OBSERVATIONS ON USING EXPANDED CLAY TO CONTROL THE EXPANSION CAUSED BY ALKALI-SILICA REACTION

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Abstract

Previous research indicated that mortar and concrete mixtures showed less expansion caused by alkali-silica reaction (ASR) when fine lightweight aggregates (FLWAs) were incorporated into the mixtures. However, these results were limited, and conflicting mechanisms and theories exist. Therefore, a research program was initiated to explore the mechanism(s) by which FLWA may mitigate ASR. In this program, one commonly used FLWA, an expanded clay, was incorporated. Tests methods including the ASTM C289 test, the ASTM C1260 test and the ASTM C1293 test were used to investigate the effectiveness of the expanded clay in ASR mitigation when combined with aggregates of known reactivity. In these mixtures, fine normal weight aggregate were replaced by the expanded clay at 25% and 50% by volume, which are the replacement levels typically used for internal curing. Preliminary results indicated that the expanded clay showed effectiveness in mitigating ASR in the ASTM C1260 test when they were in either pre-wetted or oven-dried conditions. However, more expansion was observed when the expanded clay was pre-wetted. Pore solution analysis results indicated that the expanded clay in the mixture with Spratt could reduce the alkalinity of the pore solution, especially the mixture showed reduction in alkalinity even at early ages (7 days to 28 days). SEM analysis revealed that infilling reaction products were deposited into the pores of the expanded clay. More investigation is still needed to understand the composition of the reaction products deposited into the pores of the expanded clay.

Keywords: ASR, expanded clay, ASTM, expansion, infilling reaction product

1   INTRODUCTION

Fine lightweight aggregates (FLWAs) are manufactured by heating raw materials under high temperature (1100 to 1200 °C), by which the carbonaceous minerals in raw material are decomposed and released to form an internal porous structure. Due to this specific structure, FLWAs have a high absorption capacity and low specific gravity. Because of these two features, FLWAs are usually applied to reduce the load of the structures or be used for liquid storage. When being used in concrete, FLWAs can be pre-wetted before mixing in the concrete to provide internal water inside the concrete, thus preventing shrinkage and refining microstructure of the concrete [1-4]. Due to the benefits provided by FLWAs and internal curing technique, concrete shows better performance in chloride and moisture intrusion resistance as well as ASR mitigation. Current research indicated that with FLWAs incorporated in mortar and concrete mixture, reduction in the ASR-caused expansion was observed [5-8]. These findings revealed that incorporating FLWAs in concrete can be used as a possible strategy to mitigate ASR.

Previous studies discussed several possible mechanisms, by which FLWAs can be used to mitigate ASR. However, there was a lack of agreement on the dominant mechanism. According to these studies, a few assumptions were proposed. Shin et al. showed that expansion caused by ASR was reduced when expanded shale, expanded clay and expanded slate were used to partially replace the reactive aggregates in mortar mixture [5]. Therefore, it was suspected that the expansion was reduced due to the reduction of reactive aggregate in mortar mixture. Collins et al. [9] proposed that the internal curing water in the porous aggregate could be used to dilute the pore solution in concrete. Research by Elsharief [1] indicated that a refinement of the interfacial transition zone (TTZ) was observed in the mortar mixture with either the oven-dried expanded shale or the pre-wetted expanded shale. This observation supported the finding that

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expanded shale can benefit concrete in limiting moisture and chemical intrusion [5, 10, 11]. Therefore, due to this refinement of concrete microstructure, less moisture intrusion could help with controlling moisture absorption by ASR gel. Boyd et al. [7, 12] found that the expanded shale in concrete were filled up with reaction products, which was likely to be ASR gel deposit. This observation indicated that the porous structure of the expanded shale provided space to accommodate ASR gel, thus offset the potential expansion. The pozzolanic reactivity of expanded shale and expanded clay were also investigated by grinding to micro scale. Due to the high content of silica and alumina, ground expanded shale and expanded clay showed a superior pozzolanic reactivity in mortar/concrete mixtures [5, 13, 14]. These findings could also be supportive evidences to explain why FLWAs can mitigate ASR.

However, these research results and assumptions were obtained from limited experimental tests, especially most of the results were from ASTM C1260 tests and very limited ASTM C1293 tests. The lack of data support led to some unexplained results and confounding explanations on how FLWAs can mitigate ASR in concrete. Therefore, if we want to use FLWAs as a potential strategy to mitigate ASR, a more comprehensive research program is needed to illustrate if FLWAs can be used for ASR mitigation as well as the mechanism behind it. In this study, one of the commonly used FLWAs, an expanded clay was used for investigation.

2   RAW MATERIALS
Cement
    The cement used in this study was from Montreal, Quebec, Canada. This cement was used in both ASTM C1260 and ASTM C1293 tests. Table 1 shows the chemical composition of the cement.

FLWAs
    One commercially used FLWA (expanded clay) was used in this study. The material properties are exhibited in Table 2.

Other aggregates
    Two coarse aggregates (CA1 and CA2) and two fine aggregates (FA1 and FA2) were used to create a comprehensive mixture matrix. The type, reactivity and source of each aggregate were listed in Table 3.

3   EXPERIMENTAL METHODS
ASTM C289
    ASTM C289 was used to evaluate the reactivity of a specific aggregate by immersing ground aggregates in 1.000 N NaOH. The aggregate was crushed and ground into particles of 150 μm ~ 300 μm. Prepared aggregates were immersed in 1.000 N NaOH for 24 hours at 80±1 °C, and then the dissolved silica and alkalinity reduction were measured for its potential ASR reactivity. CA1, FA1, FA2 and the expanded clay were tested.

ASTM C1260
    ASTM C1260, referred as the acerbated mortar bar test (AMBT), is a quick reactivity test on aggregates in mortar bars [15]. Mortar bars with a dimension of 25 mm × 25 mm× 285 mm were cast. The water-to-cement ratio was fixed at 0.42, and the sand-to-cement ratio was 2.25. The mortar bars were cured at an ambient temperature for 24 hours and then transferred to the DI water at 80°C for another 24 hours. Subsequently, the mortar bars were exposed to 1.0 N NaOH at 80°C. Length change of the mortar bars was periodically tracked up to 14 days afterwards. The threshold for reactivity evaluation is 0.10%. Due to the high absorption capacity of expanded clay, it was pre-conditioned in two methods: pre-wetting and oven-drying. The pre-wetted and the oven-dried expanded clay were used to replace the normal weight aggregate by 25%, 50% and 100% in volume respectively. Mixture with 100% oven-dried expanded clay was not cast due to the cohesive issue with the mortar. Table 4 shows the mixture matrix.

ASTM C1293
    ASTM C1293, which is also referred as the concrete prism test (CPT), is a test on aggregate reactivity in concrete prism. In each mixture, the reactive proportion is either a coarse or a fine aggregate (based on the results of ASTM C1260) [16]. Therefore, the ASR reactivity of this specific aggregate can be evaluated. For this test, concrete prisms in a dimension of 75 mm × 75 mm × 285 mm were cast. After curing for 24

hours, the prisms were transferred to a bucket with water on the bottom. Then the bucket was placed in a chamber at 38°C. Length change of concrete prisms were measured periodically up to one year (two years for mitigation test). By using the aggregates listed in Table 3. There are two combinations used for the control group as listed in Table 5. In addition, for each control mixture, the expanded clay was used to replace the fine aggregates at 25% and 50% replacement level in volume.

Pore solution alkalinity analysis

Pore solution analysis was used to evaluate how FLWAs influence the pore solution chemistry in the concrete mixtures. Samples for pore solution analysis were obtained from ASTM C1293 prisms, and the samples were cut from one prism subjected to the same environmental conditions as other concrete prisms, which were measured periodically (at the age of 7 days, 28 days, 84 days, 180 days and 365 days). Afterwards, samples were manually crushed into particles smaller than 2.38 mm (U.S. No.8 sieve size). Aggregates were picked out of the crushed sample and then the material left were introduced to a compression system. After being applied with a high pressure, the squeezed liquid was collected, and the pH was measured within four hours of extraction.

SEM analysis

SEM analysis was used as an option to examine the microstructure of the mortar with the expanded clay. Backscattered images of polished sections were acquired for observation. Through SEM analysis, samples were observed for: 1) The formation and origination of ASR gel; 2) Change of pore structure of the expanded clay; 3). If the ASR gel were deposited into the pores of the FLWAs.

An FEI QUANTA 600F environmental SEM was used for the analysis. Samples for analysis were all 5 mm thick, which were cut by a precision saw from mortar bars cast in ASTM C1260. These samples were soaked in an isopropyl alcohol for 72 hours with a solution change after first 24 hours to arrest the continuation of the hydration [17]. After being stabilized in epoxy, the samples were subsequently roughly polished with a #500 sandpaper to expose the surface of the mortar, and then finely polished for 8 hours with 9μm, 3μm, and 1μm micro silicon grit. Meanwhile, the dispersion oil-based lubricant was applied.

4   RESULTS
4.1 Mechanical properties

Compressive strength was tested for the mixtures in different groups. Figure 1 shows the average compressive strength of three tested specimens in each mixture. In the CA2-FA1 mixture, the compressive strength of the control was 5480 psi. The mixtures with 25% expanded clay was slightly lower than that of the control, and the mixture with 50% expanded clay showed a very similar compressive strength with the control. For the CA1-FA2 mixture, the compressive strength of the control was 5430 psi. Similarly, a slight reduction on compressive strength was observed on the concrete specimens with 25% expanded clay. No significant differences in compressive strength was observed on the concrete specimens with 50% expanded clay when comparing with the control.

4.2   ASTM C289 test results

Results of ASTM C289 test are shown in Figure 1. In the test, CA1, FA1, FA2 and expanded clay were tested. According to the test results, CA1 and FA2 were all categorized as non-reactive and FA1 was categorized as potentially reactive. However, based on results of ASTM C1260 test and ASTM C1293 test, both CA1 and FA1 were categorized as reactive. Therefore, ASTM C289 failed to detect the reactivity of CA1 and underestimate the reactivity of FA1. Compared to the FA1, expanded clay showed a more significant reduction in alkalinity while a similar amount of dissolved silica was detected. Therefore, if the fine aggregates in concrete/mortar were partially replaced by expanded clay, it can be expected that a higher amount of available alkalis to be consumed. Although the ASTM C289 test did not accurately predict the reactivity of aggregates investigated, the amount of alkali consumption of expanded clay was evaluated. This finding could be used to indicate the alkali binding ability of the expanded clay.

4.3   ASTM C1260 results

Results of ASTM C1260 test are shown in Figure 2. For the control mixture, with 100% very highly reactive fine aggregate, an expansion of 0.66% was observed. As described in section 2, the expanded clay was pre-conditioned in two ways: pre-wetted and oven-dried. In both mixture groups, it showed a reduction

in the ASR-caused expansion. It was also noticed that the expansion decreased with the increase of the expanded clay proportions. The mixtures with 100% pre-wetted expanded clay showed an expansion below the 0.10% threshold, which indicated the expanded clay is not ASR-reactive.

In addition, mixtures with pre-wetted expanded clay showed more expansion than the mixtures with oven-dried expanded clay. This difference was observed in all replacement levels, except the mixture with 100% oven-dried expanded clay (not able to be cast). There are two possibilities to explain this observation: 1). The internal water residing in the expanded clay due to pre-wetting were released from the pores and used by ASR gel for moisture absorption; 2). The oven-dried expanded clay can intake the alkalis into its pores, while in the mixture with pre-wetted expanded clay, the pores were occupied by the internal water. Therefore, the alkali binding and absorption ability was impaired.

Results indicated that by using ASTM C1260 test, the efficacy of the expanded clay in controlling ASR expansion can be evaluated. However, the pre-conditioning methods of expanded clay can significantly influence the expansion of mortar bars. The role of internal water is still in need to be investigated.

4.4   ASTM C1293

In Figure 3, the length change of concrete prism with expanded clay up to one year is shown. Figure 3(A) shows the mixture with non-reactive coarse aggregate with very highly reactive fine aggregate. The expansion at one year was 0.61% for the control group. With 25% expanded clay incorporated, the expansion was reduced to 0.33%. Further, with 100% expanded clay, the expansion was reduced to 0.07%. In general, the expansion reduced by 89.4% when 50% expanded clay was incorporated. Although the expansion of mixture with 50% expanded clay was still beyond the 0.04% threshold, it was significantly reduced compared with the control mixture.

In Figure 3(B), it shows that the expansion change of concrete mixtures with a highly reactive coarse aggregate and a non-reactive fine aggregate. An expansion of 0.15% was observed for the control mixture at one year. Concrete mixture with 25% expanded clay showed an expansion of 0.09% at one year, and 0.03% for the mixture with 50% expanded clay. In total, a reduction of 81% in the expansion was observed when 50% expanded clay was incorporated. In this group, the mixture with 50% expanded clay showed the expansion lower than the reactivity threshold, which indicated ASR was mitigated by using expanded clay. However the test results up to two years is still desired to verify if 50% expanded clay can mitigate ASR for a longer term.

Through ASTM C1293 test, expanded clay was very effective in reducing ASR-caused expansion by replacing either reactive portion or non-reactive portion in concrete. This also indicated that ASR was controlled by expanded clay due to multiple mechanisms. Based on what was found, aggregate dilution effect is not sufficient to explain the reduction in the expansion when a non-reactive portion was replaced.

4.5   Alkalinity of pore solution

Samples of extracted pore solution were obtained from the concrete prisms in ASTM C1293 test. The pH of pore solution of both concrete groups was shown in Figure 4. It was interesting to notice that the pH of pore solution was increased when the expanded clay was used to replace the reactive portion in the concrete mixture (CA2-FA1). However, in the mixture with Spratt (CA1-FA2), the alkalinity reduction was observed when expanded clay were incorporated. This reduction in pH was observed when the non-reactive portion was replaced. More alkalis were consumed with a higher dosage of expanded clay, which was indicated by a decrease of pH from 13.3 to 12.7. However, different as expected, a reduction in pH was not observed when more expanded clays were incorporated in the mixture with very highly reactive fine aggregate (CA2-FA1). The reason of this increase in pH still remains unknown.

4.6 SEM analysis

Samples for SEM analysis were obtained from mortar bars cast in ASTM C1260 test. Figure 5 shows a reactive particle in the control mixture with the ASR gel inside the aggregate after a 14-day exposure to 1.0 NaOH and 80°C. Due to the high reactivity of FA1, the ASR gel was observed in both cement paste and reactive aggregates. The gel formed when alkalis in pore solution reacted with the reactive silica in aggregates. As seen in Figure 5, when the ASR gel formed on the surface of the aggregate, it caused the cracks of aggregates by swelling due to the water absorption. Then the gel exuded into cement paste and caused the cracking of cement paste, further led to the expansion of the mortar bars (As seen in Figure 2).

In Figure 6, images for the mortar mixture with 100% pre-wetted expanded clay are shown. Figure 6(A) shows an expanded clay particle of 300~500 μm. It was observed that from the rim of the particle, many of the pores in this particle were filled up with reaction products. Due to the infill of the reaction products, the ITZ between the expanded clay and cement was minimal as what was observed by Elsharief [1]. However, some microcracks within the expanded clay particle were observed. In Figure 6(B), an expanded clay particle of 100~150 μm was observed. In this particle, the majority of the pores were filled up with this infilling reaction product. The expanded clay were well bound with cement paste around it. No microcracks were observed in the expanded clay particle.

5   DISCUSSION

Mechanical properties

Concrete specimens of mixture with 25% expanded clay showed slight reduction on compressive strength when comparing with the control. However, this reduction was not observed in the mixture with 50% expanded clay. When lightweight aggregate were used in the concrete, an increase in modulus of elasticity was reported in the research by Al-Khaiat and Haque [19]. Based on this finding, it is likely that with the incorporation of the expanded clay, the decrease of modulus of elasticity helped with the improvement on resistance to the potential expansion caused by ASR.

ASR mitigation efficacy

According to the results of ASTM C1260 test and ASTM C1293 test, expanded clay showed effectiveness in controlling expansion caused by ASR. In the ASTM C1260 test, neither of 25% nor 50% replacement level were able to reduce the expansion to below the threshold when either pre-wetted expanded clay or oven-dried expanded clay were incorporated. Only 100% pre-wetted expanded clay successfully controlled the expansion caused by ASR. In addition, the pre-conditioning method of the expanded clay can significantly influence its ASR mitigation efficacy. It was likely that the internal water residing in the pores of expanded clay reduced the chances of alkalis being bound and absorbed by the expanded clay particles. In addition, in the ASTM C1293 test, neither of 25% nor 50% replacement level could reduce the expansion below 0.04% when the expanded clay was incorporated in the mixture with CA2-FA1 (very highly reactive). But for the concrete mixture with CA1 (Spratt)-FA2, by incorporating 50% expanded clay, concrete prisms showed expansion below 0.04%. In both groups, expanded clay showed similar efficacy in reducing the expansion at 50% replacement level (89% vs. 81%). Therefore, the mitigation efficacy was largely determined by the ASR reactivity of the reactive aggregates in concrete. It is likely that by incorporating 50% expanded clay, the ASR caused by moderate reactive aggregate (0.04%~0.12% expansion in ASTM C1293) or some of highly reactive aggregate (0.12%~0.24% expansion in ASTM C1293) can be fully mitigated [18]. For aggregates with higher reactivity, a different mitigation technique might be suggested.

Alkalinity reduction

Expanded clay exhibited high alkali consumption when compared with the two reactive aggregates used in this study, CA1 and FA1. This was also observed in the pore solution of concrete mixture with CA1-FA2. A very clear reduction in alkalinity was observed. Collins indicated that the pore solution dilution can be caused by the internal water residing in the pores of FLWAs [9]. However, based on the observation in this study, this assumption was less likely, as a more severe expansion was observed when the expanded clay were pre-wetted. One possible explanation for this observation could be the pozzolanic reactivity between the expanded clay particles and alkalis in cement paste. Therefore, the alkalis were bound in the C-S-H due to the reaction. However, more studies are still needed to validate this assumption. On the other hand, the fact that alkalinity in pore solution of concrete mixture with CA2-FA1 (very highly reactive) increased with the rise of replacement level remains unknown.

Infilling reaction products

According to the observations in SEM analysis. Infilling reaction products were observed in mortar bar samples with 100% expanded clay. This phenomenon was also observed in previous studies [6-8]. In these studies, the reaction product was claimed as the ASR gel, which deposited in the pores of FLWAs. Based on the observations in Figure 6(A) and (B), the reaction product seemed to have a very strong bond with the expanded clay particle, which might be caused by the dissolving of the expanded clay. If this is true,

there could be two explanations for this observation: 1) the infilling products are the ASR gel filled into the expanded clay. However, the ASR gel continued to react with expanded clay particle until the pores of the particle were filled up; 2) the formation of the infilling reaction product was due to the reaction between expanded clay particles, the alkalis in the pore solution and surrounding cement paste, and then the reaction products grew and agglomerate until the pores of the expanded clay were filled up.

6   CONCLUSION REMARKING

In this study, an expanded clay was used to replace the fine normal weight aggregates in both mortar and concrete mixtures. By incorporating 25% and 50% expanded clay in volume, expansion caused by ASR was reduced in both mortar and concrete specimens. The applicability of current ASTM standards was evaluated and test results were compared with previous studies. In addition, these results were also used to evaluate the possibility of assumptions proposed in previous studies. A few conclusions were drawn based on the findings:

1) Expanded clay were effective in reducing the expansion caused by ASR in both mortar and concrete specimens.

2) The efficacy of the expanded clay could be determined by the methods of pre-conditioning, reactivity of the aggregates and replacement level.

3) Expanded clay can consume alkalis in pore solution of the concrete mixture when the non-reactive fine aggregate was replaced.

4) Infilling reaction products were found in the pores of expanded clay particles, and the expanded clay particles were well bound with the infilling reaction products as well as surrounding cement paste.

7   REFERENCES

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TABLE 1: Major chemical composition in cement (%).

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	Na_2O_{eq}	SO_3	LOI
19.61	4.38	2.76	62.21	2.72	0.28	0.84	0.83	3.76	2.60

TABLE 2: Materials properties of FLWAs used.

FLWAs	Absorption capacity (%)	Specific gravity
Expanded clay	30.75	1.07

TABLE 3: Coarse and fine aggregates used in the study

	ID	Type	Reactivity	Source
Coarse aggregate	CA1	Crushed limestone (Spratt III)	Highly reactive	Ontario, Canada
	CA2	Crushed limestone	Non-reactive	Washington, USA
Fine aggregate	FA1	Siliceous river sand	Very highly reactive	Oregon, USA
	FA2	Crushed limestone	Non-reactive	Texas, USA

TABLE 4: Mixture matrix for AMBT (6 mixtures including one controls. Mixture with 100% oven-dried expanded clay was not cast due to the low flowability)

	Aggregate	FLWA	Treatment	Replacement level		
Control	FA1	N/A	Oven-dried	-		
1	FA1	Expanded Clay	FA1: Oven-dried FLWAs: Pre-wetted	25%	50%	100%
2	FA1	Expanded Clay	FA1: Oven-dried FLWAs: Oven-dried	25%	50%	-

TABLE 5: Mixture matrix for CPT

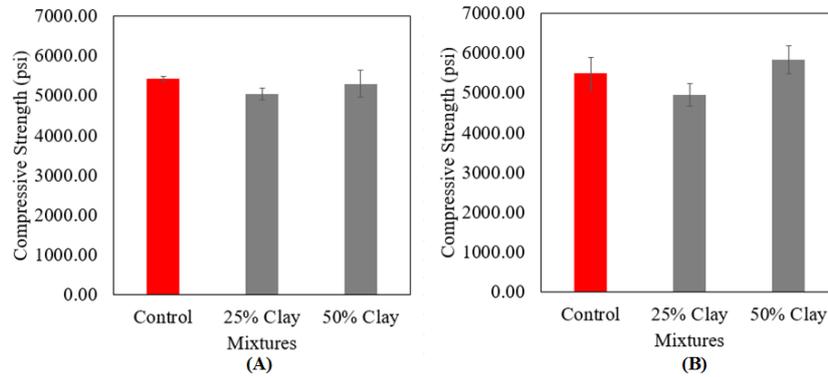|	Coarse aggregate		Fine aggregate	
Group	Aggregate type	Reactivity	Aggregate type	Reactivity
1	Spratt III (CA1)	Highly reactive	Crushed limestone (FA4)	Non-reactive
2	Crushed limestone (CA3)	Non-reactive	Siliceous river sand (FA1)	Very highly reactive

FIGURE 1: Compressive of concrete specimens: (A). Non-reactive limestone coarse aggregate with very highly reactive fine aggregate; (B). Highly reactive coarse aggregate (Spratt) with non-reactive limestone.
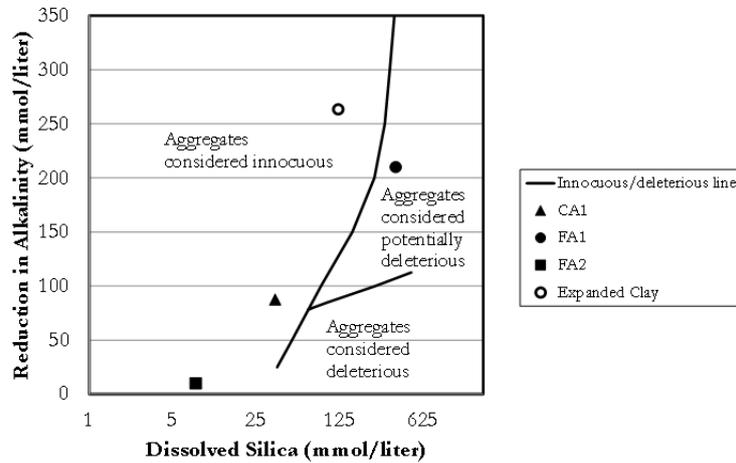
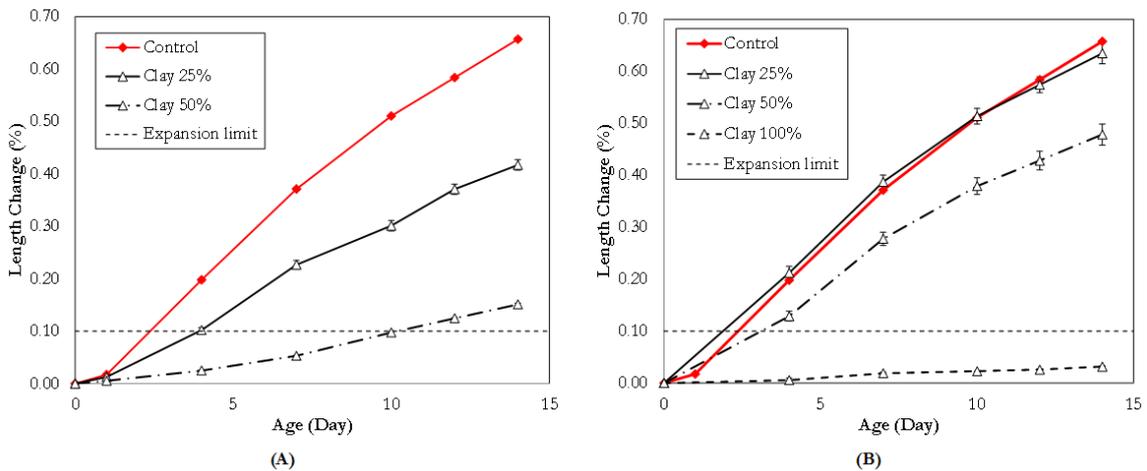

FIGURE 2: Results of ASTM C 289 test.

FIGURE 3: Results of ASTM C 1260: (A). FLWAs were pre-wetted before mixing; (B). FLWAs were oven-dried before mixing.
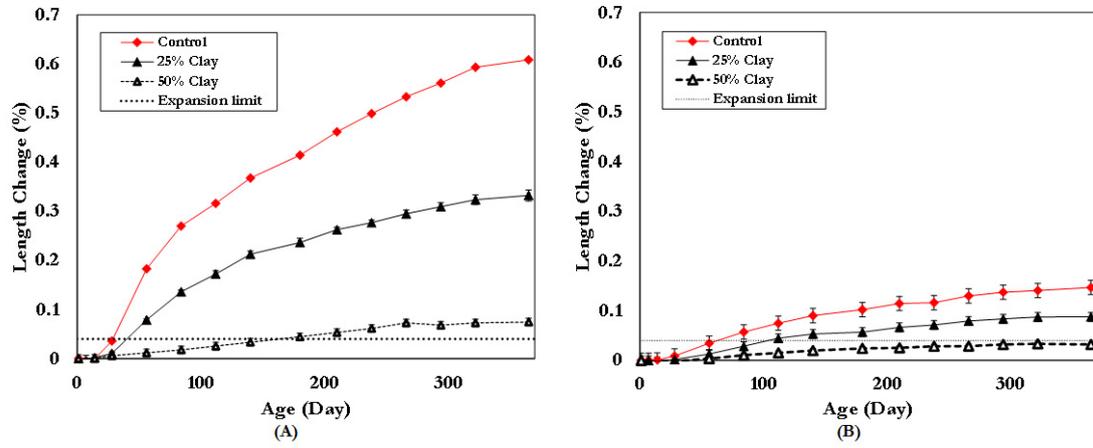
FIGURE 4: Length change of concrete prisms to two years: (A). Non-reactive limestone coarse aggregate with very highly reactive fine aggregate; (B). Highly reactive coarse aggregate (Spratt) with non-reactive limestone.
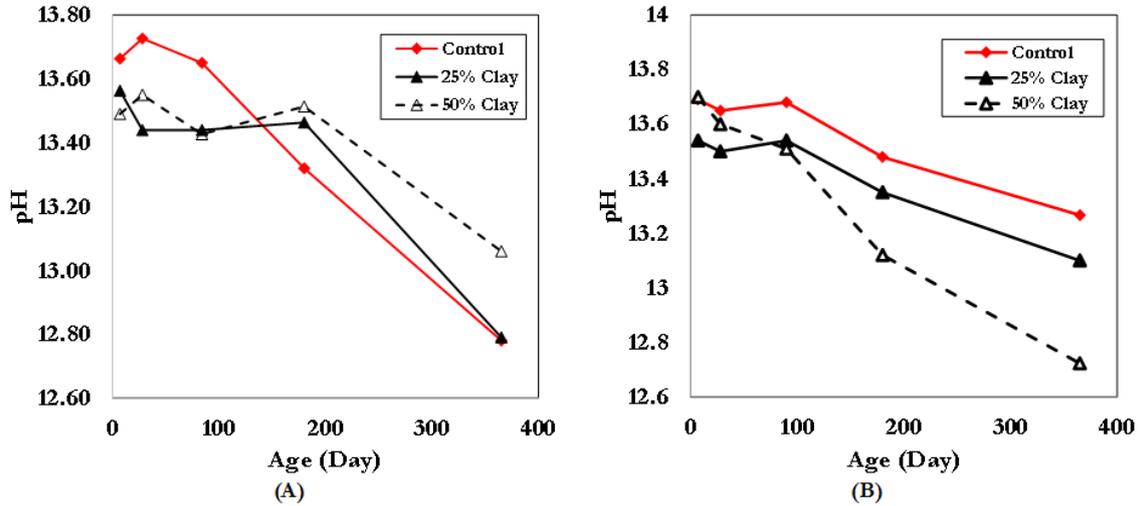

FIGURE 5: pH of pore solution in concrete prism: (A). pH of the pore solution of concrete mixture with non-reactive coarse limestone and very highly reactive fine siliceous aggregate; (B). pH of the pore solution of the concrete mixture with highly reactive coarse aggregate (Spratt) and non-reactive fine limestone.
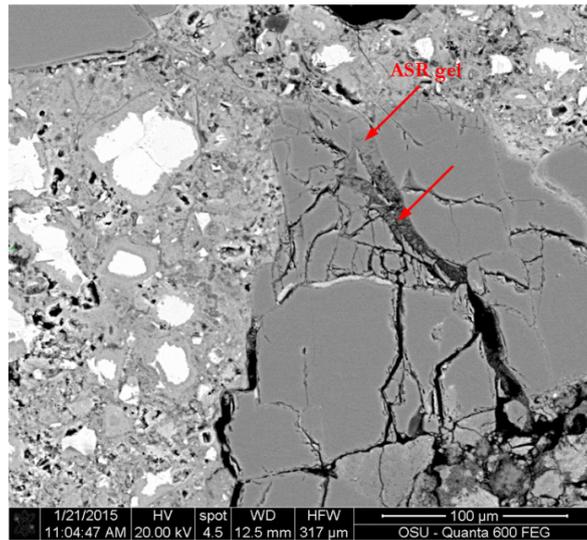
FIGURE 6: A reactive particle in the control mixture with ASR gel in it (sample from control mortar mixture after 14 days exposure to 1.0 N NaOH and 80°C).
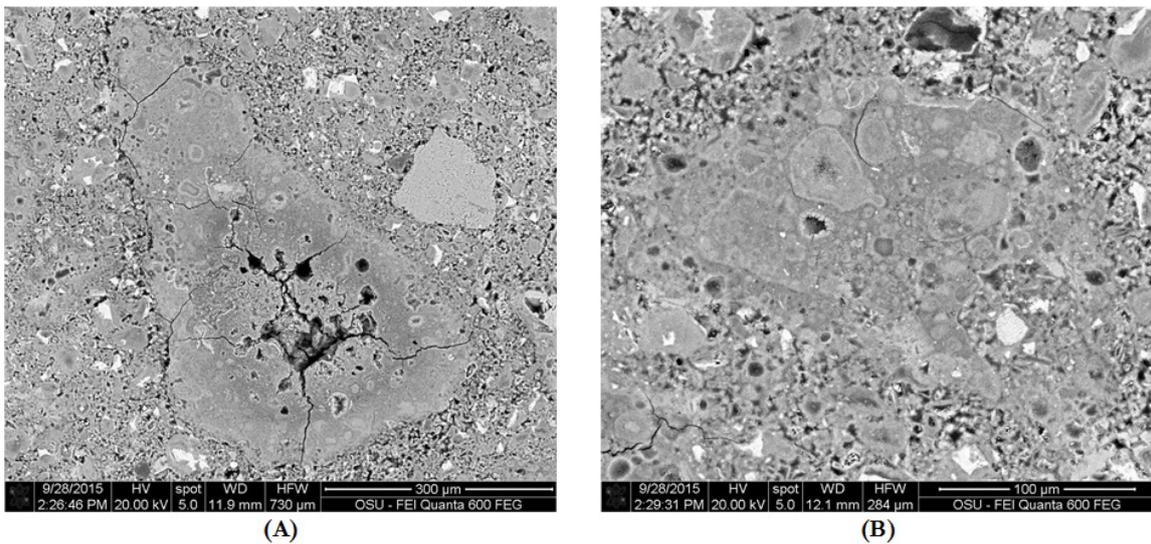

FIGURE 7: SEM images of mortar mixture with 100% expanded clay: (A). A partially reacted particle of 300~500 µm after 14 days exposure to 1.0 N NaOH and 80°C; (B). A fully reacted particle of 100~150 µm after 14 days exposure to 1.0 N NaOH and 80°C.