

Performance of alternative pozzolans – Alkali Silica Reactivity

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Abstract

With recent changes in the generation of power in the United States, including Florida, there has been a significant reduction of coal-derived utilities. The availability of Class F fly ash has diminished, and it is likely that location-specific shortages will become more frequent in the near future. It is imperative to find alternatives to coal fly ash that are abundant and suitable for use in concrete. The purpose of this proposed research is to make significant advancements with respect to the use of pozzolans available in the state of Florida, USA with a goal of more wide-spread acceptance throughout North America. This research investigated alternative pozzolans for cement replacement such as post-consumer waste glass, sugarcane bagasse ash, and ground volcanic rock for use in concrete. Long term effects of alkali-silica reaction in concrete were evaluated using ASTM C1293 which were then compared to mortar bar specimens prepared in accordance with ASTM C1567.

Keywords: alkali-silica reaction; alternative pozzolans; ASTM C1293; ASTM C1567; pozzolans

1. INTRODUCTION

Over the last 10 years, the production of energy has transitioned from coal fired power plants to alternative energy sources such as natural gas [1–3]. As a result, the production of coal fly ash has decreased and caused a shortage in the supply of this valuable by-product [3–8]. The concrete industry has grown to depend on fly ash due to the improvements and advantages associated with its use. In order to alleviate the decreased supply, several alternatives have been investigated, such as the use of impounded fly ash and alternative supplementary cementitious materials (SCM)[2,4,9,10]. For an SCM to be viable in the concrete industry, it must be available in sufficient quantities to meet demand, be able to adequately react with cement, and provide economic benefit (either immediately on the material cost or through reduced maintenance from increased concrete durability and service life) [2]. The recent demand for fly ash in the United States alone can range from approximately 15-30 short tons per year [1]. The alternative materials that were a part of this investigation include sugarcane bagasse ash (SCBA), post-consumer ground waste glass (GG), and ground volcanic rock (VR).

One of the main durability mechanisms of deterioration in concrete is Alkali-Silica Reaction (ASR). Traditionally, ASR was found to be problematic in aggregates and was originally referred to as alkali-aggregate reactivity (AAR). With the implementation of new SCM such as glass, the mechanism of ASR has expanded past a reactive aggregate to a potentially reactive SCM [2,11–14]. Therefore, when determining the adequacy of an alternative SCM, resistance to ASR should be evaluated as part of the durability performance of the material. While there are several procedures to evaluate the ASR mitigation of SCM, they each have advantages and limitations. The international evaluation methods ASTM C1260, ASTM C1567, and ASTM C1293 were used to evaluate the potential for each SCM to reduce ASR expansion.

2. BACKGROUND

Alkali-silica reaction (ASR) is known to cause extensive amounts of damage in concrete if not accounted for. This degradation mechanism involves a reaction between the alkalis in the pore solution of the cementitious system and reactive silica in the presence of water. The alkalis break down the silica and produce an absorptive gel that expands [15–20]. The expansion of the gel is what induces tensile stresses into the concrete and causes cracking [21,22]. In order to reduce or eliminate expansion, one of the three components in the reaction must be controlled. If the gel is kept from absorbing water, the alkalis are removed, or if the silica is non-reactive (or finely dispersed in low quantities), the expansion caused by ASR will be eliminated [23–25].

When cement hydrates, calcium silicate hydrate (C-S-H), calcium hydroxide (CH), and heat are produced. The presence of alkalis in the system, typically sodium and potassium, contribute to the formation of the gel. A common mitigation method to reduce the potential for ASR is the incorporation of a pozzolan. A pozzolan is a finely dispersed material that is typically high in silica, which when added into a cementitious system, reacts with the CH produced from initial hydration to form additional C-S-H. The additional C-S-H increases the strength of the concrete as well as densifying the concrete microstructure, which reduces water transport in the hardened concrete [2]. The addition of pozzolans can also result in alkali binding [18,21,26,27], which reduces the alkalis available to react; therefore reducing expansion [2,19,20]. Some common pozzolans include fly ash, silica fume, and slag cement [2,28–32]. Pozzolans are typically waste materials and are widely used in the concrete industry due to the benefits listed above in addition to their reduced cost [2,10,33,34].

2.1 Testing background

2.1.1 ASTM C1260 and C1567

The standardized test method for the rapid determination of ASR in aggregate, ASTM C1260 was first published in 1994. At 24 hours after mixing, the mortar bars are measured, submerged in water, and placed in an 80°C oven for 24 hours. The bars are then measured again to establish a baseline reading and immersed in 1N NaOH at $80.0 \pm 2.0^\circ\text{C}$. The samples are measured a minimum of three times within 14 days. The measurements can be extended past 14 days with at least one reading performed per week. Mixtures that have an expansion of more than 0.20% are considered to be potentially deleterious and are recommended to be evaluated using ASTM C1293 [35].

Due to the increased popularity of SCM in the production of concrete, ASTM C1567 was published in 2004 in order to evaluate the ASR expansion caused by combinations of SCM and known reactive aggregates. It is important to note that ASTM C1567 declares an alkali limit of 4.0% sodium oxide equivalent in the SCM being evaluated [36]. This limit is imposed because with high-alkali SCM replacements, the alkalinity of the pore solution in the mortar bars can be higher than the soak solution resulting in leaching of the alkalis from the mortar bars rather than alkali ingress [37–40]; additionally, using glass specifically has been shown to release alkalis in humid alkaline environments such as cement paste [41]. The result of this potential alkali leaching from the specimen is that observed expansion may be underestimated if the alkali content of the SCM exceeds the 4.0% limit. Therefore, it is recommended to use ASTM C1293 (discussed in the following section) for SCM with alkali contents exceeding 4.0% instead, as there are no alkali limits for the SCM in this method. The main advantage of ASTM C1260 and ASTM C1567 is the relatively short time they take for evaluation.

2.1.2 ASTM C1293

ASTM C1293 was first published in 1995 and requires the creation of concrete specimens rather than mortar. The purpose of the test is to evaluate the expansion caused by ASR in a concrete mixture. The method is widely used to evaluate if an aggregate is reactive or to evaluate if an SCM is adequate for reducing or eliminating expansion caused by ASR [2,21,40,42–44]. This method requires that the concrete be measured over a two-year period for the evaluation of the performance of concrete amended with SCM and one year when evaluating the potential reactivity of an aggregate (in the absence of SCM). Rather than being immersed in sodium hydroxide in ASTM C1260 or ASTM C1567, the specimens are placed above water, with a wicking material along the sides of the container, at $38.0 \pm 2^\circ\text{C}$ to maintain high humidity. Additionally, the alkalis are incorporated into the mixing water rather than being provided through submersion in solution like in ASTM C1567. A high alkali cement is required to be used with the addition of sodium hydroxide in the mixing water to bring the NaO₂ equivalent to

1.25% by mass of cement. Failure is reached if expansion exceeds 0.04% before two years when evaluating SCM and one year when evaluating aggregate [45].

2.2 Materials

2.2.1 Portland Cements

Three different types of portland cements were used for the two evaluation methods (ASTM C1567 and ASTM C1293). An ASTM C150 Type I/II and an ASTM C595 Type IL cement were used for ASTM C1567, while a High-Alkali (HA) Type I/II cement was used as per ASTM C1293 requirements [45]. The normal-alkali Type I/II cement is an ordinary portland cement, the Type IL cement is ordinary portland cement with approximately 11% limestone content as determined by thermogravimetric analysis, and the HA cement has an elevated amount of alkalis compared to the normal-alkali Type I/II cement.

2.2.2 Sugarcane Bagasse Ash (SCBA)

Sugarcane bagasse ash is produced through the incineration of sugarcane bagasse (processed pulp) and is a seasonal by-product in the production of sugar, meaning the supply is inconsistent. However, suppliers often impound excess material for later use rather than landfilling or land applying to crops. This study investigated two types of SCBA that were processed using different incineration methods. The ash from Facility A (SCBA-A) was incinerated, stored in an ash pond impoundment, and later collected, while the ash from Facility B (SCBA-B) was incinerated at a higher temperature and collected after incineration, bypassing the wet storage. The temperature at which SCBA is incinerated can affect the mineralogy of the resultant ash. When burned above 700 °C, the silica can begin to transform from α -quartz to form cristobalite [46,47]. The difference in incineration temperatures alone cause drastic changes between each SCBA, which lead to different reactions within the system. The production of SCBA from these plants is significantly lower than a coal burning facility producing fly ash and thus, the supply is not globally available. SCBA does have the potential to be a local solution in areas with abundant sugar production such as Brazil [48–50], India [50–52], and Thailand [50,53]. Another potential roadblock to the viability of SCBA as a cement replacement is the decrease in workability it causes. In order for SCBA to be used at higher replacements, admixtures are recommended, which could offset the economic benefits of its use. This reduction in workability is generally caused by incomplete incineration leading to high amounts of carbon.

2.2.3 Ground Volcanic Rock (VR)

Ground volcanic rock is produced by grinding volcanic rock to a particle size close to that of cement. It is often colloquially mistaken for volcanic ash as they are both produced in volcanic eruptions. However, the main difference between both materials comes from their respective cooling after eruption. Because of the natural difference in particle size, the volcanic rock cools at a slower rate than the volcanic ash, which alters the structure, producing a crystalline material as opposed to the amorphous volcanic ash. While volcanic rock is ground to a fine particle size, it is still crystalline in nature. The potential supply for VR is much higher than that of SCBA, but it is a solution that is highly location-dependent.

2.2.4 Ground Waste Glass (GG)

Ground waste glass is typically recovered from waste streams through recycling programs and is processed by cleaning and crushing collected waste glass. The advantage of using GG as a cement replacement is the global accessibility. Glass waste is globally ubiquitous; this availability can help reduce the shipping costs associated with using GG in concrete. The global recycling rate of glass is approximately 30% due to the fact that sorting coloured glass is rather expensive, which leads to most of it being crushed, and landfilled [2,5]. The abundant supply of glass and relatively low cost allow for a cost-effective SCM with significant supply to help offset the demand of the concrete industry [2,5].

2.2.5 Reactive Aggregate

The reactive aggregate used for all of the testing was acquired from the Jobe mine in El Paso, Texas, which is known to be reactive [54–56]. This is a reactive fine aggregate, which required no crushing for use in ASTM C1567 and was used as the fine aggregate for ASTM C1293.

3. TESTING PROCEDURES

X-ray fluorescence (XRF) was used to determine the chemical composition of the materials used for evaluation; the materials were analysed using glass beads fused at 1100 °C and then analysed with a Rigaku Supermini wavelength dispersive XRF.

Loss on ignition of each material was performed using a Perkin Elmer TGA 4000 thermogravimetric analyser using nitrogen as a drying gas.

X-ray diffraction (XRD) was performed to determine the crystalline composition of each material, as well as determine the amorphous content using corundum as an internal standard. The materials were analysed in powder form using a backfilled sample holder with a Panalytical X'Pert Powder XRD using copper radiation; the resultant scans were analysed using Profex v3.12.1.

The ASR susceptibility/mitigation potential of each material was evaluated using both ASTM C1567 (short-term, accelerated testing) and ASTM C1293 (long-term testing).

4. RESULTS

4.1 X-Ray fluorescence

The XRF results are summarized in Table 4.1, which shows the differences between the materials utilized. One of the key differences between SCBA-A and SCBA-B can be observed in the silica, calcium, alkali content, as well as loss on ignition (LOI). The calcium content is higher, while the silica content is lower in SCBA-B than in SCBA-A. Additionally, the alkalis in SCBA-B are higher than in SCBA-A. These differences are most likely caused by the processing of both materials. The changes to the calcium are most likely associated with the addition of gypsum, which can help reduce in emissions, the differences in silica content could possibly be caused by the use of a fluidized bed during processing, while the lower alkali content of SCBA-A is a result of leaching during the impoundment period [57,58]. The lower silica content in SCBA-B is partially explained by the additional alkalis, low LOI, and additional gypsum/lime added for emissions reduction. Additionally, it is possible for the relative silica content to be increased in biomass residuals depending on the pretreatment [59]. The differences in the chemical composition of each SCBA are exaggerated by the differences in LOI (determined by thermogravimetric analysis). The approximately double mass loss in SCBA-A skews the normalized percentages reported for XRF. The GG has a high alkali content due to the “soda” in soda-lime glass, which is the most common form of waste glass [2]. The alkali content is in excess of 13% which renders the results obtained in ASTM C1567 invalid. A portion of the research community has used the method to evaluate glass without acknowledging this; this evaluation is included to verify whether this 4.0% alkali limit is valid or invalid for waste glass for comparison to ASTM C1293.

Table 4.1: Chemical compositions of the materials used based on XRF and Loss on Ignition (LOI) results.

Compound	I/II Cement	IL Cement	HA Cement	SCBA-A	SCBA-B	GG	VR
CaO	65.3%	65.6%	61.6%	9.6%	25.4%	10.7%	6.1%
SiO ₂	20.0%	20.4%	20.6%	81.5%	55.4%	70.6%	60.8%
Al ₂ O ₃	5.0%	4.9%	5.1%	1.4%	1.8%	1.7%	16.3%
Fe ₂ O ₃	3.4%	3.7%	3.2%	1.0%	1.3%	0.3%	7.3%
MnO	0.0%	0.0%	0.1%	0.1%	0.1%	0.0%	0.2%
MgO	0.8%	0.9%	3.4%	2.5%	3.9%	2.1%	3.2%
TiO ₂	0.3%	0.2%	0.4%	0.1%	0.1%	0.1%	0.6%
Na ₂ O	0.1%	0.1%	0.2%	0.2%	0.5%	13.5%	3.1%
K ₂ O	0.3%	0.3%	0.9%	1.3%	4.3%	0.4%	1.3%
P ₂ O ₅	0.4%	0.1%	0.0%	1.8%	1.7%	0.0%	0.1%
SO ₃	3.2%	2.6%	3.2%	0.6%	4.1%	0.2%	0.1%
Na ₂ O Eq.	0.30%	0.30%	0.79%	1.06%	3.33%	13.76%	3.96%
LOI	2.3%	4.9%	2.3%	20.6%	11.2%	NA*	-0.2%**

*LOI was not performed on GG due to the elevated temperature causing melted beads to form and destroy crucibles.

** VR experienced slight mass gain due to oxidation at high temperatures

4.2 X-Ray Diffraction

The X-ray diffraction results are presented in Appendix A. A limitation of X-ray diffraction is the inability to identify amorphous components. For that reason, the XRD scan for glass is not presented, as glass is amorphous silica and did not show crystalline phases. The mineralogy of the three cements are similar, with some variation in peak intensities (for instance, there is more calcium carbonate in the Type IL cement as expected). There are however large differences between the SCBA-A and SCBA-B, which can be attributed to the differences in processing of each material. Burning biomass at different temperatures and rates alone can alter the formation of various crystalline components [46,47]. The addition of other compounds with respect to the reduction of emissions also introduces another aspect of variability. As expected, the VR has a relatively low amorphous content of approximately 10%, which validates the crystalline formation during slow cooling of the volcanic rock.

4.3 ASTM C1567

Figure 4.1 shows a comparison of the ASTM C1567 results through 28 days of exposure for Type I/II cement and Type IL cement. The expansion in the Type IL cement system is approximately 0.05% greater than that of the Type I/II cement system at 28 days; however, both systems exceed the testing threshold for potentially deleterious mixtures within three days of exposure. Both systems have a similar rate of expansion.

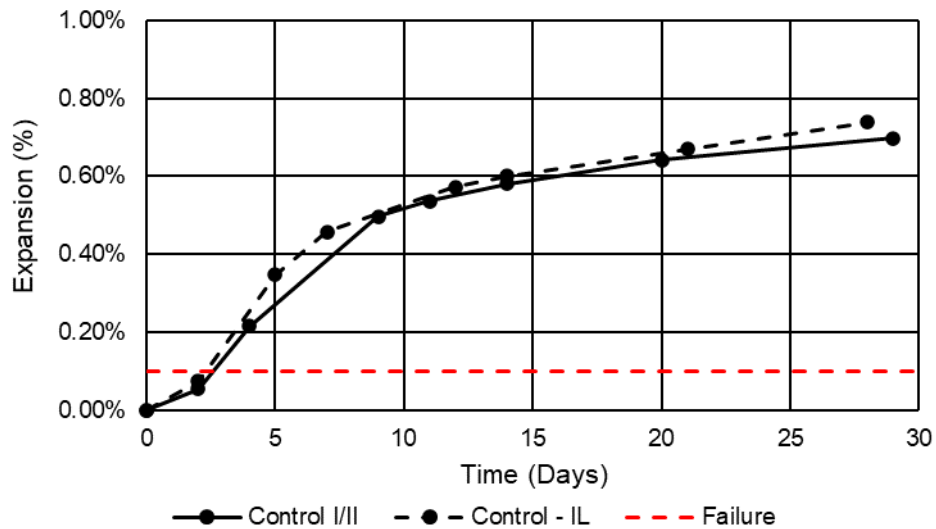


Figure 4.1: ASTM C1567 results for Type I/II and IL cement.

Table 4.2 summarizes the ASTM C1567 results in the systems with Type I/II and Type IL cement replacements while Figure 4.2 and Figure 4-3 show a visual representation of the comparisons. Furthermore, the table includes notes on which individual mixes passed the evaluation according to ASTM C1567 at 14 days.

The trend observed regarding GG as a Type I/II cement replacement shows the expansion was reduced with replacements of 30 and 40% as they are both below the failure threshold of the test method at 14 days. This trend is reported often in the literature; however, as most ground glass has an alkali content that exceeds the 4.0% limit, these values are erroneous and should not be used to qualify mixtures. Once measured past the 14-day benchmark indicated in the test method, the expansions of the high replacement mixes exceed 0.10%. When used as a Type IL cement replacement, all three replacement of GG decreased the expansion in the system to below 0.10% at 14 days. Every GG replacements exceeded the 0.10% expansion threshold when the measurements were extended to 28 days.

The 10% replacement of SCBA-A had a higher amount of expansion when compared to control, but as the replacement increased, the total expansion decreased. The same general trend is observed with the SCBA-B, in that as replacement increases, expansion decreases. However, the mix incorporating 30% SCBA-A had a much lower expansion. Even with increasing performance with higher replacement percentages, all six of the SCBA mixes failed at 14 days. While higher replacements may be able to reduce expansion further, it would likely require large doses of admixtures to allow for workable mixes. The total expansion results for SCBA-A and SCBA-B as a Type IL cement replacement are lower than the Type I/II replacements. However, neither material at any replacement reduced expansion below the 14 day expansion limits. SCBA-A is more effective in reducing expansion at higher replacements.

The use of VR as a Type IL cement replacement was also evaluated at 20%. This mix exhibited expansion exceeding the maximum expansion well before the 14 day minimum, but did decrease the overall expansion compared to the control mixture.

Table 4.2: Summary of ASTM C1567 results for each cementitious combination:

Cement	SCM	14-Day Expansion	28-Day Expansion	14-Day Passed
I/II	Control	0.68%	0.82%	NO
I/II	20% GG	0.15%	0.27%	NO
I/II	30% GG	0.05%	0.17%	YES 1
I/II	40% GG	0.04%	0.14%	YES 1
I/II	10% SCBA-A	0.75%	0.86%	NO
I/II	20% SCBA-A	0.59%	0.70%	NO
I/II	30% SCBA-A	0.27%	0.32%	NO
I/II	10% SCBA-B	0.65%	0.78%	NO
I/II	20% SCBA-B	0.59%	0.69%	NO
I/II	30% SCBA-B	0.49%	0.58%	NO
IL	Control	0.60%	0.74%	NO
IL	20% GG	0.09%	0.25%	YES 1
IL	30% GG	0.08%	0.21%	YES 1
IL	40% GG	0.05%	0.11%	YES 1
IL	10% SCBA-A	0.61%	0.74%	NO
IL	20% SCBA-A	0.54%	0.62%	NO
IL	30% SCBA-A	0.25%	0.32%	NO
IL	10% SCBA-B	0.56%	0.67%	NO
IL	20% SCBA-B	0.51%	0.62%	NO
IL	30% SCBA-B	0.45%	0.56%	NO
IL	20% VR	0.42%	0.49%	NO

¹ The observed 14-day expansion is below the testing threshold; however, the alkali content of the SCM is above the 4.0% limit rendering the results invalid.

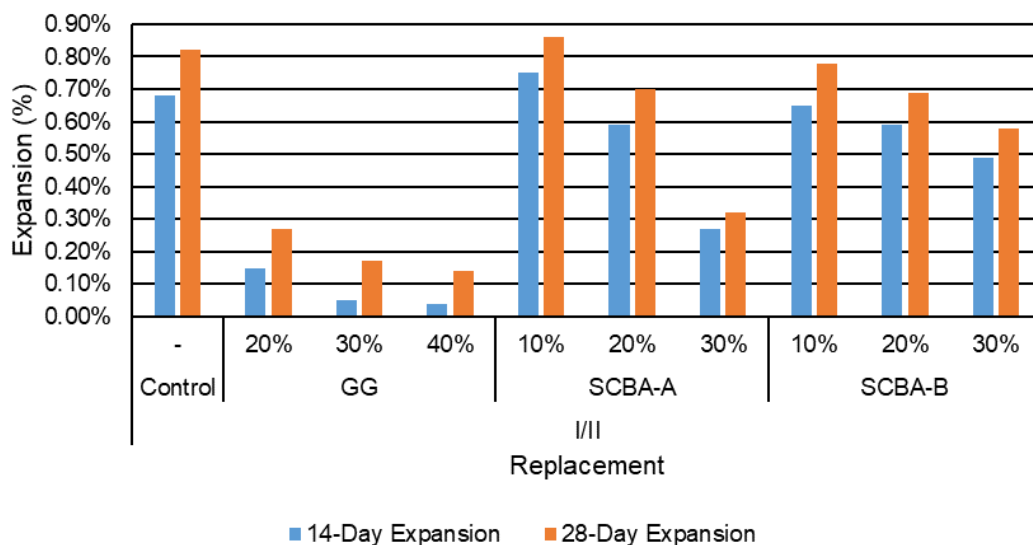


Figure 4.2: Expansion values at 14 and 28 days for Type I/II cement systems.

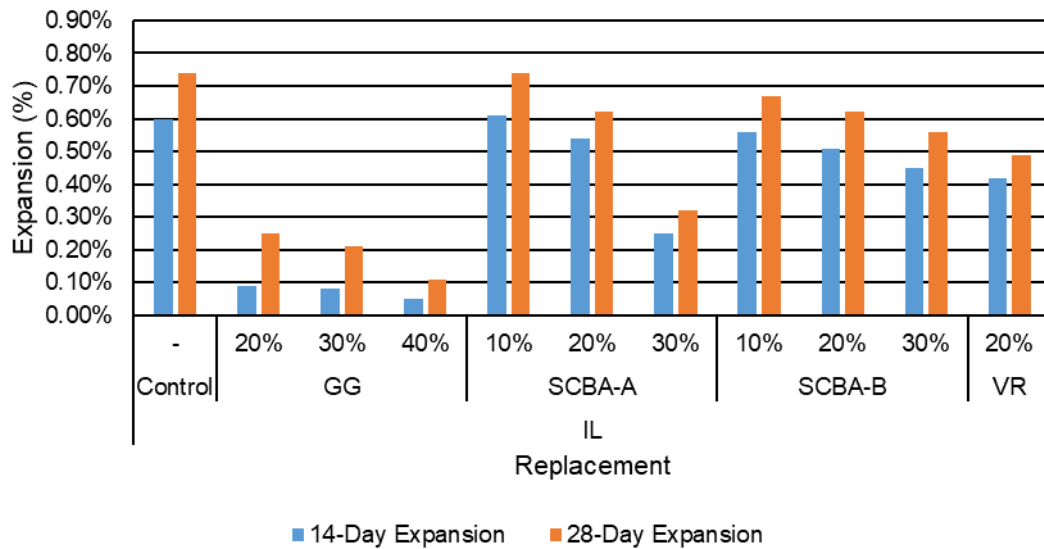


Figure 4.3: Expansion values at 14 and 28 days for Type IL cement systems.

4.4 ASTM C1293

Figure 4.4 shows the current ASTM C1293 results for the samples containing GG and VR as HA cement replacements. All of the replacement percentages have exceeded the expansion limits before the conclusion of the test (24 months). While there was a decrease in expansion compared to control, the VR and GG mixes failed to reduce expansion below the threshold limits before the two-year period.

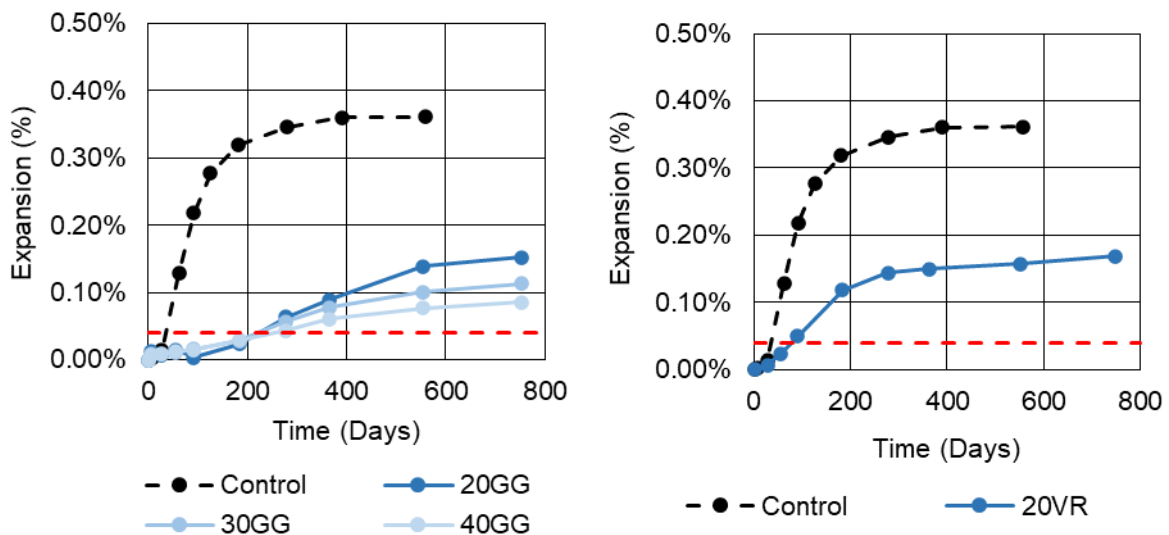


Figure 4.4: ASTM C1293 results with 20-40%GG (left) or VR (right) as HA cement replacement

Figure 4.5 presents the results from ASTM C1293 for SCBA-A and SCBA-B. The results indicate that SCBA-B is inadequate at reducing expansion caused by ASR, with expansion being increased for the samples with a 20% replacement. SCBA-A on the other hand, shows a distinct decrease in expansion with a 20 and 30% replacement. The 20% replacement exceeds the limits at 550 days of exposure. The expansion in the specimen with a 30% replacement surpassed the limit on the final measurement.

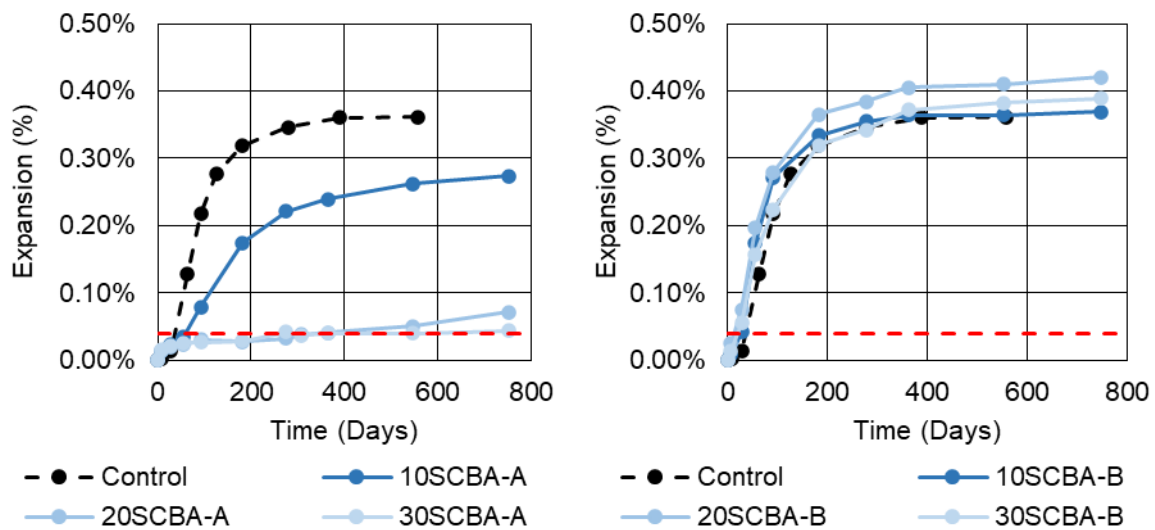


Figure 4.5: ASTM C1293 results utilizing 10-30%SCBA-A and SCBA-B as HA cement replacement

5. DISCUSSION

When comparing both types of cements to each other, the Type IL cement had a lower amount of expansion caused by ASR than the Type I/II cement throughout most of the evaluation period. Similarly, the mixtures evaluating the SCM using Type IL cement performed better than the mixtures using Type I/II cement. The reason for the significant difference between the cements is likely due to the limestone filler present in the Type IL cement. Monocarbonate formation is dependent on the amount of calcite present in the cementitious system [60,61]. The increased formation of stable monocarbonate results in an increased stability in the cementitious system. The limestone addition is also typically inter-ground with the cement, which results in a finer particle size than the cement. This can contribute to the densification of the concrete, which would then reduce the water transport in the system as well [60].

The only material that mitigated ASR in the accelerated mortar bar evaluation method was GG. However, the standard states that using materials with an alkali content exceeding 4.0% (GG contains more than 13%) “may underestimate the expansion of cementitious systems...”; therefore, the results are not reliable for this material and test method. When extended past the 14 day evaluation period to 28 days though, the expansion surpassed 0.1% by the 21 day measurement. Similar trends are observed in other publications which used ASTM C1567 to evaluate the ASR performance of GG [62–66]. The leaching in ASTM C1567 is accounted for in the immersion solution, which resupplies alkalis into the system. In the case of materials with alkali contents above 4.0%, the 1N NaOH solution is not enough to resupply the alkalis lost through leaching, causing a decrease in alkalis in the cementitious system; and therefore unconservative levels of expansion. It is specified in ASTM C1567 to use ASTM C1293 to evaluate SCM with high alkali contents, such as glass in order to produce more reliable results. The only other material with a significant reduction in expansion was SCBA-A, particularly at a 30% replacement, but the expansion still exceeded the 14-day limit. Similar overall trends were observed by [50,67,68], in which it was concluded that SCBA decreases expansion caused by ASR. The SCBA used in those studies is closest to SCBA-A, based on the chemical composition. However, there are differences between the results in each study comparatively. The results shown in [50,67] are rather similar, which could be due to the use of similar reactive aggregates. An overall expansion of approximately 0.15% at 14 days and 0.20% at 28 days was observed in the control for both publications. The behavior of the SCBA in each publication is also rather similar. The expansion of the control specimens was lower than the expansion observed by [68] and the results presented in Figure 4.1. Using the common general trends observed in each publication, it can be concluded that SCBA has the potential to reduce expansion caused by ASR. The scope of ASTM C1567 states that the purpose of the method is to evaluate combinations of aggregates and SCM. Broad determination of an SCM’s performance is not applicable due to variability in the level of reactivity different aggregates have [69,70]. Each mix in this study failed ASTM C1293. However, each material with the exception of SCBA-B has shown a decrease in expansion as the replacement increases. The SCBA-A significantly reduced expansion caused by ASR at replacements greater than 10%. While the use of SCBA-A can reduce

expansion considerably, the increased replacements cause a significant decrease in workability. The improved durability could help offset the increased cost of admixture dosages required to mitigate decreased workability. The specimens using GG have all exceeded the expansion limits, which verifies the unreliability in using ASTM C1567 for high alkali SCM. Additionally, the SCBA-A performed better in ASTM C1293 than in ASTM C1567, while the SCBA-B performed worse when comparing the results of both methods. A summary of the results for both evaluation methods is shown in Figure 5.1. The failure condition for both tests is shown with the bottom left area representing a pass condition for both tests. The mixes left of the ASTM C1567 failure condition all have GG and are therefore invalid to conclude on. The data points closest to the failure conditions for ASTM C1293 are SCBA-A mixes with a 30% replacement. However, the failure threshold was still surpassed for that mixture.

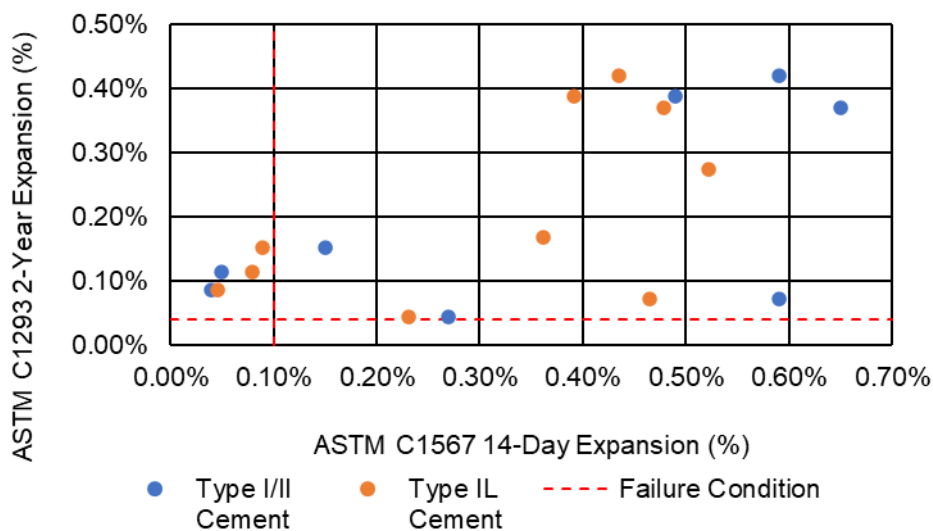


Figure 5.1: Comparison of results between ASTM C1567 and ASTM C1293.

6. CONCLUSIONS AND RECOMMENDATIONS

To further the acceptance and use of alternative supplementary cementitious materials, the performance of concrete with respect to ASR was evaluated using different methods. Due to the limitations of ASTM C1567, high alkali glass cannot be evaluated with this method and must be evaluated using ASTM C1293. Both types of SCBA and the VR failed to reduce expansion caused by the Jobe sand below the ASTM C1567 limit of 0.10%. All of the materials failed ASTM C1293. ASTM C1567 and ASTM C1293 address the issue of evaluating the mitigation potential of SCM's using different types of aggregates by clarifying that the results are only representative of the combinations being tested. Therefore, results should be interpreted in terms of mitigating the specific aggregate used, rather than forming general conclusions [36,45]. It is recommended that further testing be performed to compare the effect of different aggregate reactivity with various SCM.

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8. APPENDIX A

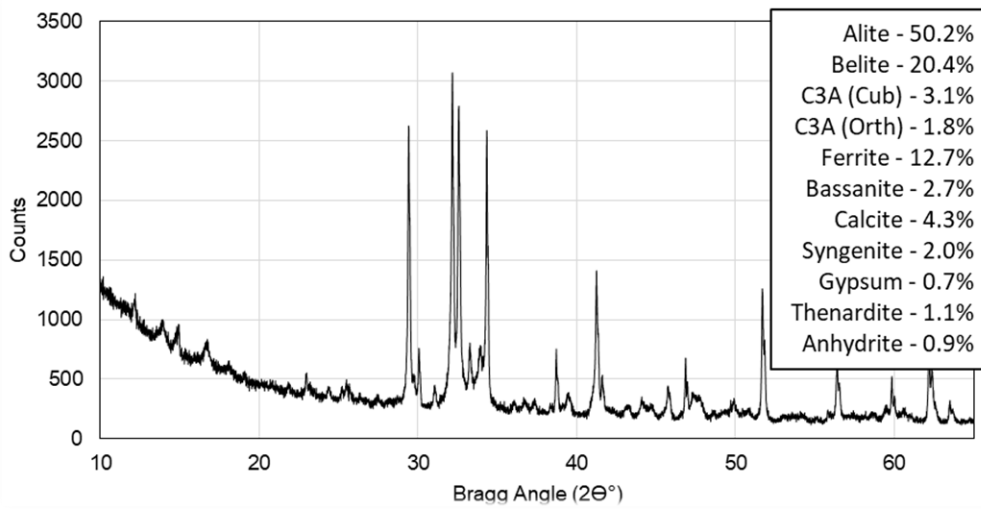


Figure 8.1: X-ray diffractogram for Type I/II cement.

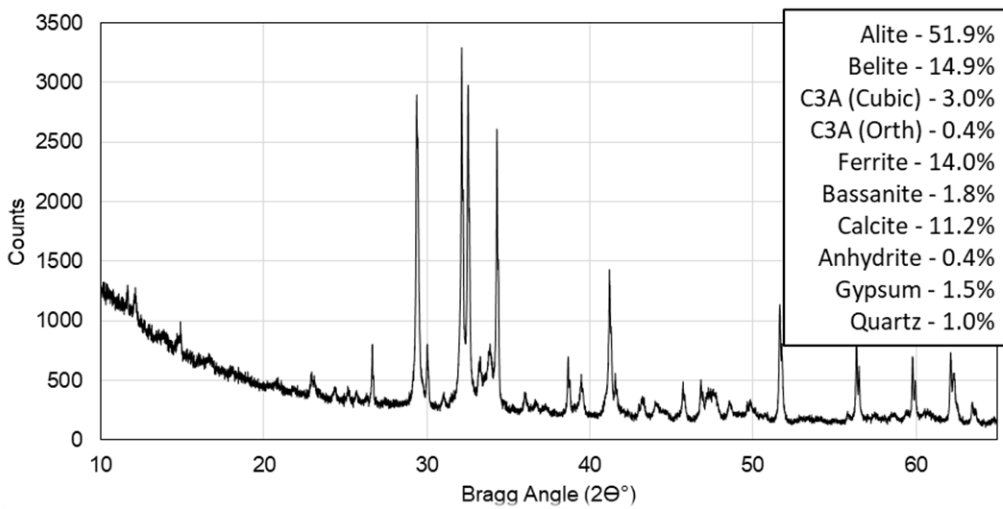


Figure 8.2: X-ray diffractogram for Type IL cement.

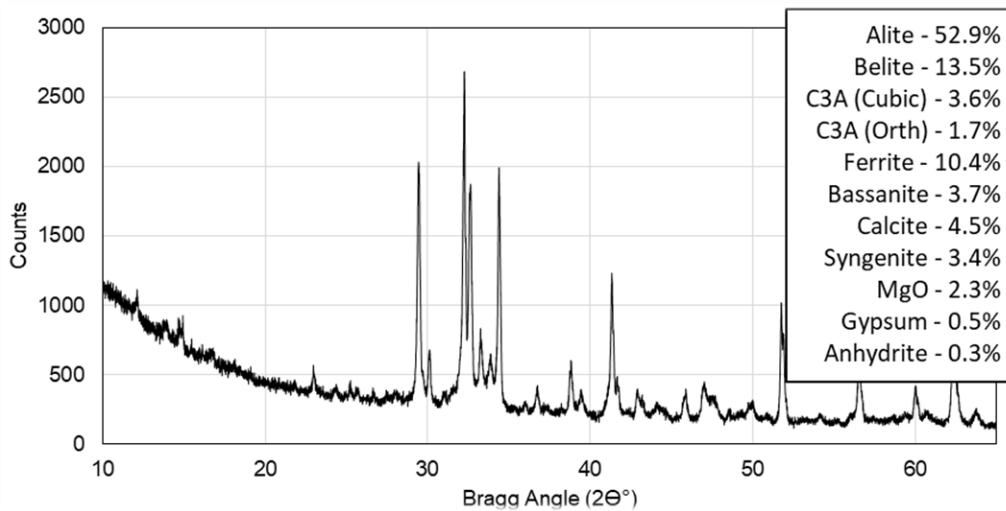


Figure 8.3: X-ray diffractogram for Type HA cement.

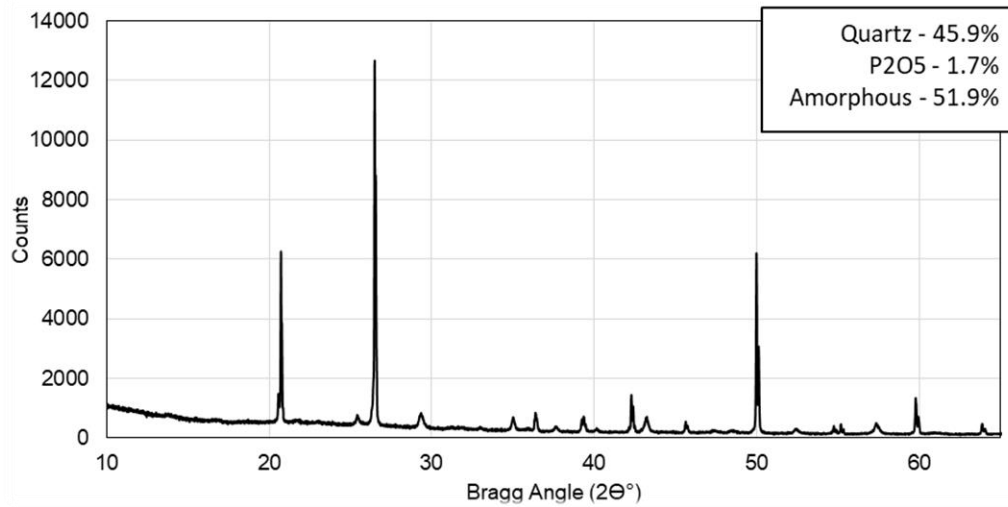


Figure 8.4: X-ray diffractogram for SCBA-A.

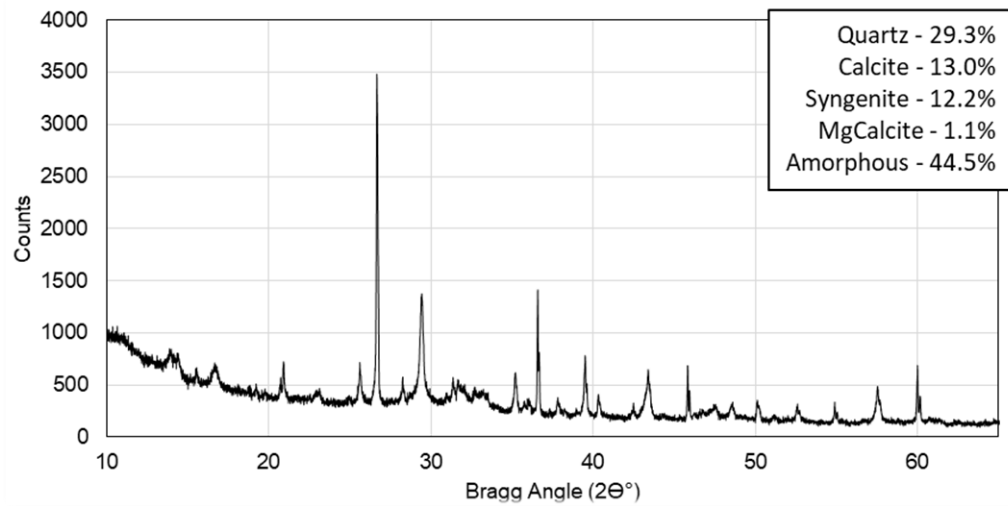


Figure 8.5: X-ray diffractogram for SCBA-B.

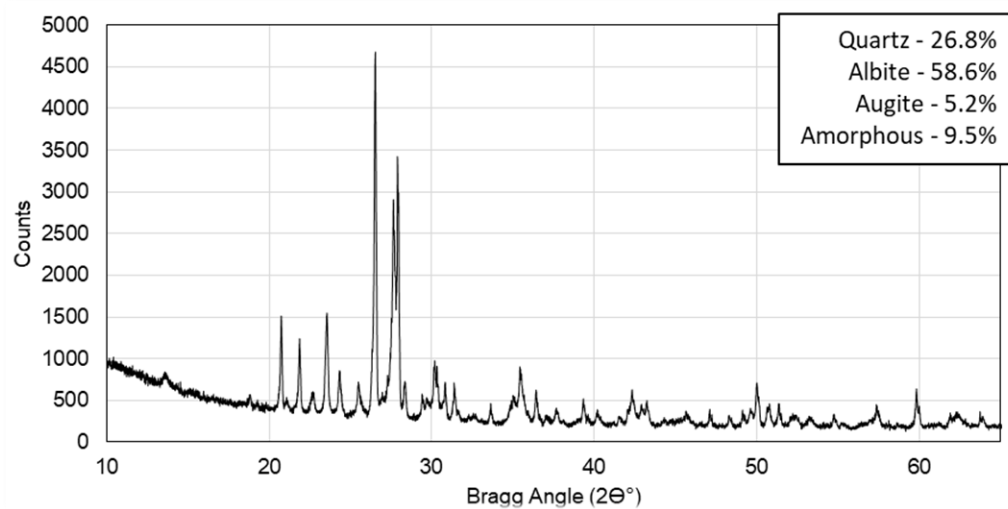


Figure 8.6: X-ray diffractogram for VR.