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Evaluation of Delayed Ettringite Formation through Physical, Mechanical, and Microstructural Assays

by D. Schovanz, F. Tiecher, N. P. Hasparyk, S. Kuperman, and R. T. Lermen

This experimental study evaluated the influence of delayed ettringite formation (DEF) on physical and mechanical properties and also microstructural characteristics of concretes cast in laboratory for 6 months. The results show that DEF can generate high expansions, in the order of 1%, and reduce mechanical properties, such as compressive strength, tensile strength, and the modulus of elasticity by more than 50%. Already in the 28th day, the presence of ettringite could be identified in the concrete matrix. The results of the study indicate high premature deterioration of the concrete and, consequently, its disqualification for use after the thermal cure adopted.

Keywords: delayed ettringite formation (DEF); durability; expansion.

INTRODUCTION

Delayed ettringite formation (DEF) has affected concrete structures in some countries. This phenomenon occurs in the hardened concrete due to the chemical reaction between the sulphated phase, that remains from the cement hydration, with the water from concrete and the environment surrounding the structure.[1-5] Its phenomenon depends on the constituent materials and the curing exposure. The final product of this reaction is the delayed ettringite crystals in the matrix of concrete, which fill pores and the paste/aggregate transition zone, thus inducing an increase in the internal pressure of the matrix, which culminates with expansions and an volume increase of the concrete elements along with microcracks. Thus, this process can lead to damages of mechanical properties of concrete, thereby affecting, in some cases, the functionality and the service life of the structures.[3,5-7]

There is a challenge to diagnose DEF precisely due to existence of combined attacks that can occur, thus making analysis even more complex, such as alkali-silica reaction (ASR) and DEF. Some studies suggest physical, mechanical, and microstructural investigations of concretes for the assertive diagnosis. Several authors state that compressive strength, although an important indicator of the quality of the material, is not as affected by DEF as are stiffness, tensile strength and the modulus of elasticity.[1,3,5,8-18]

Jeong et al.[19] evaluated the modulus of elasticity and compressive strength and found that both are highly likely to be proportional, as the results of these tests depend on the physical condition of the test specimen due to whether or not there are microcracks. However, the modulus of elasticity has been shown to be an important indicator of there being DEF, and its decrease is proportional to the increase in the percentage of expansion.[10,17]

In the design of conventional reinforced concrete structures, tensile strength is not a widely used parameter. Generally, it is assumed to be approximately 10% of compressive strength. However, this parameter can be greatly influenced by chemical degradation mechanisms, such as DEF and ASR, rather than compressive strength. Thus, tensile strength may add important information in the case of moderate levels of expansion.[10,20,21]

The evaluation of expansions over time in concrete subjected to DEF at laboratory is accessible, as it is an easily mastered methodology, but it can take time to obtain a conclusive result.[13,21] Expansion is one of the main evidences of the DEF process, because the neo-formation of ettringite in the voids causes local tensions, thereby generating expansion of cementitious paste and consequent cracking.[22] Nonetheless, microcracks in concrete can also be induced by other mechanisms such as thermal shrinkage, freezing-and-thawing cycles, ASR, or loading action. Therefore, this parameter should not be used alone to diagnose pathological manifestations, such of DEF.[1,5]

Although chemically different from each other, both ASR and DEF require moisture to drive the expansion in the concrete. In the case of ASR, expansion only occurs because moisture is present, because the product of the reaction is hygroscopic. In DEF, the presence of moisture nurtures internal chemical reactions in the cement matrix, thereby forming new products that occupy the interstitial spaces in the contours of the aggregates and in the paste, and leads to the expansion process hence degrading the structure.

As yet there are no standardized and normalized assay methods to assess the developmental potential of DEF—that is, that bring specific laboratory conditions. Authors who take up this topic adopt different methods, which makes it difficult to compare results.

This paper aims to verify the potential damage to the physical, mechanical and microstructural characteristics of concretes affected by DEF by adopting a specific curing method adapted from several studies already published on this subject.

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Table 1—Properties of cement

Mechanical properties			Chemical properties	
Characteristics		Value	Characteristics	Value
Compressive strength (MPa)	1 day	25.6	Al_2O_3, %	4.46
	3 days	38.6	SiO_2, %	18.86
	7 days	44.4	Fe_2O_3, %	2.75
	28 days	54.1	CaO, %	61.46
Physical properties			MgO, %	3.93
Specific mass (g/cm^3)		3.09	SO_3, %	3.28
Thermal expansion (mm)		0.50	Loss on ignition, %	3.37
Setting time	Initial (h:min)	03:10	Insoluble residue, %	0.76
	Final (h:min)	04:00	Na_2O, %	0, 23
Normal consistency, %		29.3	K_2O, %	0, 85
Blaine fineness (cm^2/g)		4.520	Na_2Oeq, %	0, 79
No. 200 residue, %		0.03	Hydration heat (J.g^{-1}) for 41 hours	324.42
No. 325 residue, %		0.30	Na_2Oeq. = 0.658.K_2O + Na_2O	

RESEARCH SIGNIFICANCE

Delayed ettringite formation is a complex pathological phenomenon and considered insufficiently researched. Nevertheless, there are several cases of concrete structures being deteriorated by DEF worldwide in the last years; however, there is lack of standard for testing the susceptibility to DEF. The purpose of this study is to present the results from an experimental program to study DEF. The testing method (thermal curing and exposure environment) developed and adopted had shown promising and an important step for improving the understanding of the pathology, contributing for the adoption of preventive measures, the concrete technology and durability of structures.

EXPERIMENTAL STUDY

To carry out this study, concrete specimens cast with cement consumption of 470 kg/m^3 were submitted to specific conditions that favored the development of DEF, with thermal curing at 85°C for 12 hours after mixing. Specimens (SPs) were prepared for testing for up to 168 days to evaluate the following properties: compressive strength, tensile strength, modulus of elasticity and stiffness damage index (by the stiffness damage test [SDT]). Physical evaluations such as expansion and mass were monitored along time. Visual inspection and microstructural investigation were also undertaken using scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS).

Materials

The characteristics of the cement used in this study are described in Table 1. This cement is Brazilian, equivalent to type III cement (ASTM C150) and does not contain any type of mineral admixture. Its characteristics suggest favor to DEF occurrence, such as a high Blaine fineness, between 3330 and 4635 cm^2/g, a sulfate percentage (SO_3) from 2.6 to 3.6%, and a SO_3/Al_2O_3 ratio greater than 0.50. In addition, cement have presented a high hydration heat, above 270 J.g^{-1}.

Both coarse and fine aggregates chosen for the study fall into the category of potentially innocuous to ASR, according to previous assays performed with the accelerated mortar bar test (AMBT) (ASTM C1260; NBR 15577-4: 2018[23]). The expansions obtained at the end of the assays for fine and coarse aggregate were 0.09% and 0.10%, respectively. The other characteristics of aggregates used to produce concretes are described in Table 2.

It was used a polyfunctional chemical admixture to cast all concretes composed of lignosulfonate and polycarboxylate. Its main physical properties are: density = 1.187 g/cm^3, pH = 5.41; solids content = 43.97%.

Mixture design, curing process, and environment exposure condition of concretes

The characteristics of the concretes prepared for this research are shown in Table 3.

In this study, a specific curing methodology was used based on some methods used in the studies by Rashidi et al.,[16] Giannini et al.[24] and Kchakech et al.,[25] considering the relevant changes to the objective of this research.

The method used can be visualized schematically in Fig. 1 and basically consists of the following steps: a) after casting, the concrete specimens remained at room temperature for 6 hours (25°C) covered with plastic film to keep their moisture; b) the specimens were subjected to thermal curing by immersion in water, which was heated from 25 to 85°C for 6 hours (heating rate of 10°C/h); c) maintenance of the specimens immersed in water at 85°C for 12 hours; d) cooling of the immersion water at the same rate as heating (10°C/h) to a temperature of 38 ± 2°C; and e) storage in the immersion condition at 38 ± 2°C for 168 days.

Table 2—Characteristics of aggregates

Aggregate	Property	Result
Fine	Origin	Natural/cava
	Maximum aggregate size	4.75 mm
	Fineness modulus	2.56
	Specific mass	2.05 g/cm^3
	Water absorption	3.0%
	Powdered material	1.3%
Coarse	Origin	Granite—crushing
	Maximum aggregate size	19.00 mm
	Fineness modulus	6.72
	Specific mass	2.68 g/cm^3
	Water absorption	0.40%
	Powdered material	0.32%

Table 3—Characteristics of concrete

Properties	Results
Water-cement ratio	0.46
Modulus of fineness	4.809
Natural sand, 4.75 mm (%)	46.9
Coarse aggregate, 19 mm (%)	53.1
Polyfunctional admixture (%)	0.6
Slump (mm)	110
Entrained air (%)	1.5
Cement (kg/m^3)	471
Mix design (unit)	1:1.6:1.9:0.46

Evaluation of concrete physical properties

Visual inspection of concretes was performed on specimens after mechanical testing (at all ages). The analysis consisted of verifying superficial anomalies associated with DEF, such as efflorescence and cracking.

Mass variation and expansion were monitored over time in prismatic specimens (75 x 75 x 285 mm), and every 7 days, a total of 24 readings up to the age of 168 days.

Evaluation of concrete mechanical properties

Mechanical properties were evaluated using cylindrical SPs (100 x 200 mm) at the ages of 7, 28, 56, 84, and 168 days. Assays were performed to determine axial compressive strength, diametral tensile strength, secant modulus of elasticity and stiffness damage index (SDI). For each assay three specimens were tested. All tests were performed by using national standards (ABNT NBR 5739:18[26]; ABNT NBR 7222:11[27]; ABNT NBR 8522:17[28]).

The SDI assay was performed based on the method presented by Martin et al.,[12] Sanchez et al.,[10] and Giannini et al.,[24] in which the stress and strain ratio is verified due to the controlled loading of 0.1 MPa/s, performed in five loading cycles. Applying load usually corresponds to 40% of the axial compressive strength of concrete at 28 days. However, the maximum application of the load recommended by the researchers is 10 MPa. The SDI of concrete consists on the relation between stress and strain due to a cyclic loading that is controlled at 0.1 MPa/s. The applied load represents 40% from compressive strength at 28 days and five loading cycles are performed being limited to 10 MPa. The final result represents a correlation between dissipated energy during the first cycle of loading (hysteresis area) and the plastic deformation after the five loading and unloading cycles.[10,24]

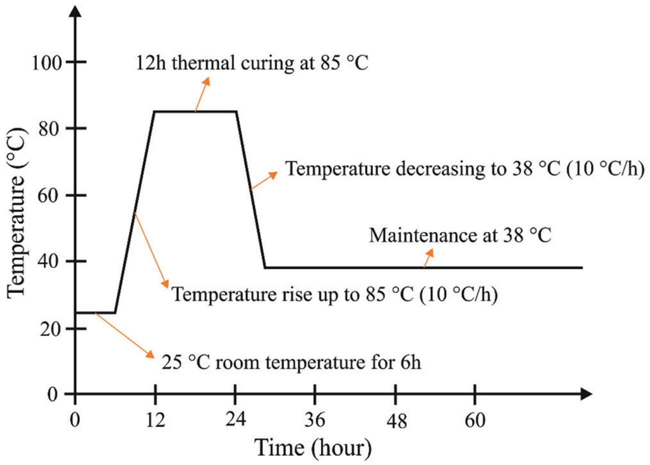

Fig. 1—Curing process and environment exposure condition.

Concrete microstructural analysis

The microstructure of the concretes was evaluated by using SEM with X-ray EDS.

The analyses were performed in samples with fracture surface on fragments taken from the nuclei of the specimens submitted previously to mechanical tests (after sputter gold coating) and the images were obtained using a secondary electron detector (SE).

EXPERIMENTAL RESULTS AND DISCUSSIONS

Physical properties

The average expansions over time are shown in Fig. 2. Specimens began to show higher levels of expansion at 35 days (0.13%), reaching 168 days with 0.98% expansion. In the studies by Amine et al.,[6] Martin et al.,[12] Fu et al.,[29] and Giannini et al.[24] on DEF, expansions at 168 days were also in the order of 1%.

The analysis of mass over time had also shown from day 28 of the assay a large increase along time (Fig. 2). At 168 days the mass increase was approximately 2%.

Al Shamaa et al.,[30] Zhang et al.,[15] and Sanchez et al.[10] indicate that variations in mass of this order are indicative of manifestations coming from DEF. Figure 3 shows the correlation between expansions and the gain in mass of the specimens over time, especially from 42 days.

Mechanical properties

The analysis of the compressive strength of concrete over time shows a drop in this property from 28 days to the final age of the assay (Table 4 and Fig. 4). Most studies on DEF point to the poor sensitivity of compressive strength, which is the opposite of what was found in this research. Many studies point out that compressive strength will only be affected when expansion percentages are around 2.0%.[24,31,32]

Table 4—Mechanical properties of concretes

Properties		Age (days)				
		07	28	56	84	168
Compressive strength (MPa)	Mean	37.91	33.07	29.08	27.44	16.87
	Standard deviation	0.08	0.20	4.20	0.56	0.13
Tensile strength (MPa)	Mean	2.74	2.54	1.74	1.73	1.23
	Standard deviation	0.56	0.43	0.08	0.20	0.21
Modulus of elasticity (GPa)	Mean	27.43	21.40	15.95	8.70	7.65
	Standard deviation	1.22	1.20	0.45	1.50	0.05

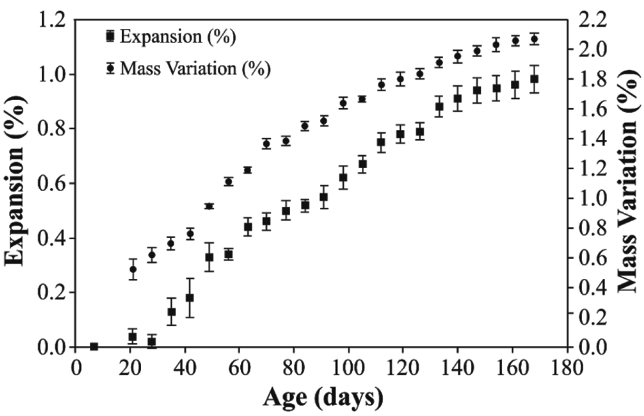

Fig. 2—Average expansions and mass variation of concrete prisms over time.

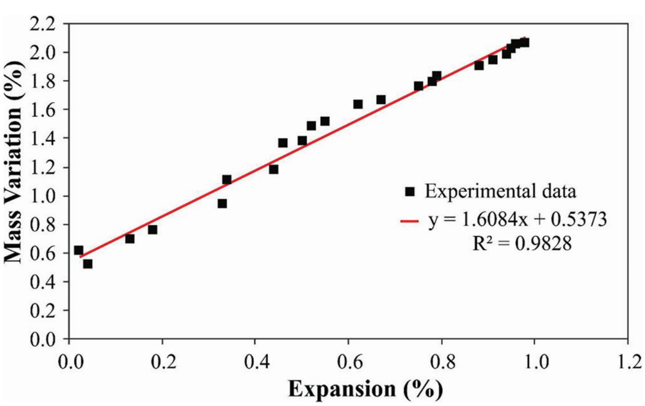

Fig. 3—Expansion versus mass variation over time.

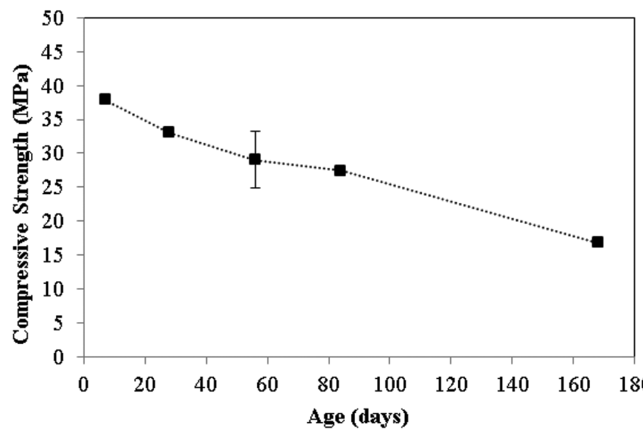

Fig. 4—Results of axial compressive strength over time.

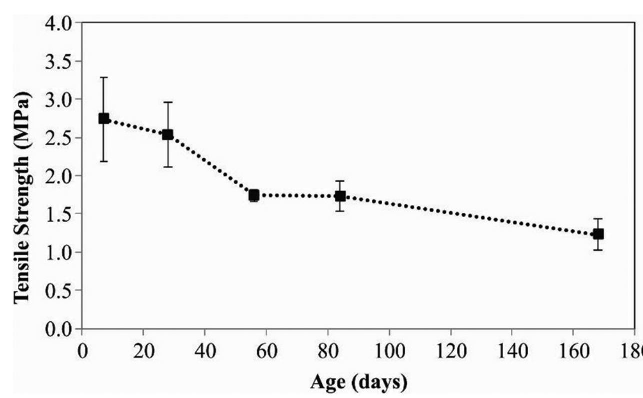

Fig. 5—Results of tensile strength by diametral compression over time.

However, in this paper, the negative reflexes in this property already occurred at 56 days (0.34% of expansion). At 168 days expansions were approximately 1%.

For the ages of 56, 84, and 168 days there was a reduction of approximately 23%, 27%, and 55%, respectively, in relation to the 7-day strength—that is, at 168 days the concrete lost over half of its compressive strength (Fig. 4). Expansions for the same ages have reached values of 0.34%, 0.52%, and 0.98%, respectively. These results are consistent with some studies such as those by Sanchez et al.[10] and Giannini et al.,[24] although each researcher had tested specific mixture designs, with different cement contents.

It should be noted that in the study by Giannini et al.[24] the results of the concrete's modulus of elasticity showed an approximate reduction of 75%, ranging from 23.10 to 5.80 GPa until reaching 1% expansion. In the present study, the reduction of the modulus of elasticity was similar, 72% (from 27.43 to 7.65 GPa), approximately. Regarding expansions, according to data from Sanchez et al.,[10] for expansion levels between 0.30 and 0.40%, the reduction in the modulus of elasticity was between 50 to 65%, while from this research and similar levels of expansion the behavior indicates an estimated decrease of approximately 35% for modulus.

Tensile strength was also sensitive to damage caused by DEF, as can be seen in Fig. 5. Between 7 and 168 days the reductions were in the order of 55% at almost 1% of expansion. Historically, concrete structures affected by DEF tend to be even more sensitive to the drop in tensile strength. Some studies indicate that this property can be reduced by up to 65%.[10,33]

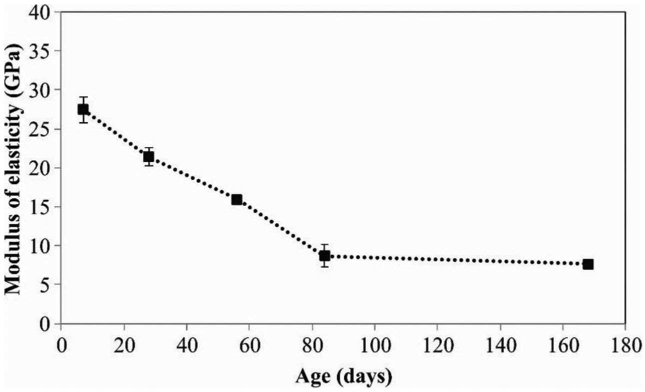

Fig. 6—Results of modulus of elasticity over time.

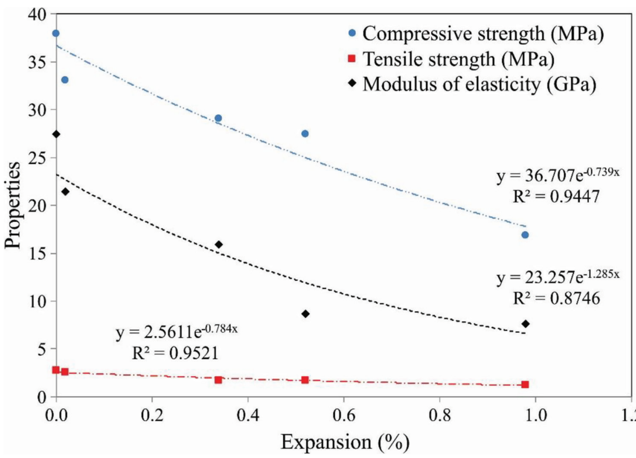

Fig. 7—Results of axial compressive strength, tensile strength, and modulus of elasticity versus expansion.

The modulus of elasticity of the concrete was also significantly affected by DEF (Fig. 6). The most significant drop in this property have occurred between 28 and 84 days, approximately 60%. Over time and up to 168 days the modulus of elasticity has reached only 7.65 GPa, with a decrease of 72%, indicating serious deterioration. Negative effects on the elasticity modulus were found in several studies, even before expressive expansions.[10,24,33] Melo[34] and Diamond[35] in their studies also found reductions in the elasticity modulus, but only at more advanced ages, and close to 1 year.

Figure 7 shows the relationship between average expansions and the average data of axial compressive strength, tensile strength and modulus of elasticity over time.

It is clear an inversely proportional behavior between the expansions and the mechanical properties—that is, while the percentage of expansion increases, all the mechanical properties investigated are reduced. The most affected properties by DEF from this study were compressive strength and modulus of elasticity.

The SDI of concrete is affected by the occurrence of cracking, according to literature.[36,37] The SDI results obtained in this study (Fig. 8) show that as the ettringite neoformations increases, expansions increase and also the damage to the cement matrix, given greater plastic deformations and lower stiffness, both detected by this assay. In general, this is a gradual process over time, bearing in mind the age evaluated in this study.

Comparing the results of SDI with those of modulus of elasticity, it is possible to notice a decrease of 72% in the modulus and an increase of the SDI to 0.52. Giannini et al.[24] observed a similar behavior (decreases in the order of 75% for the modulus of elasticity and an increase of 80% in the SDI, up to 0.3). This behavior demonstrates that the damage caused by DEF can severely affect structural stiffness.

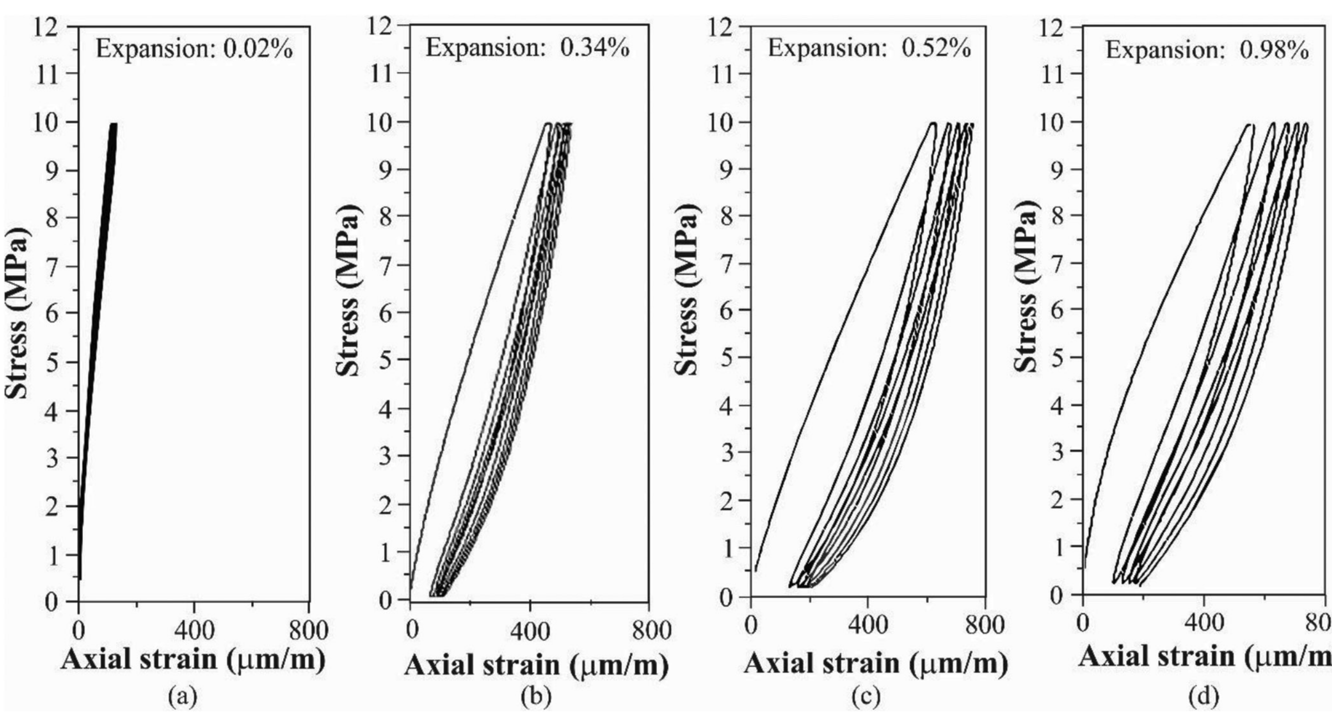

Fig. 8—Results from SDT test over time: (a) 28 days; (b) 56 days; (c) 84 days; and (d) 168 days.

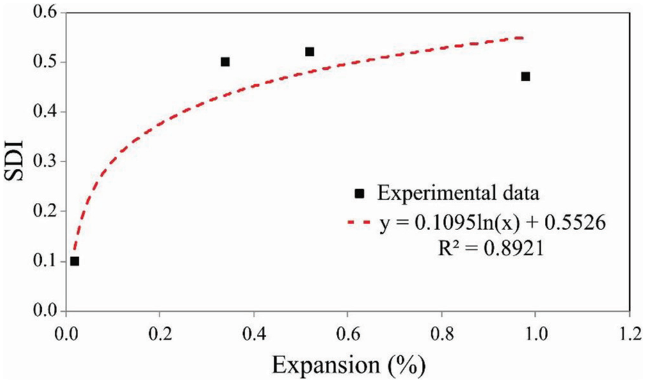

Fig. 9—SDI versus expansion.

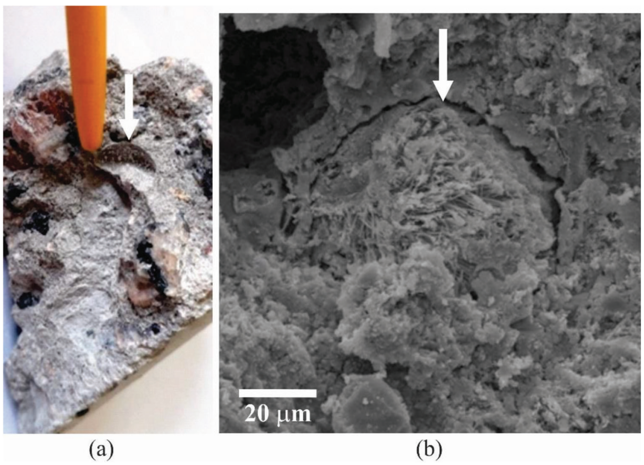

Fig. 10—Visual inspection and SEM micrograph on Day 28 of assay.

In Fig. 9, what can be verified is that for an SDI variation of between 0.10 and 0.52, the expansion reaches its maximum value of approximately 1%. However, from Day 56, there is a tendency for the SDI value to stabilize, with expansions below 0.4% at this age. In other words, for expansion rates above 0.4%, the SDI parameter was not as sensitive, even for significant gains in expansion (between 2 and 6 months).

Visual inspection and microstructural evaluation

Based on the visual inspection and microstructural analysis by means of SEM/EDS, the presence and increase of the amount of ettringite in the concrete over time was observed, thus corroborating the mechanical results of this research.

Figures 10 to 13 show images obtained from fragments taken from concrete after the mechanical tests. It is noteworthy that the ettringite was only identified, as from day 28, as white depositions in the voids of the concrete fragments (Fig. 10(a)) in the visual inspection, which was confirmed by SEM/EDS, by observing fragile areas in the paste and due to the presence of these newly formed crystals (Fig. 10(b)). Over time, it was possible to identify white efflorescences in the voids and in the paste/aggregate interface (Fig. 11(a)), which was corroborated by the SEM/EDS due the presence of several agglomerations of ettringite crystals, and also because of the fragilized paste/aggregate interface, which had neo-formed deposits. Microanalysis by EDS confirmed the presence of DEF due to the chemical elements of calcium, sulfur and aluminum being identified (Fig. 11(b) to (c)).

At 84 days, the presence of ettringite in the concrete fragments inspected was more expressive and evident (Fig. 12(a)) and the analysis of the microstructure enabled it to be observed that the paste had been completely taken by the crystals, with loss of adhesion in the paste/aggregate interface, besides powdery areas at some points (Fig. 12(b)). Those aspects became even more evident at 168 days, when efflorescences were present and disseminated in all concrete fragments that were analyzed under SEM, in the voids and in the cement matrix (Fig. 13(a) to (b)) and also on the aggregate (Fig. 14(a)), leading to expansion and cracking in the transition zone (Figure 14b).

CONCLUSIONS

The concrete produced in this research was susceptible to DEF based on the methodology adopted during the curing period, exposure condition and the materials used. The various forms of analysis carried out let it be verified that from 28 days, delayed ettringite had already formed. From this age, as a result of such analysis, it can be concluded that:

Both expansion and mass variation increased over time due to increased DEF, and at 168 days reached expansions of the order of 1%, associated with a 2% variation in mass.

With regard to the mechanical properties, compressive strength decreased approximately 55% at 168 days, thereby contradicting several previously published studies. For this same age, the tensile strength and the modulus of elasticity also suffered high reductions, in the order of 55% and 72%, respectively, which reinforces their sensitivity as to microcracks. A concrete that loses more than half of its properties will not perform adequately over time and its useful life is over.

Studies involving the use of the SDT assay showed consistency with the other results obtained. The SDI parameter presented quantitative data confirming the existence of DEF, there being 0.52 damage to concrete stiffness at 168 days.

Visual inspections, followed by microstructure analyses have indicated since 28 days several characteristics of DEF, with a progressive increase of ettringite over time. There were evidenced several areas containing massive ettringite, agglomerations of neo-formed ettringite, lack of adherence between aggregate and paste, as well as a powdery matrix of evaluated concretes.

Based on all of the above, as a global conclusion of this research it can be stated that a concrete containing cements with no admixture, with similar characteristics to the one presented in this paper, can reach high expansions and of the order of 1%. This high level of expansion could be noticed considering the high temperature achieved in the thermal curing (85°C) as well as the specific exposure environment of concrete (water immersion at 38°C). All of these are conditions to cause great negative reflexes on the mechanical properties above 50% (compressive strength, tensile strength and modulus of elasticity), which affect the durability and service life of concrete. Therefore, it is not recommended for use without the necessary steps and precautions and the adoption of preventive measures.

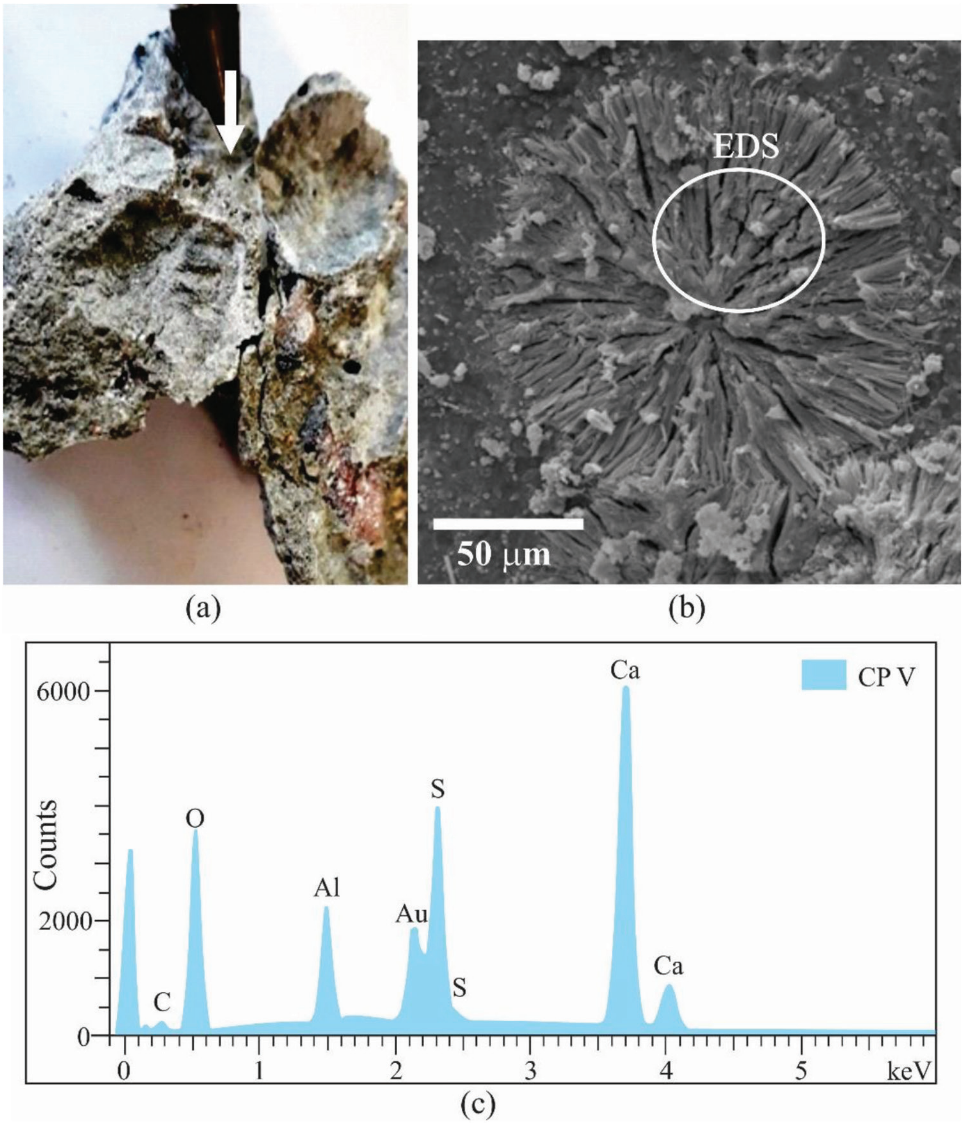

Fig. 11—Visual inspection, SEM micrography, and EDS on Day 28 of assay.

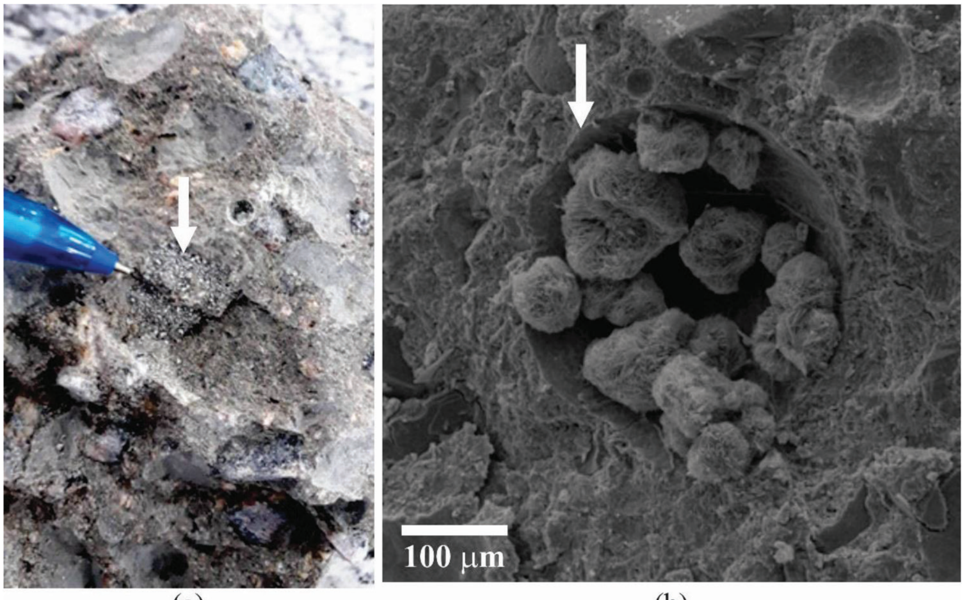

Fig. 12—Visual inspection and SEM micrograph on Day 84 of assay.

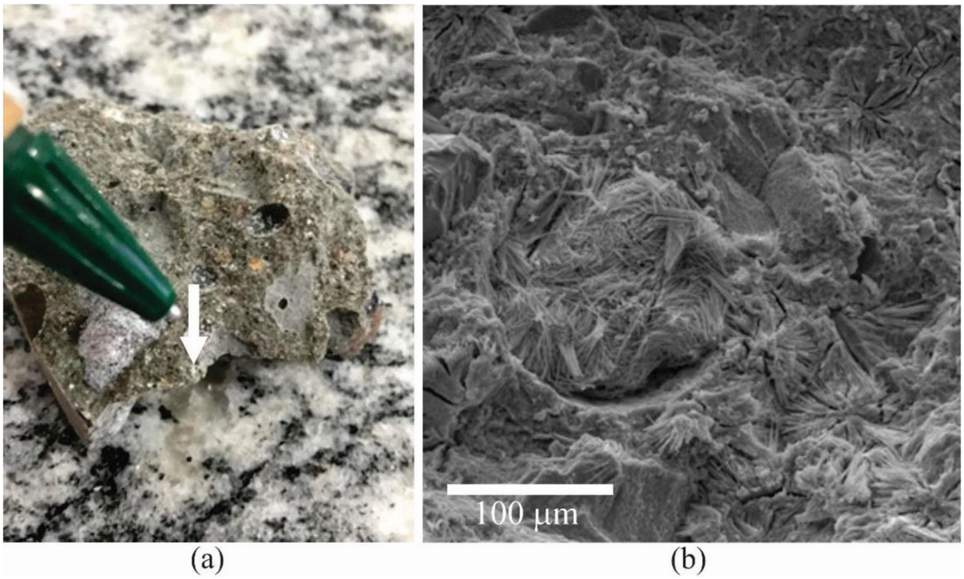

Fig. 13—Visual inspection and SEM micrograph on Day 168 of assay.

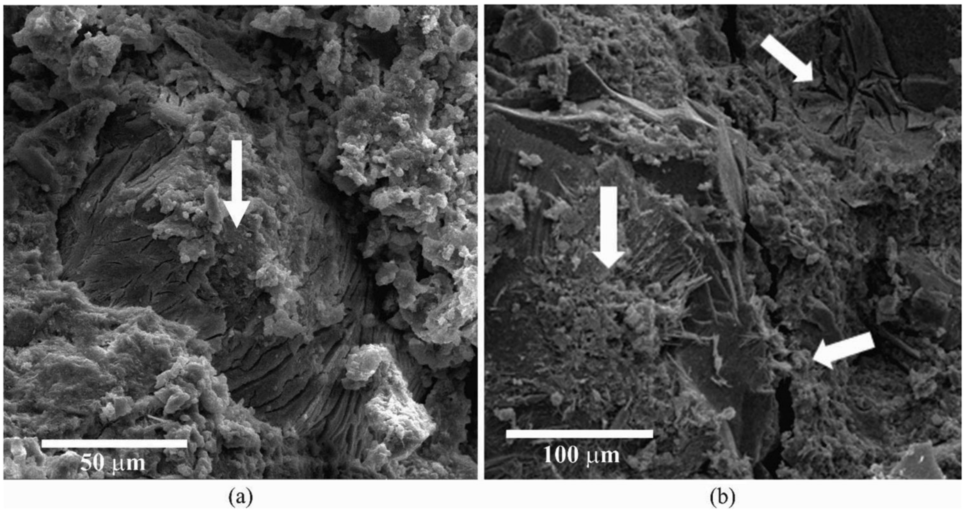

Fig. 14—SEM micrographs on Day 168.

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