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Influence of curing time on the fire performance of solid reinforced concrete plates

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ABSTRACT

When reinforced concrete elements are subjected to high temperatures, such as in a fire, they are susceptible to physical and chemical changes that cause spalling, thereby undermining their performance under such conditions. It is known that the age and the internal moisture content of concrete are factors that contribute to this event, but the intensity of spalling is not yet a consensus. This study aimed to assess the influence of age and internal moisture on the performance of concrete walls at high temperatures. Therefore, 6 real-scale walls were built with dimensions of 3.15×3.00 m, with the same composition of concrete, for tests in a vertical furnace under the ISO 834 curve, for ages of 7, 14, 28, 56, 84 and 830 days. Moisture was measured as per the electrical resistivity of concrete. It was noted that walls with ages above 84 days showed no spalling whatsoever, due to the internal moisture of concrete. The most severe spalling took place at 14 days, thus evidencing that pore interconnectivity and hydrated cement crystallization can contribute as well.

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1. Introduction

The performance of concrete structures with regards to high temperatures can be assessed through the analysis spalling severity levels of the section and mechanical strength loss [1]. Some phenomena support this analysis, such as (a) physical and chemical changes in cement paste and aggregates and (b) thermal incompatibility between these two [2]. Each mechanism develops within a specific temperature range, whereas internal moisture and degree of hydration of concrete remain as decisive factors.

The phenomenon of spalling is related to the heat flowing out of the fire and the distribution of temperature along the element. Heat flow is linked to the structure's heat rate, and, consequently, the duration of the fire. As for temperature distribution, the most influential factors are cement type, aggregates, additives, geometry and transverse section of the element, paste saturation degree, age, water/cement ratio, presence of cracks and degree of concrete porosity [3]. So, this is a complex phenomenon that justifies studies that analyze variables that should be evaluated separately to attain a more assertive study.

For concrete elements with structural function subjected to loading, the spalling is mainly influenced by the nominal stress and the concrete strength, so that the moisture content has a minor effect on the phenomenon. Therefore, concrete

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is more susceptible to spalling when required for compressive stress [4,5]. However, for concrete elements without structural function, with only sealing function, the stress is of lower intensity and, therefore, the moisture content is more relevant for the phenomenon.

For a thermo-hydraulic and thermomechanical matter, spalling comprises a phenomenon that promotes the loosening of layers of the surfaces of concrete exposed to high temperatures. It is a semi-destructive phenomenon that derives from the nonuniform distribution of heat along the section [6,7] and the amount of evaporable water of concrete [8]. It occurs after the sudden release of energy and can even be explosive. The internal pressure caused by free water vapor contained within concrete, along with the formation of cracks and transverse displacement of the system, all of which are consequence of an internal stress state, can contribute to spalling [8].

Moisture content is directly proportional to concrete electrical resistivity, which is influenced by water saturation degree, and concrete porosity and salinity [9,10]. Other authors state that the mechanism of spalling does not depend on the piece's stress state [11], but that is not a consensus [2]. Binding conditions turned out to be more influential than external loading, mainly in terms of the restraint to the thermal expansion generated by the heat [12]. Studies on real fire cases have shown that columns, slabs and walls are most susceptible to spalling because they are subjected to a higher degree of thermal expansion restraint [13]. Therefore, bigger samples behave differently from those evaluated in smaller scale, deserving more research focus.

Factors such as surface heating rate, internal free water and porosity do not necessarily ensure the existence of spalling in every heat-exposed concrete and section [1]. Spalling occurs when (I) surface heating rate is about 3 °C per minute [3], (II) cement paste permeability is low, below $5 \times 10^{-15} \text{ m}^2$ [12] and (III) pore saturation degree is high, between 2% and 3% of the mass of concrete [2]. The nature and particle-size distribution of the coarse aggregate contribute as well [14–16]. FIB Bulletin № 38 [17] even puts age, maximum temperature and heating rate of the piece, shape and size of the transverse section, presence of cracks, steel rate, reinforcement arrangement, presence of fibers and the intensity of loading of the structural element as factors to be researched on.

Neville [18] states that concrete, when wet, behaves as a semiconductor, that is, it presents electrical resistivity of about 102 Ωm. When dry, it is capable of presenting electrical resistivity of about 109 Ωm. For Polder [19], though, increases in temperature result in smaller resistivities due to higher ionic mobility and higher ion-ion or ion-solid interaction.

Table 1 – Amount of materials.

Description	Quantity (kg/m ³)
Cement	350
Fine sand	273
Regular sand	637
Gravel	952
Superplasticizer	2.2
Water	187

Table 2 – Wall naming.

Wall number	Testing age (days)
W1	7
W2	14
W3	28
W4	56
W5	84
W6	830

This scenario reveals few researches on the influence of moisture content and age on the fire performance of real-scale reinforced concrete elements. Therefore, this study aims to analyze reinforced concrete plates, with concrete of different ages being subjected to high temperatures. Therefore, 6 real-scale walls analysed, with the same composition of concrete, for ages of 7, 14, 28, 56, 84 and 830 days.

2. Methods

In order to conduct this study, reinforced concrete plates were crafted with dimensions of 3.15 × 1.00 m and 0,1 m of thickness. For each test, three concrete plates had to be used to form a wall with dimensions of 3.15 × 3.00 m. The 18 plates built totalized a set of 6 walls that were subjected to the fire resistance test at ages of 7, 14, 28, 56, 84 and 830 curing days. The plates were previously instrumented to measure electrical resistivity, hence allowing the measurement of internal moisture content.

Table 1 presents the amount of materials used to craft the plates, whereas **Table 2** names the walls under analysis and **Table 3** shows the strengths of concrete up to the age of 84 days.

High early strength Portland cement was used, which had specific gravity of 3120 kg/m³. The admixture added to concrete was a synthetic superplasticizer based on polycarboxylate polymers with density of 1120 kg/m³. The steel used to craft the pieces was ribbed with yield strength of 500 MPa (5×10^8 Pa) and diameters of 5.0 mm (0.005 m), 8.0 mm (0.008 m) and 12.5 mm (0.0125 m). **Fig. 1a** shows the reinforcements in reinforced concrete plates.

To read electrical resistivity, starting from the Wenner four-pin method, 4 flexible conductors with 8.0 mm (0.008 m) of diameters were placed on the middle plate of each wall, 0.075 m away from the corners and with 0.1 m of spacing from each other, as depicted in **Fig. 1b**. Electrical resistivity was read for walls W1 to W6.

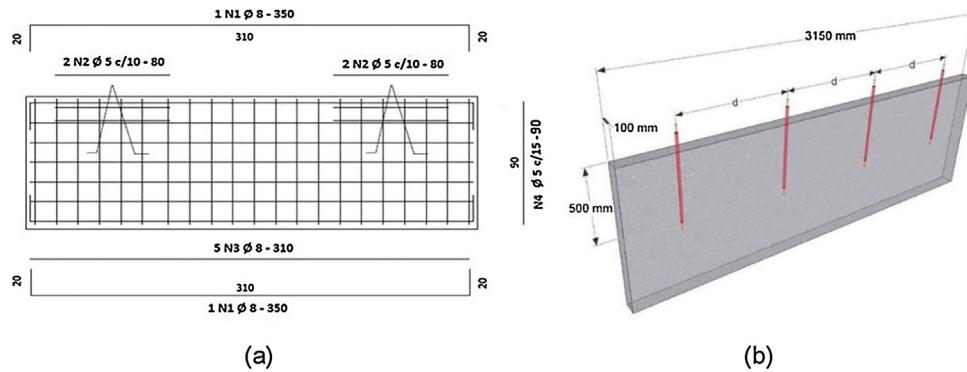
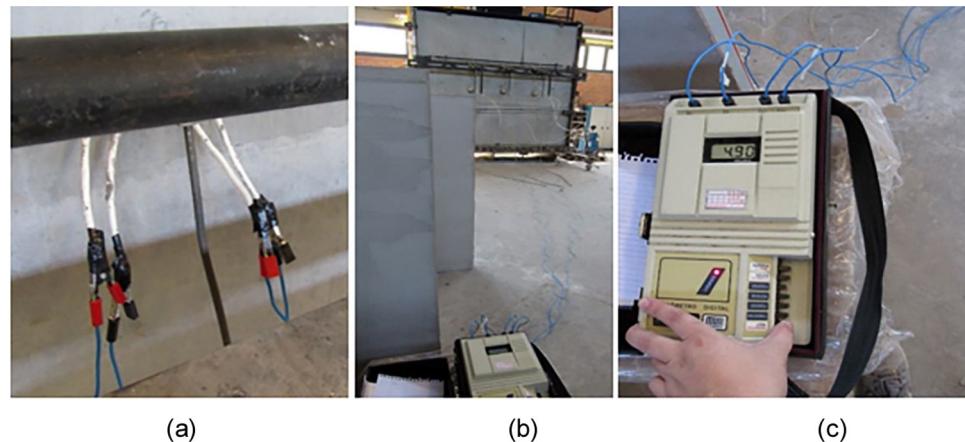
Still regarding the electrical resistivity analysis, at the end of the conductor cables installed on the plates, a Megabráss-branded MTD-20 KW digital ground tester was attached, as depicted in **Fig. 2**. Eq. (1) was then used to determine the electrical resistivity of the plates.

$$\rho = \frac{4\pi R}{1 + \frac{2D}{\sqrt{D^2+4d^2}} - \frac{D}{\sqrt{D^2+d^2}}} \quad (1)$$

where, ρ is the electrical resistivity (Ωm), R is the resistivity read by the equipment (Ω), D is the distance between electrodes (m) and d is the depth of the electrode (m). Concrete

Table 3 – Compressive strength.

Curing age	Sample	Compressive strength (MPa)	Potential compressive strength (MPa)	Standard deviation (MPa)
7 days	1	40.0 (4×10^7 Pa)	40.0 (4×10^7 Pa)	1.56 (1.56 × 10 ⁶ Pa)
	2	37.8 (3.78×10^7 Pa)		
14 days	1	43.5 (4.35×10^7 Pa)	43.5 (4.35 × 10 ⁷ Pa)	1.70 (1.7 × 10 ⁶ Pa)
	2	41.1 (4.11×10^7 Pa)		
28 days	1	59.9 (5.99×10^7 Pa)	59.9 (5.99 × 10 ⁷ Pa)	0.07 (7 × 10 ⁴ Pa)
	2	59.8 (5.98×10^7 Pa)		
56 days	1	70.8 (7.08×10^7 Pa)	70.8 (7.08 × 10 ⁷ Pa)	0.14 (1.4 × 10 ⁵ Pa)
	2	70.6 (7.06×10^7 Pa)		
84 days	1	71.5 (7.15×10^7 Pa)	75.0 (7.5 × 10 ⁷ Pa)	2.47 (2.47 × 10 ⁶ Pa)
	2	75.0 (7.5×10^7 Pa)		

**Fig. 1 – Drawing of plate (a) reinforcement and (b) conduits.****Fig. 2 – Details of (a) connection of the devices to the sample, (b) connection of the devices to the digital ground tester, and (c) digital thermometer used to read electrical resistivity.**

internal moisture, which is affected by this parameter, was determined by the previously prepared smaller-scale samples with dimensions of $0.1 \times 0.1 \times 0.2$ m, demonstrated in Fig. 3a and b. These prototypes served as a means to calibrate the equipment and guide the real-scale process.

The fire resistance tests were performed in itt Performance technological institute, at Unisinos (University of Vale do Rio dos Sinos), in Brazil. The concrete plates were installed on a movable metal frame with dimensions of 3.15×3.00 m (Fig. 4a). The plates were cured on the frame and were attached to the vertical furnace (Fig. 4b) on the day of testing. The furnace had a fire-exposure area of 2.50×2.50 m

and used gas burners. Fig. 4c depict the wall attached to the furnace.

The test followed the standard time–temperature curve described by ISO 834-1 [20]. To seal the system and bind the plates, fire-resistant sealant was used to preserve airtightness and thermal insulation.

The test method followed NBR 10636 [21], ASTM E119 [22], AS 1530 [23] and BS 476 [24]. During the test, three criteria were evaluated: (a) thermal insulation, (b) smoke and airtightness and (c) structural stability. The walls were tested without loading due to the real use of the concrete plates in the buildings, so that they have exclusively the sealing function.



Fig. 3 – Detailing of small-scale prototypes to determine resistivity of real-scale.

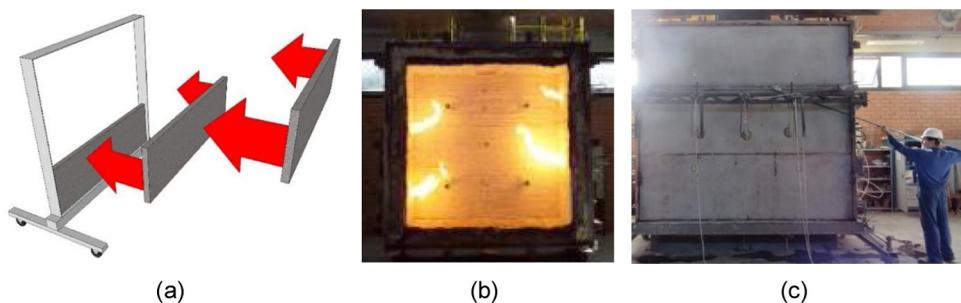


Fig. 4 – Sample assembling procedure.

The fire-resistance rating (FRR) was then determined based on these requirements. Whenever one of these requirements was not met, the test would be halted, with the exception of thermal insulation. It was possible to identify cracks on the external face of the samples with the aid of a thermographic camera.

Regarding thermal insulation evaluation, 9 thermocouples were placed on the unexposed face of the walls. As per BS 476 [24], the sample fails to meet this requirement when the arithmetic mean of the external temperature surpasses 140 °C, or individually if any thermocouple surpasses 180 °C, whichever occurs first. As for airtightness, failure occurs when there is a crack or opening that allows the passage of hot gases and smoke, hence setting ablaze the cotton wad. The structural stability requirement refers to the total or partial collapse of the samples. Deformations were assessed every 10 min with the aid of a laser measuring tool positioned on the center of the wall.

The sample was continuously monitored by type K thermocouples with 1.5 mm (0.0015 m) of diameter on the fire-exposed face, and type T thermocouples with 0.7 mm (0.0007 m) of diameter on the unexposed face.

3. Results and discussion

Every wall was exposed to fire for 240 min. Table 4 demonstrates the standard requirements of thermal insulation, airtightness and structural stability managed during the test, along with the displacements measured at the center of the samples.

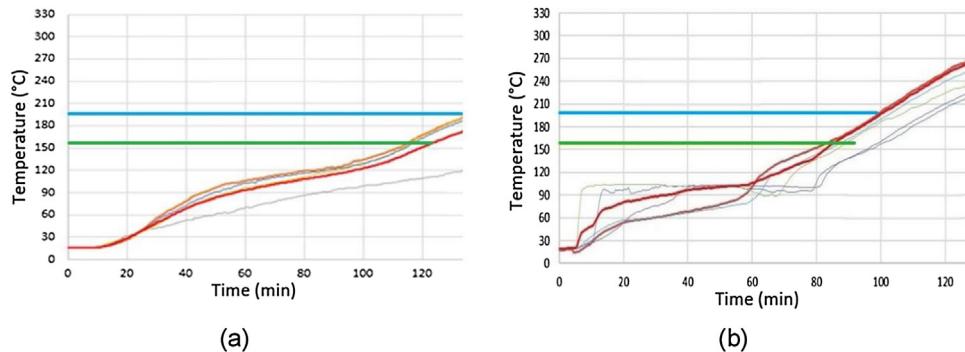
It should be noted that W6 attained an FRR lower than that of W5, despite its longer curing time. This result can be explained by the decrease of porosity, due to cement hydration and the consequent filling of concrete voids, changing the structure of the pores of the paste [25]. This suggests that there is a heat transfer mechanism that stands out among the others, which is thermal conduction. Fig. 5a and b show the temperature evolution of each thermocouple positioned on non-fire face during the test of samples W5 and W6, respectively, where the green line is the thermocouple average failing temperature, the blue line is the individual failing temperature

Some references state that, in concrete with less porosity, heat transfer by thermal conduction is intensified, thus mitigating the mechanisms of radiation and convection. Rigão [26] and Gil et al. [27] highlights that thermal conductivity depends on the density of the materials of the mixture. In case of lower void ratios, then the structure will have greater thermal conductivity values. It is also known that thermal conduction makes heat flow more rapidly along the material than the mechanisms of radiation and convection, which do not occur in solid mediums, as per Rosemann [28]. Due to the major action of heat transfer, the thermal insulation of concrete is negatively influenced, as noted.

Therefore, because W5 and W6 did not exhibit spalling and, consequently, a loss of wall thickness, the heat transfer mechanism was not accelerated. Thus, the porosity of the walls is a significant factor to analyze the thermal insulation of the samples. Because W6 had a much longer cure time than W5, it obtained a higher degree of hydration and smaller porosity, allowing heat transfer to occur faster. Fig. 5 corroborates

Table 4 – Comparison between fire resistance tests.

Wall	Testing age (days)	Thermal insulation (min)	Airtightness (min)	Structural stability	Displacement measured (m)
W1	7	104.0	240	Unaffected	0.045
W2	14	86.5	240	Unaffected	0.060
W3	28	97.5	240	Unaffected	0.072
W4	56	95.5	240	Unaffected	0.078
W5	84	123.5	240	Unaffected	0.069
W6	830	91	240	Unaffected	0.079

**Fig. 5 – Temperature evolution during the test of (a) W5 and (b) W6.****Table 5 – Magnitude of spalling and resistivity.**

Wall number	Severity levels of spalling		Resistivity (Ωm)
	In area (m^2)	In percent (%)	
W1	1.64	26.25	72.8
W2	2.72	43.49	93.2
W3	0.54	8.61	310.7
W4	0.38	6.07	506.2
W5	0.00	0.00	644.0
W6	0.00	0.00	1048.1

this analysis due to the steeper increase in the non-fire face temperature of W6 compared to W5.

In general samples with longer curing ages showed smaller areas of spalling. After 84 days of curing, concrete spalling did not occur, owing to the higher degree of hydration that decreases free water. This result is in consonance with the main references that study the phenomenon, which evidence that, in laboratory fire resistance tests that revolve around concrete samples, prototypes with curing age of more than 90 days may disregard spalling [1]. This is therefore stressed in the results shown in Table 5, which discloses the degree of spalling and its correlation with resistivity.

As for the 7-day old plates, most of the water that intensifies spalling suffers from low cement hydration and ends up presenting greater interconnectivity between cement paste pores. This fact made it easier for water to evaporate and was followed by severe spalling due to the low tensile strength of concrete at that age. At 14 days, cement hydration was higher, with an interlacing of cement hydration crystals yielding less interconnectivity between pores, which, despite the amount of free water that remained, underwent higher spalling than at 7 days. At 28 days, the degree of hydration and the interlacing of hydrated cement crystals was even higher, although

the smaller amount of free water that remained ended up hindering spalling. This line of thought justifies the performance of older plates, showing that high cement hydration and presence of free water are fundamental to comprehend the phenomenon, and that they can be ignored at ages above 84 days.

Fig. 6a-f demonstrate the overall appearance of the samples that were tested, whereas Fig. 7a-e f comprise the thermographic images at 15 min of testing, when most of the samples presented large cracks. The maximum temperature recorded, as shown in Fig. 7, on W1 was 64 °C, W2 was 81 °C, W3 was 76 °C, W4 was 76 °C, W5 was 62 °C and W6 was 64 °C.

The resistivity reading for concrete plates presented results that were coherent with the bibliography. Shekarchi et al. [29] even affirm that electrical conductivity is related to the number of ions dissolved in the water present in the interconnected pores of the paste. Therefore, with smaller moisture contents as a result of the curing time, the system provides less water for the electrical conductivity phenomenon and ends up producing higher resistivity values.

There was a difference of 217.5 Ωm between the sample with 14 days and the one with 28 days of curing owing to the cement paste being more intensely hydrated. At the first ages, namely 7 and 14 days, resistivity was low, leading to the higher spalling levels of walls W1 and W2. There was also a significant increase of resistivity for the sample with 28 days of curing, which is noted by a peak of same magnitude in the spalling measured.

4. Conclusions

The performance of concrete plates under high temperatures is influenced by spalling, as plates that undergo higher levels of it also achieve shorter thermal insulation times. Spalling

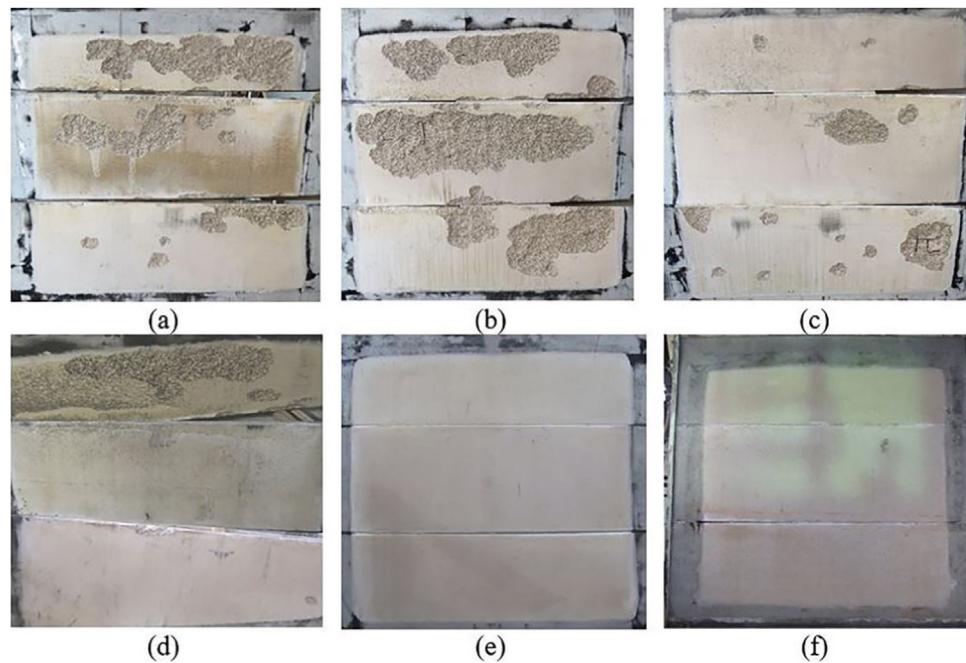


Fig. 6 – Appearance of wall (a) W1, (b) W2, (c) W3, (d) W4, (e) W5 and (f) W6.

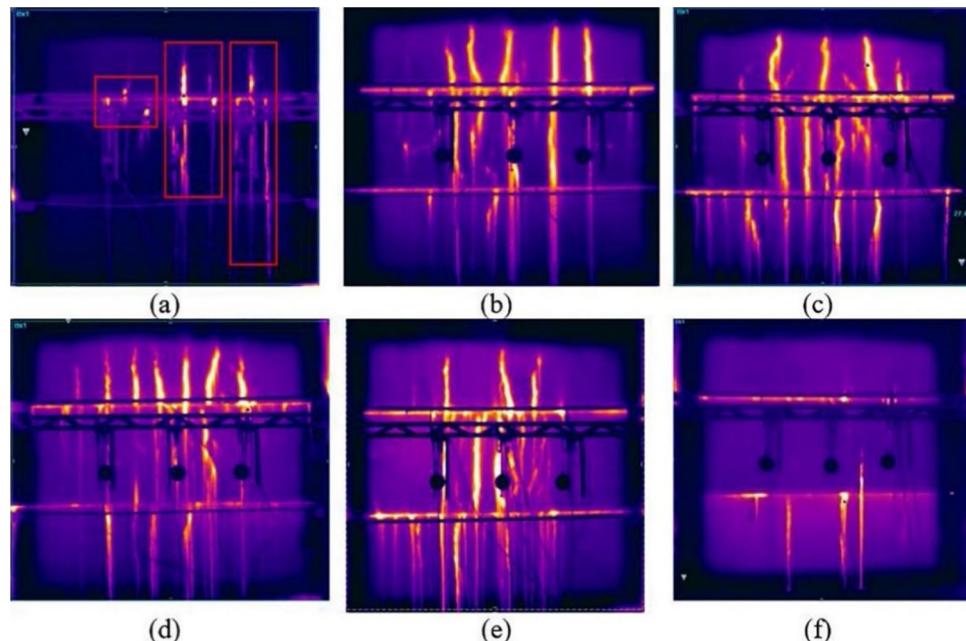


Fig. 7 – Thermographic images of (a) W1, (b) W2, (c) W3, (d) W4, (e) W5 and (f) W6.

is related to the degree of hydration of cement, justified by the presence of free water in the pores, even though internal moisture tends to decrease, thus decreasing free water in the pores at more advanced ages. Still, moisture content should not be treated as the sole factor that has effect in the analysis of spalling of reinforced concrete elements subjected to high temperatures.

At 7 days, low mechanical strength and high interconnectivity between concrete pores, both of which result from the

low interlacing of the paste's hydrated crystals, seem to be the most relevant factors. At 14 days, the spalling caused by the degree of hydration of cement seems to be the main factor regarding thermal insulation. After 84 days of curing, the phenomenon of spalling can be disregarded from the analyses because some of the conditions required to trigger this phenomenon are no longer in effect.

Electrical resistivity may become a suitable tool to measure the likelihood of spalling for prototypes of concrete exposed

to high temperatures, presenting variation that are apparently influenced by the materials that compose concrete. Thus, as the measured electrical resistivity values increase, the probability of spalling may decrease due to the lower moisture content and, consequently, provide a better fire performance of the structure.

Conflict of interest

The authors of this papers attest that there is no conflict of interest.

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.jmrt.2019.12.081>.

REFERENCES

- [1] Robert F, Colina H, Debicki G. A durabilidade do concreto mediante ao fogo. In: Ollivier J-P, Vichot A, editors. Durabilidade Do Concreto. 1 ed São Paulo: IBRACON; 2014. p. 509–58.
- [2] Khouri GA. Effect of fire on concrete and concrete structures. Progress in structural engineering and materials, Vol. 2 ed. John Wiley & Sons; 2001. p. 429–47.
- [3] Fu Y, Li L. Study on mechanism of thermal spalling in concrete exposed to elevated temperatures. Mater Struct 2011;44(1):361–76.
- [4] Zheng WZ, Hou XM, Shi DS, Xu MX. Experimental study on concrete spalling in prestressed slabs subjected to fire. Fire Safety J 2010;45(5):283–97.
- [5] Hou Xiaomeng, Kodur VKR, Zheng Wenzhong. Factors governing the fire response of bonded prestressed concrete continuous beams. Mater Struct 2015;48(9): 2885–900.
- [6] Jansson R. Fire spalling of concrete: theoretical and experimental studies. [Civil Engineering PhD]. Stockholm: KTH Vetenskap Och Konst; 2013.
- [7] Fernandes B, Gil AM, Bolina FL, Tutikian BF. Thermal damage evaluation of full scale concrete columns exposed to high temperatures using scanning electron microscopy and X-ray diffraction. DYNA (Medellín) 2018;85:123–8.
- [8] Wang G, et al. Fire safety provisions for aged concrete building structures. Procedia Eng 2013;62:629–38.
- [9] Lafaste JF. Evaluation non destructive de l'état d'endommagement des ouvrages en béton armé par mesure de résistivité électrique. [Civil Engineering PhD]. Université de Bordeaux; 2002.
- [10] Plooy Rdu, Dérobert X, Villain G, PalmaLopes S. Development of a multi-ring resistivity cell and multi-electrode resistivity probe for investigation of cover concrete condition. NDT E Int 2013;54:27–36.
- [11] Morita T, et al. An experimental study on spalling of high strength concrete elements under fire attack. Fire Safety Sci 2000;6:855–66.
- [12] Kalifa P, Menneteau FD, Quenard D. Spalling and pore pressure in HPC at high temperatures. Cement Concrete Res 2000;30(12):1915–27.
- [13] Costa CN. Dimensionamento de vigas de concreto armado em situação de incêndio. [Civil Engineering PhD]. São Paulo: Polytechnic School, Universidade de São Paulo; 2008.
- [14] Pan Z, Sanjayan JG, Kong DLY. Effect of aggregate size on spalling of geopolymers and Portland cement concretes subjected to elevated temperatures. Const Building Mater 2012;36:365–72.
- [15] Ehrenbring HZ, Quinino U, Oliveira LS, Tutikian BF. Experimental method for investigating the impact of the addition of polymer fibers on drying shrinkage and cracking of concrete. Struct Concr 2019;20:1064–75, <http://dx.doi.org/10.1002/suco.201800228>.
- [16] Pacheco F, Souza R, Christ R, Rocha C, Silva L, Tutikian BF. Determination of volume and distribution of pores of concretes according to different exposure classes through 3D microtomography and mercury intrusion porosimetry. Struct Conc 2018;19:1419–27, <http://dx.doi.org/10.1002/suco.201800075>.
- [17] Fédération Internationale. Du Betón (fb). Fire design of concrete structures – materials, structures and modeling – state-of-art report. Lausanne: Bulletin d'information 38; 2007. p. 97.
- [18] Neville AM. Propriedades do concreto. 523p. 5.ed. Porto Alegre: Bookman; 2016.
- [19] Polder RB. Test methods for on site measurement of resistivity of concrete – a RILEM TC-154 technical recommendation. Const Building Mater 2001;15:125–31.
- [20] International Organization for Standardization (ISO). Fire-resistance tests – elements of building construction – part 1: general requirements, ISO 834; 1999.
- [21] Associação Brasileira de Normas Técnicas (ABNT). Paredes divisorias sem função estrutural - determinação da resistência ao fogo: método de ensaio, NBR 10636; 1989.
- [22] ASTM E119-18a. Standard test methods for fire tests of building construction and materials. West Conshohocken, PA: ASTM International; 2018.
- [23] Australian Standard, Sidney AS 1530: methods for fire tests on building materials, components and structures; 2005.
- [24] British Standard, London BS 476-3: fire tests on building materials and structures. Classification and method of test for external fire exposure to roofs; 2004.
- [25] Santos L. Avaliação da resistividade elétrica do concreto como parâmetro para a previsão da iniciação da corrosão induzida por cloretos em estruturas de concreto. [M.Sc. dissertation]. Brasília: Civil and Environmental Engineering Department, Universidade de Brasília; 2006.
- [26] Rigão AO. Comportamento de pequenas paredes de alvenaria estrutural frente a altas temperaturas. Santa Maria, 2012. [M.Sc. dissertation]. Santa Maria: Civil and Environmental Engineering Graduate Program, Universidade Federal de Santa Maria; 2012.
- [27] Gil A, Pacheco F, Christ R, Bolina FL, Khayat KH, Tutikian BF. Comparative study of concrete panels' fire resistance. Aci Mater J 2017;114:755–62.
- [28] Rosemann F. Resistência ao fogo de paredes de alvenaria estrutural de blocos cerâmicos pelo critério de isolamento térmico. [M.Sc. dissertation]. Florianópolis: Civil Engineering Graduate Program, Universidade Federal de Santa Catarina; 2011.
- [29] Shekarchi M, Tadayon M, Chini M, Hoseini M, Alizadeh R, Ghods P, et al. Predicting chloride penetration into concrete containing silica fume, with measuring the electrical resistivity of concrete. In: 4th International Conference on Concrete Under Severe Conditions. CONSEC'04. Proceedings of the 4th CONSEC Congress. 2004.