

The combined role of alkali silica reaction and delayed ettringite formation in durability loss of concrete structures

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Abstract

Durability of concrete as a structural material is key in its worldwide use for buildings and infrastructure, with the lifetime service of concrete greatly impacting its economic, environmental, and social costs. Causes of durability loss in some concrete structures can be attributed to the alkali-silica reaction (ASR) and delayed ettringite formation (DEF). Both are chemical reactions with the potential of deleterious cracking, expansion, and strength loss in affected elements. Significant overlap exists in the factors contributing to ASR and DEF in concrete structures, with widely reported evidence of deleterious DEF frequently occurring in conjunction with mild or moderate ASR. For precast concrete, binder and temperature limits based on mortar experiments are used during curing to minimise DEF risk. The role of other constituents in concrete specimens, notably the aggregate, have been overlooked. This paper investigates the role of ASR in the susceptibility of concrete to DEF. Concrete specimens were manufactured under conditions that promoted deleterious ASR and DEF, including aggregate reactivity, binder composition, and curing temperature. Linear expansion and compressive strength were measured over 1-year. Only concrete systems subject to heat-curing and elevated alkali and sulfate contents showed deleterious effects.

Keywords: alkali-silica reaction, concrete durability, delayed ettringite formation, alkali content, sulfate content, heat curing

1. INTRODUCTION AND LITERATURE REVIEW

The durability of concrete as a construction material is an important issue for the global usage of concrete and cementitious materials in modern built infrastructure, and is strongly linked to the economic, environmental, and social costs to source, assemble, and maintain such structures during their service life [1-3]. Two known causes of durability loss in concrete are the alkali-silica reaction (ASR) and delayed ettringite formation (DEF), which are separate chemical reactions that have the potential to cause deleterious expansion, microcracking, and strength loss in affected structures.

1.1 Delayed Ettringite Formation

DEF is a form of internal sulfate attack in concrete, with the primary mechanism being the temporary decomposition of the sulfate mineral ettringite during early cement hydration and late age reprecipitation of ettringite in hardened concrete as an expansive phase [4, 5]. Deleterious DEF is of most concern in the manufacture of precast concrete with the use of hot steam to accelerate the strength gain of concrete elements. The combination of external heating and internal heat of cement hydration has the potential to increase and sustain temperatures which prevent early ettringite formation, increasing risk of DEF [6-8]. Worldwide, reported cases of deleterious DEF are uncommon, which can be attributed to both the unlikely combination of necessary conditions (early heating, high temperatures, sulfate, etc.) and the limited availability of information regarding known cases [9-12].

Internal sulfate attack attributed to DEF was first reported in precast railway sleepers or ties subject to sustained heating in western Europe in the 1970's [13, 14]. Via electron microscopy, distinct needle-like

ettringite crystals were observed in microcracks and linked to deleterious expansion. Other investigations suggested that ASR was the culprit, with ettringite formation as a contributing and/or non-deleterious side-product [8].

From the microstructural view, DEF is characterised by the formation of a solid ettringite phase along the interfacial transition zone (ITZ) between the cement paste and aggregate particles. The expansive ettringite dislocates aggregates from surrounding paste material and creates tensile stress in the bulk concrete, with potential for microcracks and deleterious failure [8, 15].

In an Australian context, reported instances of DEF are rare, which is attributed to high local manufacturing standards of cement clinker and construction engineering [12, 16-18]. Local research by Shayan since the 1990's has highlighted the role of aggregate reactivity in DEF process and observed deleterious cracking [8, 16, 19, 20]. Recent work by Ramu has investigated the role of cement composition in mortars, with alkali content and molar ratio of SO_3/C_3A being dominant factors in ettringite formation [4, 17, 21, 22].

1.1.1 Early Ettringite Formation

Early ettringite formation (EEF) is a normal process in ordinary cement hydration (Equation 1). Sulfate (SO_3, \bar{S}) in the form of gypsum ($CaSO_4 \cdot 2H_2O, C\bar{S}H$) is inter-ground with clinker during the production of cement, which upon mixing with water will react with the aluminate phase ($(CaO)_3 \cdot Al_2O_3, C_3A$) to form ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O, C_6A\bar{S}_3 \cdot 32H$)



Ettringite acts to inhibit the initial hydration of aluminate and prevent the negative phenomena of 'flash-setting' from occurring in fresh concrete. As the bulk concrete material is still plastic at this stage, it can safely accommodate the additional expansive phase of ettringite [3, 5, 23].

1.1.2 Factors Influencing DEF

The primary factors of DEF can be separated into processing and reactant factors, depending on their relation to the formation of ettringite. Processing factors relate to the stability of ettringite and similar phases in the concrete material, which is significantly affected by the solubility of sulfate in the pore solution. They include; elevated alkali content, elevated temperature, and sustained heating. Reactant factors are associated with availability of reactant constituents for ettringite formation, their stoichiometric ratios, and the relative stability of related hydration products (e.g.; sulfo-aluminate and carbo-aluminate phases). They are principally the aluminate and sulfate content of the cement binder, and their respective molar ratios [10, 11, 18].

The pessimum composition of cements for deleterious DEF have been reported to be high alkali (1.00 – 1.25 % w/w Na_2O_{eq}), high sulfate (4.0 – 5.5 % w/w SO_3). For curing, conditions that promote DEF are extended time (≥ 8 hrs) at elevated temperatures (> 70 °C) [4, 18, 21]. These conditions are used in the design of relevant guidelines and practices, especially for the precast manufacturing industry. Many of these findings are based on laboratory experiments with mortar specimens, and thus may not be appropriate for true concrete elements [6, 17, 18, 20].

For hydrated cement, ettringite exists in an equilibrium with other species, specifically monosulfate phases (AF_m) and aqueous sulfate ions. When the pore solution is saturated with sulfate ions, ettringite formation is favoured. (Equation 2).



Increasing alkalinity and temperature increases the solubility of sulfate ions in pore solution and drives the decomposition of ettringite. Reducing either will in turn decrease sulfate solubility, and under saturated conditions will drive the re-precipitation of ettringite as a solid phase with potential for deleterious DEF [24-28].

1.1.3 Mitigation of Deleterious DEF

Mitigation of deleterious DEF and associated risk involves multiple strategies; binder content limits (alkali and sulfate content), temperature thresholds during precast manufacture, and the use of supplementary cementitious materials (SCMs) in the mix design [5, 10, 19]. Currently there are a limited number of

specifications within Australia that explicitly mention risk of DEF in concrete structures, such as Qld MRTS 70 and NSW T HR CI 12002 ST specifying core temperatures for concrete elements (precast or in-situ) be limited to < 75 °C.

1.2 Alkali-Silica Reaction

Alkali-silica reaction (ASR) is a chemical reaction in concrete involving alkaline pore solution and non-crystalline silica material, forming an expansive ASR gel phase with the potential for deleterious expansion, microcracking, and strength loss. Risk of deleterious ASR is of interest in all major concrete structures, as the slow progression of ASR can be undetected or misidentified by external inspection and is difficult to halt or reverse in damaged material [12, 29].

For ASR, the primary reaction is the dissolution of exposed silica by free hydroxide ions, with the alkaline pore solution serving as the reaction medium. Overall, this step is the breaking of weak $Si - O - Si$ bridge bonds and the dissolution of SiO_2 (Equation 2).



Following this is the formation of a silica sol, subsequent gelation, swelling of the ASR gel when exposed to water, and then potential for cracking and expansion [29] (Equation 4).



From the microstructural view, ASR is characterised by the formation of amorphous silica gel from within reactive aggregate particles, following with longitudinal cracks from aggregate material towards the cement paste. The expansive gel creates tensile stress in bulk material, with potential for to microcracks and deleterious failure.

1.2.1 Factors Influencing ASR

Primary factors of ASR involve alkali, reactive aggregates, and moisture [30]. Alkali species (Na, K) in the pore solution elevated hydroxide ion concentration which dissolve exposed silica, and become imbedded in ASR gel to increase its hygroscopic properties. Aggregates containing amorphous or semi-crystalline silica material supply silicon dioxide for ASR gel formation. Water acts as a reaction and transport medium and causes significant swelling in any formed ASR gel.

1.2.2 Mitigation of Deleterious ASR

Mitigation strategies for deleterious ASR include; alkali limits in the binder composition and concrete mix design, screening of reactive aggregates (as classified by local standards), and the use of SCMs in the mix design [31]. In an Australian context, SA HB 79: 2015 serves as a comprehensive starting resource for risk of ASR in concrete infrastructure, including strategies for initial design, monitoring and treatment of affected elements [32].

1.3 ASR & DEF

The combination of ASR and DEF in affected concrete structures can be synergetic, resulting in significant expansion and microcracking compared to ASR or DEF in isolation [7, 8, 10, 20]. This is attributed to the process of ASR gel formation consuming alkali species from the pore solution, reducing alkalinity and decreasing the solubility of sulfate towards the precipitation of solid ettringite [24, 28]. Additionally, minor cracking from either DEF or ASR can act to increase mobility of reactant species (Na, K, S) and allow water ingress, creating ideal conditions for more significant deleterious reactions to occur in the bulk material [5, 8, 10].

In Australia, multiple instances of deleterious ASR have been identified in both historical and modern structures [32]. By contrast, cases of deleterious DEF in Australia have only been reported in conjunction with mild or moderate ASR [12, 16]. Local research by Shayan from the 1990's onward has investigated the link between ASR and DEF in cementitious materials, with findings supporting the theory that ASR is the primary cause of cracking in concrete [8, 20].

1.3.1 Aims and Objectives

Worldwide reported research into deleterious DEF has focused on modified cement systems in mortar specimens, with industry guidelines for true concrete materials based on these findings. In Australia, cases of deleterious DEF have only been reported in conjunction with mild or moderate ASR. The role of the aggregate has been overlooked in the mechanisms of DEF in concrete and potential for deleterious expansion. Identifying the origins of DEF and ASR-influenced DEF in concrete will aid in the development for future guidelines for the manufacture of precast heat-cured concrete elements towards reducing costs and risk of failure. This paper, therefore, aims to investigate the process of DEF and identify the roles of ASR reactive aggregates, binder composition, and curing temperature in the susceptibility of concrete systems to deleterious DEF. The principal objectives of this work are to investigate the role of cement composition and curing conditions in promoting deleterious DEF in concrete with inert aggregates, and to investigate the role of ASR reactive aggregates on the susceptibility of concrete to deleterious DEF.

2. EXPERIMENTAL

2.1 Materials

For the manufacture of concrete specimens, locally sourced materials were used. The binder was an Australian supplied, commercially available, general purpose (GP) cement. Elemental oxide composition of the cement was determined by XRF analysis (Table 2.1). The phase content of the cement was estimated by modified Bogue's phase calculations (Table 2.2).

Table 2.1: Chemical Composition of GP cement, via XRF.

Component	L.O.I.	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	Na ₂ O	K ₂ O	Cl	Total	Na ₂ O _{eq}
Content (w/w %)	4.0	64.1	19.4	4.9	3.0	1.2	2.6	0.17	0.45	0.037	100.1	0.47

Table 2.2: Phase Composition of GP cement, via modified Bogue's phase calculation.

Phase	Alite (C ₃ S)	Belite (C ₂ S)	Aluminate (C ₃ A)	Ferrite (C ₄ AF)
Content (w/w %)	52.7	15.9	7.9	9.1

Potable tap water was used for cement mixing and preparation of saturated limewater. An alkali-free superplasticiser was used as admixture for sufficient workability during cement mixing. To promote deleterious DEF expansion, the chemical content of the cement was altered by addition of sodium hydroxide (*NaOH*) for alkali content to 1% w/w *Na₂O_{eq}* (4.5 kg/m³), and gypsum (*CaSO₄ · 2H₂O*) for sulfate content to 4% w/w *SO₃* (18 kg/m³), corresponding to previously reported pessimum for deleterious DEF [4, 21].

Aggregates used were selected for potential ASR reactivity. Coarse aggregate 1 was basalt material, and coarse aggregate 2 was dacite material. They were classified respectively as non-reactive and potentially reactive, according to AS 1141.60.2. Gradings of 20mm and 10mm were combined in a 3:1 ratio. Fine aggregate was a river sand and classified as non-reactive according to AS 1141.60.1.

2.2 Method

Concrete prisms of size 70×70×285mm were manufactured using AS 1012.2 as a guide for mixing and AS 1141.60.2 for design of prisms. The mix design used was based on common practices for the

Australian precast manufacture industry, specifically the high cement content, and low w/c ratio (Table 2.3).

Specimens were subject to two different curing conditions; ambient (23 °C) and heat-cured, with 3 prisms for each set (6 total). Heat-cured specimens were stored in sealed plastic bags with a damp cloth for moisture, placed in a programmable oven, then subject to a set temperature cycle of 4 hrs at 30 °C, heat ramp of 30 °C/hr to 90 °C, isothermal for 12 hrs at 90 °C and then cool to ambient (Figure 2.1). This cycle mimicked the internal temperature of a large precast concrete element [18].

After demoulding, prisms (ambient, heat-cured) were stored in separate limewater tanks at fixed temperature conditions (23 ± 1 °C). Linear length of prisms was measured at day 1, day 7 (as reference point), day 28, then monthly thereafter, using AS 1141.60.2 as a guide.

Compressive strength tests were carried out at 28-days (100×200mm cylinders, ×2 samples) and 1-year (75mm cubes, ×2 samples, cut from measured prisms) according to AS 1012.9. Cylinders were stored in limewater at 23 °C.

Table 2.3: Concrete mix design

Property	Cement content	Coarse aggregate	Fine aggregate	w/c ratio	Batch size	Slump
Detail	450 kg/m ³	1190 kg/m ³	640 kg/m ³	0.40	100 kg	120 mm

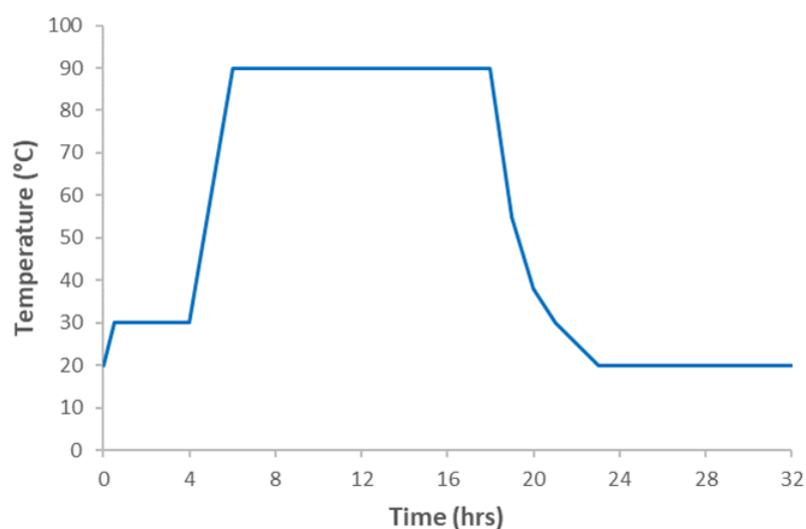


Figure 2.1: Set temperature program of concrete specimens during heat-curing. This cycle was designed to mimic the internal temperature profile of a large precast element. The heat-curing stages were: pre-set (4 hrs, 30 °C), ramp (30°C/hr up to 90 °C), soak (12 hrs, 90 °C), cool and demould, then store at ambient (23 °C).

3. RESULTS AND DISCUSSION

Concrete specimens were manufactured as described, with variation of conditions of binder (control or cement as received vs. pessimum composition), aggregate (non-reactive vs. reactive), and curing conditions (ambient vs. heat-cured) as relevant factors for ASR, DEF, and ASR-DEF. Linear expansion of prisms were monitored over 1-year, and compressive strength was measured at 28-days and 1-year.

For the concrete systems investigated, the naming scheme is as follows. “DEF-only” systems only have inert or non-reactive aggregates, and thus have limited ASR reactivity. “ASR-DEF” systems have reactive aggregates present, and thus have potential for ASR to occur. “Pessimum” systems had the

chemical composition of the binder elevated to 1.00% Na_2O_{eq} (N) and 4.0% SO_3 (S), while “Control” systems used the cement binder as received with 0.47% Na_2O_{eq} and 2.6% SO_3 (complying to the alkali and sulfate limits used in Australia).

An arbitrary threshold for deleterious expansion was set at 0.03% of total linear length, based on the conventional Australian accelerated ASR aggregate reactivity test methods (AS 1141.60.2). This threshold is conservative compared to other international standards, including ASTM (ASTM C1293, 0.04%) and RILEM (RILEM AAR-3, 0.05%). Note that the method used varied from conventional concrete prism test methods, with significant changes in environment (samples were immersed in water at 23 °C) and binder alkali content ($1.00\% < 1.25\% Na_2O_{eq}$).

3.1 Role of Alkali, Sulfate, and Temperature in DEF in Concrete

Concrete specimens were manufactured with conditions to promote deleterious DEF related to curing (ambient vs. heat-cured) and chemical composition of the binder (control or as received vs. pessimum or elevated alkali and sulfate). Linear expansion of prisms was monitored over 1-year (Figure 3.1).

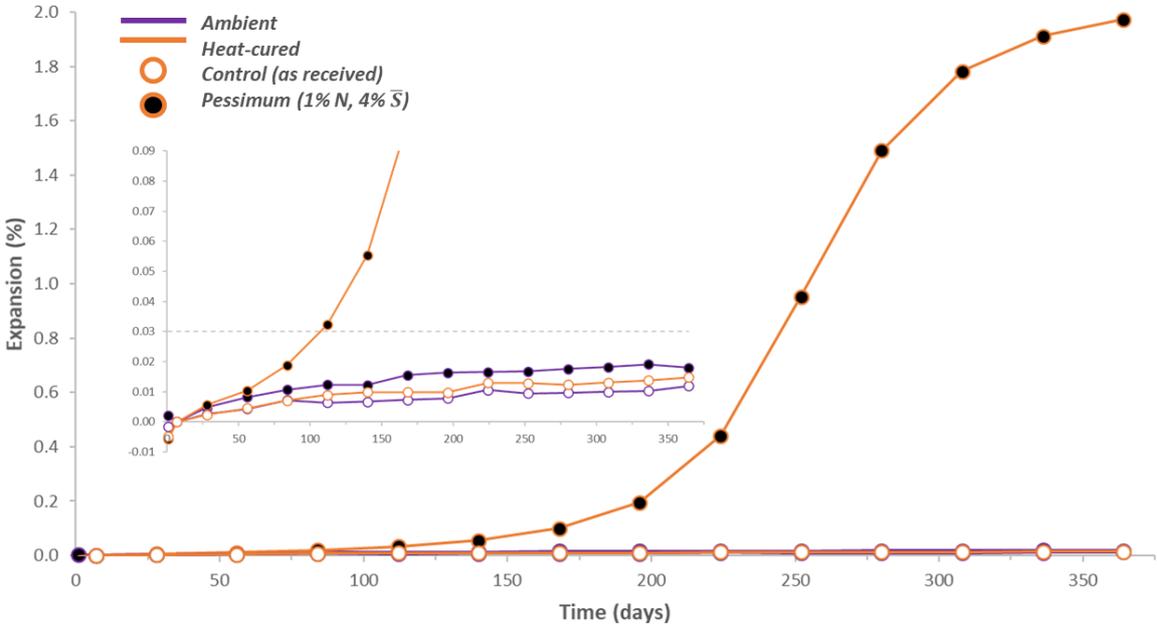


Figure 3.1: Linear expansion of concrete prisms with non-reactive aggregates, and insert (middle left) of small scale. Concrete systems with pessimum conditions (1% alkali, 4% sulfate) and heat-curing showed deleterious expansion. Expansion in all other systems was non-deleterious after 1-year.

The only concrete systems that showed deleterious expansion were those subject to pessimum conditions and heat-curing, which is attributed to DEF. All other concrete systems (ambient cure/control, ambient cure/pessimum, heat-cured/control) did not show deleterious expansion. This supports the theory that temperature, elevated alkali, and elevated sulfate are all essential factors for deleterious DEF to occur, and without these conditions present the associated risk is low.

Observed expansion for prisms with pessimum binder conditions and subject to heat-curing was significant, reaching an overall change of $\approx 2.0\%$ of total length at 1-year. This expansion slowed in the last few months of measurement but has not stopped and is expected to continue beyond the 1-year mark. The amount of overall expansion is severe but comparable to reported instances of deleterious expansion associated with DEF, ranging from 1.2 – 1.8% [9, 20, 21]. From the smaller view (Figure 3.2 insert), expansion was early (100 – 150 days) and large ($> 0.09\%$), similar to other laboratory studies of DEF [11, 19, 21], which is attributed to the rapid (≥ 90 days) re-precipitation of expansive ettringite from the pore solution in hardened bulk concrete material [4, 13].

For non-deleterious systems, the order of expansion was (from highest to lowest); heat-pessimum > ambient-control > heat-control. The causes of this are linked to the effects of increased alkali (increasing water uptake) and sulfate (gypsum as an offset to early shrinkage) [1, 3], with minor contribution increased porosity in heat-cured specimens compared to ambient, as expected with precast concrete [4, 8].

3.2 Role of Aggregate Reactivity in DEF in Concrete

Concrete specimens with potentially reactive aggregates were manufactured with conditions to promote deleterious ASR-DEF related to curing (ambient vs. heat-cured) and chemical (control or as received vs. pessimum or elevated alkali and sulfate). Linear expansion of prisms was monitored over 1-year (Figure 3.2). The full expansion of concrete specimens was also compared between systems with and without reactive aggregates (Figure 3.3).

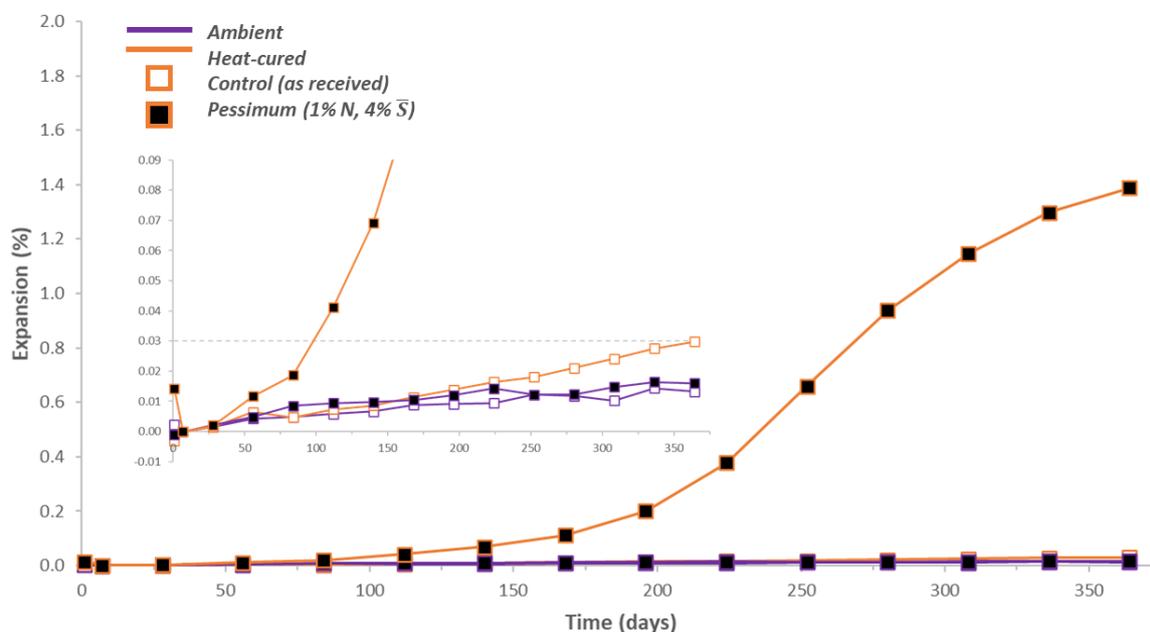


Figure 3.2: Linear expansion of concrete prisms with reactive aggregates, and insert (middle left) of small scale. Concrete systems with pessimum conditions (1% alkali, 4% sulfate) and heat-curing showed deleterious expansion. Expansion in all other systems was non-deleterious after 1-year, but heat-cured control was borderline.

Similar to the previously discussed DEF-only systems, only concrete specimens subject to heat-curing and pessimum conditions showed deleterious expansion and strength loss. The initial expansion profile was also early and large, with no significant differences between systems occurring until 225-days, where the DEF-only overtakes the ASR-DEF. The overall expansion was less than the non-ASR system, up to 1.4% at 1-year, and similarly the rate of expansion has slowed but is still ongoing.

One potential cause for this difference is the progression of ASR slowing the DEF process in affected concrete specimens. The reactive silica present could be acting as a weak SCM, slowing transport of reactants by pore blocking, dispersing formed ettringite away from the ITZ, and creating micro-voids for ettringite to precipitate freely without restraint. Literature concerning the efficacy of SCMs such as fly ash to mitigate deleterious DEF [18], and the potential use of finely ground reactive aggregate as SCM [31, 33] give evidence to this explanation.

The heat-cured with control (binder as received, without alkali and sulfate addition) did show expansion, but was but not crossing the deleterious threshold (0.03%) at 1-year.

The heat-cured with control system did show slow expansion, but as this change did not cross the 0.03% threshold at 1-year, it was classified as non-deleterious. As this expansion is still ongoing, it is expected it will cross the deleterious limit beyond the 1-year mark. This expansion was attributed to increased porosity in the heat-cured concrete, allowing water transport of alkali species and the formation of expansive ASR gel from available aggregate material.

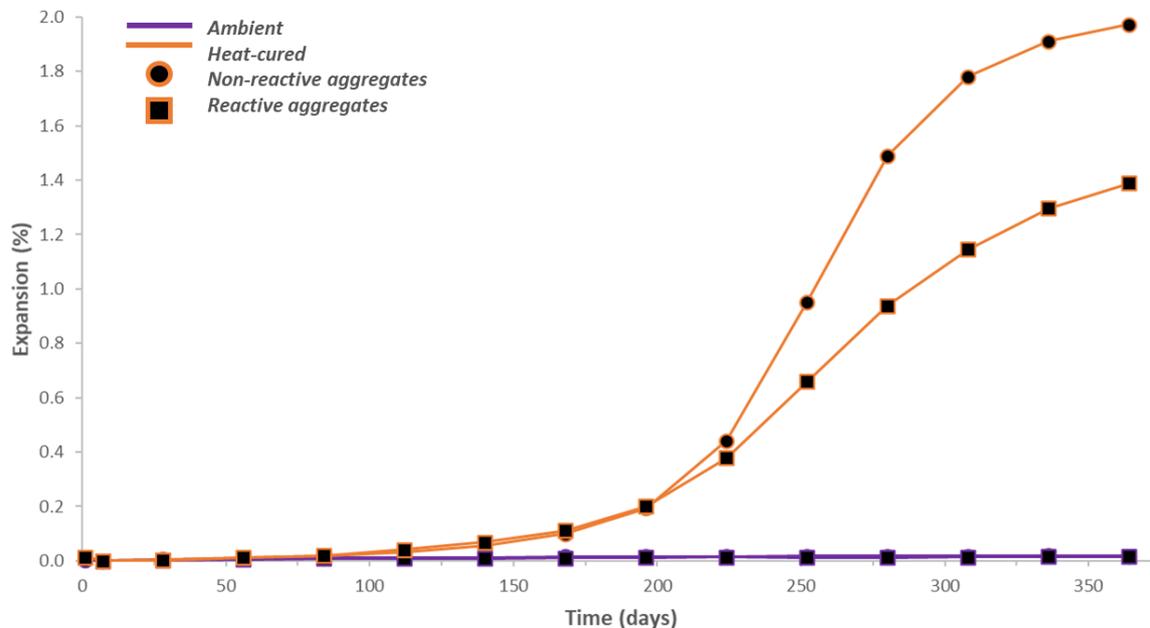


Figure 3.3: Linear expansion of concrete prisms, comparing systems with and without ASR-reactive aggregates, in the presence of elevated alkali and sulfate content. Only concrete systems with heat-curing and pessimum conditions showed deleterious expansion, all other systems did not. ASR and non-ASR systems showed similar early expansion, but the DEF-only system reached 2.0% while the ASR-DEF system reached a lower 1.5% at 1-year. Expansion for both systems has slowed but is still ongoing at 1-year.

3.3 Influence of ASR & DEF on Concrete Strength

Compressive strength of specimens was tested at 28-days and at 1-year (Figure 3.4). For concrete systems with heat-curing and pessimum conditions, observed strength loss was severe and correlated with deleterious expansion. For 28-day measurements, strength was marginally lower in all heat-cured specimens compared to corresponding ambient specimens. This was expected, as precast manufacture of concrete sacrifices late strength (≥ 28 -day) for early strength (1-day) [1, 7].

For DEF-only system (without reactive aggregate) with deleterious expansion (heat-cured with pessimum) the observed strength change was in decline, going from 45.0 MPa at 28-days to 24.0 MPa at 1-year (-42% gain). This is a severe loss of strength when compared to counterpart systems which all had observed gains in compressive strength from 28-day to 1-year, for the ambient with pessimum (from 54.0 to 78.5 MPa, 48% gain) and heat-cured with control (from 33.0 to 55.5 MPa, 68% gain).

Similarly, for the ASR-DEF system (with reactive aggregate) with deleterious expansion (heat-cured with pessimum), the strength loss was a change of 43.0 MPa at 28-days to 37.5 MPa at 1-year (-13% gain). This lesser decline when compared to the DEF-only strength loss corresponds to the differences in the amount of expansion observed in both deleterious systems. For the counterpart ASR-DEF systems, the changes were towards increasing strength, for ambient with pessimum (from 57.5 to 80.0 MPa, 39% gain) and heat-cured with control (from 46.0 to 76.0 MPa, 65% gain).

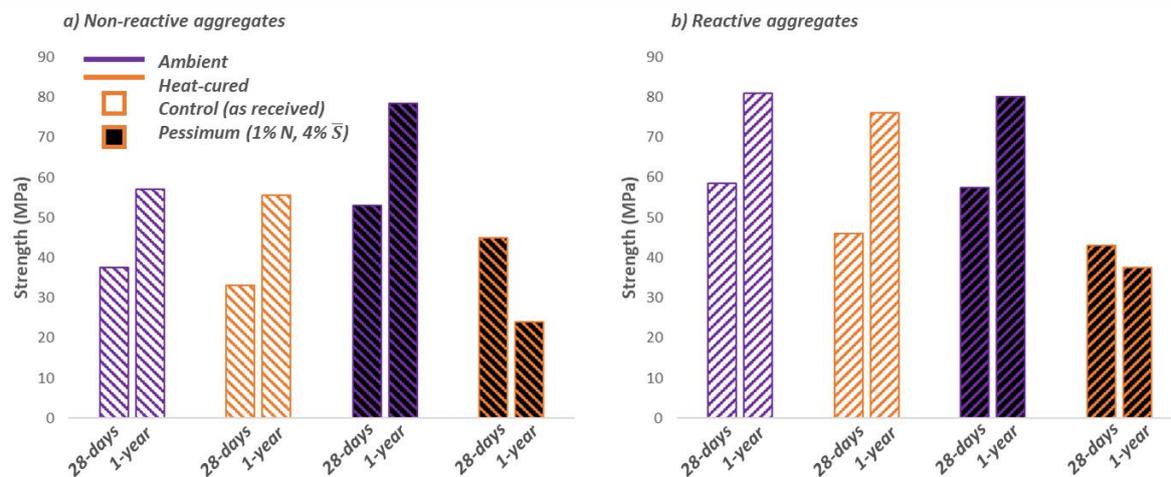


Figure 3.4: Compressive strength of concrete specimens. Comparing non-reactive aggregates (a) with reactive aggregates (b), and tests at 28-days (left bar) with 1-year (right bar). Significant strength loss at 1-year was observed in concrete systems with heat-curing and pessimum conditions. All other systems had acceptable strength gain.

4. CONCLUSIONS

An investigation of the role of alkali-silica reaction in the susceptibility of concrete to delayed ettringite formation has been done by experimental study with concrete systems. The role of alkali, sulfate, temperature and aggregate reactivity have been assessed with regards to deleterious DEF and ASR-DEF in concrete prisms. The key findings of this investigation are presented as follows.

Deleterious DEF is not observed in concrete prisms prepared with Australian produced cement, which is attributed to low alkali and sulfate contents. Cement or binder systems with elevated alkali and sulfate contents increase the risk of deleterious DEF. A combination of binder compositions, of elevated alkali (1% $\text{Na}_2\text{O}_{\text{eq}}$) and sulfate (4% SO_3) contents, and curing conditions, of high temperature (90 °C), sustained heat-curing (12 hrs), are necessary for deleterious DEF to occur in concrete. Concrete systems without these conditions did not exhibit deleterious expansion after 1 year of monitoring.

Aggregate reactivity influences expansion due to DEF, but only in the presence of heat-curing and pessimum conditions. Observed expansion was early and large in both ASR-DEF and DEF-only systems. At 1-year, the DEF-only system showed more expansion than the ASR-DEF system, with both significantly more than non-deleterious systems without heat-curing or pessimum conditions. Significant loss of compressive strength was observed in ASR-DEF and DEF-only concrete systems with deleterious expansion.

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