Evaluation of the Correlation Between ASR Expansion and Pozzolanic Reactivity of Ternary Concrete Systems Containing Glass Powder

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Abstract. Glass powder (GP), as supplementary cementing materials (SCMs) in concrete manufacturing, is characterized by two controversial facts. Post-consumer GP is mostly made of silica in an amorphous phase, which provides high pozzolanic reactivity potential. However, its high alkali content (≈13% Na_2O_{eq}) has a negative effect on concrete expansion by engaging alkali-silica reactions (ASR), which differentiates GP from other conventional SCMs. Both perspectives are the subject of multiple debates concerning the possibility of alkali release into the concrete pore solution over the long term. As well as how the synergistic combinations of GP and other SCMs could be the key to this issue. The combined use of GP and other SCM with higher silica/alumina contents offers a promising alternative to reduce the expansion of concrete to the normative lower limits. In this context, this paper focuses on studying the mechanism that prevails by linking the expansion of binary and ternary concrete and the pozzolanic reactivity of the respective GP/SCM blends by isothermal calorimetry based on the R^3 method. All to establish a potential dosage that will allow the broad use of GP as SCM. The expansion of concrete prisms made with reactive aggregates and different percentages of GP and metakaolin, silica fume, fly ash or blast furnace slag was measured for over 6 years. The synergy was observed in specific blends, as the GP/metakaolin and GP/silica fume samples systematically decreased concrete expansion in comparison with the binary prisms. Accordingly, calorimetry results reported an increased reactivity for the same systems, showing a wide correlation within both parameters.

Keywords: Glass powder · ternary blend concrete · alkali-silica reaction · pozzolanic reactivity · supplementary cementitious materials

1 Introduction

Construction-related environmental issues have mobilized extensive research on recyclable materials to replace others that require high energy costs and owe a significant carbon footprint [1]. In this perspective, sustainable hydraulic binders, pozzolans, have been used to reduce the cement content in the second most used material on earth,

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concrete [2, 3]. Due to its wide availability and enhancement of concrete's long-term mechanical and durability, the glass powder (GP) is one of the most recently standardized pozzolans [4, 5].

GP used as supplementary cementitious materials (SCM) in concrete manufacturing can be characterized by two controversial facts. The high alkali content of post-consumer glass (≈13% Na2Oeq) has the potential to generate alkali-silica reactions (ASR), which differs GP from other conventional SCMs [6–9]. However, recent studies suggest that GP effectiveness on suppressing concrete expansion is enhanced when associated with other binders [10–15]. Essentially, ternary blends are designed to improve the attenuation capacity of a SCM at a nominal dosage level that doesn't compromise concrete mechanical behavior [10]. In this scenario, this paper aims to investigate the potential synergy of 6 years old binary and ternary concrete prisms containing GP combined with Metakaolin (MK), Silica Fume (SF), Fly ash (FA) or Blast furnace slag (BFS) by means of ASR expansion test.

On the other hand, the difficulty remains in overcoming the clinker influence issue on the assessment of the studied SCMs reactivity. Common testing practices to assess the level of reactivity, such as the Fratinni method and the Modified Chapelle Method, generally have limitations, poor correlation with resistance development, and are not easily reproducible [16–18]. In this context, the RILEM TC 267-TRM R^3 model has been used as a tool to isolate the reactivity of those systems by conceiving a paste capable of reproducing the concrete pore solution and solid phase [19, 20]. Its scope was validated for SCM of certain prescribed origins, such as calcined clays and by its accurate correlation with the 28-day compressive strength correlation. It is, although, uncertain if the R^3 method could reach long term accuracy for lower pozzolanic reaction kinetics, as for FA and BFS. Therefore, the reactivity of 13 ternary and 6 binary blends was tested by isothermal calorimetry according to the R^3 method. The cumulative heat release of each sample was then correlated to the 2 and 6 years concrete prisms expansion data for a long term reactivity evaluation of the GP blended mixes.

2 Materials

2.1 Chemical Composition of the SCM

The same SCMs were used for both the expansion concrete prisms tests and the isothermal calorimetry R^3 pastes. The concrete prisms studied in this paper were previously cast by Fily-Paré (2021) [14] and the specifications of the used materials figures as follows: post-consumer ground GP from a local recycling facility in the province of Québec (Canada); MK from Georgia (USA); SF from a ferrosilicon plant also located in the province of Québec (Canada); grade 80 BFS from Ontario (Canada); class F FA from Alberta (Canada). The concrete prisms were cast using a 0.94% Na_2O_{eq} alkali content GU type cement, from Québec (Canada), boosted to a 1.25% Na_2O_{eq} by addind NaOH in the mix water A highly reactive Spratt limestone and manufactured high-purity limestone sand were used as coarse and fine aggregate. As for the R^3 model pastes, potassium hydroxide (KOH) with purity $\geq$ 85.0%, potassium sulfate (K_2SO_4), 99.43% purity, and purified limestone ($CaCO_3$) along with portlandite (Ca(OH)2) were used.

The chemical composition and the loss of ignition (LOI) of the above binder materials were obtained by major elements fusion followed by ICP-OES analyses and are presented in Table 1.

Table 1. SCMs chemical composition as determined by ICP-OES.

Oxide	GP*	SF	MK	FA	BSF	OPC
SiO_2	68.98	93.98	50.81	53.63	37.09	18.70
CaO	10.88	0.54	0.06	11.96	37.05	60.81
Al_2O_3	2.09	0.23	43.86	24.17	11.61	5.00
Fe_2O_3	0.46	0.14	0.48	3.71	0.42	3.70
MgO	1.09	0.25	0.05	1.07	12.17	2.70
MnO	0.021	0.034	0.004	0.048	0.326	0.047
Na_2O	12.49	0.12	0.27	2.44	0.42	0.25
K_2O	0.66	0.57	0.25	0.69	0.5	1.05
Na_2Oeq	12.92	0.49	0.43	2.89	0.75	0.94
TiO_2	0.066	0.005	1.415	0.665	1.088	0.228
L.O.I	1.71	3.2	1.84	0.80	1.80	1.90

2.2 Concrete Prisms Specimen Confection

The concrete specimens investigated in this study were produced in 2016 for the experiments carried out by Fily-Paré (2021). The binary and ternary mixtures were cast and tested in accordance with CSA A23.2-28A standard practice, with slight modifications in the prism dimensions to avoid alkali leaching (10 cm^2 transversal section). All concrete mixtures were made with 420 kg/m^3 of cementitious materials, 1054 kg/m^3 of coarse reactive aggregate and between 677 and 732 kg/m^3 of non-reactive fine aggregate, and with a water-to-binder ratio of 0.42.

Concrete mixtures were made with 15–50% of cement replacement. The GP content was fixed at 10 and 30% for a 5–10% SF or 5–15% MK ranged percentage. Meanwhile, mixes incorporating 15–30% FA or 20–40% BFS ranged in their GP content from 10 to 20% (Table 2). Binary mixes were made using 10, 20 and 30% of GP. The percentages were selected according to the specificities of each SCM and their optimum cement replacement level without compromising the workability and mechanical properties of the concrete.

A total of 13 ternaries and 3 binaries samples will be analyzed in the present study.

Table 2. GP/SCM content in concrete prism dosage

GP (%)	FS (%)		MK (%)	
	5	10	5	15
10	10GP5SF	10GP10SF	10GP5MK	10GP15MK
30	30GP5SF	30GP10FS	30GP5MK	-
GP (%)	FA (%)		BFS (%)	
	10	30	20	40
10	10GP15FA	10GP30FA	10GP20BFS	10GP40BFS
20	20GP15FA	-	20GP20BFS	-

2.3 Mix Design of the R^3 Test

In order to access the pozzolanic reactivity of both binary and ternary systems, the mix design of the pastes was based on the one proposed in the R^3 test method. This test was originally designed by Avet et al. (2016) [21] and consists of a group of viable and robust techniques adapted to measure the chemical reactivity of calcined clays. According to the literature [17, 20, 22, 23], conventional pozzolanic reactivity tests have low reproducibility, accuracy and often show poor correlation with the strength development of mortars and concretes [15, 20, 24, 25]. Therefore, the R^3 model is recommended by the RILEM TC 267-TRM (2018) and committee as the most effective for a correlated strength development [17].

The basic principle of R^3 test method is to use a simplified model to isolate the reactivity of a pozzolan from that of the Portland cement [21]. For this purpose, a mix of the GP, as the main pozzolan, a second SCM, $Ca(OH)_2$ and $CaCO_3$ in a 0.3 M potassium (K) alkaline solution (pH around 13.6) was designed. The solution R^3 is composed of fixed proportions of KOH and K_2SO_4 dissolved in deionized water. The solids are added in proportions of 11.11 g SCMs, 33.33 g of $Ca(OH)_2$, and 5.56 g of $CaCO_3$, as presented in Table 3, resulting in a water/binder ratio of 1.2.

Table 3. Mix design for R^3 model paste.

Solution			Solids		
K2SO4(g)	KOH (g)	DW (g)	CaCO3	Ca(OH)2	SCM
1.2	0.24	60	5.56	33.33	11.11

For the R^3 pastes, the SCM parcel of the paste was fractioned into a total of 13 different SCM/GP ratios according to the concrete's mix design, as shown in Table 4.

Table 4. GP/SCM ratios for binary and ternary pastes

Mixture	SCM/GP	GP (g)	FA (g)	BFS (g)	SF (g)	MK (g)
Binary	-	11.11	11.11	11.11	11.11	11.11
10GP15FA	1.50	4.44	6.67			
10GP30FA	3.00	2.78	8.33			
20GP15FA	0.75	6.35	4.76			
10GP20BFS	2.00	3.70		7.41		
10GP40BFS	4.00	2.22		8.89		
20GP20BFS	1.00	5.56		5.56		
10PV5FS	0.50	7.41			3.70	
10GP10SF	1.00	5.56			5.56	
30GP5SF	0.17	9.52			1.59	
30GP10SF	0.33	8.33			2.78	
10GP5MK	0.50	7.41				3.70
10GP15MK	1.50	4.44				6.67
30GP5MK	0.17	9.52				1.59

3 Methods

3.1 Isothermal Calorimetry Measurements

The calorimetry tests were carried out using a Calmetrix isothermal calorimeter I-Cal HPC 2000 with two logging channels. The baseline of the calorimetry was measured with 30 g of inert quartz in each acquisition channel for 4 days. Firstly, the SCM blends were weighed and mixed with the $Ca(OH)_2$ and $CaCO_3$ in the 125 ml plastic calorimeter vessels until no visual agglomeration was observed. Then, the R^3 solution was prepared in a magnetic stirrer, set to 40 °C, for about 30 min to ensure homogenous dissolution. Both solids mix and R^3 solution were placed in an oven at 40 ± 1 °C for at least 2 h, until reach thermal balance. The liquid and solid phases were then hand-mixed for 2 min using a plastic stirrer that remained inside the container afterwards to avoid mass loss. Immediately after, about 43.2 g of paste for each sample was sealed and placed inside the calorimeter. The temperature was fixed to 40 °C and the data acquisition was carried out for 7 days.

The heat flow and, by extension, the cumulative heat of the sample were calculated by eliminating the first 1 h due to the temperature loss during the mixing time at environmental temperature ± 23 °C, which intemperate with the calorimeter signal stabilization causing an initial negative flow. The results of calorimetric measurements were normalized to the SCM mass in the samples.

3.2 Concrete Prisms Test – Expansion Due to ASR

Concrete prisms were cast and stored in accordance with CSA 23.2-28A. The length-change measurements were performed without pre-cooling of the test specimens. A total of four large prisms were made for each concrete mixture and All specimens were regularly monitored for mass and length variations over a 6-year period.

4 Results and Discussion

4.1 Cumulative Heat Measurements

Binary Mixtures. The Fig. 1 shows the cumulative heat normalized per gram of SCM for the binary R^3 pastes. It is important to note that the key parameters that influence the kinetics of the pozzolanic reactions, such as the temperature, the pH and the portlandite content were the same for every sample, at the initial state of the test. The phases C-S-H, C-A-S-H, AFm, and AFt precipitate resulting in a strong exothermic heat release, which occurs mainly in the first 24 h of reaction [23, 26]. It's clear that, in that interval, the MK and GP samples were the most reactive, releasing respectively 94% and 87% of the total heat accumulated in 7 days. For those samples and for the OPC, the behavior post-peak displays an achieved state of equilibrium as the curves flatten, which possibly means that the amount of amorphous silica in solution have precipitated as C-S-H and C-A-S-H. Meanwhile, the hydraulic binder BFS, as well as the pozzolans SF and FA released only 52%, 39% and 20%, respectively. The first two showed very similar post-peak behaviors transcribed into exponential growth curves, which differentiates of the almost linear one observed for FA. Although the surface aspect and shape of the SCMs were not evaluated in the present work, it is constated in the literature that the surface aspect and form of the SCM particles are one of the main factors that dictate their degree of reaction [27]. The spherical smooth shape of FA is known to delay the pozzolanic reaction to a large extent, as the silica is well-tangled in its round balanced structure [28]. The FA curve tendance shows no sign of heat release stabilization in 7 days, which suggests that its reactivity can only be fully assessed by different long-lasting tests.

The results shown in the graph of the Fig. 1 seem to agree with those obtained in previous studies [19, 23].

Ternary Mixtures. The cumulative heat normalized per gram of SCM for the combined GP/SCM mixtures are presented in Fig. 2a-d. Overall, the presence of GP appears to reduce heat release in all ternary systems, when comparing the amount of heat emitted individually by GP and other SCMs. Furthermore, it can be observed that the increase in GP/SCM ratio reflects directly in the stabilization of heat release for all ternary samples.

For GP/FA group (Fig. 2a), the GP seemed to even block the later reaction as displayed in the ternary sample's post peak curve inclination (after 24 h). In the other hand, the R^3 protocol is adapted for short term reactivity analyses and it seems not to be compatible with the FA used in this work. For instance, the 10GP30FA sample shows a potential tendency to surpass the GP's reactivity in the long term. Similar behavior is noticed in the GP/SF group (Fig. 2b), although the GP/SF samples seem to have reached thermodynamic equilibrium by the end of the 5th day.

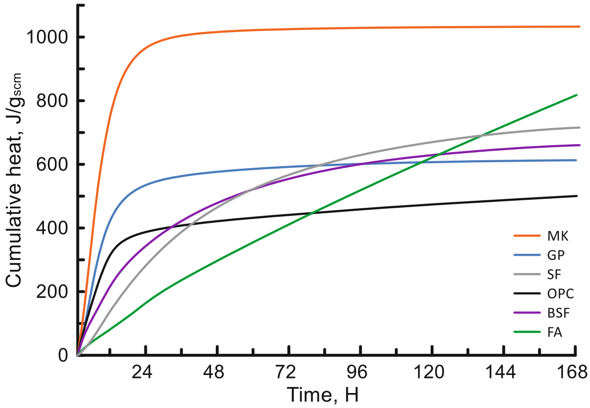

Fig. 1. Cumulative heat curves for R^3 binary pastes.

The scenario changes when we observe the GP/BFS and GP/MK groups, as we can pinpoint a synergistic effect. In the first group, the reactions are happening even after the peak, which indicates that the addition of BFS to the system provides a deceleration in the kinetics, meaning that the portlandite is being consumed slowly for the precipitation of the C-S-H. The 10GP40BFS and 10GP20BFS surpassed GP's heat release on the 4th day of reaction showing slight growth tendance, whilst the GP has reached equilibrium after 3 days.

The MK analyzed in this study has liberated the highest amount of heat which is reflected in the GP/MK mixtures as 10GP15MK and 10GP5MK having the highest accumulated heat liberation of all ternary mixtures, surpassing GP since the beginning of the reaction. If we consider that the R^3 method was originally conceived to evaluate the calcined clay's reactivity, it is very possible that the 0.3 M K matrix is accelerating the dissolution of the reactive phases ($>$90% $SiO_2 + Al_2O_3$) of its similar, MK. Being the series that arrived quickest in the plateau.

4.2 Pozzolanic Reactivity and ASR Expansion

When analyzing the binary mixes containing GP and OPC, the expansion decreases as the glass content increases 10% GP (0.149%), 20% GP (0.145%) and 30% GP (0.110%), whilst OPC (0.173%) [14] in 6-years expansion for GP samples and 2-years expansion for OPC. This fact by itself indicates that the Na_2O_{eq} content is not the only factor at stake in ASR expansion dynamic, as the GP alkali content is 10 times higher than that of the boosted cement (1.25% Na_2O_{eq}). Rather is how and when those alkalis are released in the pore solution and how much is absorbed by the C-S-H.

The only mixes that showed expansion below the 0.040% two years threshold were ternary concretes: 30GP10SF, 10GP15MK, 10GP30FA, 20GP15FA and 10GP40BFS (Fig. 3a,b). The expansion behavior seems to match those obtained in the studies carried out by [5, 10, 12]. Within those mixtures, considering the cement replacement level (25%), the most efficient is the 10GP15MK (0.040%), which is also the R^3 ternary

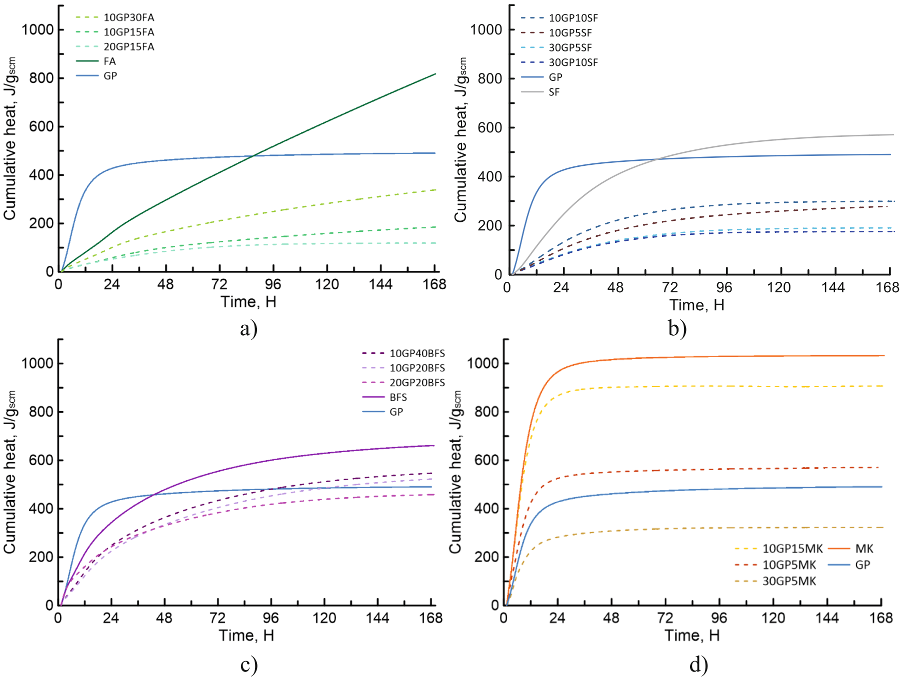

Fig. 2. Cumulative heat curves for R^3 model ternary pastes: a) GP/FA; b) GP/SF c) GP/BFS; d) GP/MK.

paste that released the highest amount of cumulative heat in the isothermal calorimetry test. By replacing 40% of the cement, the lowest expansion was achieved for 30GP10SF (0.023%), when compared to 10GP30FA (0.040%) and even to 50% cement replacement 10GP40BFS (0.032%). A correlation between the last two mixtures' expansion and their R^3 measured reactivity is also possible, as they were both previously mentioned as the samples showing a tendency of growth in the cumulative heat curve.

Although all the above-mentioned mixtures remained close to the threshold and show a sign of stabilization in 6-year expansion measurements, the only system capable of containing it under 0.040% was the 30GP10SF. The latter has also shown a stabilized expansion from 2 to 6 years.. Furthermore, by increasing only 5% of the SF content, the expansion went from 0.089% (30GP5SF) to 0.022% (30GP10SF), which could suggest that, in those systems, there may be chemical/physical conditions that limit the leaching of alkalis from the glass. Also, the Ca/Si ratio in the C-S-H produced by the pozzolanic reactions is lower than that of the cement hydration which increases the alkali absorption on the C-S-H structure. In Fig. 4a,b it is possible to compare the 30GP10SF ternary concrete's surface and cross-section to those of a highly damaged binary concrete (20GP) in 6 years of test measurements.

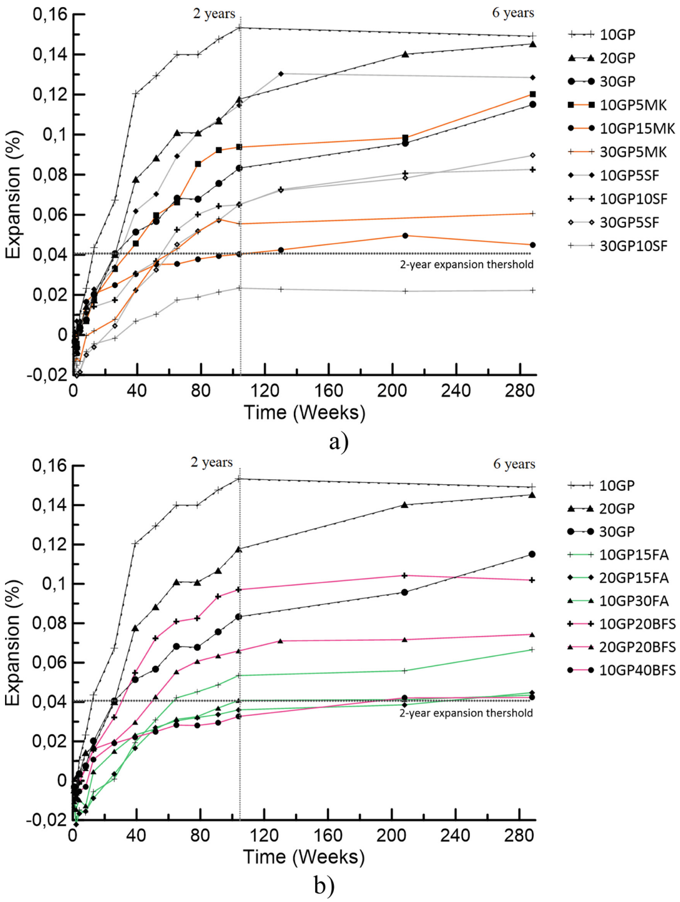

Fig. 3. 6-year expansion for binary and ternary concrete prisms: a) GP/MK and GP/SF; b) GP/FA and GP/BFS.

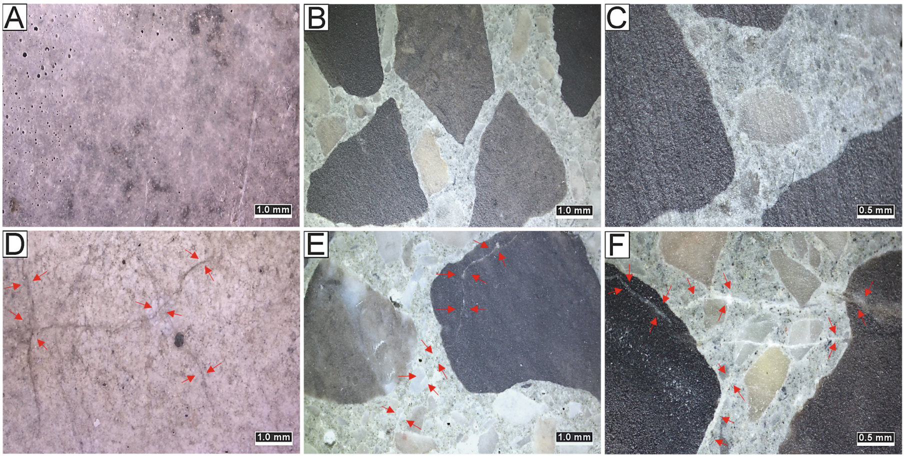

Fig. 4. High and low 6-year expansion concrete prisms samples: a) 30GP10SF undamaged surface; b), c) 30GP10SF cross-section with no visible cracks; d) 20GP surface showing typical ASR cracking; e), f) 20GP cross-section with ASR gel-filled cracks in the aggregates radiating into the matrice.

5 Conclusion

The R^3-test method for evaluating hydraulic binders and pozzolan's reactivity seem to correlate highly with the specific kinetic gradients of each SCM and it corroborates its conception purpose, the 28-day compressive strength correlation. For instance, the MK and GP pastes reached thermodynamic equilibrium within 2 h whilst the exothermic reactions kept progressing for BFS, SF and mainly FA, showing no immediate signs of stabilization. The GP/MK system synergy was observed as a consequence of the correlation between a significant improvement in the reactivity of their respective ternary mixtures and the 2-year expansion measurements. In addition, the 10GP15MK was the ternary concrete with the lowest cement replacement level to achieve the lowest expansion in 6 years. The GP/FA was the sole group in which the expansion of all 3 different ternary concretes was kept below 0.07% throughout the whole test interval. The 20GP15FA and 10GP30FA samples have displayed a particularly similar expansive behavior, which suggests that, at over 35% of cement replacement, there are no significant changes in the ASR mechanisms (alkali leaching and dissolution of reactive silica) for a 0.75 to 3 FA/GP ratio. Although limited investigations concerning the synergistic effect of ternary blends containing GP to mitigate ASR were done, this study helps to understand that the degree of expansion in concrete with GP is related to the pozzolanic and alkali activation of the GP/SCM systems. Therefore, for low cement replacement levels, the high reactivity of metakaolin combined with GP reports reduced expansion levels. While for higher substitution levels, FA blends are shown to be more effective in mitigating expansive reactions. According to what was reported in the present work, the reactive ASR potential of GP with high alkali content can be balanced if it is employed in combination with highly pozzolanic SCM, and can keep the expansion close to the normative limits after 6 years of expansion.

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