Accelerated Test for Assessing the Potential Risk of Alkali-Silica Reaction in Concrete Using an Autoclave

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Abstract

To rapidly assess the potential risk of alkali-silica reaction (ASR) in concrete, an accelerated test using an autoclave by adopting multi-cycle 80°C steam warming at atmospheric pressure is proposed. The influence of autoclave steam warming temperature, cycles/duration, and alkali dosage on expansion of mortar bars and concrete prisms was evaluated. Mechanical properties of concrete under accelerated ASR test were investigated. Furthermore, SEM-EDS analysis confirmed ASR products and indicated that the expansion is caused by ASR. The expansion limits considered for classifying aggregates were discussed. The experimental results demonstrated that the period required for assessing the potential risk of ASR in concrete (dacite aggregate in this study) can be shortened to 37 days.

Keywords: Durability, Alkali silica reaction (ASR), Expansion, Accelerated ASR test, Autoclaving

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1. Introduction

Alkali-silica reaction (ASR) is one of the most harmful long-term distresses for reinforced concrete structures as observed worldwide [1]. This chemical reaction between certain forms of reactive siliceous minerals in aggregates and alkali metal hydroxides in the pore solution, produces an alkali-silica gel that swells by imbibing water from surrounding environment. This swelling causes expansion and cracking of the concrete, leading to reduction in mechanical properties, and ultimately resulting in the lower durability and performance of the affected structure [2]. Deleterious ASR in hardened concrete occurs under the following conditions. Aggregates need to have a certain amount of reactive siliceous minerals; sufficient alkalis should exist in concrete pore solution; adequate moisture and calcium ions should be available [3]. It can take 10-15 years for ASR damage to be developed in structures [4-6]. Hence, in order to ensure that the aggregate is suitable for concrete, it is required to be assessed for its ASR potential, prior to be used in the concrete mix.

For assessing the reactivity of aggregates, currently there are four accelerated laboratory test methods available, namely, accelerated mortar bar test (AMBT), concrete prism test (CPT), accelerated concrete prism test (ACPT) and ultra-accelerated autoclave test. The potential reactivity of the aggregates can be identified within a few days if the autoclave test is used whereas the duration of CPT can be up to one year. The AMBT, such as the standard test AS 1141.60.1 [7] and ASTM C1260 [8], as well as the RILEM recommended test method RILEM AAR-2 [9], involves crushing of the aggregate to specific grading and immersion of specimens in 1M NaOH solution at 80°C. Although AMBT can provide results within 21 days by adopting AS 1141.60.1 and within 16 days if ASTM C1260 is used, both false positive and false negative results had been reported [10, 11]. By examining the accuracy of different test methods, Sirivivatnanon et al. [12] concluded that the improved AS 1141.60.1 is a relatively reliable accelerated test method in screening 'reactive' and 'slowly reactive' aggregates, while ASTM C1260 was found to be more reliable in classifying the non-reactive aggregates. With evidence from field performance, CPT test is currently recognised as the most reliable test method for identifying the reactivity of aggregates and the most reliable performance test for assessing the potential risk of ASR in concrete [12]. The long test duration of CPT, which takes one year for ordinary Portland cement (OPC) concrete mixes and two years for mixes with Supplementary Cementitious Materials (SCMs) for mitigation purpose, however, limits its application in practice. In addition, mixes used in real structures often have mix proportions different to the specific aggregate grading, cement contents and water-to-cement ratio as specified in AS 1141.60.2 [13], ASTM C1293 [14] or recommended by RILEM AAR-3 [15], and hence the CPT also has its limitations. ACPT refers to accelerating the CPT by increasing the testing temperature from 38°C to 60°C [16-19]. Among the ACPT methods, RILEM AAR-4.1 [19] recommends a test duration of at least 20 weeks. In addition, alkali leaching in ACPT is of concern [20]. The standard miniature concrete prism test AASHTO T 380-18 [21], allows classification of deleterious alkali-silica reactivity of aggregate within 8 weeks, by immersing the 50 × 50 × 285mm concrete prisms in 1M NaOH solution at 60°C. For slowly reactive aggregates, the test duration is extended to 12 weeks. In ultra-accelerated autoclave tests carried out to date, the mixes were boosted with alkalis between 1.5 to 4.0% by mass of cement and maximum temperatures used by different researchers varied between 111°C and 150°C. For mortar bars, duration of autoclaving was 2 to 6 hours, while for concrete prisms, duration of the autoclaving was between 4 to 24 hours [22-26]. However, in spite of extensive research on ASR over a period of approximately 80 years, there is still a need for a reliable and rapid test method for testing the reactivity of aggregates. Also, there is a need for a reliable performance test method that can rapidly assess the potential risk of ASR [3].

For the purpose of rapidly testing concrete mixes as used in the field and for studying reinforced concrete elements by accelerating ASR within a short period of time, an ultra-accelerated test method is needed. This paper introduces the development of a novel accelerated autoclave test method for assessing the potential risk of ASR. In the first phase, experiments are conducted on mortar specimens to accelerate ASR within a short period of time by using an autoclave to investigate the influence of different temperatures, duration/cycles and different alkali dosage on expansion. The experimental program and test results are introduced in section 2 and section 3. In the second phase, then, this novel accelerated test method is applied to concrete specimens. Expansion of concrete prisms, external and internal cracking are recorded. Microscopic observation under scanning electron microscope (SEM) confirmed that the expansion occurs in the accelerated test is caused by ASR. Tests on mechanical properties of concrete specimens under accelerated ASR test method are conducted. Details are provided in section 4 and 5.

2. Phase I experimental program – mortar specimens

2.1. Materials and mix proportions

A general-purpose Portland cement with an alkali content of 0.50% Na_2O_{eq} was used in the mixing as binder material. The chemical composition of the general-purpose Portland cement was examined by X-ray fluorescence and is provided in Table 1, in terms of oxides (wt.%). Equivalent alkali content (Na_2O_{eq}) of cement was calculated as Na_2O+0.658*K_2O. In addition, a technical grade sodium hydroxide (NaOH) pellets with purity of 98% was added into the mixing water and pre-dissolved in a fraction of the mixing water before mixing, to adjust the alkali contents (Na_2O_{eq} by mass of cement) in the mortar.

Table 1

Chemical composition of general-purpose Portland cement (wt.%).

	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	K_2O	N_2O	LOI	Na_2O_{eq}
GP cement	64.18	19.67	4.78	3.10	2.37	0.91	0.41	0.23	4.09	0.50

Two types of aggregates were used in this study, one is the highly reactive aggregate, namely dacite aggregate, and another is the non-reactive aggregate (basalt aggregate) as a reference to demonstrate the efficacy of the proposed accelerated test method. In terms of the non-reactive basalt aggregate, the expansion of the 1-year concrete prism test (CPT) performed by Cement Concrete & Aggregates

Australia (CCAA) was 0.007%, which is below the expansion threshold of 0.03% for classifying reactive and non-reactive aggregate according to AS 1141.60.2, or expansion limit of 0.04% as per ASTM C1293. The dacite aggregate is crushed from the dacite coarse aggregate and the non-reactive aggregate is crushed from the non-reactive basalt coarse aggregates. Both two types of crushed aggregates have the same grading as shown in Fig. 1. It is worth to mention that, for reference specimens, crushed non-reactive aggregates, general-purpose Portland cement and 3.5% alkali loading (Na_2O_{eq} by mass of cement) were used in the mix proportion, while for reactive specimens, crushed reactive dacite aggregate, general-purpose Portland cement were adopted. To study the influence of alkali loading on mortar bar expansions, different alkali loadings (2.5%, 3.0% and 3.5% Na_2O_{eq} by mass of cement) were used in the reactive specimens.

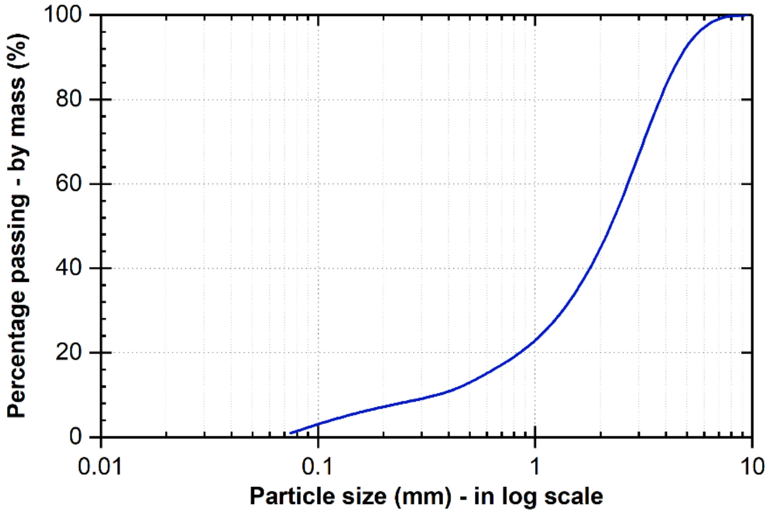


Fig. 1. Grading curve of crushed aggregates

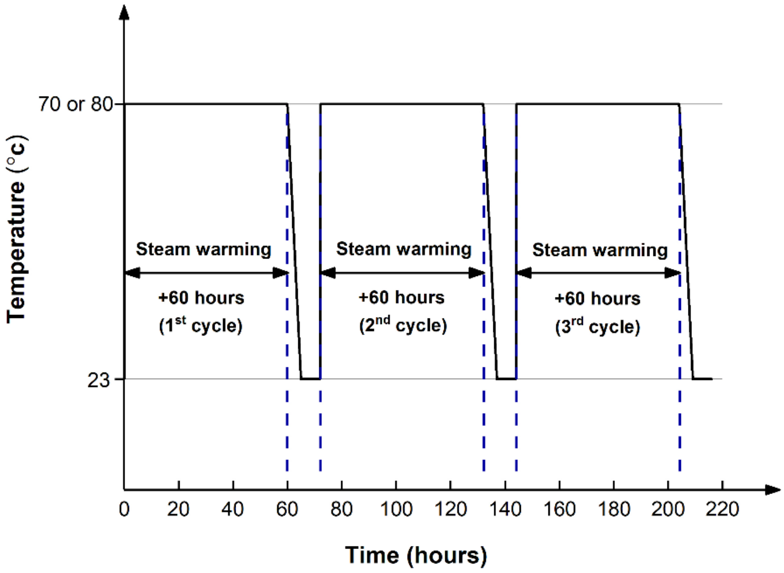
The mix proportions for preparing mortar specimens adopted in this study is one part of cement to 2.25 parts of dacite aggregates or non-reactive aggregates with the maximum particle size 4.75mm. Water-cement ratio is 0.4, instead of 0.47 as stated in the AS 1141.60.1. Also, in order to boost the alkali content in the mortar specimens, sodium hydroxide (NaOH) pellets were added to the mixing water 24 hours prior to the mixing to increase the alkalis of different mortar specimens to 2.5%, 3.0% and 3.5% Na_2O_{eq} by mass of cement.

According to previous study performed by Fournier et al. [27], for mixes with the total alkali content below 2.5% (1.0% and 1.25%), the expansion of mortars bars made with four different aggregates after 4 hours of autoclave treatment at 130°C was smaller than 0.05% and was similar to each other, regardless of the reactivity of aggregates. Also, Abe et al. [28] studied the influence of alkali loading (1.5%, 2.5% and 3.5%) on expansion of mortar bars with an autoclaving temperature of 100°C. At 1.5% alkali loading, very small expansion was observed for mortar bars with reactive aggregates, but at 2.5% alkali loading, considerable expansion was observed, and at 3.5% alkali loading, the expansion of reactive aggregate mortar bars increased even further. A 2.5% alkali loading was proposed by Abe et al. [28] in the 100°C autoclave test. Therefore, in this study, to accelerate ASR in a short period, tests were performed on specimens with the total alkali content above 2.5%.

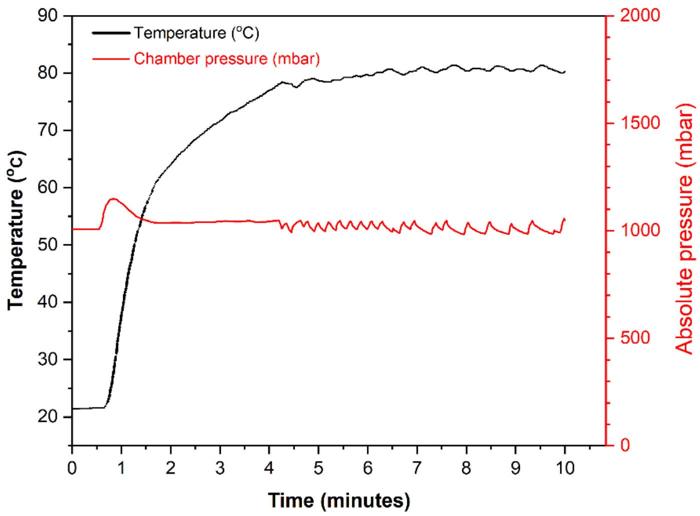
2.2. Specimen fabrication and steam warming procedure

The mortar mixing procedure follows ASTM C305 [29]. After casting, the mortar specimens (25 × 25 × 285mm) with embedded stainless-steel studs at two ends were kept in 23°C 90%RH humidity cabinet for 24 hours then demoulded and the initial measurements (length and mass) were conducted. The initial length of the mortar bars was measured by a digital comparator. The specimens were then cured in 23°C 90%RH humidity cabinet for another 24 hours, thereafter, the specimens were taken out and put into an autoclave for steam warming.

In order to accelerate ASR, a testing regime of 3 cycles at a specific temperature was adopted, using an autoclave with a chamber volume of 153 litres (Zirbus LVSA 50/70). To study the influence of different temperature on the expansion of mortar bars, two temperature conditions, 70°C and 80°C, were applied. Each cycle of steam warming duration is 60 hours and totally three cycles of steam warming were applied. The pressure inside the autoclave chamber is kept at atmospheric pressure. Fig. 2(a) shows the temperature-time relationship of steam warming for three cycles. Fig. 2(b) shows the change of temperature and corresponding absolute pressure in the autoclave chamber when 80 °C steam warming is applied. It can be seen that the autoclave chamber is rapidly heated up to 80°C in less than 10 minutes. The temperature quickly stabilizes at 80°C with minor variations and the pressure is maintained at 1000mbar. This is achieved by the independent steam pot of the autoclave injecting steam into the chamber automatically and controlling the temperature while keeping the chamber in a fully saturated condition during the entire test period. At the end of each cycle, the expansion change of the mortar bars, due to the accelerated ASR were recorded and the next cycle was applied.



(a). Time-temperature cycles adopted for conditioning the specimens



(b). Characteristic temperature and absolute pressure in autoclave chamber with 80°C steam warming (at the beginning of each cycle)

Fig. 2. Temperature and pressure in the autoclave chamber adopted for the multi-cycle accelerated test

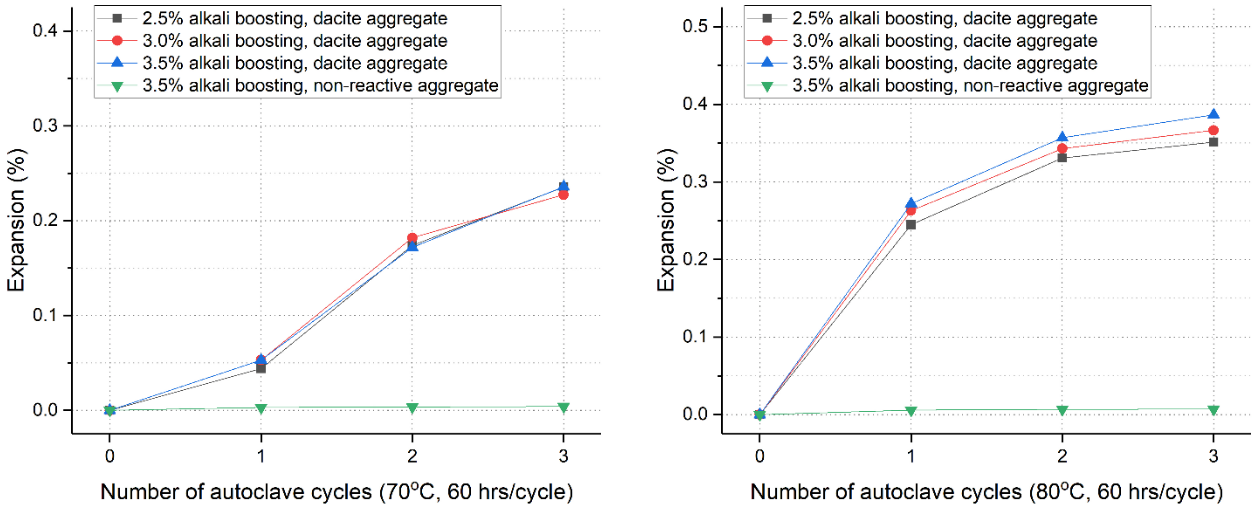
2.3. Expansion measurements

Initial lengths of the mortar bars were measured and recorded using a digital comparator after demolding. After each cycle of steam warming in the autoclave, the specimens were taken out, immediately stored in sealed plastic bags for 2 hours to cool down to room temperature at 23 ± 2°C and the length measurements were conducted. In this process, the specimens were kept in a saturated condition and drying and/or carbonation were avoided. Changes in length were used to calculate the expansion of the specimens after 1, 2 and 3 cycles of autoclave steam warming.

3. Results and discussion for phase I experiments

3.1. Expansion of mortar bars

Expansion of the mortar bars from the time of demoulding and after 3 cycles of autoclave steam warming is shown in Fig. 3. After 3 cycles of 70°C steam warming in the autoclave, a total expansion of about 0.24% was recorded. After 3 cycles of 80°C steam warming, total expansion reached 0.35% to 0.39%. In addition, it was observed that expansion increased with the increasing of steam warming duration/cycles. However, this parameter has no effect on non-reactive basalt aggregate specimens. Besides, as can be seen from Fig. 3(b), for the control non-reactive basalt aggregates (3.5% alkali loading), when subjected to the 80°C autoclave cycles, there was minimal expansion. The environment in the autoclave is fully saturated and hence no drying shrinkage is possible. The autogenous shrinkage due to cement hydration is very small and the net result of specimens with non-reactive basalt aggregate was a small expansion (less than 0.02%). In a previous study, performed by Shayan et al. [30], also found minor expansions (about 0.02%) of mortar bars made with non-reactive basalt aggregates immersed in 1 M NaOH solutions (80°C). The influence of alkali content and the influence of steam warming temperature will be discussed in the following sections.



(a). 70°C (b). 80°C

Fig. 3. Expansion of specimens with reactive and non-reactive aggregates in autoclave test

3.2. Influence of total alkali content (2.5%, 3.0%, 3.5%)

At 80°C steam warming cycles, the expansion of reactive dacite aggregate mortar bars increased with the increasing of total alkali content from 2.5% to 3.5% Na_2O_{eq} (see Fig. 4). However, at 70°C autoclave steam warming cycles, this trend is not significant (see Fig. 3(a)). For non-reactive aggregate mortar bars, total alkali content to 3.5% Na_2O_{eq} level only has negligible effect on the expansion of the specimens.

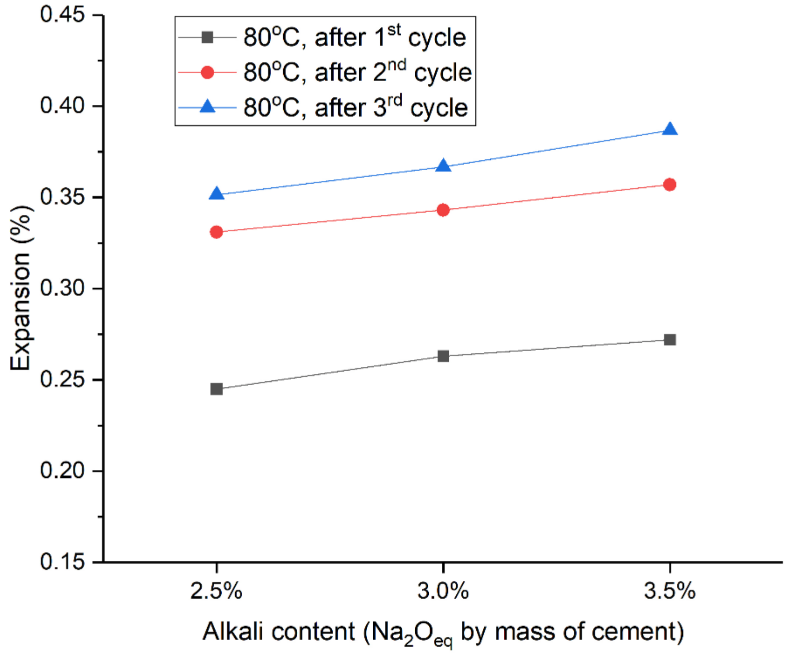


Fig. 4. Expansion – Alkali content relationship (80°C steam warming)

By performing autoclave test for ASR using a large cooking pressure pot at 100°C to 130°C, Nishibayashi et al. [31] reported that expansion of the reactive specimens increased with increasing of total alkali content of the mixes when alkali content was over 1.0% Na_2O_{eq} and decreased when alkali content was higher than 3.0% Na_2O_{eq}. The maximum expansion occurred when alkali content was about 2.5% Na_2O_{eq}. However, the authors suggested a total alkali content of 1.5% Na_2O_{eq} in the proposed test procedure. Through chemical analysis, the authors also confirmed that the reaction products were the same as that from the normal accelerated test at 38°C and 100% RH. Fournier et al. [27], however, observed that expansion of reactive specimens was significantly improved when the alkali content was raised from 2.5% Na_2O_{eq} to 3.5% Na_2O_{eq}, but this parameter had no effect on the behaviour of non-reactive aggregates. The authors proposed a rapid autoclave mortar bar test method with 3.5% Na_2O_{eq} alkali loading. Wood et al. [26] investigated the influence of alkali loading on the expansion of mortar bars with a 130°C autoclaving temperature and a 5 hours autoclaving duration. The authors reported that 2.5% Na_2O_{eq} alkali loading failed to distinguish the potential reactivity between non-reactive and reactive aggregates, while increasing the alkali content from 3.5% to 4.5%

had minimal effect for promoting expansion of mortar bars. Therefore, Wood et al. [26] recommended a 3.5% Na_2O_{eq} alkali loading for autoclave mortar bar test.

3.3. Influence of temperature on expansion (70°C, 80°C)

The influence of steam warming temperature (70°C and 80°C) on expansion of mortar bars is studied. For reactive dacite aggregate specimens after 3 cycles of autoclaving, when the steam warming temperature in the autoclave was increased from 70°C to 80°C, it was observed that the average expansion of mortar bars was improved by a factor of 1.49, 1.61 and 1.64 for mixes with 2.5%, 3.0% and 3.5% alkali content, respectively. Hence, temperature increase from 70°C to 80°C can significantly accelerate the ASR for reactive aggregates. This suggests that steam warming temperature at 80°C is more efficient than testing at 70°C for rapid ASR testing by using an autoclave.

3.4. Microscopic observation for assessment of ASR

Microscopic examination under SEM on samples from mortar bars subjected to 3 cycles of 80°C steam warming in an autoclave is conducted. ASR gel is Ca-Alkali(Na,K)-Si-H_2O in general. As H_2O is unable to be detected by scanning electron microscope and energy dispersive spectrometry (SEM-EDS), Ca, Na, K and Si are the indicators that ASR gel is present [32-35]. Fig. 5 shows the cracks through aggregates and ASR gel formation after 3 cycles in the autoclave. ASR gel around aggregate was observed as well and Fig. 6 demonstrates the ASR gel at aggregate-cement paste interface.

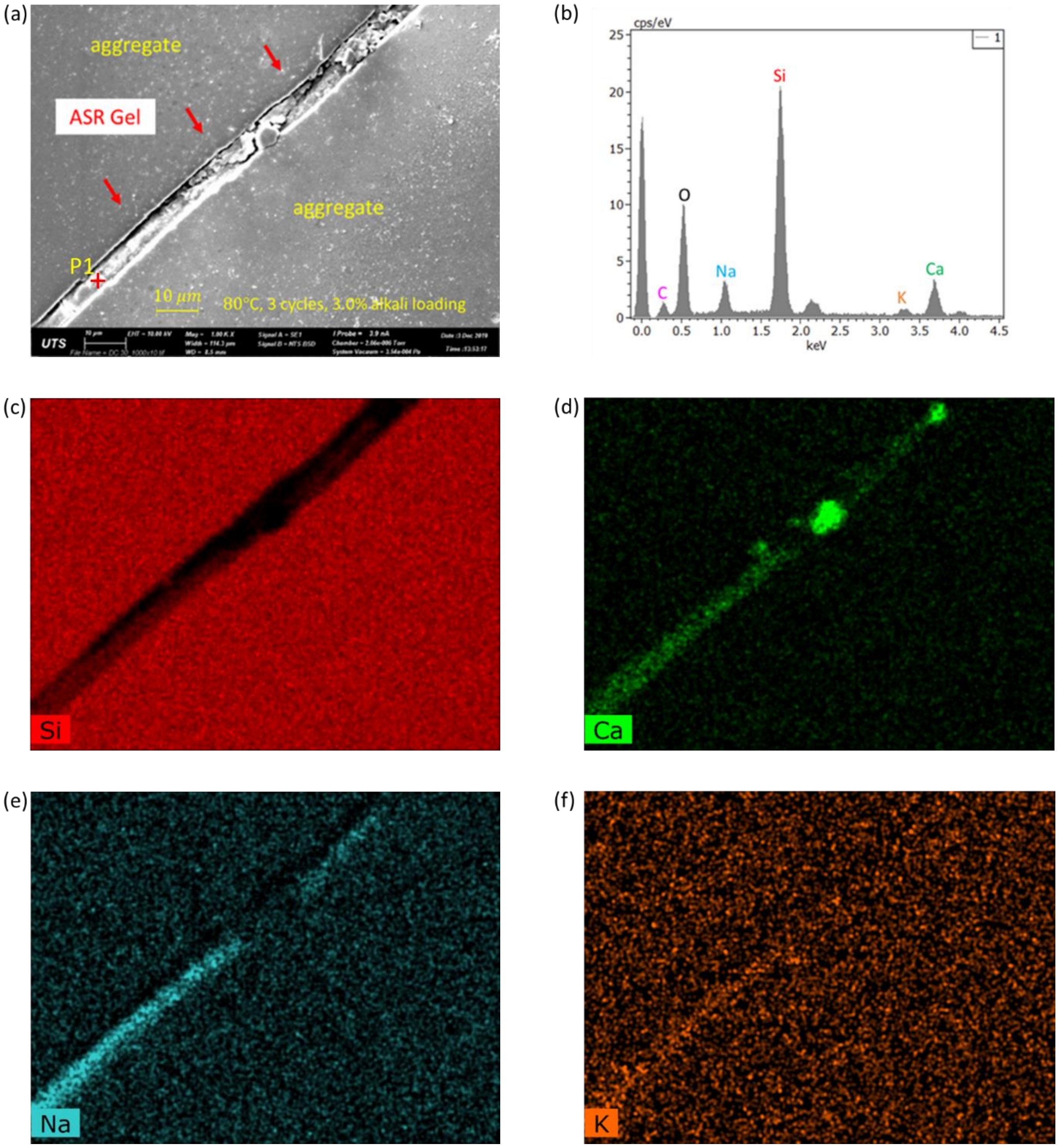


Fig. 5. SEM-EDS maps of the ASR gel within an aggregate

(a). Image by SEM (backscatter electron beam, C-coated sample);

(b). EDS spectra correspond to P1; (c)-(f). SEM-EDS maps

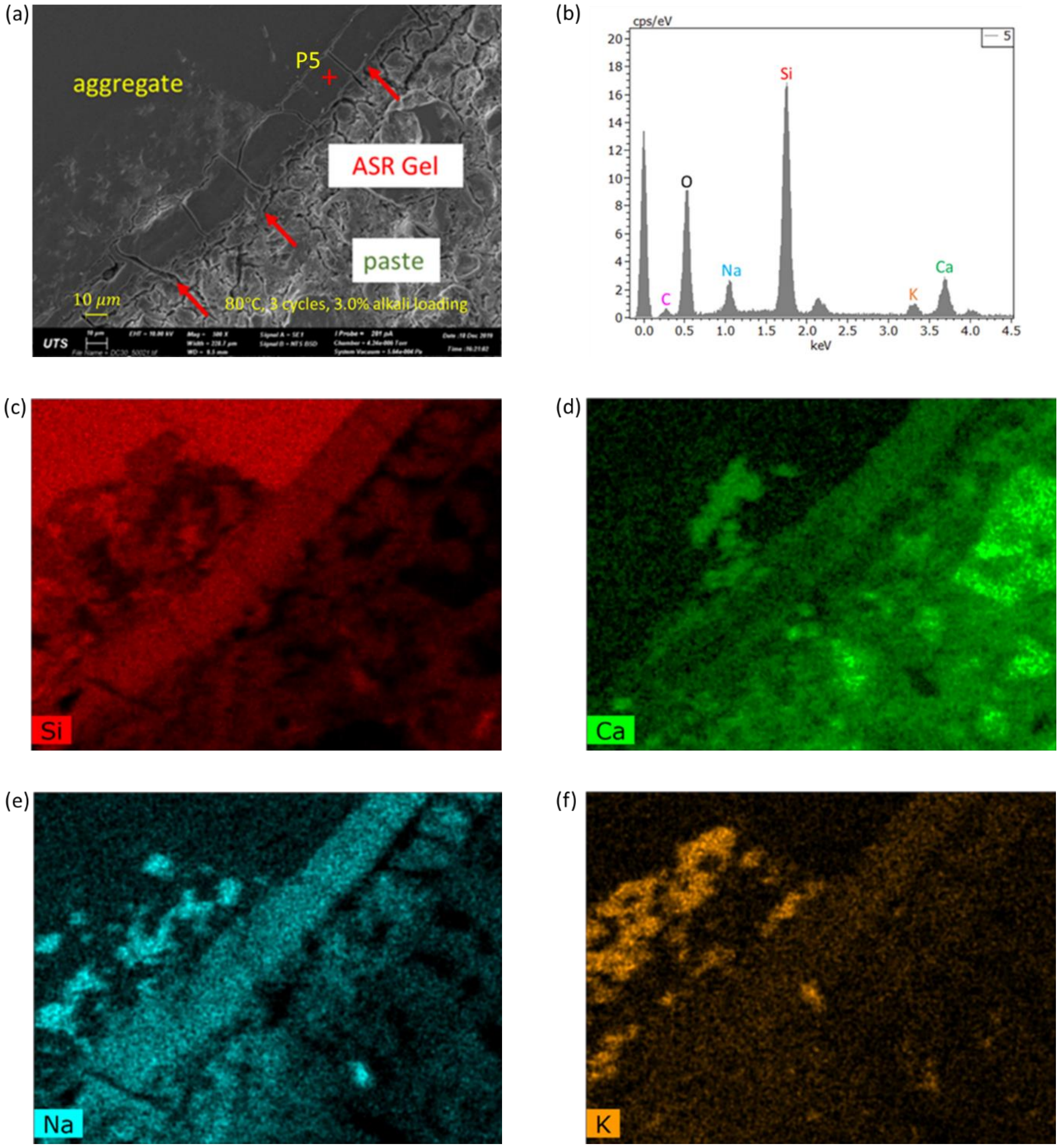


Fig. 6. SEM-EDS maps of the ASR gel at aggregate-cement paste interface
(a). Image by SEM (backscatter electron beam, C-coated sample);
(b). EDS spectra correspond to P5; (c)-(f). SEM-EDS maps

3.5. Summary of phase I experimental program

Through the phase I experimental program on mortar bar specimens, a new accelerated test for ASR using an autoclave is established. Main findings and the proposed test procedure are summarised as follows:

(1) Under various test conditions, expansion values of the mortar bars made with reactive dacite aggregates increased with increasing of steam warming temperature and autoclave duration/cycles. Tests at 80°C steam warming temperature demonstrated that expansion values of the reactive Dacite aggregate mortar bars increased with the increasing of total alkali content of the mix.

(2) The proposed final test procedure consists of 80°C maximum temperature, 60 hours duration per cycle and a total number of three cycles of steam warming using an autoclave. In this accelerated test, it is recommended to use 2.5% Na_2O_{eq} (by mass of cement) alkali loading for the mortar and concrete mixes.

(3) The proposed test method is suitable for preliminary test to identify the potential reactivity of aggregate. However, more tests on different aggregate types which include highly reactive, moderate reactive and slowly reactive aggregates need to be conducted. Correspondence between the test results obtained from the new accelerated autoclave test and the expansion of field concrete needs further investigation.

(4) Based on test results of mortar bar specimens at phase I, it is expected that the proposed test method could also be applied to concrete mixes. Through accelerated autoclave test, significant expansion within a short test duration is expected for concrete specimens which could simulate long-term expansion in concrete structures subjected to severe exposure conditions.

4. Phase II experimental program – concrete specimens

4.1. Materials and mix proportions

In all concrete mixes, a non-reactive fine aggregate, Sydney sand, was used. As for the coarse aggregate, the highly reactive dacite aggregate with maximum particle size of 20mm was used in the concrete mixes. The grading information of dacite coarse aggregate and Sydney sand is illustrated in Fig. 7. The mix proportions used for concrete specimens is given in Table 2. Following the accelerated test procedure proposed in phase I, two representative alkali loading levels at 2.5% and 3.0% Na_2O_{eq}

by mass of cement were applied to the concrete mix. The interest of phase II was to extend the proposed method to evaluate the potential risk of ASR on concrete expansion and mechanical properties, and the expansion of concrete prisms with non-reactive basalt aggregates was so small (70 micro-strain) according to the 1-year concrete prism test (CPT) performed by Cement Concrete & Aggregates Australia (CCAA), hence mixtures with reactive dacite aggregates were tested.

Although previous results revealed that the expansion increases with the total alkali content of the mix containing reactive aggregates [28], the mechanical properties of the concrete can be significantly reduced by the increasing of the alkali content with addition of sodium hydroxide [36]. Therefore, for studying the mechanical properties of plain and reinforced concrete affected by ASR, a lower alkali content is preferred. In this study, the 2.5% alkali loading was chosen to investigate the change of mechanical properties under the accelerated test condition. To compensate for the loss of compressive strength caused by alkali boosting, a low w/c ratio was chosen for the initial mix without boosting so that after high levels of alkali boosting, the remaining strength values were maintained at reasonable levels. Thus, the mix used in the current investigation was different to what was recommended in ASTM C1293, AS 1141.60.2, RILEM AAR-3 or RILEM AAR-4.1.

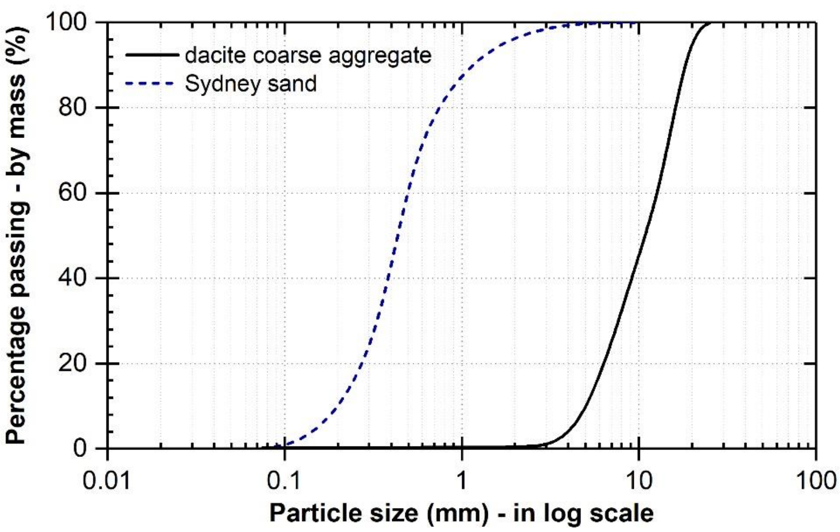


Fig. 7. Grading of fine aggregates (Sydney sand) and coarse aggregates (dacite aggregate)

Table 2

Concrete mix proportions

Material	Concrete mix	
	With 2.5% Na_2O_{eq} boosting (kg/m^3)	With 3.0% Na_2O_{eq} boosting (kg/m^3)
20mm dacite aggregate	1160	1160
Sydney sand (non-reactive)	620	620
Cement	520	520
Water	192.5	192.5
NaOH pellets added	13.69	17.12
Na_2O_{eq} (Calculated)	13.0	15.6

4.2. Specimen fabrication and steam warming procedure

Sodium hydroxide pellets with purity of 98% were used to raise the alkali content in the concrete mix to 2.5% and 3.0% by mass of cement. The NaOH pellets were dissolved in mixing water 24 hours before the mixing and cooled down to room temperature at around 23°C, afterwards, solutions were stored in sealed plastic containers ready for use in concrete mixing. A 70 litres horizontal pan mixer was used to mix the fresh concrete, following AS 1012.2:2014 [37]. For the 3.0% Na_2O_{eq} boosting mix, three prisms with embedded stainless-steel studs were cast, while for the mix with 2.5% alkali loading, twelve cylinders and three prisms were cast. The size of the cylinders was 100mm (diameter) by 200mm (height), and that of the prisms was 75 × 75 × 285mm.

Specimens were demolded after one day from casting, and then the initial length and mass of the prisms were recorded for expansion and mass change measurement. Thereafter, all specimens were stored in a humidity cabinet with temperature of 23°C and relative humidity of 90% RH. At the age of 28 days, three cylinders were tested for the modulus of elasticity and then compressive strength, in accordance with AS 1012.17 and AS 1012.9, respectively. Meanwhile, the length and mass of all the prisms were measured.

The accelerating tests started at the age of 28 days, using the same equipment as mentioned in Phase I. All prisms and the remaining nine cylinders were placed in the top and bottom baskets (see Fig.9), respectively. The same autoclaving cycles used in section 2.2 was adopted to accelerate ASR of concrete at the temperature of 80°C, 60 hours per cycle and three cycles in total. At the end of each cycle, three cylinders were taken out from the autoclave for testing of modulus of elasticity and compressive strength, whilst the expansion and the mass of the prisms were measured. Following the results obtained from phase I, the focus of phase II was extended to evaluate the potential risk of different levels of ASR expansion on concrete performance. As such, to obtain different levels of expansion, a series of cycles were performed. It is worth to mention that the mass was recorded immediately after the specimens were taken out, while the length of the prisms was measured after the specimens were cooled down to room temperature at 23°C. After the mass measurement, the prisms were stored in sealed plastic bags for 6 hours to cool down to room temperature at 23 ± 2°C, and then the length measurements were conducted. In this process, the specimens were kept in a saturated condition and drying and/or carbonation were avoided.

5. Results and discussion for phase II experiments

5.1. Expansion and mass change

Fig. 8 shows the expansion of the concrete prims after 28 days of curing in the humidity cabinet and after each cycle of autoclaving. A slight shrinkage approximately 190 micro-strain was observed at the age of 28 days. The expansion increases with the autoclaving cycles. The expansion of prisms with 2.5% Na_2O_{eq} boosting was 0.05% after one cycle, while this value was raised to 0.18% after three cycles. Similar phenomenon was confirmed by the results obtained from specimens with 3.0 % Na_2O_{eq} boosting, expansion increasing from 0.07% to 0.22% after 3 cycles. In addition to the autoclaving cycles, the expansion was increased with the alkali content. Moreover, the effect of alkali content on expansion under the same cycle was enhanced by the increasing of cycles in the scope of this study. As shown, when only one cycle (60 hours) was applied, the difference of expansion between two mixes was 0.02%, while that difference reached 0.04% after three cycles (180 hours). Effects of more cycles on the expansion of specimens needs further investigation.

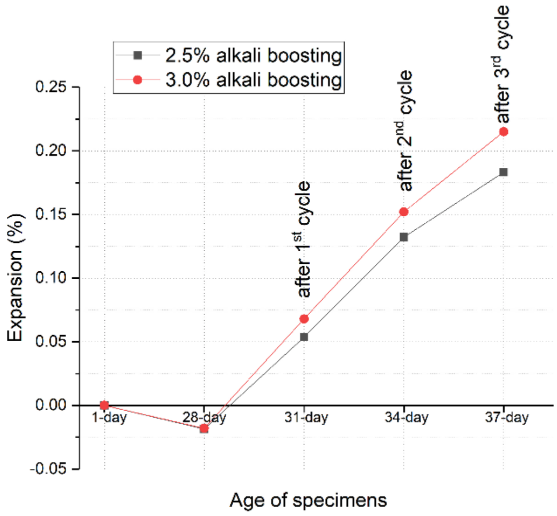


Fig. 8. Expansion of concrete prisms after 3 cycles in the autoclave

As mentioned before, the ASR expansion is caused by gel swelling after absorbing water. Different mass gain would be expected in prisms with different expansion. The mass change of prisms measured immediately after being taken out from the autoclave is shown in Fig. 9. This operation was to reduce the influence of moisture evaporation on the mass measurement. The results showed that the mass of prism increases with increasing autoclaving cycles, and the prisms with higher alkali loading exhibited higher mass gain, which agreed well with the expansion results in Fig. 8. Interestingly, after 28 days of curing in the humidity cabinet, a slight mass gain of about 0.05% was observed when there is no expansion in prisms, which could be attributed to the continued hydration of the cement during this period. As can be seen, in the first two cycles, the mass gain of prisms increased significantly with the increasing of autoclaving cycles, from about 0.05% to 0.38% for specimens with 2.5% alkali loading, and to 0.46% for specimens with 3.0% alkali content, respectively. However, this trend was slowed down afterwards, as minor mass gain was recorded for specimens with 2.5% and 3.0% alkali loading when the third cycle was applied. It should be mentioned that the hydration of the unreacted cement possibly continued during the autoclaving cycles, which could contribute part of the mass again. Therefore, the measured mass gain could be attributed to the combination of continued hydration and ASR gel formation.

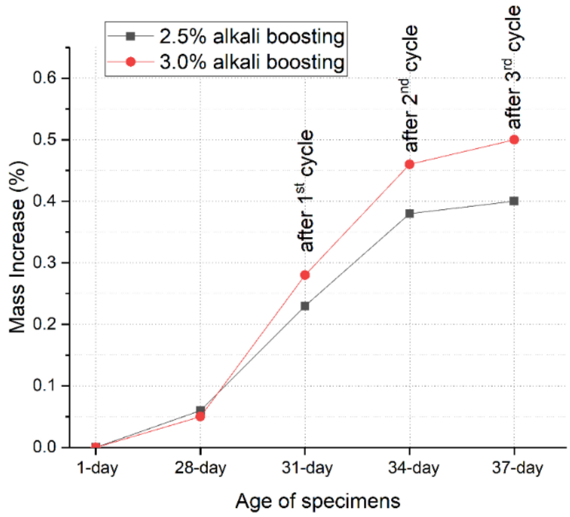


Fig. 9. Mass change of concrete prisms after 3 cycles in the autoclave

5.2. Cracking of concrete specimens and microscopic observation for assessment of ASR

The crack patterns of concrete cylinders and prisms at the end of third autoclaving cycle, with 2.5% Na_2O_{eq} boosting, are shown in Fig. 10. Some white exudations were found on the surface of concrete specimens. In order to analyse the ASR products that cause expansion and cracking, samples from prism with 2.5% Na_2O_{eq} boosting experienced 3 autoclaving cycles were subjected to SEM/EDS examination. As shown in Fig. 11(a), similar morphology as reported in [38, 39] of typical rosette ASR products were observed. Fig. 11(b) illustrates the EDS spectra of the observed ASR products. The microscopic examination indicates that the expansion of the concrete specimens is caused by ASR.

Fig. 10. External cracks on cylinders and prism after 3 cycles in the autoclave

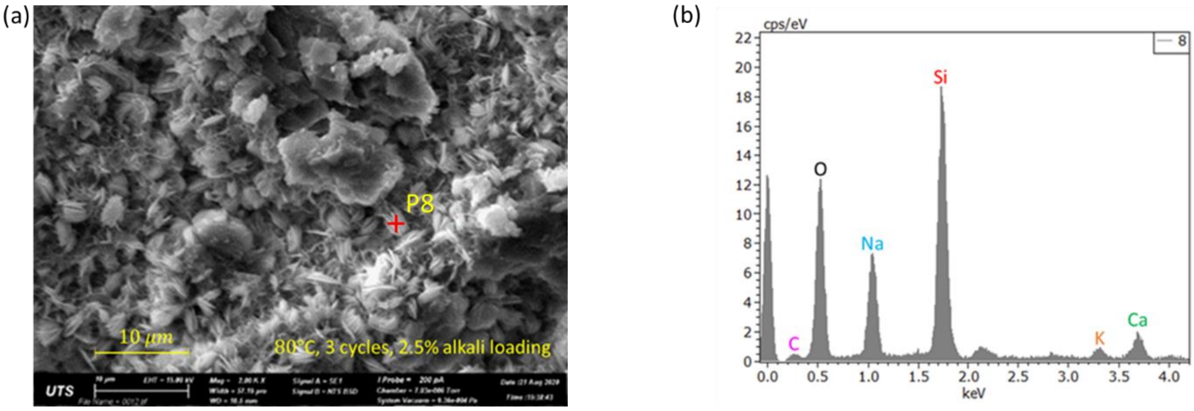


Fig. 11. SEM image of ASR products and EDS spectra correspond to P8 area (C-coated sample)

5.3. Mechanical properties of concrete under accelerated ASR test

Fig. 12 presents effects of autoclaving cycles on compressive strength and elastic modulus of cylinders with 2.5% alkali boosting. The compressive strength increased until the end of the second cycle and showed a decreasing trend thereafter. It can be observed that the compressive strength initially increased with the increasing of expansion at relatively low expansion level in this study, which was consistent with previous findings [40, 41]. As can be seen, up to expansion level about 0.13%, the compressive strength was improved by 15.3% and 23.6% after the first and second cycle respectively, with respect to the 28-day compressive strength. Thereafter, the compressive strength showed a decreasing trend while expansion keeps increasing. The gain in compressive strength dropped to 16.7% as the expansion developed to 0.18% at the end of the third cycle. It is expected that the compressive strength will continue to decrease with the increasing of expansion in concrete. This trend had already been reported by Gautam et al. [40]. The authors observed a maximum reduction of 4-6% in compressive strength at 365-day compared with 28-day value when expansion reached 0.24-0.35%.

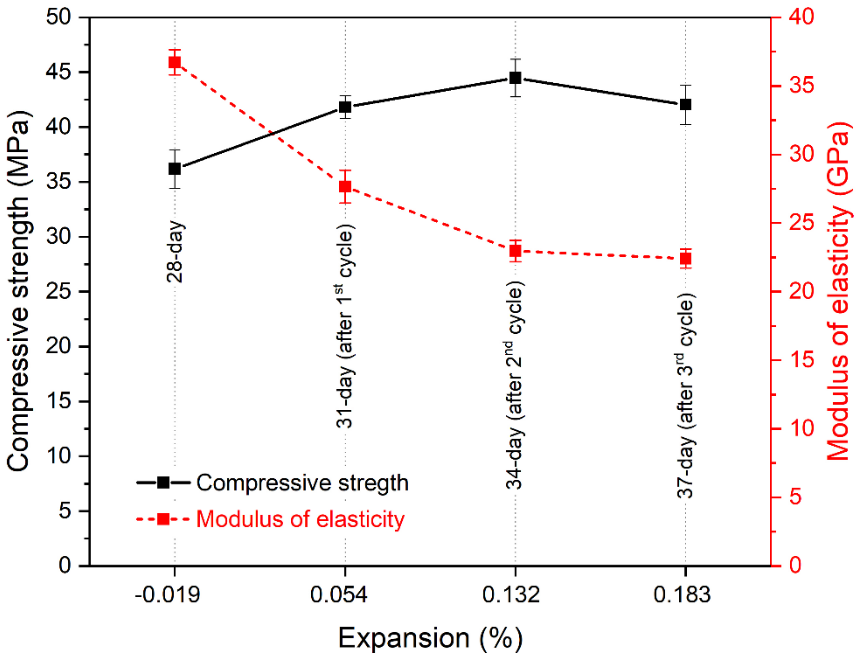


Fig. 12. Change in compressive strength and modulus of elasticity of concrete cylinders after 3 cycles in the autoclave

Modulus of elasticity is generally regarded as the most sensitive mechanical property affected by ASR. Prior to compressive strength tests, three cylinders was tested after each cycle to obtain the modulus elasticity and the results were presented in Fig. 12. The modulus of elasticity was systematically reduced with autoclaving cycles. With respect to the modulus elasticity at 28 days, a reduction of 24.5%, 37.9% and 39.0% was recorded after 1, 2 and 3 cycles, respectively. The degradation of the modulus elasticity is due to the micro-cracking caused by accelerated ASR.

It is well known that ASR affects modulus of elasticity and tensile strength of concrete more significantly than the compressive strength. In the current tests, it is clear that the modulus of elasticity is systematically reduced due to ASR expansion. Meanwhile, at the expansion levels measured, the effect on compressive strength does not indicate a clear reduction. Further investigation on splitting tensile strength was conducted. Fig. 13 shows the splitting tensile strength of cylinders subjected to 1, 2 and 3 cycles of 80°C autoclave treatment. It can be seen from Fig. 13 that the splitting tensile strength increased initially with the autoclaving cycles and after 2 cycles, it showed a decreasing trend. These results are similar to the results reported by Larive [41] and Esposito [42]. However, at high expansion levels, previous study showed that the splitting tensile strength decreased significantly [42].

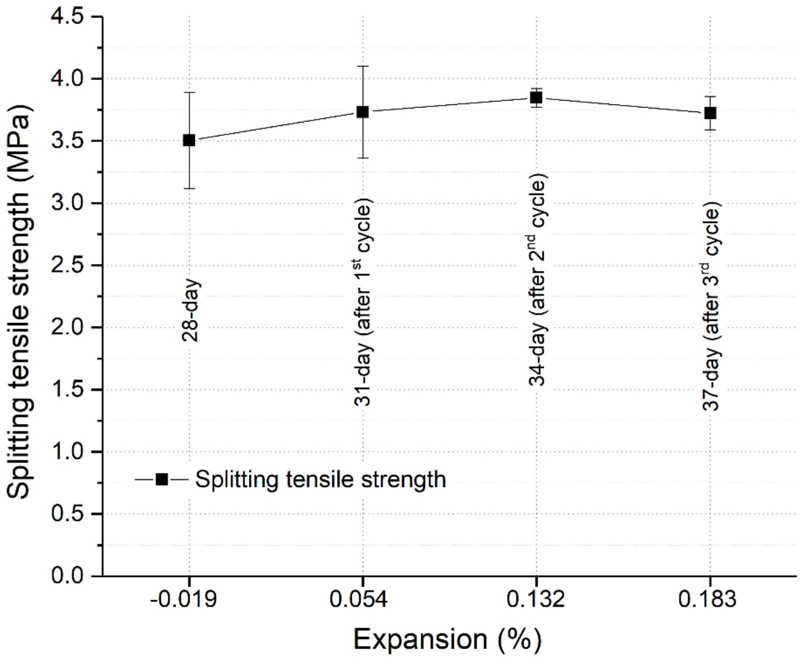


Fig. 13. Splitting tensile strength of cylinders (without autoclave treatment and with 80°C autoclave treatment, dacite aggregate cylinders with 2.5% alkali boosting)

6. Aggregate reactivity classification

For the expansion limits to classify the aggregates according to the proposed autoclave expansion test, the same limits given in ASTM C1778-20 [43], which is shown in Table 3, can be referred to:

Table 3

Aggregate reactivity classification (ASTM C1778-20)

Aggregate reactivity level	Reactivity description	1-year expansion (CPT, ASTM C1293)	14-day expansion (AMBT, ASTM C1260)
R0	Non-reactive	<0.04%	<0.10%
R1	Moderately reactive	≥0.04%, <0.12%	≥0.10%, <0.30%
R2	Highly reactive	≥0.12%, <0.24%	≥0.30%, <0.45%
R3	Very highly reactive	≥0.24%	≥0.45%

The comparison of dacite aggregate mortar bar expansion results of autoclave test and AMBT is shown in Fig. 14 whereas the comparison of concrete prism expansion results of autoclave test and CPT is shown in Fig. 15.

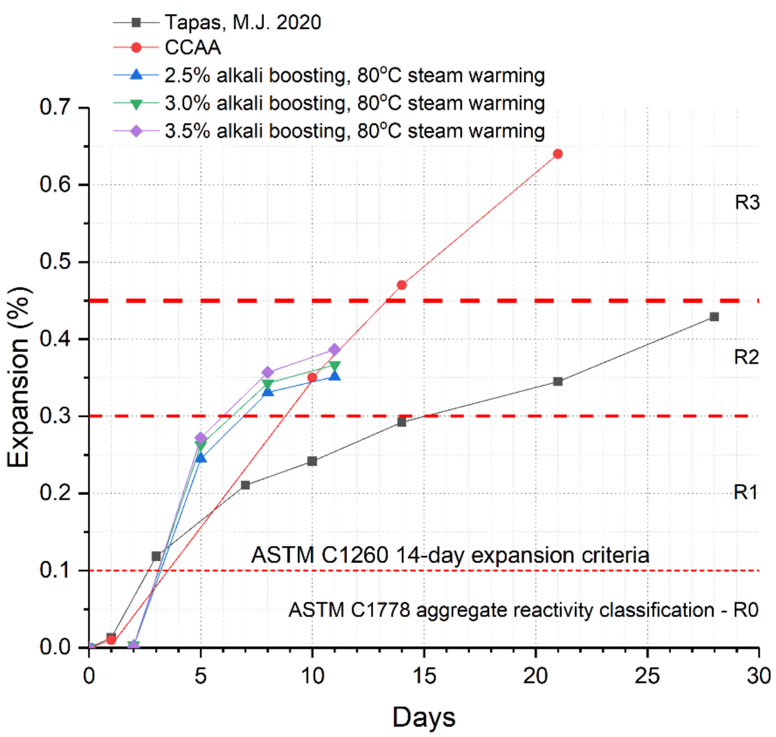


Fig. 14. Comparison of expansion results between 80°C autoclave treatment with alkali boosting and AMBT for dacite aggregate mortar bar

As can be seen from Fig. 14, expansion of mortar bars made with dacite aggregate and 2.5% alkali loading reaches 0.35% after 3 cycles in the autoclave, which is greater than 0.3% while is less than 0.45%. Hence, the tested dacite aggregate can be classified as 'highly reactive'. The average 14-day AMBT results of dacite aggregate, performed by Cement Concrete & Aggregates Australia (CCAA) and Tapas [44] are at the upper and lower limits of 'highly reactive' classification as shown in Fig. 14.

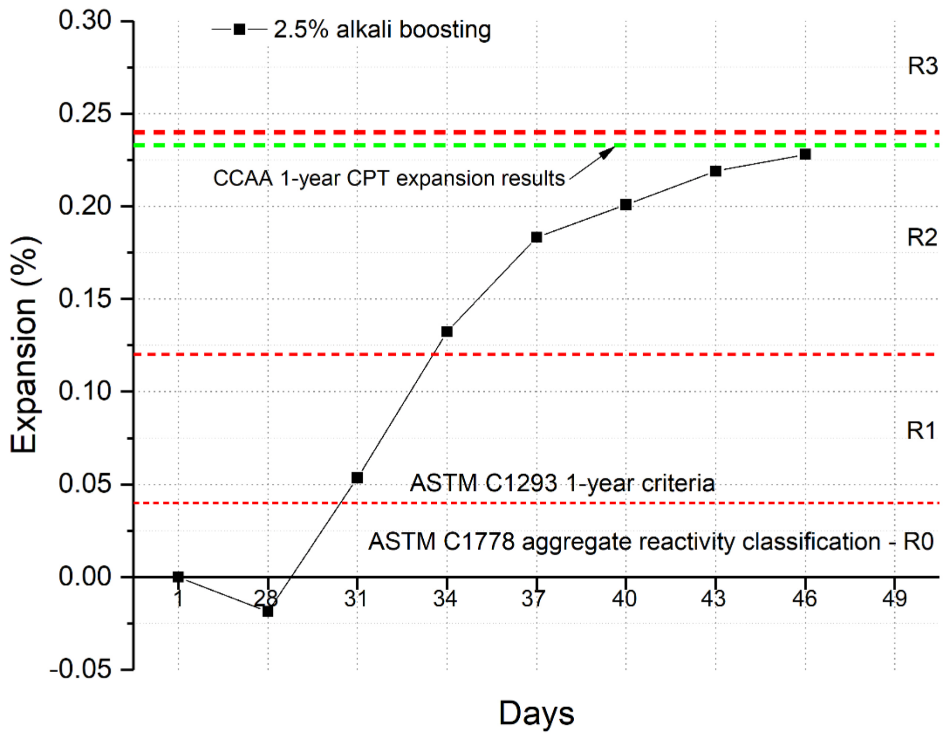


Fig. 15. Comparison of expansion results between 80°C autoclave treatment with 2.5% alkali boosting and CPT for dacite aggregate prism

Fig. 15 shows the comparison of concrete prism expansion result (with dacite aggregate and 2.5% alkali loading) after 3 cycles in the autoclave (at the age of 37 days) and the 1-year CPT result (finding from CCAA), also classifies the dacite aggregate as 'highly reactive'. Fig. 15 also illustrated the expansion after a total number of 6 cycles in the autoclave (at the age of 46 days). The average expansion of the prisms reaches 0.228% after 6 cycles, which is an increase of 26.7% compared to the expansion obtained after 3 cycles (0.18% at the age of 37 days). Hence, for 'highly reactive' aggregates, with 3 cycles, the reactivity of the aggregate can be classified. However, for slowly reactive aggregates, more cycles (e.g., 6 cycles) are needed.

7. Conclusions

An accelerated test method for assessing the potential risk of alkali-silica reaction in concrete using an autoclave is proposed in this study: a testing regime of 3 cycles of steam warming at 80°C using an autoclave, with 60 hours per cycle. Basing on the method, the effects of temperature, alkali loading and autoclaving cycles on the ASR expansion of mortar bars and concrete prisms were investigated. Afterwards, SEM-EDS analysis was conducted to confirm the ASR products. Furthermore, the method was adopted to study the mechanical properties degradation caused by ASR in concrete. Although the mortar test is not much faster than the AMBT, the 37-day concrete test is a marked improvement over the 12-month CPT in testing time. The main conclusions can be drawn as follows:

(1) For mortar bars containing dacite aggregate subjected to autoclaving cycles, the expansion was slightly affected by increasing alkali loading from 2.5% to 3.5% when the steam warming temperature was at 70°C, and the maximum expansion was 0.23% at the end of three cycles. While the expansion was significantly increased by increasing the alkali content as the temperature was elevated to 80°C, and a maximum expansion of 0.39% was recorded in a short period.

(2) Although increasing the alkali content and autoclaving temperature could achieve higher expansions, to study the mechanical properties of ASR affected concrete, controlling alkali content in concrete and autoclaving temperature is necessary, because high alkali content by addition of NaOH and high autoclaving temperature affect the mechanical properties of OPC concrete. Therefore, an alkali content of 2.5% by mass of cement and a temperature of 80°C are recommended for using autoclaving cycles to test ASR.

(3) As revealed by the expansion measurements and mechanical properties assessment performed on concrete, the multi-cycle autoclave test method appears to be suitable for investigating ASR

deterioration of actual concrete mixes within a short period of time. For concrete prisms containing dacite aggregate with 2.5% alkali boosting, a maximum expansion of 0.18% was achieved after 3 cycles of autoclaving. The modulus of elasticity systemically decreased with the increasing expansion. Compressive strength and splitting tensile strength, however, showed an initial increase under low expansion, followed by a reduction at higher expansion levels.

(4) Potential degradation of mechanical properties and their effect on bond deterioration, and residual load capacity (in terms of flexural strength and shear strength) of ASR affected reinforced concrete structures can be investigated using the proposed accelerated ASR test method in an autoclave.

Acknowledgement

This research is funded through an Australian Research Council Research Hub for Nanoscience Based Construction Materials Manufacturing (IH150100006) with the support of Cement Concrete & Aggregates Australia. The SEM-EDS analyses were performed by Dr Marie Joshua Tapas and the authors are grateful for her contribution.

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