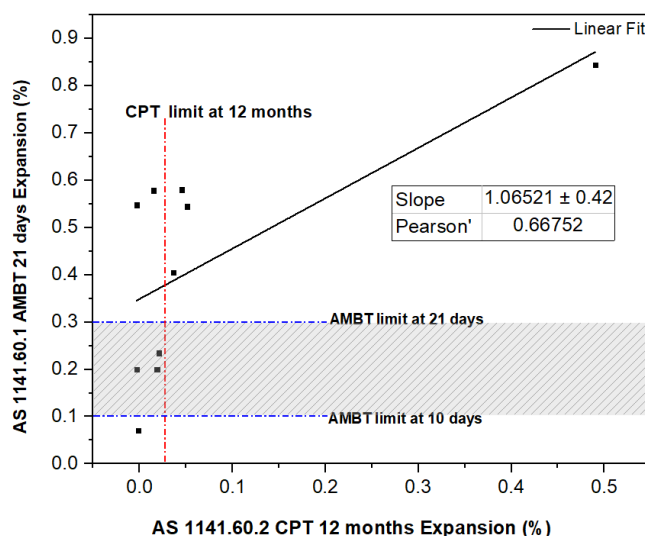


Figure 2 Correlation between AMBT and CPT test methods.

A graphical representation of the correlation between the AMBT and CPT results for the 10 mix formulations studied in this work is given in Figure 2. Concrete prisms for CPT were prepared with a non-reactive aggregate sourced from Australia. Four mixes were observed to exceed both the expansion limits for AMBT and CPT, suggesting confirmatory results for both tests and indicative of the potential reactivity of those aggregates in field structures. Likewise, one non-reactive mix was also consistently identified by both test methods. Conversely, at the 21-day limit, AMBT falsely identified 2 non-reactive aggregates as reactive, and another 3 as slowly reactive (shaded region). This outcome is attributed to the high temperature (80°C) and excess alkali (1M NaOH) test conditions employed in AMBT. Additionally, the extended 21-day test duration used in the AS 1141.60.1 AMBT method possibly increases the exposure of non-reactive aggregates containing even minimal amounts of susceptible silica to show some level of expansion in the high temperature-alkali test environment, resulting in a false positive indication of reactivity. Consequently, the outcome of this study calculates a 50% reliability for the AS 1141.60.1 AMBT test method in correctly predicting the potential reactivity of aggregates. Furthermore, a correlation coefficient of 0.67 was obtained at the stipulated expansion limits and ages suggesting a reasonable correlation between the results of both expansion test methods. In another study on the correlation between AMBT and CPT compared to the actual field performance of the respective aggregates [19], a strong correlation of $r = 0.87$ was also observed between AMBT and CPT.

3.2.2 CPT and ACPT

The statistical correlation between CPT at 38°C after 12 months and ACPT (CPT at 60°C) after ages 1 to 6 months for a total of 26 concrete mixes is presented in Table 4.

Table 4 Correlation between 12 months CPT and ACPT results at different ages

ACPT Age (Months)	False-negative results in ACPT (%)	False-positive results in ACPT (%)	Pearson's correlation Coefficient (r)
1	23	---	0.74
2	15	---	0.87
3	8	---	0.89
4	8	4	0.89
5	8	4	0.90
6	4	4	0.89

Overall, a positive correlation was calculated at all ACPT ages assessed. A similar Pearson's correlation coefficient of approximately 0.9 was found between 12 months CPT and ACPT from ages 2 to 6 months. Considering the suggested test ages of 3 months [10] and 5 months [11], the correlation coefficients are statistically equal (± 0.01) at both ages. Nonetheless, at 5 months, the percentage accuracy of ACPT is lower (88%) than at 3 months (92%) as ACPT may also provide false-positive results. Thus in this study, the results demonstrate that the best correlation between CPT and ACPT was achieved at 3 months of ACPT. This outcome is also represented in the correlation plot shown in Figure 3.

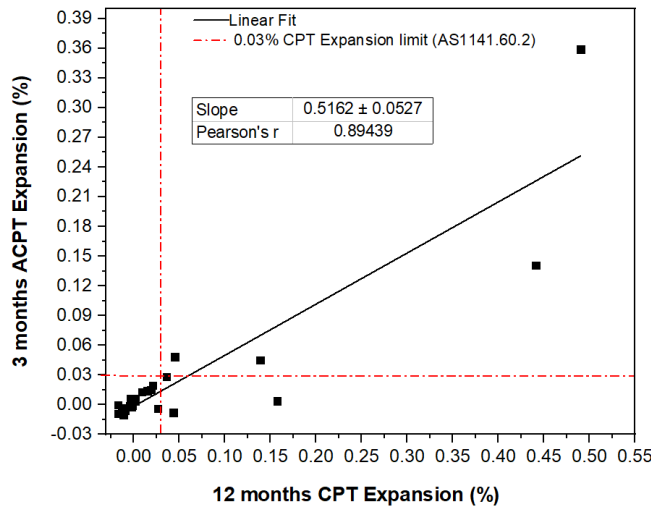


Figure 3 Correlation between 12 months CPT and 3 months ACPT expansion tests.

Figure 3 indicates a well-correlated linear relationship between CPT and ACPT at 3 months. By applying the 0.03% expansion limit for AS 1141.60.2 to ACPT at 3 months, a reliable indication of an aggregate's reactivity (expansion) at 12 months of standard CPT can be achieved. Furthermore, out of the 26 concrete mixes studied, the 2 mixes that were incorrectly identified by the ACPT method can also be observed. It has been reported that concrete prisms tested under ACPT conditions tend to exhibit lower expansions than prisms tested under CPT conditions [20]. Consequently, some studies suggest that considering that the ACPT method is an accelerated version of an already accelerated test method (CPT), an acceptance limit lower than the limit stipulated for the standard CPT test, following a series of comprehensive studies, may be appropriate when considering the ACPT method as a short-term alternative to CPT for assessing the ASR potential of aggregates [10, 21].

3.3 Effect of Alkali Leaching on Correlation between CPT and ACPT

The amount of alkali leached during CPT and ACPT as determined using MP-AES is illustrated in Figure 4. Three concrete mixes (M1-M3) were selected to study the effect of temperature on alkali leaching and its corresponding impact on the correlation between CPT and ACPT. The results shown represent the approximate leachate amount for a single prism average; that is, the measured value divided by 3 (3 prisms in a pail).

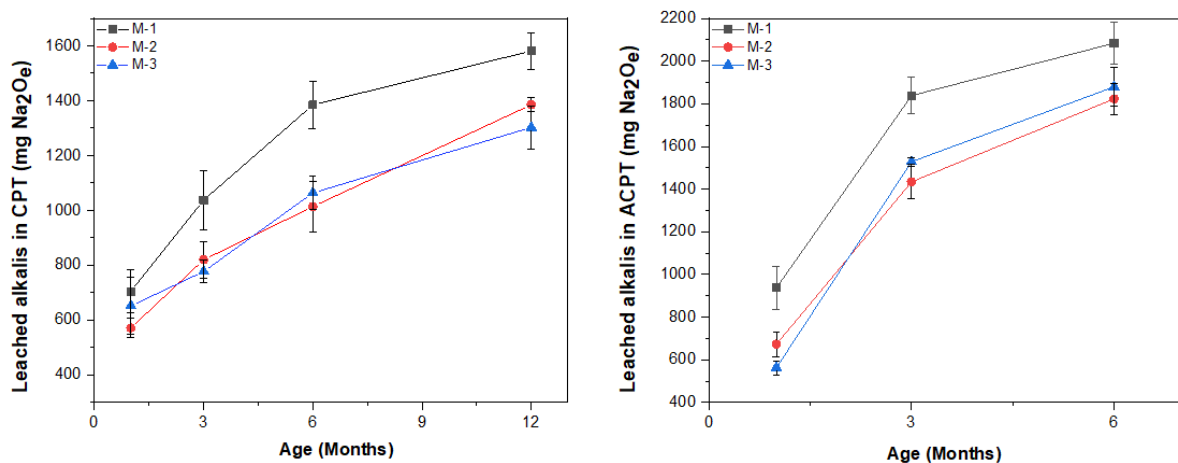


Figure 4 Alkali leaching from prisms tested under CPT (38°C) and ACPT (60°C) condition. *Note: All concrete prisms have initial alkali content of $5.25 \text{ kg/m}^3 \text{ Na}_2\text{Oe}$.*

Generally, an increase in alkali leaching was observed with increasing age and temperature for both CPT and ACPT. Nonetheless, the relative difference in the leachate amount decreased towards the end of the test period approaching 12 months and 6 months for CPT and ACPT, respectively (Table 4), even

though an increase in expansion was recorded for these prisms (unpublished work). The continued expansion observed for these concrete prisms is likely because the amount of alkali leached was not enough to reduce the overall alkali content of the concrete below the alkali threshold of the aggregates [15]. The proportion of alkalis leached, expressed as a percentage of the total alkali of $5.25 \text{ kg/m}^3 \text{ Na}_2\text{O}_e$ (alkali content in cement + NaOH in mix water) supplied, is also presented in Table 5. The values in the table represent the average of the 3 different prisms shown in Figure 4 at the corresponding ages.

Table 5 Alkali leaching (Na_2O_e) for concrete mixes during CPT and ACPT expansion tests

Assumptions	Test Method	Cumulative Percentage of leached alkali (%)			
		1 month	3 months	6 months	12 months
Total Alkali: 5.25 kg/m^3 Alkali per prism: 8.86 g	CPT	7	10	13	16
	ACPT	9	18	22	-

The percentage leached alkalis measured in this study was found to be lower than the typical 25 to 40% that has been reported in literature for the CPT method [4, 22]. This can be associated with the differences in the reactivity levels of the aggregate used in the concrete mix. Considering that a proportion of the total alkalis available in the concrete is potentially bound in hydrates and ASR reaction products, and therefore cannot be leached, it can be concluded that the reactivity of the aggregate plays a pivotal role in the amount of alkalis leached from the concrete prisms; that is, highly reactive aggregates that form more ASR products consume more alkali, reducing the amount of alkali available to be leached. This alkali leaching behaviour has also been confirmed by Bavasso et al. [23]. Ultimately, the results in Table 5 show that 18 % alkali leaching at 3 months of ACPT correlates to the 16% alkali leaching that occurs after 12 months of CPT. This further corroborates the outcome of correlation data, suggesting the potential application of ACPT at 3 months as an alternative to CPT at 12 months.

Further, the influence of temperature and alkali content on the expansion of CPT and ACPT was also investigated to determine the potential effect of these test parameters on the correlation outcome of the test methods. A plot of alkali content versus temperature for 2 selected aggregates, RT and AS, as shown in Figure 5, confirms that an increase in alkali content significantly increased the expansion of the aggregates at both 38°C and 60°C . It is also evident that an increase in temperature results in a decrease in overall expansion as a consequence of the increased alkali leaching, as discussed in Figure 4 and Table 4. This outcome is consistent with reports in literature [20].

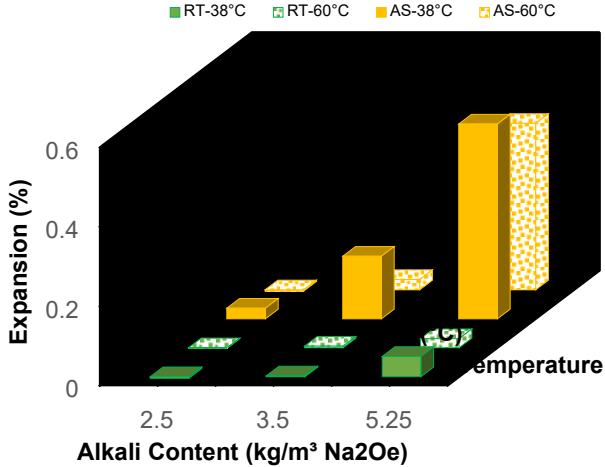


Figure 5 Effect of varying alkali and temperature on measured expansion

4. Conclusions

In this study, the correlation and interactions between the different test methods for assessing ASR, categorised as petrography, chemical tests and physical expansion methods, has been investigated using Pearson’s correlation method. Additionally, the factors that potentially affect the reliability and

correlation of the expansion test methods were also studied. Based on the 4 aggregate sources studied, and the respective test methods used in this work, the following conclusions can be drawn:

- There is a consistency in the reactivity prediction of aggregates by the different test methods. However, the reactivity classification of aggregates by petrography varies slightly from the results obtained in CPT as this technique relies on the skill of the petrographer and available information on the aggregates' field performance, which can vary with location.
- The ASTM C 289 chemical test, although withdrawn, may provide results that are comparable to CPT after 12 months, with a correlation coefficient (r) of 0.85. However, due to the inconsistency reported with this method, it is recommended for use a complementary test method.
- A positive correlation ($r= 0.67$) exists between AMBT after 21 days and CPT after 12 months. Nonetheless, AMBT produces a notable percentage of false indication on aggregates reactivity owed to the conservative test conditions. This emphasises the need to employ additional concrete testing test methods for aggregates that fail the 10-day expansion limit of 0.1% in AMBT.
- There is a strong positive correlation between CPT after 12 months and ACPT after 3 months. This suggests the potential use of ACPT as a reliable substitute for CPT where early results are required for decision making. However, further benchmarking tests are required to assess the tests efficacy for different types of aggregate sources and to establish a suitable acceptance limit for ACPT.
- Alkali leaching occurring in concrete prism tests is noteworthy. The extent of alkali leaching in concrete prism tests is intensified with increasing temperature. The percentage of alkali leaching after 3 months of ACPT is comparable (18%) to the alkali leached out after 12 months of CPT (16%), supporting the correlation of the ACPT to CPT after 3 months.
- An increase in alkali content results in higher ASR expansion. However, for higher temperature systems, a decrease in expansion was measured. This is associated with the increased alkali leaching that occurs at higher temperatures. Although alkali content and temperature affect the rate expansion, there is no interaction between these two factors. That is, a change in alkali content is not dependent on temperature and vice versa.

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