Computers and Concrete, *Vol. 20, No. 2 (2017) 197-204*
DOI: https://doi.org/10.12989/cac.2017.20.2.197

Effect of fineness of high lime fly ash on pozzolanic reactivity and ASR mitigation

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(Received March 21, 2016, Revised April 11, 2017, Accepted April 12, 2017)

Abstract. Typically, high lime fly ash (Class C) has been characterized as a fly ash, which at lower replacement levels is not as effective as the low lime (Class F) fly ash, in mitigating alkali-silica reaction (ASR) in portland cement concrete. The influence of fineness of Class C, obtained by grinding virgin fly ash into finer particles, on its pozzolanic reactivity and ASR mitigation performance was investigated in this study. In order to assess the pozzolanic reactivity of mortar mixtures containing virgin or ground fly ashes, the strength activity index (SAI) test and thermo-gravimetric analysis (TGA) were conducted on the mortar cubes and paste samples, respectively, containing virgin fly ash or two ground fly ashes. In addition, to evaluate any improvement in the ASR mitigation of ground fly ashes compared to that of the virgin fly ash, the accelerated mortar bar test (AMBT) was conducted on the mortar mixtures containing different dosages of either virgin or ground fly ashes. In all tests crushed glass aggregate was used as a highly reactive aggregate. Results from this study showed that the finest fly ash (i.e., with an average particle size of 3.1 microns) could increase the flow ability along with the pozzolanic reactivity of the mortar mixture. However, results from this study suggested that the fineness of high lime fly ash does not seem to have any significant effect on ASR mitigation.

Keywords: class C fly ash; fineness; pozzolanic reactivity; ASR; mitigation

1. Introduction

Alkali-Silica Reaction (ASR) is one of the most common deterioration mechanisms in concrete. It is caused by the reaction between reactive silica, typically found in some aggregates, and alkalis present in the pore solution of the cement paste. The product of ASR reaction is a hygroscopic gel, which in presence of moisture swells causing cracks and deterioration in the matrix of concrete.

Typically supplementary cementitious materials (SCMs) are used as cement replacement materials to suppress the alkali-silica reaction in concretes containing reactive aggregates. Among all of the conventional SCMs, fly ashes are widely used to control ASR; however fly ashes also have diverse chemical composition depending on the source of coal and the operational characteristics of the power plants, which can affect its effectiveness in mitigating ASR. ASTM C618 recognizes two general classes of fly ashes (Class F and Class C) based on their chemical composition. Fly ashes containing high levels of silica, alumina and iron oxide together (> 70%) are categorized as Class F fly ashes and those containing the sum of silica, alumina and iron oxide between 50% and 70% are categorized as Class C fly ashes. Alternatively, Class F fly ashes also contain much lower levels of calcium oxide (lime) content (typically less than 15%) compared to Class C fly ashes. Due to the reactive nature of the glass present in Class C fly ash, it reacts faster and exhibits higher early age strengths in concrete compared to Class F fly ashes at equivalent dosage levels in concrete. On the other hand, due to the lack of calcium in the alumino-silicate glass present in Class F fly ashes, the early age strength is one of the main concerns when Class F fly ash is used in concrete (Helmuth 1987). Several studies have been carried out to evaluate the beneficial influence of Class C and Class F fly ashes on concrete properties (Nie *et al.* 2015, Yoo *et al.* 2015, Shaikh and Supit 2015, Simčič *et al.* 2015, Uysal and Akyuncu 2012 and Sumer 2012). In terms of durability properties, and particularly with regards to ASR mitigation, the use of Class F fly ash has been shown to offer more benefits compared to the Class C fly ash at equivalent and typical dosage levels that do not negatively influence early-age properties of concrete to a significant extent (Gebler and Klieger 1986, Thomas *et al*. 2011, Du *et al*. 2012, Folliard *et al*. 2012, Roskos *et al*. 2012, Harish and Rangaraju 2011).

A study by Shafaatian *et al.* (2013) was carried out to study the possible mechanism in which class C or class F fly ash can mitigate ASR distress in mortars containing reactive aggregate. Alkali dilution, alkali binding, mass transport reduction, increase tensile strength, altering ASR gel and reducing aggregate dissolution rate were found to be the potential mitigation mechanism for fly ash. While vast majority of studies have concentrated on the ASR mitigation performance of fly ash, some studies were carried out to address the influence of fineness or chemical composition of fly ashes. A study by Chindaprasirt *et al*. (2005) was carried out to evaluate the effect of fineness of fly ash on the strength and porosity of the cement paste containing different dosages of fly ash with average particle

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http://www.techno-press.org/?journal=cac&subpage=8
ISSN: 1598-8198 (Print), 1598-818X (Online)

size of 19 and 6.5 microns. It was found that the paste specimen containing finer fly ash showed higher compressive strength and lower porosity. A study by Venkatanarayanan and Rangaraju (2013) was carried out to decouple the individual effects of fly ash fineness and its chemical composition on the mitigation of ASR distress in mortars. In this study, a range of fly ashes containing different lime contents were used as SCMs and mortars prepared with reactive aggregates were evaluated in the accelerated mortar bar test method. It was found that the mortar bars containing ground fly ash with finer particle size showed significantly less expansion than the corresponding virgin fly ashes with coarser particle size distribution. In addition, the fineness of fly ash was found to have as significant of an impact as the chemical composition of fly ash.

A study by Shon *et al*. (2007) was carried out to evaluate the influence of fly ash chemical composition on the ASR mitigation performance of several Class C or Class F fly ashes using the accelerated mortar bar test method. It was found that the oxide content of the fly ash ($SiO_2+Al_2O_3+Fe_2O_3$) is one of the main factors that can influence the ASR mitigation of fly ashes. In this study, different sources of fly ashes with the sum of silica, alumina and iron oxide contents ranging from 58.2% to 83.7% were used as a portland cement replacement. These fly ashes had alkali contents ranging from 0.09% to 1.47% Na_2O_{eq}. It was found that the fly ashes containing higher oxide ($SiO_2+Al_2O_3+Fe_2O_3$) content and lower alkali content were more effective in mitigation ASR distress in concrete.

Another study by Shehata *et al*. (1999) was carried out to evaluate the influence of fly ash chemical composition on the chemical composition of paste pore solution. It was found that the use of fly ash containing high dosage of alkalis increases the alkalinity of the pore solution, thus exacerbating ASR induced expansion in concrete prisms subjected to the concrete prism test. Aydin *et al*. (2010) carried out a study to evaluate the influence of fly ash fineness on mechanical properties and ASR mitigation performance of mortar or concrete specimens. It was found that although the compressive strength of the concrete specimens containing finer fly ash was significantly higher than that of the concrete specimens containing coarser fly ash, no differences was observed in the degree of ASR mitigation offered by either finer or coarser fly ash in the mortar bar tests.

Based on the previous studies, it can be broadly concluded that Class C fly ash (with its higher lime content) has lower ASR mitigation performance compared to Class F fly ash at equivalent dosage levels. Therefore, high dosage levels of Class C fly ash are required to effectively control the ASR expansion in concrete containing reactive aggregate (typically more than 30% replacement level). However, it should be acknowledged that the minimum threshold level of cement replacement required to effectively mitigate ASR is dependent not only on the properties of the specific fly ash, but also on the degree of reactivity of aggregate, alkali level of cement, among other factors such as mixture proportions.

The objective of this study is to clearly discern the influence of fineness of Class C fly ash on the degree of pozzolanic reactivity and its ability to mitigate ASR. In this study the fineness of the Class C fly ash was varied by grinding a virgin fly ash to two different levels of fineness in a planetary ball mill. The average particle size for each of the three fineness studied in this investigation were 13.7 microns (virgin), 5.6 microns (G1) and 3.1 microns (G2) microns. In these ASR mitigation studies, an ASR-prone aggregate derived from crushing recycled glass bottles was used as the aggregate material in the mortar mixtures.

2. Experimental work

2.1 Materials

2.1.1 Cementitious materials

In this study, an ASTM C150 Type I portland cement with high-alkali content (Na2Oeq=0.88%) was used. A Class C fly ash (CaO = 29.8%) with a specific gravity of 2.77 was used as a supplementary cementitious material (SCM). To investigate the influence of fineness of the Class C fly ash on its ability to effectively mitigate ASR, the virgin fly ash was ground to two levels of fineness in the lab using a Retsch planetary ball mill (PM 100) at a speed setting of 250-rpm for 30 min (G1) and 90 min (G2). The particle size distributions of the virgin and ground fly ashes (i.e., G1 and G2) are shown in Fig. 1. The average particle size (d50) of the virgin, G1 and G2 fly ash are 13.7, 5.6 and 3.1 microns, respectively. The chemical compositions of the portland cement and fly ash are given in Table 1.

2.1.2 Fine glass aggregate

Glass aggregate with an oven-dry specific gravity of 2.42 and an absorption value of 0.03%, produced from crushing and washing waste soda bottles of different mixed colors was used in this study. Previous studies carried out by Afshinnia *et al*. (2015) showed that the glass aggregate derived from crushing recycled glass bottles is an ASR-prone aggregate. Therefore, fine glass aggregate was selected in order to show the ASR mitigation performance of virgin or ground fly ashes. In addition, recycled glass is one of the waste materials that usually disposed of in landfills. Therefore, by incorporation of this recycled material in concrete as an aggregate replacement, waste glass can be easily eliminated from landfills.

Table 1 Chemical compositions of cement and fly ash

Chemical composition (%)	Cement	Class C Fly ash
SiO_2	19.45	31.31
Al_2O_3	4.85	18.64
Fe_2O_3	3.79	5.49
CaO	61.37	29.85
MgO	2.92	5.54
SO_3	3.30	2.55
Na_2O_{eq}	0.88	2.1

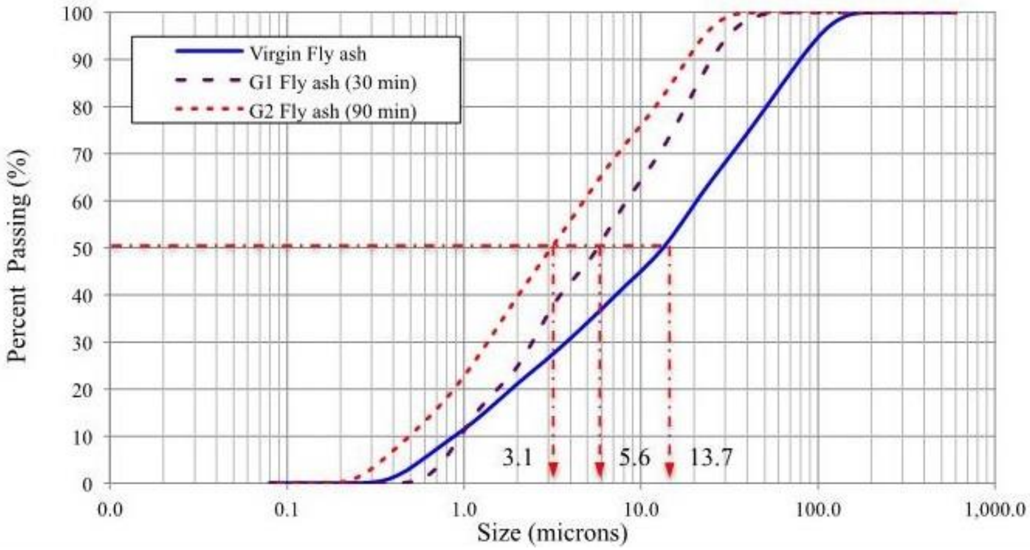


Fig. 1 Particle size distribution of virgin and grinded fly ashes

Table 2 Mixture proportions of material used in preparation of mortar cubes and mortar bars containing virgin or grinded fly ashes (used for the Flow test, Strength activity test and the AMBT test)

Mixture ID	Cementitious Materials (grams)					Glass aggregate (Retained on Sieve # - grams)					W/C
	Portland cement	Virgin fly ash	Fly ash G1	Fly ash G2	Dosage Level (%)	#8	#16	#30	#50	#100	
Control	375	0	0	0	0	85	211	211	211	127	0.47
10F	337	38	0	0	10	85	211	211	211	127	0.47
20F	300	75	0	0	20	85	211	211	211	127	0.47
30F	263	112	0	0	30	85	211	211	211	127	0.47
40F	225	150	0	0	40	85	211	211	211	127	0.47
10FG(1)	337	0	38	0	10	85	211	211	211	127	0.47
20FG(1)	300	0	75	0	20	85	211	211	211	127	0.47
30FG(1)	263	0	112	0	30	85	211	211	211	127	0.47
40FG(1)	225	0	150	0	40	85	211	211	211	127	0.47
10FG(2)	337	0	0	38	10	85	211	211	211	127	0.47
20FG(2)	300	0	0	75	20	85	211	211	211	127	0.47
30FG(2)	263	0	0	112	30	85	211	211	211	127	0.47
40FG(2)	225		-	150	40	85	211	211	211	127	0.47

2.2 Test procedure and mixture proportion

2.2.1 Flow (Workability)

In order to evaluate the influence of fineness of fly ash on the workability of the mortar mixtures, the flow test was conducted on the mortar mixtures as per ASTM C1437. Additionally, control mix without fly ash was prepared as a reference mixture. Three samples were tested for each mixture.

2.2.2 Strength activity index

Pozzolanic reactivity of each mixture containing virgin or ground fly ashes was evaluated by studying the 28-day strength activity index of control and fly ash-dosed mortar cubes as per ASTM C311. Three specimens were tested for each mixture.

2.2.3 Pozzolanic reactivity evaluation using Thermo-Gravimetric Analysis (TGA)

Pozzolanic reactivity of the virgin or ground fly ash was examined using Thermo-Gravimetric Analysis (TGA). The consumption of calcium hydroxide due to pozzolanic reaction of fly ash can be tracked by monitoring the decomposition of calcium hydroxide upon heating. In this test method, the mass loss within a specific temperature range is studied to quantify the amount of calcium hydroxide present in the cement paste. Typically, significant mass loss occurs in cement pastes between 440-520℃ which corresponds with the decomposition of calcium hydroxide. Also, mass loss can occur between 600-780℃ that corresponds with the decomposition of calcium carbonate. In order to compare the workability and pozzolanic reactivity of the virgin fly ash with ground fly ashes, mortar mixtures containing virgin or ground fly ash were prepared with 20% fly ash as a portland cement

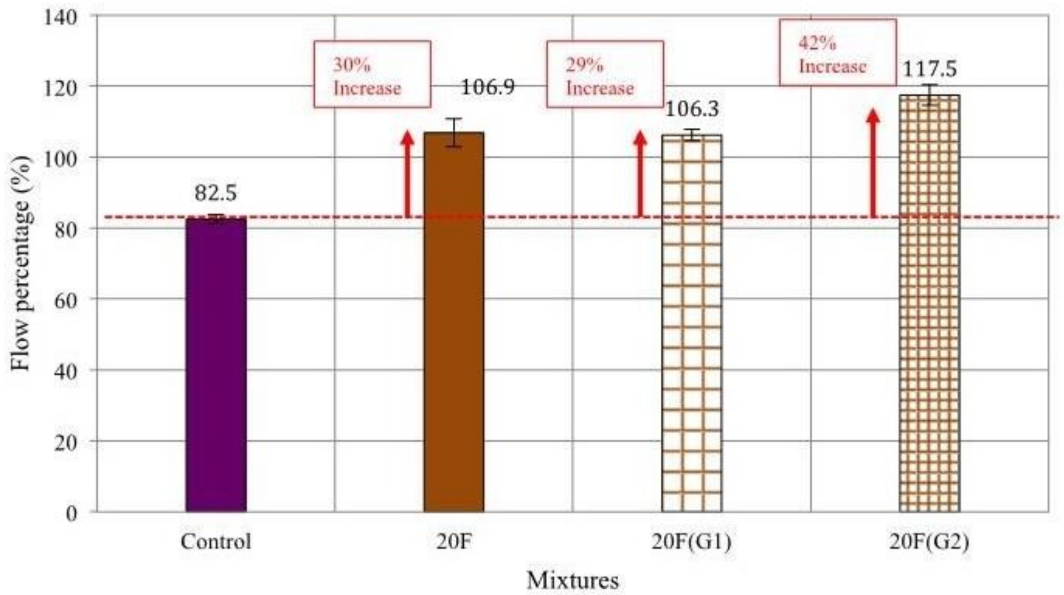


Fig. 2 Flow percentage of the mortar samples containing virgin or grinded fly ashes

replacement material. For TGA studies paste specimens containing equivalent proportions of cement and fly ashes were cast and cured similar to the mortar specimens.

2.2.4 ASR mitigation evaluation using Accelerated Mortar Bar Tests (AMBT)

To evaluate the effect of grinding on the ASR mitigating performance of fly ash, accelerated mortar bar tests as per ASTM C1260 and C1567 were conducted wherein granular crushed glass aggregate was used as a highly reactive aggregate. Thus, in mortar mixtures, cement was replaced by virgin or ground fly ash at levels of 10%, 20%, 30% and 40% by mass of cement. Three mortar bars were cast for each mortar mixture.

The mixture proportions of constituents that were used in preparation of test specimens for flow test, strength activity index and the AMBT test are given in Tables 2. Paste specimens (i.e., without aggregate) for TGA studies were prepared using the cementitious blends containing portland cement and fly ashes with water to cement ratio of 0.4. No chemical admixture was used in this study.

3. Results and discussion

3.1 Flow test

Fig. 2 shows the flow values of mortar mixtures containing virgin and ground Class C fly ashes. As shown in Fig. 2, regardless of the fly ash fineness (i.e., virgin fly ash or ground fly ashes), all mortar mixtures containing fly ash showed significantly higher flow values compared to that of the control mixture (without fly ash). However, among all mortar mixtures containing fly ash, the higher flow value was achieved in the mortar mixture containing the finest fly ash (i.e., G2). For instance, the flow percentage of the mortar mixtures containing 20% fly ash (G2) was 42% higher than that of the control, while this value for the mortar mixture containing virgin or G1 fly ash were 30% and 29% higher than that of the control, respectively. These results suggest that by using the finer high lime fly ash in mortar mixtures, better workability of the mortar mixtures can be achieved.

The ball bearing effect caused by the spherical shape of the fly ash particles provides a lubricant effect within the mortar or concrete mixtures and consequently enhances the workability of the mixture. As shown in Fig. 2 the workability of the mixtures containing fly ash (both virgin and ground fly ashes) was significantly higher than that of the control, which indicates the positive influence of using fly ash in mortar mixtures. The workability of the mortar mixture containing ground fly (G1) ash was comparable to that of the mortar mixture containing virgin fly ash. However, when the fly ash (G2) was used, the workability of the mixture increased. The incorporation of more spherical particles along with the small size of these particles (i.e., 3.1 microns) compared to the virgin fly ash, improved the workability of the mixture.

3.2 Strength activity index

In order to evaluate the influence of fly ash fineness on the pozzolanic reactivity of fly ash, the 28-day strength activity index values of the mortar cubes containing 20% virgin or ground fly ashes were determined.

As shown in Fig. 3, the 28-day strength activity index of the mortar mixtures containing G1 fly ash is almost comparable with that of the mortar mixtures containing virgin fly ash, both of which are significantly below that of the control mixture. For instance, the 28-day strength

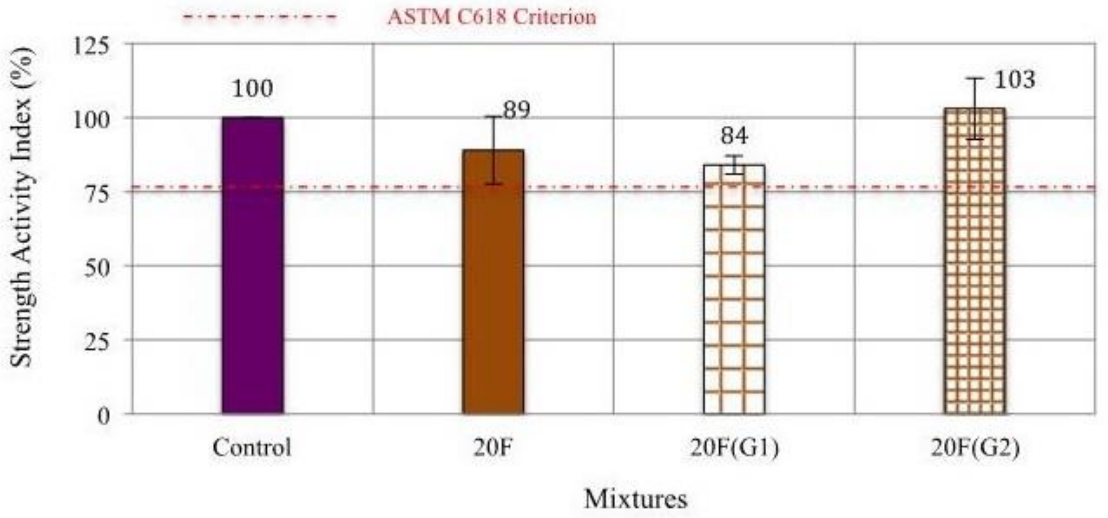


Fig. 3 28-day strength activity index of mortar mixtures containing virgin or grinded fly ashes

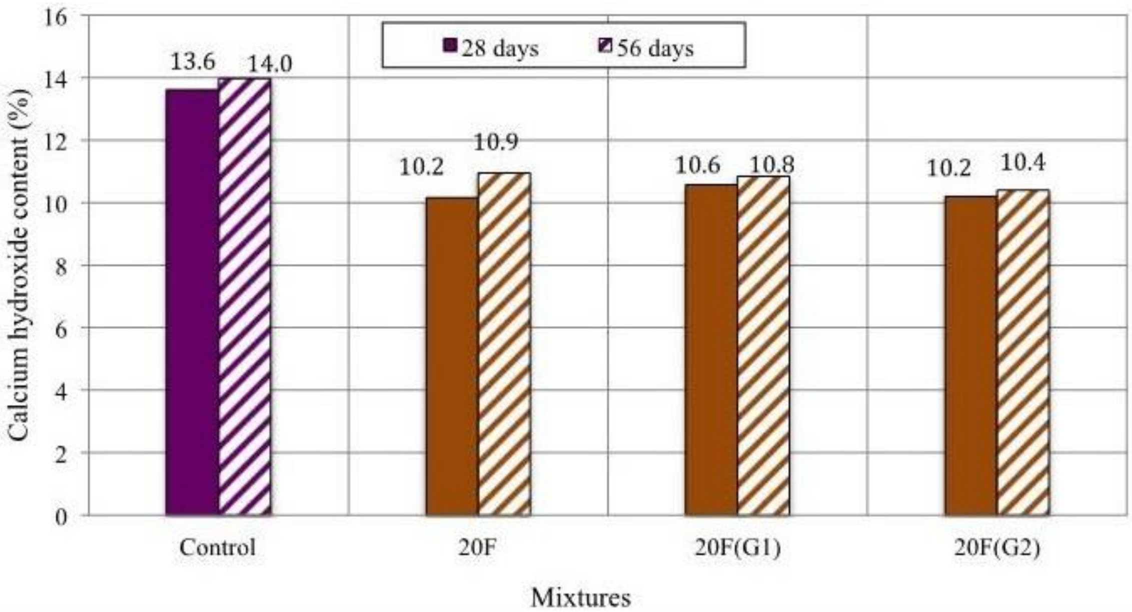


Fig. 4 Calcium hydroxide content (%) in specimens containing virgin or grinded fly ashes at 28 and 56 days

activity index of the mortar mixtures containing 20% virgin fly ash is 89%, while this value for the mortar mixtures containing 20% G1 fly ash is 84%. The use of G2 fly ash, the finest of all the three fly ashes used in this study, showed significantly higher 28-day strength activity index at 103% compared to the other two fly ashes, although the SAI of G2 fly ash mixture was above that of control mixture. This behavior was also observed in the flow test wherein the mixture containing G2 fly ash showed better flow results than other mixtures. The authors believe that the strength activity index results can be compared with the flow test results. Better flow ability of the mixture may directly affect the compaction of the mixture, which enhanced the strength of the mortar specimens.

These results suggest that the use of high lime fly ash with an average particle size of 3.1 microns can improve the compressive strength of the mixtures even at a modest replacement level of 20% by mass of cement.

3.3 Pozzolanic reactivity evaluation using Thermo-Gravimetric Analysis (TGA)

In order to evaluate the effect of fly ash fineness on the pozzolanic reactivity of fly ash, the TGA test was conducted on the paste specimens at 28 and 56 days. The calcium hydroxide content of the paste sample is shown in Fig. 4. The dosage of the SCMs in all the specimens was kept constant (i.e., 80% portland cement and 20% fly ash). Therefore, to eliminate the dilution effect the calcium hydroxide content of the samples should be compared with 80% of the calcium hydroxide content in control paste (i.e., 10.8% and 11.2% for 28 and 56 days, respectively).

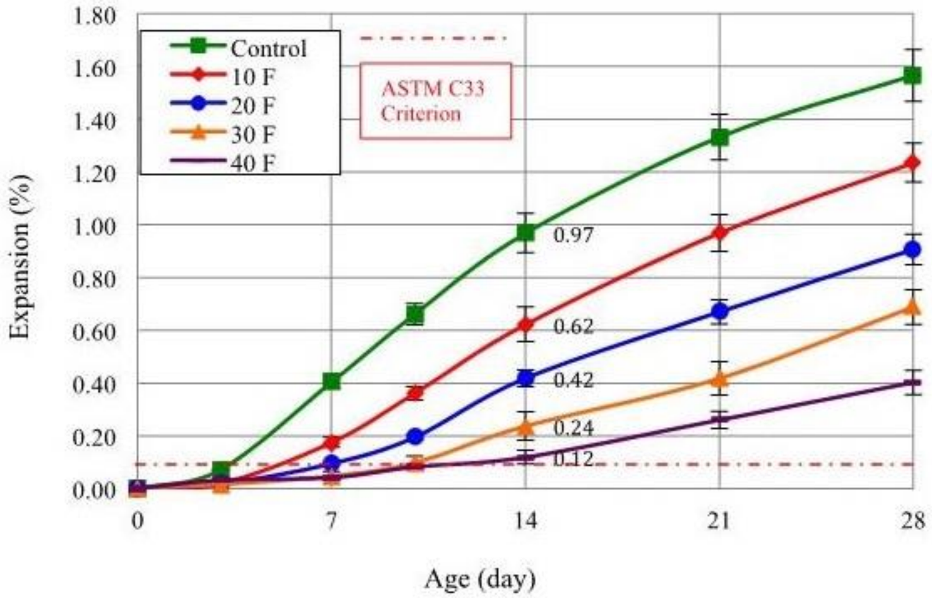


Fig. 5 Expansion behavior of the mortar bars containing fly ash

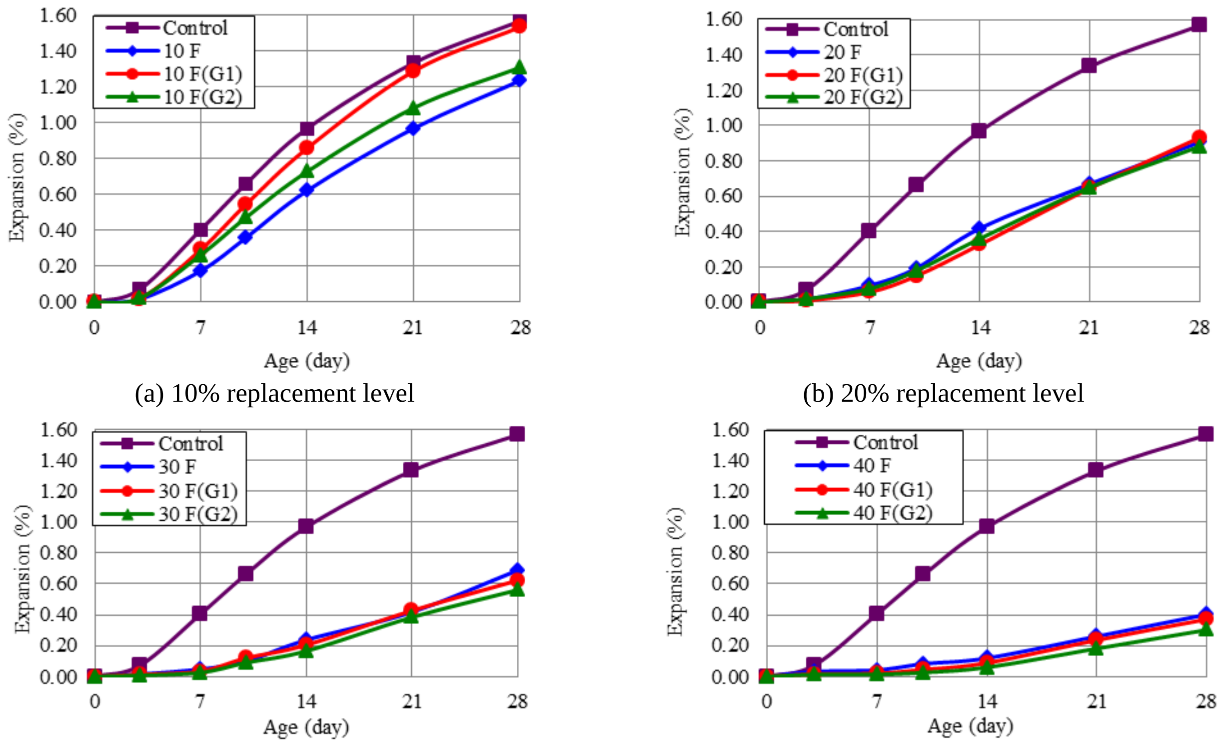
As shown in Fig. 4, the 28-day calcium hydroxide content of the paste samples containing either virgin or ground fly ashes were only slightly less than that of the control. For instance, the 28-day calcium hydroxide content of the paste samples containing G1 or G2 fly ashes were 10.6% and 10.2% for the paste samples containing G1 and G2 fly ashes, respectively, compared to that of control specimen at 10.8%. While the possibility exists that Class C fly ash may contain free lime (CaO) that produces calcium hydroxide of its own, in terms of the TGA results the fineness of high lime fly ash does not appear to have a significant effect on the calcium hydroxide content of the paste samples at 28 days. Similar results were observed in 56-day calcium hydroxide content of the paste samples.

3.4 ASR mitigation evaluation using Accelerated M ortar Bar Tests (AMBT)

Fig. 5 shows the expansion behavior of the mortar bars containing reactive glass aggregates in the accelerated mortar bar tests in presence of different dosages of virgin Class C fly ash. The 14-day expansion values of the mortar bars containing 10%, 20%, 30% and 40% virgin Class C fly ash are 0.62%, 0.42%, 0.24% and 0.12%, respectively. Thus, in order to mitigate the ASR distress in mortar bars containing crushed glass aggregate, a dosage level of Class C fly ash greater than 40% is required. At such high cement replacement levels, the early-age strength gain may be negatively affected thus limiting the use of virgin Class C fly ashes for such purposes. The factors that likely render the ineffectiveness of Class C fly ash in mitigating ASR even at such high dosage levels are its inability to reduce the calcium hydroxide content of the cementitious paste and possibly the alkali level of the fly ash. Also, Class C fly ash typically produces C-S-H gel with higher calcium-to-silica ratio (compared to Class F fly ash). Therefore, the higher calcium-to-silica ratio within the C-S-H gel results in ineffectiveness of the C-S-H to bind alkalis throughout the pore solution. In addition; the alkali contribution from glass aggregates may be impeding the ASR mitigation ability of the fly ash. As the purpose of this study was to assess the influence of fineness of fly ash on ASR mitigation, and as the glass aggregates were common to all tests, the role of alkali contribution from glass was not investigated in this study. However, in order to consider incorporation of alkalis from glass structure, the expansion values of the mortar bars were evaluated within 28 days. However, no evidence of secondary ASR reaction was observed among the mortar bars tested in this study.

Figs. 6(a)-6(d) show the expansion behavior of the mortar bars containing virgin or ground fly ashes (i.e., G1 and G2) at 10%, 20%, 30% and 40% replacement levels, respectively. As shown in Fig. 6, regardless of level of grinding, the mortar bars containing 10% virgin fly ash showed better mitigation performance compared to that of the mortar bars containing 10% ground fly ashes. For instance, the 14-day expansion values of the mortar bars containing 10% G1 or G2 fly ash were 0.86% and 0.73%, respectively, while this value for the mortar bars containing 10% virgin fly ash was 0.62%. Therefore, in terms of mitigation performance, the fineness of fly ash when replaced as a cement replacement at low dosage level (i.e., 10%) has a negative influence on the mitigation performance of high lime fly ash. It is likely that at a low level dosage, increasing the fineness of Class F fly ash may elevate the calcium-to-silica ratio of the pozzolanic reaction product which will have minimal ability to bind alkalis in the matrix. In addition, the increased fineness may also exacerbate any alkali release from within the fly ash grains thus accelerating the expansion.

In mortar bars containing 20% fly ash, the fineness of fly ash showed slightly positive influence on the mitigation



(a) 10% replacement level (b) 20% replacement level

(c) 30% replacement level (d) 40% replacement level

Fig. 6 Expansion behavior of the mortar bars containing virgin or ground fly ash

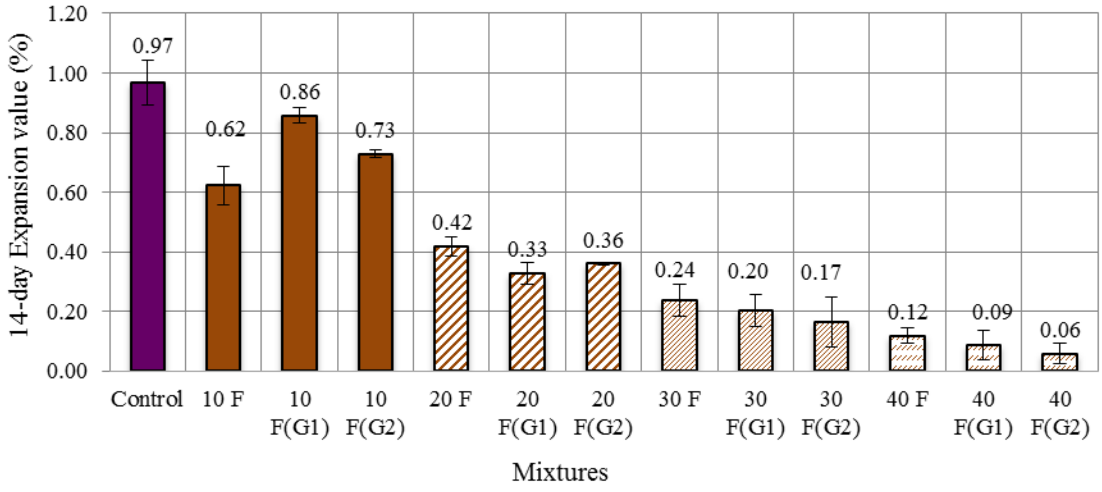


Fig. 7 14-day expansion values of mortar bars containing virgin or grinded fly ashes

performance of the fly ash. However, no significant differences were observed in the expansion results of mortar bars containing ground fly ashes with different finenesses. For instance, the 14-day expansion value of the mortar bars containing 20% virgin, G1 and G2 fly ash were 0.42%, 0.33% and 0.36%, respectively. The positive influence of grinding fly ash was also observed in 14-day expansion values of mortar bars containing either 30% or 40% fly ash. For instance, the 14-day expansion value of the mortar bars containing 30% G1 and 30% G2 fly ashes showed 17% and 29% reduction compared to that of the mortar bars containing 30% virgin fly ash. The 14-day expansion value of the mortar bars containing 40% G1 and 40% G2 fly ashes showed 25% and 50% reduction compared to that of the mortar bars containing 40% virgin fly ash. These results suggested that the fineness of high lime fly ash does not seem to have any significant effect on ASR mitigation.

Fig. 7 shows the 14-day expansion values of the mortar bars containing virgin or ground fly ashes. As shown in Fig. 7, with an exception of mixtures containing 10% virgin or ground fly ashes, among all other replacement levels, the 14-day expansion values of mortar bars containing ground fly ashes were less than that of the mortar bars containing virgin fly ash, indicating the influence of fineness of fly ash on its mitigation performance. Also, it is clear from Fig. 7 that among all mortar mixtures investigated in this study, the mortar mixtures containing 40% G1 and 40% G2 met the requirements of 0.10% expansion or less at 14 days as recommended by ASTM C33 specification.

4. Conclusions

The following conclusions are drawn based on the research conducted in this investigation on the pozzolanic reactivity and mitigating ability of high lime fly ashes with different fineness:

- Mortar mixtures containing finest fly ash among the three fly ashes used in this study showed better flow result compared to other mixtures.
- The mortar mixtures containing the finest Class C fly ash (i.e., G3) exhibited the highest level of strength activity index among the three fly ashes evaluated. However, the TGA results did not show any significant reduction in the calcium hydroxide content as a result of enhanced fineness of the fly ash. Therefore, it can be concluded that the role of fineness of fly ash in improving the strength activity index is not significantly influenced by the pozzolanic reactivity of the fly ash. It is likely that the improvement in strength activity index is more due to densification of the cementitious matrix due to finer particle size of the ground Class C fly ash.
- In terms of ASR mitigating ability, the increasing fineness of Class C fly ash does not appear to have any significant influence on ASR mitigation.

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