ASR Resistance of Ternary Cementitious Systems Containing Silica Fume-Fly Ash Using Modified ASTM C 1260 Method

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Abstract

Supplementary cementitious materials (SCM) such as fly ash, ground granulated blast furnace slag and silica fume are now being extensively used in concrete to control expansion due to alkali-silica reactivity (ASR). However, the replacement level of a single SCM needed to deleterious ASR expansion and cracking may create other problem and concerns. For example, incorporating silica fume at levels greater than 10% by mass of cement may lead to dispersion and workability concerns, while fly ash can lead to poor strength development at early age. The combination of silica fume and fly ash in ternary cementitious system may alleviate this and other concerns, and result in a number of synergistic effects. The aim of the study was to enable evaluation of more realistic suitability of a silica fume-fly ash combination system for ASR resistance based on an in-house modification of ASTM C 1260 test method. The modification can be more closely identified with actual field conditions. In this study three different strengths of NaOH test solution (1N, 0.5N, and 0.25N) were used to measure the expansion characteristics of mortar bar made with a reactive aggregate. The other variable included longer testing period of 28 days instead of a conventional 14 days.

Keywords: alkali-silica reactivity, modified ASTM C 1260 test method, silica fume, fly ash, normality of NaOH solution

1. Introduction

Ever since alkali silica reactivity (ASR) was identified in concrete some six decades ago, innumerable reports on concrete structures exhibiting deterioration due to ASR have been documented. Parallel to this, it has also been well established now that partial replacement of portland cement by supplementary cementitious materials (SCM) such as fly ash, ground granulated blast furnace slag, and silica fume reduces the expansion due to ASR. ^{2,3)}

The concept using cement-silica fume-fly ash combination to control ASR is another step forward from the benefits that accrue from using a binary-blend cementitious system.

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Tel: +1-979-458-4148; Fax: +1-979-845-1701 E-mail address: c-shon@ttimail.tamu.edu It has been proven that silica fume and fly ash are quite effective in producing concrete with low transport properties in which they definitely have positive effect on ASR resistance by reducing the potential for ionic ingress, migration, and concentration into concrete, and subsequently controlling expansion associated with ASR. ^{4,5)}

However, both these materials have some limitations. For example, silica fume can create increases in water demand and placing difficulties, while fly ash can lead to poor strength development at early age. ⁶⁾

The use of silica fume and fly ash in ternary cementitious system can alleviate these and other concerns, and additionally result in a number of synergistic effects. Baalbaki et al. ⁷⁾ and Borsoi et al. ⁸⁾ reported durability enhancement of concrete for sulfate attack with silica fume—fly ash combinations, and Li, et al. ⁹⁾ correlated the mechanical properties and pore structure properties in this system. However, rather limited data are available on the effect of silica fume-

fly ash combination on ASR resistance.

The ASR testing program involved some important modification to the ASTM C 1260 test method because these modifications can be more closely identified with actual field conditions. ¹⁰⁾ A general criticism of ASTM C 1260 test method is the severity of test conditions. It is not uncommon for aggregates with a good field performance track record and no history of ASR to test as reactive by ASTM C 1260 method.

Over the years some limitations of this method have become apparent. First, exposing the mortar to such a high temperature of 176°F (80°C) for 14 days does not represent field temperature condition, but was deemed necessary to accelerate the test. Second, the dosage of alkali (1N) in the test solution is abnormally high for a normal structural concrete. Finally, researchers have begun to realize that making decisions on whether to accept or reject an aggregate based on 14 day expansion results may be not be very representative.

In order to alleviate the inherent limitations of this test method, the authors developed two sets of important modifications that include three different strengths of NaOH solution (1N, 0.5N, and 0.25N) to test the expansion of mortar bars. The other modification include longer testing period of 28 days instead of 14 days.

These parameters were then implemented to evaluate ASR resistance of binary and ternary cementitious blends, with varying proportions of portland cement, silica fume, and Class F fly ash.

2. Experimental

2.1 Materials

Materials used in this study included ASTM Class C silica fume, ground-granulated blast furnace fly ash (fly ash), and ASTM Type I portland cement. The chemical and physical properties of the cementitious materials are presented in Table 1.

The siliceous sand that had been earlier classified as reactive aggregate had an expansion of 0.24 percent at 14 days according to the conventional ASTM C 1260 test method. The chemical analysis yielded over 90 % silica in aggregates. Quartz was identified as the principal mineral with small amounts of calcite, microcline feldspar and other silicate minerals present. Optical properties of this aggregate indicated that the reactive siliceous component consists mainly of strained quartz with undulatory extinction, a characteristic optical property considered to be indicative of possible ASR under appropriate conditions.

The test solution used contained 40.0g of NaOH dis-

solved in 900 mL of water, diluted with additional distilled water to obtain 1.0 L of solution. A 1.0 \pm 0.01 N sodium hydroxide (NaOH) solution was prepared and standardized to \pm 0.001 N. 0.5N and 0.25N NaOH solutions were also prepared to evaluate the effect of lower alkalinity on ASR expansion results.

2.2 Mixture characteristics

Tests were conducted on binary and ternary cementitious blends with varying proportions of ordinary portland cement, silica fume, and fly ash (OPC + silica fume + fly ash). The selected replacement levels of silica fume and fly ash combinations were 10 percent, 20 percent, and 30 percent by mass of cement. A set of plain cement mortar bars was also tested in conjunction with the ternary mixes. Mortars were prepared at water-cementitious material ratio (W/CM) of 0.47. The mixture proportions are presented in Table 2.

2.3 Test procedure

Mortar bar specimens were cast in 1" x 1" x 11-1/4" moulds with a water/cement ratio of 0.47. Immediately after casting, the test specimens were covered, and placed in a moist-curing room at 23 $^{\circ}$ C for a period of 24 ± 2 hours.

Mortar bars were then immersed in water in a sealed container at room temperature for 2-3 hours and then the container was transferred to an oven kept at a constant

Table 1 Physical properties and chemical analyses of cementitious materials

| Composition | Cement | Silica fume Fly ash | |
|-----------------------------------------------------------------------|--------|---------------------|-------|
| SiO ₂ | 19.12 | 96.9 | 47.73 |
| Al ₂ O ₃ | 5.07 | 0.52 | 19.95 |
| Fe ₂ O ₃ | 3.40 | 0.14 | 4.32 |
| (SiO ₂ +SiO ₂ +Fe ₂ O ₃) | 27.59 | 97.56 | 72.00 |
| CaO | 64.73 | 0.58 | 15.45 |
| MgO | 0.64 | 0.00 | 2.54 |
| SO ₃ | 3.13 | 0.13 | 0.78 |
| Na ₂ Oe. ^a | 0.65 | | |
| Na ₂ Oe. ^b | 1 | 0.32 | 0.32 |
| Loss on Ignition | 2.26 | 1.47 | 0.05 |
| Fineness c | 95.30 | - | 20.09 |
| Specific Gravity | 3.11 | 2.34 | 2.48 |
| Initial Set, min | 150 | - | _ |
| Final Set, min | 270 | - | _ |

^a Available alkali, expressed as Na2Oe, as per ASTM C 150.

^b Available alkali, expressed as Na2Oe, as per ASTM C 311.

^c Amount retained on 325 sieve %.

temperature of $80^{\circ}\text{C} \pm 2^{\circ}\text{C}$ to avoid thermal shock to the prisms. After a 24-hour preconditioning period, the length of the mortar bars was measured, and then the mortar bars were immediately transferred to storage containers filled with a 0.25N, 0.5N, and 1N NaOH solutions maintained at 80°C . The containers were then placed in the oven at 80°C . The length change of mortar bars was periodically measured over a 28-day period instead of the normal 14-day period recommended in the ASTM C 1260 procedure.

3. Test results and discussion

3.1 Compressive strength

Compressive strength development of mortar mixes with various combinations of potland cement, silica fume, and fly ash are shown in Figure 1. It is well known that silica fume starts to contribute to the strength development as early as one day after mixing, whereas fly ash takes more

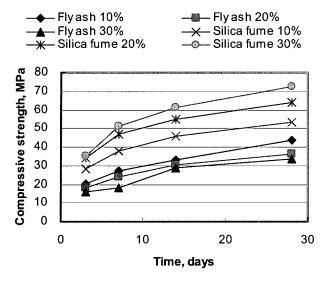
than seven to fourteen days before it makes any significant contribution to the development of strength.10) As expected, the strength of silica fume-replaced mortar was higher than that of fly ash at all ages. The strength of silica fume-replaced mortar is also greater than that of silica fume and fly ash combination mixtures. The strength development of silica fume-fly ash combination mixture is greater than that of fly ash at all ages. Thus, it is can be concluded that that the addition of silica fume to silica fume improves both early and late age strength based on the equal binder content and equal water-to-cement ratio.

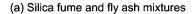
3.2 Expansion as a function of time

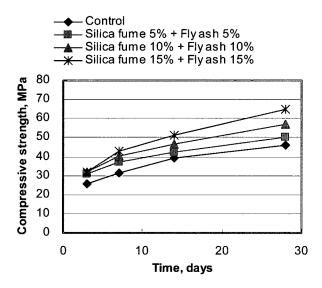
Fig. 2 shows expansions measured for mortar bars with made with three different combinations of silica fume and fly ash in different strength of NaOH solution over 28 days. The combination of 5 percent silica fume-5 percent fly ash shows inferior performance compared to other combina-

Table 2 Mortar mixture proportion and expansion results

| | Mixture | 6.11. | F1 4 1 | Expansion, % | |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------|-------------|---------|--------------|--------|
| Mixture | | Silica fume | Fly Ash | 14-day | 28-day |
| IN NaOH solution Silic | Control | - | - | 0.245 | 0.594 |
| | Silica fume 5% + Fly ash 5% | 5 | 5 | 0.130 | 0.286 |
| | Silica fume 10% + Fly ash 10% | 10 | 10 | 0.075 | 0.164 |
| | Silica fume 15% + Fly ash 15% | 15 | 15 | 0.009 | 0.020 |
| | Fly ash 5% | - | 5 | 0.202 | 0.447 |
| | Fly ash 10% | - | 10 | 0.155 | 0.321 |
| | Fly ash 15% | - | 15 | 0.119 | 0.269 |
| | Fly ash 20% | - | 20 | 0.085 | 0.187 |
| | Fly ash 30% | - | 30 | 0.036 | 0.066 |
| | Silica fume 5% | 5 | - | 0.176 | 0.381 |
| | Silica fume 10% | 10 | - - | 0.122 | 0.284 |
| | Silica fume 15% | 15 | - | 0.017 | 0.044 |
| | Silica fume 20% | 20 | - | 0.011 | 0.018 |
| | Silica fume 30% | 30 | = | 0.005 | 0.005 |
| | Silica fume 5% + Fly ash 5% | 5 | 5 | 0.130 | 0.286 |
| | Silica fume 5% + Fly ash 10% | 5 | 10 | 0.105 | 0.254 |
| | Silica fume 5% + Fly ash 15% | 5 | 15 | 0.055 | 0.106 |
| | Silica fume 5% + Fly ash 20% | 5 | 20 | 0.029 | 0.055 |
| | Silica fume 5% + Fly ash 30% | 5 | 30 | 0.007 | 0.013 |
| 0.5N NaOH solution | Control | - | = | 0.111 | 0.252 |
| | Silica fume 5% + Fly ash 5% | 5 | 5 | 0.033 | 0.074 |
| | Silica fume 10% + Fly ash 10% | 10 | 10 | 0.013 | 0.026 |
| | Silica fume 15% + Fly ash 15% | 15 | 15 | 0.007 | 0.012 |
| 0.25N NaOH solution | Control | - | - | 0.070 | 0.171 |
| | Silica fume 5% + Fly ash 5% | 5 | 5 | 0.011 | 0.018 |
| | Silica fume 10% + Fly ash 10% | 10 | 10 | 0.009 | 0.014 |
| | Silica fume 15% + Fly ash 15% | 15 | 15 | 0.005 | 0.007 |





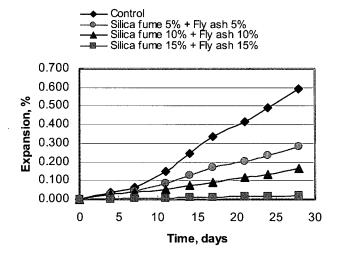


(b) Control and silica fume + fly ash mixtures

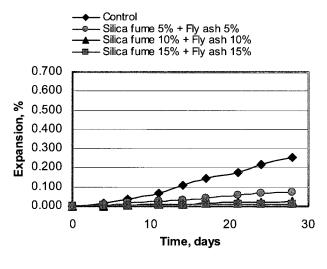
Fig. 1 Compressive strength development of different mixture

tions with higher SCM replacement percentage. For example, the mortar bars with 5 percent silica fume and fly ash in 1N NaOH solution expanded 0.13 percent, whereas those with 10 percent and 15 percent expanded less 0.1 percent at 14 days. Thus, it was barely within the potentially reactive aggregate category as per the standard ASTM C 1260 method. The rate of expansion continued to increase between 14 and 28 days, and reached as much as 0.29 percent at 28 days (this specimen will continue to be monitored over the long term). Nonetheless, significant improvement in reduction of the expansion was obtained for higher cement replacement levels.

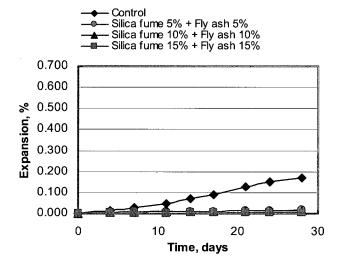
For 0.5N NaOH solution, all three ternary blends performed similarly. Expansion was minimal and reached the



(a) 1N NaOH solution



(b) 0.5N NaOH solution



(c) 0.25N NaOH solution

Fig. 2 Expansion curves of mortar bars

range from 0.01 percent to 0.07 percent at 28 days, which presents the non-reactive aggregate criterion of below 0.1 percent set by the standard. Similar results were obtained for 0.25N NaOH solution. The level of replacement had no effect on expansion with this lower strength of NaOH solution.

3.3 Effect of SCM combination

Fig. 3 compares the efficiency of different levels of cementitious materials in reducing the expansion at both 14 days and 28 days. When fly ash replacement was up to 20 percent, only limited improvement was observed, but greater reduction in expansion was noted for 30 percent replacement level at both 14 days and 28 days. Silica fume showed superior performance in reducing the expansion as the silica fume replacement levels increased.

The results demonstrate that for silica fume and fly ash combinations, 10 percent replacement did not effectively reduce the mortar bar expansion. In general, expansions were greater when the testing period was extended to 28 days¹²⁾. However, large decrease in ASR expansion was observed in the specimens with 20 percent and 30 percent combinations, and these were as low as 0.01 percent, even at 28 days expansion.

3.4 Effect of fly ash content in presence of silica fume

Fig. 4 shows the expansion behavior of selected mortar bars containing various fly ash contents when combined at a replacement level of 5 percent with silica fume. Specimens incorporating 5 and 10 percent fly ash expanded rapidly up to 28 days, which confirmed that low replacement levels of SCM are ineffective in reducing ASR expansion. 13) The addition of relatively large amounts of fly ash, that is, 15 percent or more by mass of cementitious material, reduced expansion significantly: 28 days value was less than 0.1 percent. It is not clear why the addition of larger amount of fly ash improves the ASR resistance. There possibly exists a threshold beyond which the resistance mechanism becomes effective, and this threshold can be expected to vary from one silica fume to another, or one fly ash to another. It is conjectured that the increased resistance of ternary blends may simply be a result of pore structure refinements, increased resistance to the diffusion of alkalies, and the overall reduced possibility of ASR gel formation. Further testing is underway to elucidate the mechanisms behind the ASR resistance of silica fume and fly ash combination.

3.4 Synergistic effect of ternary cementitious systems

Fig. 5 shows various levels of replacement of silica fume and fly ash. The use of low levels of a single SCM was not sufficient to control expansion. A 10 percent replacement of silica fume did not have any effect in reducing expansion,

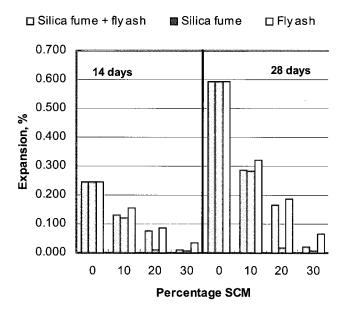


Fig. 3 Effect of supplementary cementitious materials content

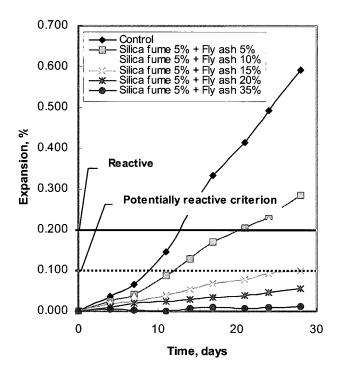
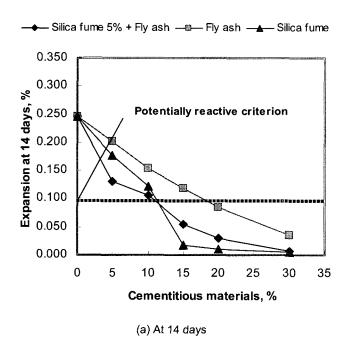
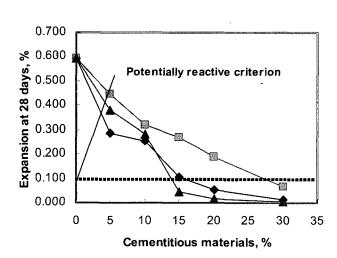


Fig. 4 Expansion curves of mortar bars in 1N NaOH solution

but significant improvements were observed for higher replacement levels.

The results support the previous findings that at a high replacement level the alkalies contributed by silica fume is less than those contributed by the portland cement. With respect to ASR resistance, little benefit is achieved from mortar bars containing only fly ash replacement of less than 15 percent. Fly ash probably requires levels of replacement in the range of 20% to meet the potentially reactive aggregate criterion of 0.1 percent at 14 days, compared with approximately 15 percent for the silica fume. Consequently, using a potentially reactive aggregate criterion of 0.1





◆— Silica fume 5% + Fly ash —

Fly ash —

Silica fume

(b) At 28 days

Fig. 5 Synergistic effect of ternary cementitious systems

percent at 14 days, 15 percent silica fume and 20 percent fly ash are the minimum amounts necessary to control expansion of mortar bars.

However, combination of lesser amount of these materials can also be very effective in controlling ASR expansion. It effects a total reduction in expansion equal to or greater than the sum of each individual SCM. For example, combination of mortar bars containing 5 percent silica fume and 15 percent fly ash was found to meet the potentially reactive aggregate criterion while 15 percent silica fume or 20 percent fly ash was sufficient to fulfill the criterion, respectively. These results indicate that a synergistic effect is present, which yields greater reduction in expansion allowing lower replacement levels of individual SCM.

3. Conclusion

The results of the ASR expansion for combination mixtures of silica fume and fly ash using the modified ASTM C 1260 test method demonstrate that using the appropriate combination mixture has the distinct beneficial effect of reducing expansion due to ASR, even when the testing period is extended to 28 days instead of the conventional 14 days in the presence of reactive aggregate. Based on the results, the following conclusion can be drawn:

- 1) Relatively high replacement levels are required to control expansion below 0.1% at 14 days when only a single SCM is used
- 2) The addition of high fly ash content with small amount of silica fume significantly improves ASR resistance.
- 3) The level of replacement did not exhibit different effect on the test results with lower strengths of NaOH solution.
- 4) The shortcoming of fly ash in terms of high level of replacement that is required to control ASR can be compensated by the incorporation of a relatively small amount of silica fume in the mixture.
- 5) The synergistic effect (reduction in expansion) was present when 5 percent silica fume and 15 percent fly ash are used in combination.
- 6) In terms of durability, the ternary blend combinations of silica fume and fly ash showed a considerable reduction in the rate of expansion compared with binary mixes containing only silica fume or fly ash. Furthermore, price difference on the basis of material costs, or appropriate mixture proportion between individual components may allow the ternary cementitious system to compete with binary system or plain portland cement. In our opinion these test results will help in the research of preventive measurement against ASR in ternary cementitious system.

References

- 1. Stanton, T.E, "Expansion of Concrete Through Reaction between Cement and Aggregate," *Proceedings of the American Society of Civil Engineers*, Vol. 66, 1940, pp.1781-1811.
- Thomas, M.D.A., "Review of the Effect of Fly Ash and Slag on Alkali-Aggregate Reaction in Concrete," Building Research Establishment Report BR 314, Construction Research Communications, Ltd., Watford, UK, 1996, pp.117.
- Ramachandran, V.S., "Alkali-Aggregate Exapnsion inhibiting Admixtures," *Cement and Concrete Composites*, Vol. 20, 1998, pp.149-161.
- 4. Xu, Z., Hooton, R.D., "Migration of Alkali Ions in Mortar Due to Several Mechanisms," *Cement Concrete Research*, Vol. 23, 1993, pp.951-961.
- Thomas, M.D.A., Hooton, R.D., and Rogers, C., "Prevention of Damage Due to Alkali-aggregate Reactions (AAR) in Concrete Construction-Canadian Approach," Cement Concrete and Aggregate, Vol. 19, 1997, pp.26-30.
- Roger, C.A. and Hooton, R.D., "Reduction in Mortar and Concrete Expansion with Reactive Aggregates Due to Leaching," *Cement Concrete and Aggregate*, Vol. 13, 1991, pp.42-49.
- Baalbaki, M, Sarkar, S.L., Aitcin, P.C., and Isabelle, H., "Properties and microstructure of high-performance Concretes Containing Silica fume, Slag, and Fly," *Proceeding of 4th International Conference on Silica Fume*, Slag, Fly Ash, and Natural Pozzolans in Concrete, Istanbul, 1992, pp.921-935.

- Borsoi, A., Collepardi, S., Coppola, L., Troli, R., and Collepandi, M., "Strength and Durability of High Performance Concrete with Pozzolanic and Composite Cement," Proceeding of 5th International Conference on Durability of Concrete, 2000, pp.159-172.
- 9. Li, D., Shen, J., Chen, Y., Cheng, L., and Wu, X., "Study of Properties on Fly Ash-Slag Complex Cement," *Cement and Concrete Research*, Vol.30, 2000, pp.1381-1387.
- Shon, C.-S., Zollinger, D.G., and Sarkar, S.L., "Evaluation of Modified ASTM C 1260 Accelerated Mortar Bar Test for Alkali-Silica Reactivity," *Cement and Concrete Research*, Vol.32, 2002, pp.1981-1987.
- Malhotra, V.M. and Mehta, P.K., "Pozzolanic and Cementitious Materials," Gordon and Breach Publishers, Amsterdam, 1996.
- Shon, C. S., Kang, S. G., and Kim, Y. S., "ASR Effectiveness of High Volume Fly Ash Cementitious Systems Using modified ASTM C 1260 Test Method," KCI Concrete Journal, Vol. 14, 2002, pp.81-85.
- 13. Shon, C. S., Zollinger, D. G., and Sarkar, S. L., "Testing the Effectiveness of Class C and Class F Fly Ash in Controlling Expansion Due to Alkali-Silica Reaction Using Modified ASTM C 1260 Test Method," ASCE Journal of Materials in Civil Engineering, in Press.
- 14. Hobbs, D.W, "Alkali-Silica Reaction in Concrete," Thomas Telford, London, 1988, pp.103-133.