Effect of Different Cements with Fly Ash on DEF Occurrence Over Time

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Abstract

This study reports an experimental program involving delayed ettringite formation (DEF) of composites with several pozzolanic cements. Mortars were prepared using a standard mix in the presence of 6 cements. DEF was induced by thermal curing (Peak of 85ºC). Laboratory assessments included expansion measurements, mechanical properties, and microstructural analyses. Results indicate that pozzolanic cements produced less damage from DEF in comparison to a cement with no pozzolan. Whereas mortars containing pozzolanic cements showed less expansions ranging between 0.10% and 0.20%, reference mortar (with no pozzolan) exhibited larger expansions, up to 0.40%). Nevertheless, the level of expansion and negative impacts on mechanical properties proved that DEF was not able to be mitigated. Furthermore, pozzolans appeared just to delay damages from DEF. The use of pozzolans in the production of Portland cements contributes to a reduction of cement and, thus, CO2, and must be considered in the mix designs of concretes improving durability in front of external agents. Nevertheless, studies on hydration heat of cements, prediction of internal temperature of concrete and also limitation of curing temperature must be considered before concrete placement in structures to avoid risks of thermal cracking and also an internal sulfate attack by DEF.

Keywords: *Delayed Ettringite Formation – DEF; pozzolanic cement; fly ash; expansion; durability.*

I. Introduction

This research evaluates whether pozzolanic cements with fly ash affect delayed ettringite formation (DEF) over time in mechanical properties and microstructure.

Only short-term lab tests have proven that pozzolanic mineral admixtures can prevent DEF [1], [2]. Long-term studies have shown that pozzolans such as fly ash cause a delay in the development of DEF [1], [3], [4]. To fully prevent DEF, controlling internal temperatures from hydration heat in massive structures or limiting thermal curing is essential [5], [4], [6].

Deleterious ettringite crystallizes in hardened concrete, with DEF occurring mainly in concretes exposed to over 60°C early on due to thermal curing or exothermic hydration [7], [8], [4]. DEF creates expansive pressures that crack concrete, compromising performance, durability, and service life. The cracks are usually interconnected and multidirectional [9].

Literature suggests that concrete expansion between 0.2% and 0.3% reduces tensile strength and elasticity. At around 1% expansion, damage is severe, significantly affecting mechanical properties and stiffness [10], [11], [12], [13], [14], [15], [16], [4], [6].

Concern over DEF-related issues has grown with rising cement use and finer binders for early strength. Cement's physicochemical properties directly affect reaction occurrence and speed. In this context, pozzolanic cements containing fly ash are available for technical applications. However, the fly ash used in these cements does not always originate from the same source and, consequently, may exhibit varying characteristics. This study aims to assess how different properties of pozzolanic cements influence the occurrence of DEF.

This paper is structured as follows: Section 2 describes the experimental program, materials, and methods used to assess the influence of pozzolanic cements on DEF occurrence. Section 3 presents the results regarding physical, mechanical, and microstructural behaviors of mortars. Section 4 discusses the microstructural assessments and their relationship with physical and mechanical performance. Section 5 offers a global analysis integrating the findings, and Section 6 concludes the study by summarizing the key outcomes and practical implications.

II. Experimental Program

In this study, mortars were produced to investigate the mitigating potential of fly ash cements. The mixtures were prepared in a ratio of 1:2.75 (cement:sand), based on ASTM C 1012 (2019) [17] and ASTM C 109/109M [18], test method where the mortars are cured until they reach a specification strength of 20.0 ± 1.0 MPa (3000 ± 150 psi), measured in cubes made from the same mortar, before immersing the bars in sulfate. The water/cement ratio (w/c) for the production of mortars was 0.485.

To evaluate sulfate attack, mortar cylindrical specimens of 5×10 cm were cast following recommendations from NBR 7215 (ABNT, 2019) [19], and also prismatic specimens of 2.5×2.5×28.5 cm were prepared according to NBR 13583 (ABNT, 2014) [20].

To induce DEF, a thermal cycle (used for concrete) specified in a Brazilian Method [16], was adopted. This method was developed based on previously published works [21], [22], [23], [24], [25], [26], [11], [12]. The composite type was adapted for this study, using mortars instead of concrete. After casting and thermal cycling, mortars underwent the following stages:

1) The mortars remained at room temperature for 6 h.
2) They were conditioned in a tank with water, starting at an initial temperature of 25°C (the water was heated at a rate of 10°C/h until it reached 85°C, which took 6 h).
3) They were kept at 85°C for 12 h.
4) The water in the tank was cooled until reaching 38°C, using the same cooling rate (10°C/h).
5) The mortar continued immersed in water at 38°C (± 2°C) for 12 months until analysis.

2.1. Materials

To assess fly ash cement behavior under DEF-triggering conditions, six Brazilian pozzolanic cements (CP IV RS, similar to American Type IP, labeled B-G) and one high early-strength cement without pozzolan (CP V, similar to Type HE, labeled X) were studied. Table 1 shows their main oxides and characteristics.

TABLE 1. Chemical characteristics of cements.

Cements	Type of pozzolan	Al_2O_3 (%)	SiO_2 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	L.I (%)	I.R (%)	SO_3/Al_2O_3	$(SO_3)^2/Al_2O_3$	H.h (J/g)
B*	Fly Ash	9.96	38.38	3.93	35.60	4.49	1.11	4.75	38.81	0.48	0.12	131.50
C*	Fly Ash	8.38	31.55	3.13	42.25	5.52	2.05	7.12	26.86	0.30	0.50	141.70
E*	Fly Ash	8.96	31.27	4.45	44.88	3.73	0.91	4.85	25.18	0.10	0.20	145.90
F*	Fly Ash	7.63	28.00	3.19	50.97	1.98	1.71	4.91	19.62	0.22	0.45	228.10
G*	Fly Ash	11.12	35.25	4.16	37.64	4.55	1.19	4.15	36.43	0.11	0.21	167.60
X	-	4.18	19.70	2.48	61.62	3.04	3.45	4.45	2.14	0.825	1.65	297.90

According to NBR 16697 (ABNT, 2018) [27], the pozzolanic cements studied have low heat of hydration, below 270 J/g at 41 h. Cement X, without fly ash, does not share this trait. Thus, pozzolanic cements do not affect initial curing products under normal conditions. The pozzolanic content and sources vary by cement type.

Table 2 shows the cements' physical characteristics. Cements E, F, and G have specific surface areas similar to Cement X (high early-strength), indicating lower-than-expected fineness. This may accelerate hydration and increase early-age temperatures.

TABLE 2. Physical characteristics of cements.

Cements	Thermal expansion (%)	Initial setting time (min)	Final setting time (min)	Specific area (cm^2/g)	Specific mass (g/cm^3)
B	0.00	5:00	6:00	5.010	2.83
C	0.00	2:45	4:30	5.730	2.79
E	0.07	3:30	4:20	4.000	2.83
F	0.00	3:10	4:50	3.990	2.93
G	0.07	3:15	5:00	3.920	2.73
X	0.01	3:20	4:40	3.970	-

The aggregate used for the production of the mortars is granitic, sourced from Goiás state (Brazil). The aggregate has a maximum dimension of 4.8 mm and a fineness modulus of 2.42. Table 3 presents the characteristics of the aggregate.

TABLE 3. Aggregate characteristics.

Characteristics	Results
Specific Mass (g/cm^3)	2.66
Powdered material (%)	16.98
Water absorption (%)	0.10

2.2. Methods

The dimensional variation and water absorption of the mortars were evaluated according to NBR 9779 (ABNT, 2012), [28]. Cylindrical specimens with dimensions of 5 × 10 cm were used for this purpose.

Six prismatic mortar specimens with dimensions of 2.5 × 2.5 × 28.5 cm were cast for each cement type. The characteristics were analyzed at 7-day intervals during the first three ages, and then at 15-day intervals from 28 days of age up to 365 days.

Linear dimensional variation was evaluated following the recommendations of NBR 13583 (ABNT, 2014) [20], but with extended exposure time. Mass variation was evaluated following the recommendations outlined in NBR 13583 (ABNT, 2014), [20].

The mechanical properties of the mortars were evaluated using cylindrical specimens with dimensions of 5 × 10 cm at the ages of 7, 28, 56, 84, 168, 252, and 365 days. The axial compressive strength test was conducted according to NBR 7215 (ABNT, 2019) [19], and the tensile strength was assessed through diametral compression, following NBR 7222 (ABNT, 2011) [29], with the specimen sized 5 × 10 cm.

The modulus of elasticity was determined by the impulse excitation method, analyzing vibration frequencies from a striker's impact. Using specimen mass and dimensions, the dynamic modulus was calculated. The direct transmission method measured ultrasonic wave velocity along cylindrical specimens. Tests followed NBR 8802 (ABNT, 2019) [31] in triplicate at 168 and 365 days.

The dynamic modulus of elasticity is calculated based on the ultrasonic wave velocity and the material's density. In this study, the dynamic modulus of elasticity (Ed) was determined using Equation 1, where ρ represents the density of the test specimen,υ is the ultrasonic wave propagation velocity, and υ is Poisson's ratio (assumed to be 0.2 for mortars).

$$Ed = p.\upsilon^2.\frac{(1+\upsilon).(1-2\upsilon)}{1-\upsilon}$$

Microstructural analyses were performed on fracture surfaces after one year using a Tescan VEGA3 SEM with SE + BSE detectors under high vacuum. Qualitative EDS microanalysis used an Oxford double EDS detector (AZtec system) at 20 kV. Neoformed compounds were examined, and line-scan mapping analyzed elemental distribution.

III. Results

3.1. Monitoring Physical and Microstructural Behavior

By evaluating expansion over time, data indicated a continuous and increased growth for all mixtures. However, the mortar produced with pure cement (called X) showed more substantial expansion, as expected, reaching 0.42% at 12 months, following a tendency toward sigmoidal behavior, as described by Portella et al. (2021) (Figure 1) [8]. These results are consistent with those reported by the literature [32], [33], [34], [35], [36], [37], [4], [6], [38], [40].

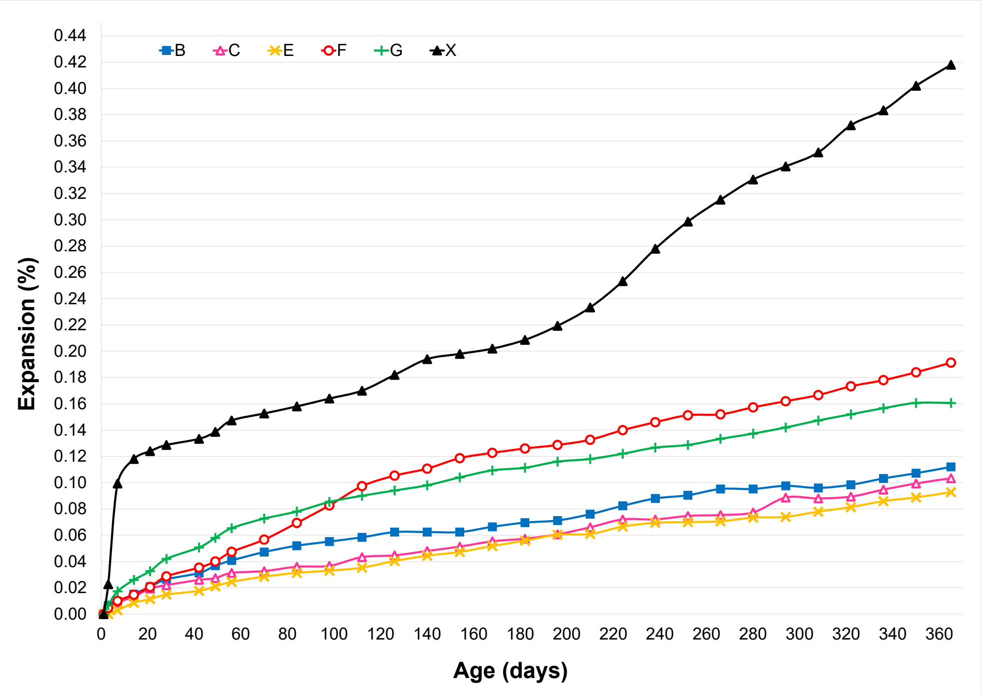


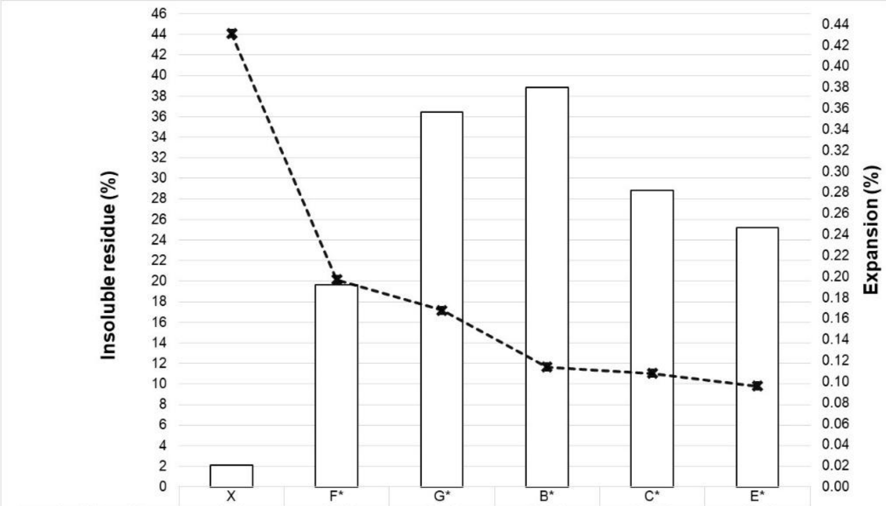
Fig. 1. Expansion of mortars over time.

Expansion levels varied among cements over time. At 12 months, fly ash cement mixtures showed 0.9% to 19% of the expansion seen in non-pozzolanic cements. Mineral admixtures reduced expansion but maintained constant growth rates. Cement F had the highest expansion (0.19%), indicating DEF, where crystals first fill pores before causing internal stress [41]. In contrast, mixtures with cements B, C, and E had the lowest expansions (below 0.10%).

No parameters currently limit DEF expansion in mortars using this method. However, NBR 16697 (ABNT, 2018) [27] requires sulfate-resistant cement to have expansion below 0.03% at 42 days per NBR 13583 (ABNT, 2014) [20]. Applying this to DEF, only C and E specimens would meet the limit at 42 days, while all cements would exceed it at 12 months, showing no stabilization trend.

3.1.1. Relationship Between Cement Characteristics and Expansions

The content of fly ash incorporated in the cement and its relationship with the percentage of expansion is shown in Figure 2.

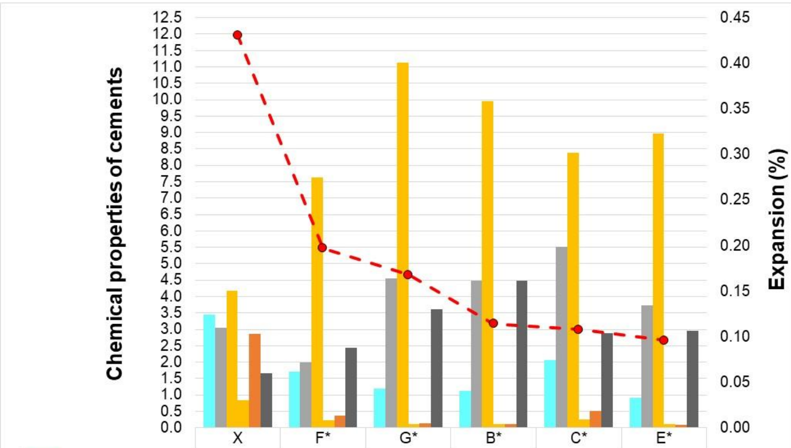


	X	F*	G*	B*	C*	E*
Isoluble residue	2.14	19.62	36.43	38.81	28.86	25.18
Expansion - 1 year	0.431	0.197	0.168	0.114	0.108	0.096

Fig 2. Insoluble residue versus expansions.

Cements with higher pozzolanic content showed lower expansion. With fly ash above 25%, expansion stayed below 0.25%, while pure cement (X) had the highest. Despite high RI content, all cements exceeded 0.10% expansion at 12 months.

Figure 3 illustrates the relationship between cement chemistry and expansion. Al_2O_3 above 8% reduced DEF-related expansion, except for Cement G. [2] also highlighted that high Al_2O_3 in pozzolans reduces DEF expansion and lowers sulfate content in mixtures.



	X	F*	G*	B*	C*	E*
SO3	3.45	1.71	1.19	1.11	2.05	0.91
MgO	3.04	1.98	4.55	4.49	5.52	3.73
Al2O3	4.18	7.63	11.12	9.96	8.38	8.96
SO3/Al2O3	0.83	0.22	0.11	0.11	0.24	0.1
$(SO_3)^2/Al_2O_3$	2.85	0.38	0.13	0.12	0.5	0.09
Total alkalis	1.66	2.43	3.6	4.48	2.89	2.96
Expansion - 1 year	0.431	0.197	0.168	0.114	0.108	0.096

Fig. 3. Chemical characteristics of cements versus expansion.

Research indicates that cements with SO_3 > 2.6%, alkali equivalent > 3 kg/m³, and MgO > 1% are prone to DEF [42], [43], with DEF expansion directly proportional to MgO content [44]. Despite all cements in this study containing MgO values above 1%, no clear correlations were found between MgO, alkali content, and DEF expansions.

The highest SO_3 content (3.45%) corresponded to Cement X (without pozzolanic admixture). For pozzolanic cements, Cement C showed the highest SO3 content (2.05%) but there was no correlation between this content and the measured expansion.

According to [32], [26], [33] and [45], the closer the SO3/Al2O3 ratio is to 1, the greater the propensity for DEF development. Cement X (without pozzolans) presented the highest SO3/Al2O3 ratio (0.83).

The ratios for the cements with fly ash were much lower and were always below 0.24 (0.1– 0.24), with no clear behavioral trend among them.

The $(SO3)^2/Al2O3$ ratio has been linked to DEF occurrence and is considered more relevant than the SO3/Al2O3 ratio. According to [33], values above 2 indicate susceptibility to DEF occurrence. In the present study, only Cement X had an $(SO3)^2/Al2O3$ above to 2, whereas the pozzolanic cements showed much lower ratios (below 0.5).

The lowest DEF expansion (0.10%) at 365 days was observed in the mortar with Cement E, which had the lowest SO_3 and SO_3/Al_2O_3 ratios, as well as a low $(SO_3)^2/Al_2O_3$ ratio. In contrast, Cement X showed higher expansions and values for all three parameters.

According to [34], partial cement substitution with fly ash reduces the SO_3/Al_2O_3 ratio, which aligns with this study and [46], who showed that replacing cement with slag and fly ash reduced DEF expansion. [33] also discussed the beneficial effect of slag and fly ash against DEF. In these studies, controlling Al_2O_3 content increased monosulfate precipitation, lowering sulfate concentration in the pore solution, which reduced sulfate absorption by C-S-H gels and DEF expansion potential.

The tested pozzolanic cements delayed the deleterious expansive process of DEF; however, their efficiencies varied.

3.1.2 Capillary Absorption

Mortars with pozzolanic cements had lower absorptions than the reference mixture (Figure 4). Over time, increased water absorption in all mixtures suggested DEF damage. Even cements with fly ash showed higher absorption due to expansion and internal microcracking from DEF, allowing more water intake.

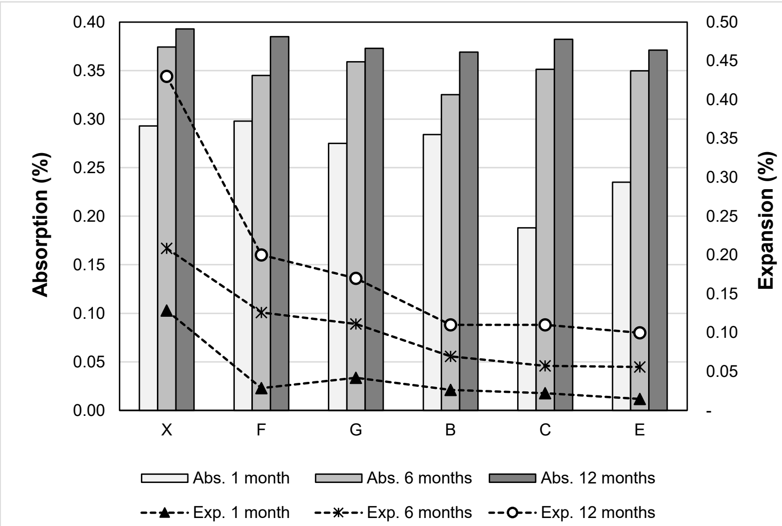


Fig. 4. Behavior of capillary absorption in relation to expansion**.**

Mineral admixtures cause physicochemical changes in cement matrices, altering microstructure and porosity through pozzolanic reactions (chemical effect). They also reduce the transition zone thickness and refine pores by filling voids with small particles (microfiller effect) [47]. In this study, all fly ash-containing cements showed similar behavior, with absorption slightly lower than Cement X, regardless of expansion rate.

3.2. Long-Term Monitoring of Mechanical Behavior

3.2.1. Tensile Strength

The mechanical properties aligned with the expansion behavior observed among the tested cements. As expansion increased, strength decreased in cements containing fly ash and without pozzolanic admixtures. Tensile strength showed significant reductions over time, reaching approximately 40% for Cement X, 20% for Cements F and G, and 10% for the others at 12 months (Fig. 5).

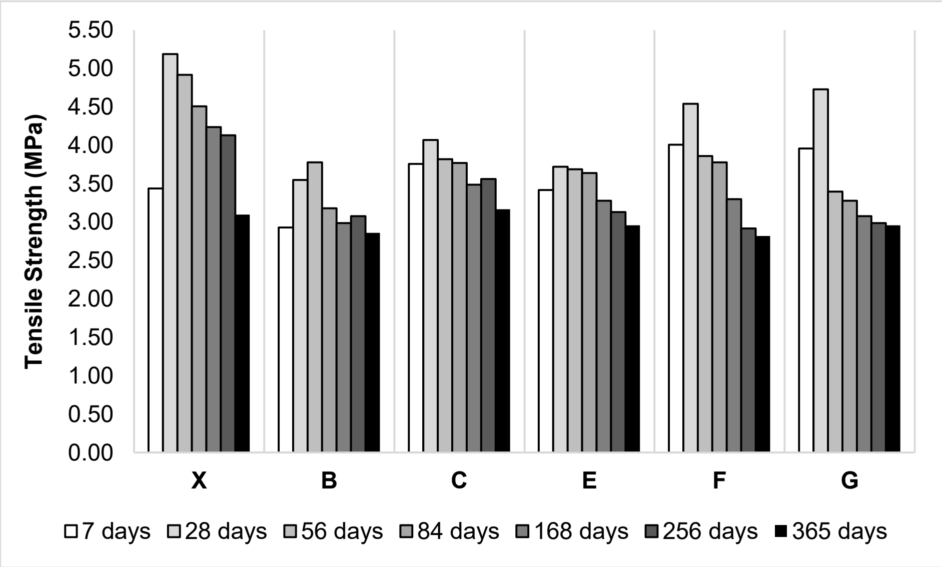


Fig. 5. Tensile strength over time.

Figure 5 also shows an initial increase in tensile strength, followed by a sustained decline over time. This trend suggests that the rapid filling of voids by ettringite crystals contributes to early strengthening, particularly within the first 28 days for most cements. This improvement persists for up to 56 days, as seen in Cement B. Beyond this period, the expansion process triggered by delayed ettringite formation (DEF) leads to a subsequent deterioration in mechanical properties.

These findings highlight concerns about the performance of specimens with cement containing fly ash, as DEF-related mechanical degradation was observed in all samples.

According to [10], concretes affected by DEF with expansions of 0.05% to 0.12% show a 65% reduction in tensile strength, while expansions around 0.20% reduce it by 70% to 80%. [11] noted that expansions of 0.2% to 0.3% significantly lower tensile strength and modulus of elasticity. [3] reported a 17% reduction in tensile strength at 252 days for fly ash concretes. [37] found that expansions above 0.45% cause significant damage, with a 50% reduction in mechanical properties. Even at 0.10% expansion, DEF deterioration starts, with a 10% reduction in tensile strength in one year.

In this study, a substantial decrease in the dynamic modulus of elasticity was evident for all tested cements. Similar to the other properties, the largest decrease (approximately 35%) occurred for Cement X, confirming the negative influence of DEF on the integrity of composites through neoformations and microcracking (Figure 6) and the advancement of DEF, along with increased expansion.

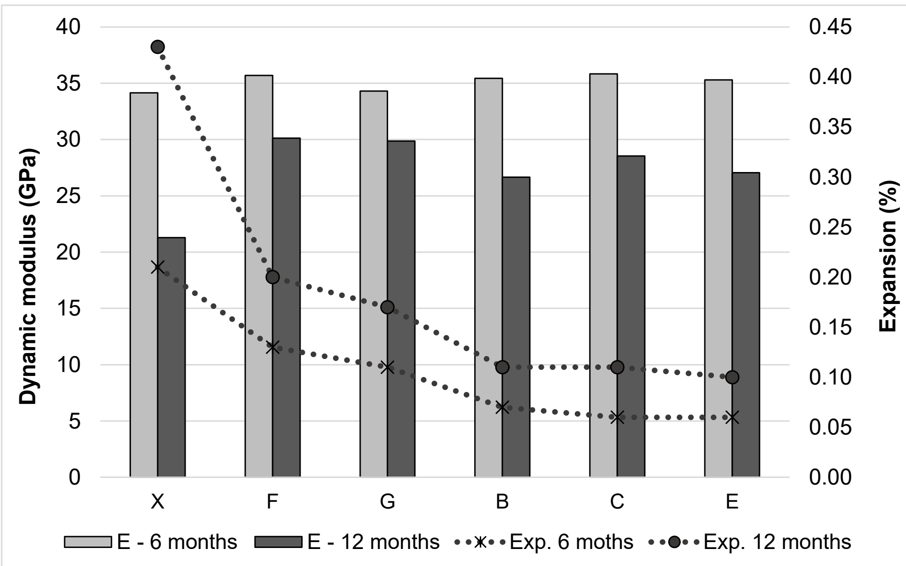


Fig. 6. 1Dynamic modulus from ultrasound and expansions.

Regarding DEF and modulus of elasticity reduction, [10] found a 20% to 50% decrease for expansions above 0.20%. [11] reported up to 75% reduction, while [14] observed reductions over 20% for expansions above 0.30%. [41] noted an 82% reduction in modulus for concretes without pozzolan affected by DEF, compared to a 10% reduction for fly ash cement concretes.

According to [48], the reduction in static modulus is more pronounced and significant than in the dynamic modulus, indicating that static modulus may be more vulnerable to damage from expansion during the latent phase of DEF.

3.2.1. Axial Compressive Strength

Compressive strength data are presented in Fig. 7. Mortars containing fly ash exhibited reductions in compressive strength of 20–30% between the ages of 56 or 84 days (depending on the cement used) and 168 days.

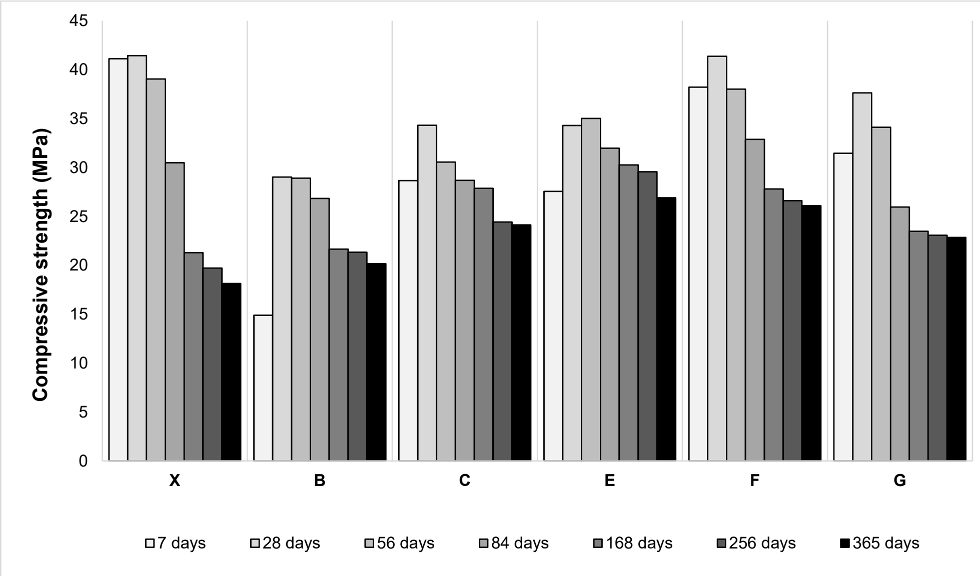


Fig. 7. Compressive strength over time.

Figure 8 shows the expansion at 3 months and 12 months for each mixture, along with average strength levels. Mortar with Cement X had the greatest compressive strength loss, decreasing by 25% at 3 months, over 50% at 6 months, and 56% at 12 months, indicating early deterioration. These results confirm the progressive expansion and detrimental effects of DEF.

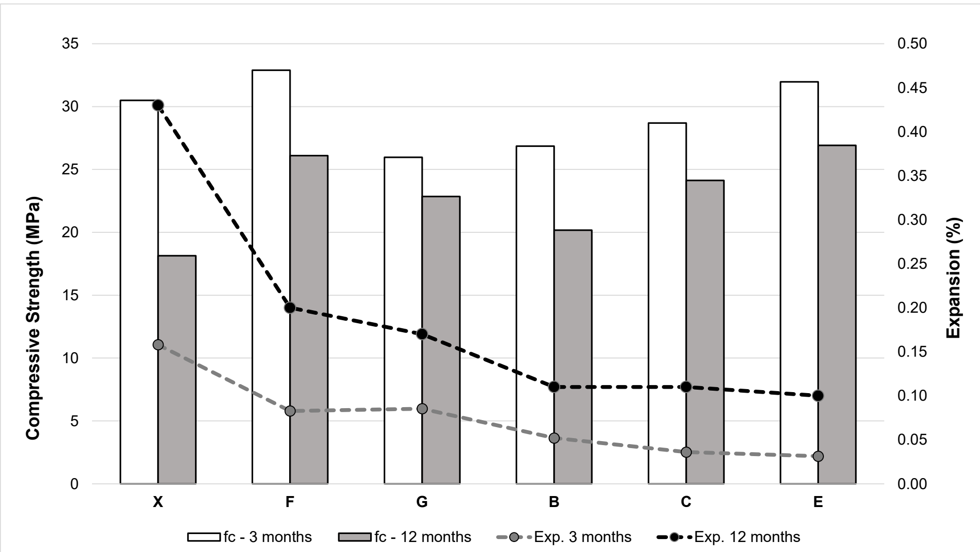


Fig. 8. Compressive strength and expansion at 3 and 12 months.

Strength continued to decline over time for all cements, though the rate slowed after 6 months. While no clear trend emerged, an inverse relationship between expansion and strength was observed, with higher expansions linked to sharper strength declines. According to [32], DEF-induced microcracking begins in the transition zone, causing significant compressive strength reductions, especially in mixtures with pozzolans.

The results of this study align with [49], who found up to 80% reduction in concrete compressive strength, and [50], who reported a 40% reduction in mortar strength by 180 days. Recent studies confirm that DEF affects mechanical properties, reducing compressive and tensile strength, as well as modulus of elasticity [3], [4].

The results of the present study align with findings by [48], who report that DEF expansion has a significant and continuous impact on reducing compressive strength as expansion progresses.

IV. MICROSTRUCTURAL ASSESSMENT OF COMPOSITES AND ITS RELATION TO PHYSICAL AND MECHANICAL BEHAVIORS

SEM analyses of the reference mixture (high expansion) were performed. Three expansion levels were identified after one year: X (0.43%), F (0.20%), and E (0.10%). DEF microstructural characteristics were observed in all composites, as well as deterioration after the period of exposure (one year), with some differences that suggest different stages of deterioration.

The E and F mixtures (with fly ash) were similar in the voids filled by neoformed AFt crystals, though they differed in morphology and expansion. The E mixture (lower expansion) had fine needle-shaped ettringite, while F showed clusters of ettringite, indicating a more advanced DEF stage (0.20%). The reference mixture, with 0.43% expansion, showed extensive deterioration, with voids filled with ettringite and powdered cement paste. These findings are illustrated in the images and EDS spectra in Figures 9 and 10.

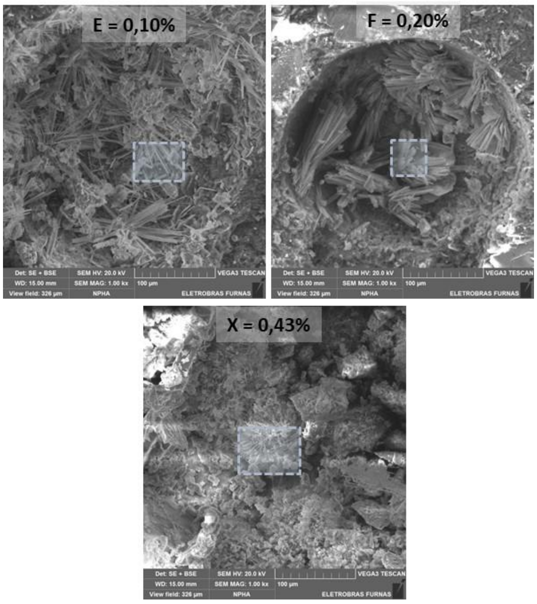


Fig. 9. Sulphate phases inside the voids of composites and areas indicative of spectra performed by EDS (Fig. 10); (in %: expansion levels at 1 year). *Note: For each cement tested (E, F and X, it is indicated the level of expansion achieved at the last age.*

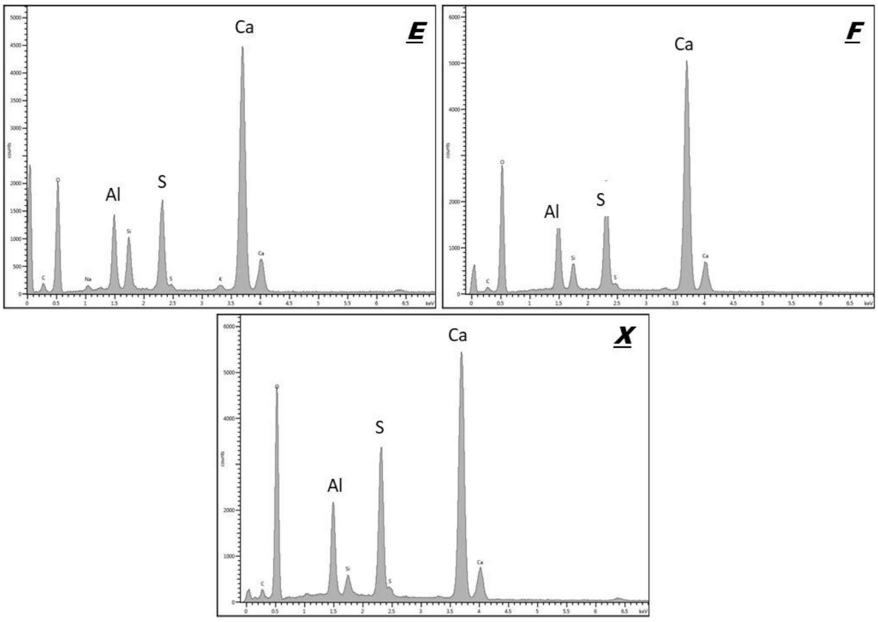


Fig. 10. Spectra from EDS microanalyses of phases identified in some voids of samples.

The cement matrices analyzed at higher SEM magnifications corroborate the previous observations of the voids in the mortar samples (Figure 11). The 0.10% expansion mixture (E) with 25% fly ash exhibited during SEM analyses a denser microstructure along cement matrix, whereas the 0.20% expansion mixture (20% fly ash) showed a fragilized cement paste because of DEF. Moreover, the 0.43% expansion of the reference material indicated macrocracking in addition to a fragile matrix with powdered material.

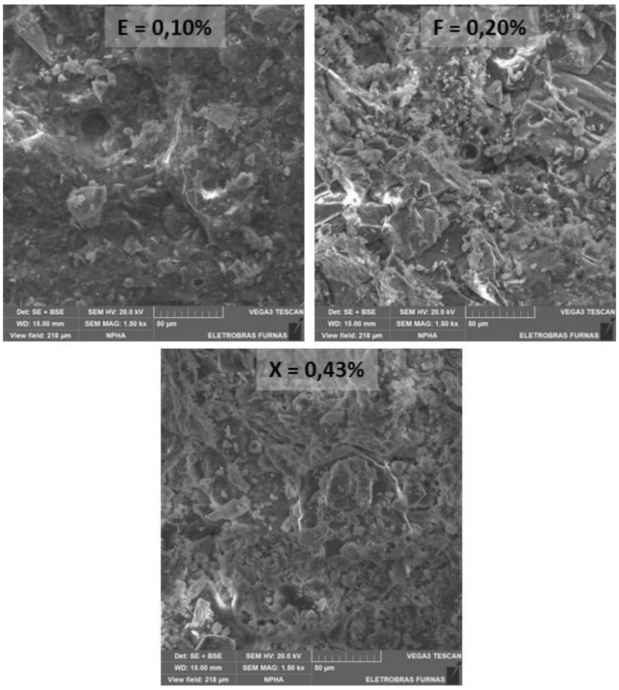


Fig. 11. SEM view of cement matrix microstructures according to expansion levels (in %) at 1 year.

At higher SEM magnifications (Figure 12), a massive structure derived from ettringite dispersed in several areas of the cement matrix in the reference (X) was identified, indicating extensive damage. All the features of the matrices indicate an abnormal microstructure and lack of integrity in terms of mechanical properties and durability.

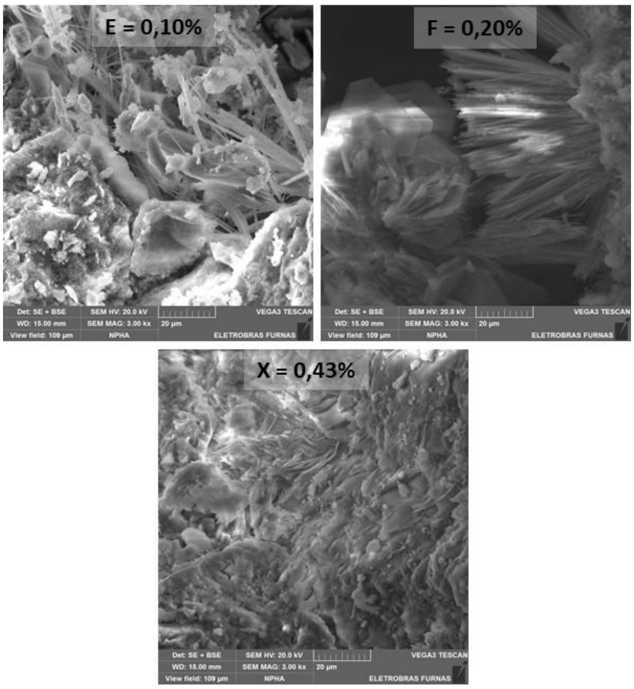


Fig. 12. Detail of cement paste with of some formations at high magnification by SEM (in %: expansion levels at 1 year).

The study by [8], also described ettringite formations in cement composites. DEF occurred over time in the pores of the mortar and concrete specimens studied, along the cement matrix, and in the interfacial transition zone (ITZ). In the cement paste specimens, DEF was detected prematurely (at one month) by SEM analyses. [8], conducted microstructural studies by SEM/EDS and detected masses of ettringite crystals, which were associated with microcracking of early-strength cement at one year.

The research conducted by [41] conducted several microstructural analyses of concretes over time and monitored the development of DEF from 28 to 365 days. The characteristics of the composites at the beginning were completely different from those after one year. At first, few crystals were present in acicular form, and, over time, the products completely filled voids and clustered as chemical reactions proceeded. As the material aged, massive formations were observed, even in pozzolanic cement, indicating DEF and deterioration, which are also associated with a substantial decrease in mechanical properties.

Several specific ITZ analyses were performed to evaluate the patterns of the neoformations next to aggregate (Agg.) particles after one year (Figures 13 to 16). Nearby all aggregate grains investigated sulfate phases exist, resulting in the loss of bond strength with the cement paste. The mixture with the least expansion (E: 0.10%) exhibited fewer crystals in the interfacial transition zone (ITZ) despite the presence of sulfate phases, but the one with 0.20% expansion (F) showed some cracking and clusters of ettringite (Et.) in the interface. The visualization of ettringite in the ITZ of the reference mortar (0.43% expansion) was impaired by the high level of microcracking and the presence of powdered materials. Furthermore, those analyses confirmed the presence of the AFt and AFm phases by EDS line-scan elemental microanalyses of the ITZ (Figures 13–15).

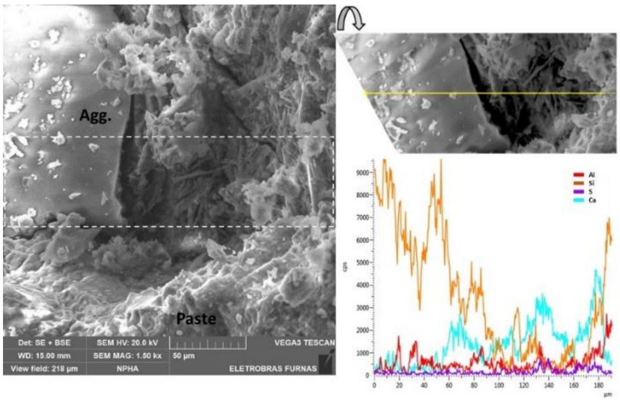


Fig. 13. SEM and line-scan mapping images with elemental distribution along TZ. Mixture E (0.10%-expansion).

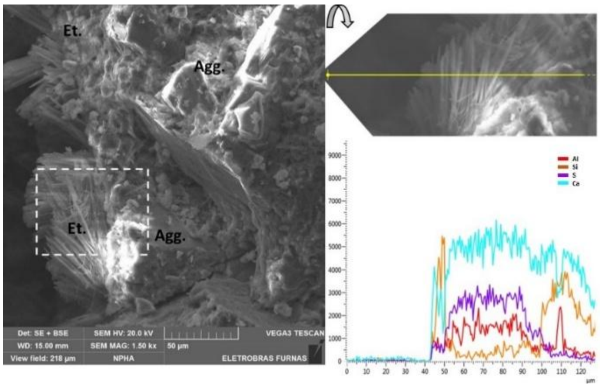


Fig. 14. SEM and line-scan mapping images with elemental distribution along TZ. Mixture F (0.20%-expansion).

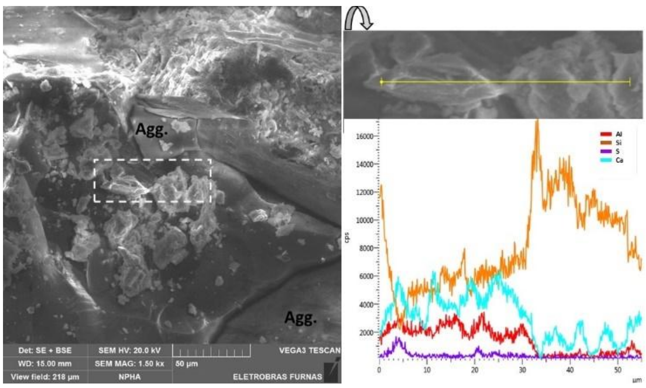


Fig. 15. SEM and line-scan mapping images with elemental distribution along TZ. Mixture X (0.43%-expansion).

The differences that were ascertained by SEM/EDS occurred due to singular cement compositions. Nonetheless, regardless of those singularities, all samples showed damage from DEF. This damage occurred due to injuries to the microstructural integrity of the composites, although in different stages of expansion and deterioration. Thus, the distinct evolution of DEF over time stemmed from the presence of mineral admixtures.

V. Global Analysis

Considering data from the literature, the results of the expansion tests, and an analysis of the evolution of mechanical properties, the mixtures could be grouped into three levels of expansion and DEF aggressiveness (Table 4).

TABLE 4. DEF: Level of aggressiveness based on one-year expansions.

Data in Percentages (%)	X	F	G	B	C	E
Expansion	0.43	0.17 a 0.20			<0.11	
Drop in compressive strength	56	37 a 40			22 a 31	
Drop in tensile strength	40	33 a 38			19 a 23	
Drop in modulus	38	13 a 16			11 a 13	
Level of aggressiveness	Severe	Aggressive			High	

Taking into consideration the adopted DEF limit of 0.04% [16], for concrete, the behavior remained the same, indicating that non-resistance could be attributed to DEF for all tested cements.

All of the mixtures with the lowest expansions (B, C, and E) contains fly ash incorporated in the pozzolanic cement. Notwithstanding, the expansions ranged from 0.10% to 0.11% and represent more than double the existing limits. Furthermore, the decrease in mechanical properties indicates that damage occurred in these three composites. Losses of up to 30% in compressive strength, in addition to the decreases observed in tension and modulus, are unacceptable. This situation indicates an important level of aggressiveness.

The second group, which exhibited expansions between 0.17% and 0.20%, experienced losses in strength of up to 40%, and a 16% decrease in modulus, indicating a high degree of aggressiveness. For expansions above 0.4%, the losses in compression are substantial, reaching nearly 60%, with notable effects also on tension and modulus, indicating severe aggressiveness.

VI. CONCLUSIONS

Based on the measured expansions, the loss of mechanical properties, the increased water absorption, and, especially, the presence of neoformed ettringite in the evaluated mixtures, the incorporation of fly ash could not prevent the occurrence of DEF.

The presence of fly ash delayed the onset of negative effects of DEF compared to the mixture without this pozzolan.

Distinct levels of expansion were measured. The reference mortar (without pozzolan) exhibited higher expansions (above 0.43%), whereas those that contained pozzolanic cements exhibited expansions ranging between 0.10% and 0.20%.

A compromise of all measured mechanical properties was observed by expressive loss percentages. The modulus of elasticity and tensile strength were more susceptible to cracking. When fly ash was present in the mixture, these variables decreased by approximately 38% and 47%, respectively. In the case of compressive strength, reductions of up to 40% were verified. Therefore, it is important to note that the Brazilian pozzolanic cements containing fly ash that were studied have not shown resistance to DEF.

Significant differences were observed in the expansion behavior of the tested cements, which can be attributed to their intrinsic physical, chemical, and thermal properties. Both the SO_3 theory and the relationships between SO_3 and Al_2O_3 demonstrated substantial effects on expansion. All cements exhibited MgO content exceeding 1%, which diminishes the likelihood of DEF development. However, the heat of hydration in most cements was found to correlate with the expansions related to DEF.

Finally, there was a relation between the content of insoluble residues and the negative effects associated to DEF, indicating that a higher fly ash content in the cement results in lesser damage. However, the microstructural evaluation revealed the occurrence of ettringite crystals in all mixtures at 12 months, varying intensity between samples.

In summary, given that expansive chemical reactions can persist for years, it is unfortunate that none of the cements tested in this study were found to be resistant to DEF. Thus, the pozzolanic cements evaluated in this research did not avoid the occurrence of DEF and in the field of thermal curing other type of precautions, especially by limiting temperature (bellow 60ºC), must be applied in order to prevent damage.

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