Carbonation of concretes containing LC³ cements with different supplementary materials

Carbonatação de concretos contendo cimento LC³ com diferentes materiais suplementares

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Abstract

Due to the clinkerization process during the Portland cement production, large amounts of CO_2 are emitted, increasing the effects related to climate change (approximately 5-10% of global CO_2 emissions come from cement production), consequently, the seek for alternatives to mitigate these high emissions are necessary. The use of supplementary cementitious materials (SCM) to partial replace of Portland clinker/cement has been the subject of different research, including the use of LC^3 cements (Limestone Calcined Clay Cements), where up to 50% of Portland clinker can be replaced, however, cement industry has already used other supplementary cementitious materials with pozzolanic activities in commercial cements. In this sense, this work evaluates the performance of concretes containing LC^3 mixtures with the presence of different SCM (silica fume, fly ash, sugarcane bagasse ash and açaí stone ash) regarding durability issues by carbonation. The results showed that all concretes with LC^3 presented higher carbonation fronts in relation to the reference concrete, with Portland cement, due to the lower availability of calcium to react with the CO_2 that penetrates into the concrete pores, so the adoption of curing procedures and coatings are recommended.

Keywords: concrete; LC^3-50; metakaolin; supplementary cimenticious materials; clinker.

Resumo

Devido ao processo de clinquerização durante a produção do cimento Portland, grandes quantidades de CO_2 são emitidas, aumentando os efeitos relacionados às mudanças climáticas (aproximadamente 5-10% das emissões globais de CO_2 são provenientes da produção de cimento), consequentemente, a busca por alternativas para mitigar esses altas emissões são necessárias. O uso de materiais cimentícios suplementares (SCM) para substituição parcial do clínquer/cimento Portland tem sido objeto de diversas pesquisas, incluindo o uso de cimentos LC^3 (Limestone Calcined Clay Cements), onde até 50% do clínquer Portland pode ser substituído, entretanto, a indústria cimenteira já utilizou outros materiais cimentícios complementares com atividades pozolânicas em cimentos comerciais. Nesse sentido, este trabalho avalia o desempenho de concretos contendo misturas LC^3 com a presença de diferentes SCM (sílica ativa, cinza volante, cinza de bagaço de cana e cinza de caroço de açaí) quanto a questões de durabilidade por carbonatação. Os resultados mostraram que todos os concretos com LC^3 apresentaram frentes de carbonatação maiores em relação ao concreto de referência, com cimento Portland, devido à menor disponibilidade de cálcio para reagir com o CO_2 que penetra nos poros do concreto, por isso a adoção de procedimentos de cura e revestimentos são recomendados.

Palavras-chave: concreto; LC^3-50; metacaolim; material cimentício suplementar; clínquer.

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Introduction

In recent years, several researches have been carried out seeking for alternatives to reduce the impacts related to climate change and global warming issues, being carbon dioxide (CO_2) one of the main gases responsible for the greenhouse effect. In this context, the Paris Agreement signed by 195 nations and European Union aims at limiting the rise on temperature up to 1.5 ºC in relation to pre-industrial levels. At this point, the clinkerization process in the cement industries is responsible for approximately 5% to 10% of global CO_2 emissions, where in general, for each ton of cement produced, 850kg to 1,100kg of CO_2 are emitted. Also from this perspective, it is estimated that cement consumption will increase, not only for new infrastructure, but also due to the need for conservation and rehabilitation of existing structures, which improves the problem related to the emission of CO_2 from cement production that contribute to environmental impacts (ANTONI *et al.*, 2012; BERRIEL *et al.*, 2016; BISHNOI, 2020; KRISHNAN; YU *et al.*, 2021; MEHTA; MONTEIRO, 2006; NAIR *et al.*, 2020; SCRIVENER *et al.*, 2018).

One of the alternatives to mitigate the CO_2 emission is related to the use of supplementary cementitious materials (SCM) to partial replace Portland clinker in cement. Nair *et al.* (2020) pointed a variation between 0.64kg to 0.81kg CO_2/kg of cement when SCM are used. In this sense, blast furnace slag and fly ash, which are residues from the steel and energy industries are the main SCM used (AVET *et al.*, 2016; DU; PANG, 2020; SCRIVENER *et al.*, 2019; ZHANG *et al.*, 2020).

Blast furnace slag has latent hydraulic properties that is, present in an alkaline environment with water, the hydration reactions leads to the formation of hydration products, analogous to that observed in the hydration of Portland clinker providing strength and durability to concrete, whereas fly ash has pozzolanic properties, leading to the formation of C-S-H from reactions with portlandite, $Ca(OH)_2$ or also represented by CH (MEHTA; MONTEIRO, 2006; NEVILLE, 2012). At this point, although the effects of both blast furnace slag and fly ash are recognized in the literature, the availability of these materials to meet the rising demand for Portland cement is insufficient globally, since all blast furnace slag and fly ash with pozzolanic properties are already used by cement industries (DU; PANG, 2020; ; NAIR *et al.*, 2020; SCRIVENER *et al.*, 2018; SCRIVENER *et al.*, 2019).

In this context, the search for alternatives to the current cement production has motivated researches regarding to the combined use of metakaolin and limestone in partial replacement of Portland clinker, where 50% replacements of Portland clinker are reported in ternary mixtures of metakaolim and limestone known as Limestone Calcined Clay Cements (LC^3-50).

Basically, LC^3-50 cements are composed of 50% Portland Clinker, 30% Metakaolin, 15% Limestone and 5% Gypsum, allowing to significantly reduce the amount of Portland Clinker present in the cement (ANTONI *et al.*, 2012; AVET *et al.*, 2016; BERRIEL *et al.*, 2016; DU; PANG, 2020; KRISHNAN; BISHNOI, 2020; NAIR *et al.*, 2020; SCRIVENER *et al.*, 2018; SCRIVENER *et al.*, 2019; YU *et al.*, 2021; ZHANG *et al.*, 2020). About this issue, the literature points out that clay calcination temperature is between 650 to 800 °C (half the temperature to produce Portland clinker) and do not involve the limestone decarbonation, mitigating CO_2 emissions in the cement industry (KRISHNAN; BISHNOI, 2020).

In fact, Berriel *et al.* (2016) point out that even applying wet production processes for the LC^3-50, the performance against global warming surpasses the most modern cement production processes today. Almenares *et al.* (2017) analyzed variations in the production of calcined clays using the infrastructure of cement industries, looking for the production of LC^3. The authors were successful in demonstrating the feasibility of producing calcined clay with properties suitable for the production of LC^3 using the infrastructure of existing cement industries. Krishnan and Bishnoi (2020) point out that clays with kaolinite content from 27% are already suitable for the production of LC^3. Scrivener *et al.* (2019) emphasizes that clays with kaolinite content between 40% and 75% lead to greater gains in mechanical strength, consequently, it is not necessary to use calcined clay with kaolinitic content above this range. Avet *et al.* (2016) presented a study where, through thermogravimetry assays, the pozzolanicity of calcined clay can be evaluated through the loss of water between 110 and 400 ºC.

In LC^3-50 mixtures, the aluminate phase present in the calcined clayconsumes Portlandite (CH) forming hemicarbonate (metastable phase) and monocarboaluminates (final stable phase) that control limestone reaction. From this mechanism a refinement of the cement matrix, enhancing the mechanical strength and durability of concrete, is obtained (AVET *et al.*, 2016; YU *et al.*, 2021).

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At this point, Rodriguez and Tobon (2020) point out that clinker hydration process present in the LC³ has an influence on the compressive strength at all ages, while the calcined clay/limestone ratio has influence only 72 hours after the beginning of hydration.

In this regard, although researches with LC³ have advanced, there are still lines of research that need contributions, such as the presence, iteration and feasibility of other SCM such as silica fume, fly ash, sugarcane bagasse ash and açaí stone ash in mixtures containing metakaolin and limestone. At this point, not only the mechanical performance, but also issues related to durability must be evaluated, since it reduces not only the costs related to maintenance and rehabilitation, but also reduces the consumption of new raw materials for the execution of these works. In this sense, concrete carbonation is an important parameter in order to evaluate the service life and durability of reinforced concrete structures by the perspective of reinforcement corrosion risk.

Reinforcement corrosion is an electrochemical process and one of the main problems associated with the degradation of field reinforced concrete structures, whether in an urban or marine environment, reducing the bearing capacity of the structures and, in extreme cases, lead to collapse, due to a decrease in mechanical properties of reinforcement as a function of the increase in the corrosion degree. In this sense, chlorides (marine environment) and/or CO_2 (urban environment) penetrate through concrete pores and, when achieve reinforcement create conditions for the beginning of the bars corrosive process after the destruction of the passivating layer that protects reinforcements in concrete (APOSTOLOPOULOS; DEMIS; PAPADAKIS, 2013; BALESTRA *et al.*, 2016; COSTA JUNIOR; PINHEIRO, 2019, KHAN; FRANÇOIS; CASTEL, 2014; MURAKAMI *et al.*, 2021; REHMAN; AL-HADHRAMI, 2014).

Electrical resistivity is a material property that describes the it's ability to handle with the charge flow through the gradient between apparent voltage and current passing, both multiplied by a constant that describes the material's characteristics (CHEN; CHANG; YEIH, 2014; SENGUL 2014; YU; LIU; CHEN, 2017). Considering that reinforcement corrosion is an electrochemical process, electrical resistivity is a non-destructive technique that allows continuous monitoring of real concrete structures in order to evaluate the probable reinforcement corrosion risk. Hornbostel, Larsen and Geiker (2013) presented an extensive literature review regarding the relationship between electrical resistivity and corrosion risk.

Sengul (2014) describes electrical resistivity as an indicator of the durability of concrete structures. Bem, Lima and Medeiros-Junior (2018) describe parameters relevant to concrete characteristics, aggregates, cement consumption, porosity, presence of SCM, and their effects on electrical resistivity. Medeiros-Junior and Lima (2016), Hou *et al.* (2017). Balestra *et al.* (2019) correlated the electrical resistivity of field reinforced concrete structures with the penetration of chlorides, potentially causing reinforcement corrosion.

Carbonation deals with the concrete pH reduction by the CO_2 action that ends up destroying the passivating film that protects the reinforcements inside the concrete, creating conditions to start the corrosive process. At this point, some of the main lines of research presented in the literature range from concrete dosage and use of SCM (BUCHER, *et al.*, 2017; SHI *et al.*, 2016; SILVA *et al.*, 2015) to numerical modeling of CO_2 penetration (PAUL *et al.*, 2018). Regarding the analysis of field concrete structures, Han, Park and Yang (2013) analyzed the carbonation front of 20-year-old coastal structures, with carbonation fronts between 3 and 15 mm being observed, whereas Talukdar and Banthia (2013) analyzed the carbonation front of buildings in different cities around the world in the 2000s, with carbonation fronts between 3 and 13 mm.

Although reinforcement corrosion is a well-documented issue in the literature, there is a gap regarding the analysis of durability considering the use of concrete with lower carbon emission cements, such as LC³-50, combined with other SCM that is an open topic in the literature. Thus, the present work fits into this perspective, seeking to analyze how the presence of SCM - Silica fume, fly ash, sugarcane bagasse ash and açaí ash - can affect both the electrical resistivity and the carbonation front of concretes containing LC³ in order to evaluate the reinforcement corrosion risk as described in next chapters.

Materials and methods

The analysis of different types of SCM, active silica, fly ash, sugarcane bagasse ash and açaí stone ash, in the electrical resistivity and carbonation front of a concrete using LC³ was performed in this work. Therefore, the reference mix was 1: 2.35: 2.85: 0.63, cement: fine aggregate: coarse aggregate: water, with slump 100 ± 20 mm with approximately 350 kg/m³ cement content. Aggregates available in the laboratory of materials of Federal University of Technology – Toledo were used in this research. The properties of the aggregates are shown in Table 1.

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Given the difficulty in obtaining clinker to perform the tests, a Portland cement CP V ARI - Analogous to ASTM TYPE III or CEM I - was used in this study, since its composition is closest of the clinker and gypsum values found in the LC^3-50. The use of Portland cement basically composed of clinker and gypsum was also performed in different works presented in the literature (DU; PANG, 2020; YU *et al.*, 2021; ZHANG *et al.*, 2020).

Table 1 – Coarse and fine aggregates characteristics.

Identification Specific mass (kg/m^3)	Maximum characteristic dimension (mm)	Fineness modulus	
Coarse aggregate	2680	9.5	-
Fine aggregate	2650	0.60	1.95

Source: The authors.

In addition, it should be noted that a reference concrete was made without superplasticizer, however, to achieve a slump greater than 80 mm in LC^3 mixtures, a dosage equal to 3.5% of policarboxylate ether-based superplasticizer (PCE) in relation to the binder mass was required due to the high specific surface of the materials as fine aggregate, limestone, cement and calcined clay. The literature (FERREIRO; HERFORT; DAMTOFT, 2017; NAIR *et al.*, 2020; ZARIBAF; UZAL; KURTIS, 2015) had already pointed out the need to use superplasticizers in LC^3 mixtures.

Finally, due to the presence of metakaolin and limestone in the mixtures with LC^3, the SCM content was standardized at 10% making up the percentage relationships shown in Table 2. In this sense, the nomenclature used LC^3-60, was adopted since it was assumed an approximately 60% replacement of clinker by metakaolin, limestone and SCM. The chemical characteristics of the materials determined by Energy Dispersive Spectroscopy (EDS) are shown in Table 3.

Using a concrete mixer, the concretes were done. For this purpose, all coarse aggregate, 50% of the water and the Portland cement (CP V ARI or LC^3 with or without the SCM, according to the percentages shown in Table 1) were mixed for one minute. Afterwards, fine aggregate and the rest of the water (containing the PCE additive in the case of LC^3) were added and the mixture continued for five minutes.

After this mixing procedure, 10×20 cm cylindrical specimens were molded and cured under saturated conditions at 23 ± 2 ºC for 28 days. After this curing period, compression tests were performed, and the results of each mixture are shown in Table 2.

The lower compressive strength of LC^3 mixtures in comparison of reference concrete can be justified due to low clinker content as expected. In addition, six specimens of each mixture were conducted for accelerated carbonation assay, Figure 1. For the carbonation, the maximum CO_2 penetration occurs when the pore saturation degree is close to 50% (CEB, 1992). In this way, to reach this saturation the specimens were placed in an oven at 80 ºC, with the mass being determined at intervals of 60 minutes up to reach the saturation degree of 50%.

Figure 1 – Accelerated carbonation assay.

Source: The authors.

In addition, a previous group of two specimens of each mixture was analyzed in order to previously verify their concrete alkalinity. In this case, specimens, one of each group identified in Table 2, with a saturation degree of 50% were not subjected to accelerated carbonation assay, being partially fractured through the splitting test and, immediately after the fracture, subjected to spraying of a phenolphthalein solution as recommended by RILEM CPC 18 (RILEM, 1988). The alkalinity was verified through the intense color observed immediately after spraying the solution, as exemplified in Figure 2. All concretes, Reference, LC^3-50 and LC^3-60, shown high alkalinity environment for reinforcement protection.

After verifying the concrete alkalinity, specimens were placed inside the carbonation chamber and subjected to a constant pressure of 20 Psi for 48 hours to promote the CO_2 penetration. For this procedure, first, after place the specimens and close the chamber, all air inside the chamber was removed using a pump vacuum for 20 minutes, being the CO_2 injected into the chamber in the sequence. After 48h, the specimens were removed from the chamber and submitted to splitting test for fracture along its length, where, a phenolphthalein solution was immediately sprayed on the newly fractured surface.

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Table 2 – Group's identification and perceptual of SCM.

Identification	(%)			SCM (%)				Concrete compressive strength (MPa)
	Portland cement	Calcined clay	Limestone	Silica fume	Fly ash	Sugarcane bagasse ash	Açaí stone ash	
Reference	100	-	-	-	-	-	-	32.11 ± 2.26
LC³-50	55	30	15	-	-	-	-	28.60 ± 1.06
LC³-60 S	45	30	15	10	-	-	-	21.55 ± 2.48
LC³-60 FA	45	30	15	-	10	-	-	22.43 ± 1.66
LC³-60 SBA	45	30	15	-	-	10	-	21.83 ± 0.98
LC³-60 ASA	45	30	15	-	-	-	10	18.34 ± 1.77

Source: The authors.

Table 3 – Main chemical elements of the cement and SCM.

Material	Si	Ca	C	S	Mg	K	Na	Al	Fe
Cement	8.58	25.35	5.45	1.83	3.55	1.39	0.15	3.23	1.42
Calcined clay	9.65	-	15.24	-	1.15	0.35	-	33.53	1.82
Limestone	1.25	24.11	14.85	-	2.19	0.26	-	-	0.19
Silica fume	44.58	-	-	-	0.64	0.63	-	-	-
Fly ash	18.87	0.85	20.57	0.71	1.58	1.90	0.57	-	1.40
Sugarcane bagasse ash	11.34	1.83	33.01	-	2.04	2.67	-	-	7.51
Açaí stone ash	2.07	2.08	50.46	3.30	2.35	9.43	-	-	0.13

Source: The authors.

Figure 2 – Concrete alkalinity verification: (a) Reference; (b) LC³-50 and (c) LC³-60 S.
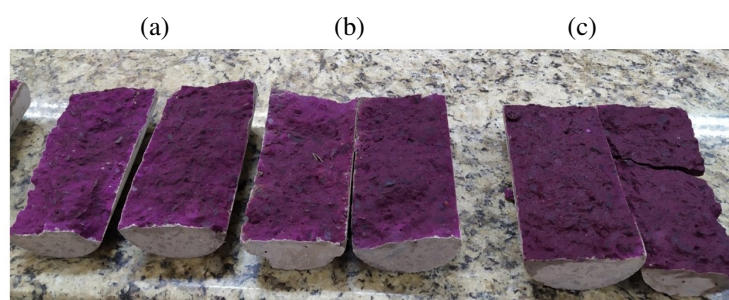
Source: The authors.

Using a metal ruler, the carbonation front of the concrete was determined, where the colourless front was measured from the specimens' surface.

Micrographs and calorimetry assays were performed in order to support the results. In this sense, cubic one mm side samples (For micrographs) and powder samples (For calorimetry assays) from uncarbonated concretes were obtained. In this sense, the cubic samples were obtained cutting the concretes, whereas, powder samples were obtained with rotary drill. The powder samples were passed in a sieve (#0.075 mm) and were analyzed in a Toledo Mettler Calorimetry from 30 °C to 900 °C with range of 10 °C/min in nitrogen atmosphere.

Results and discussion

Figure 3 shows the specimens after spraying the phenolphthalein solution for carbonation evaluation. It should be pointed in Figure 3 that when the pink color was not observed in some LC³-60 mixtures a fractured concrete with Ordinary Portland Cement (OPC) was sprayed with the phenolphthalein solution only for color comparison.

Carbonation fronts were in all cases, much higher than the carbonation fronts observed in the reference concrete. In this case, all LC³-50 and LC³-60 concretes were carbonated (pH < 9) where the colorless was observed after spraying the phenolphthalein solution, demonstrating a much lower performance compared to the reference concrete.

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Figure 3 – Concrete samples after carbonation tests Concrete samples after carbonation tests: (a) Reference; (b) LC^3-50; (c) LC^3-60 S; (d) LC^3-60 F; (e) LC^3-60 SC and (f) LC^3-60 A.

(a)   (b)   (c)

(d)   (e)   (f)
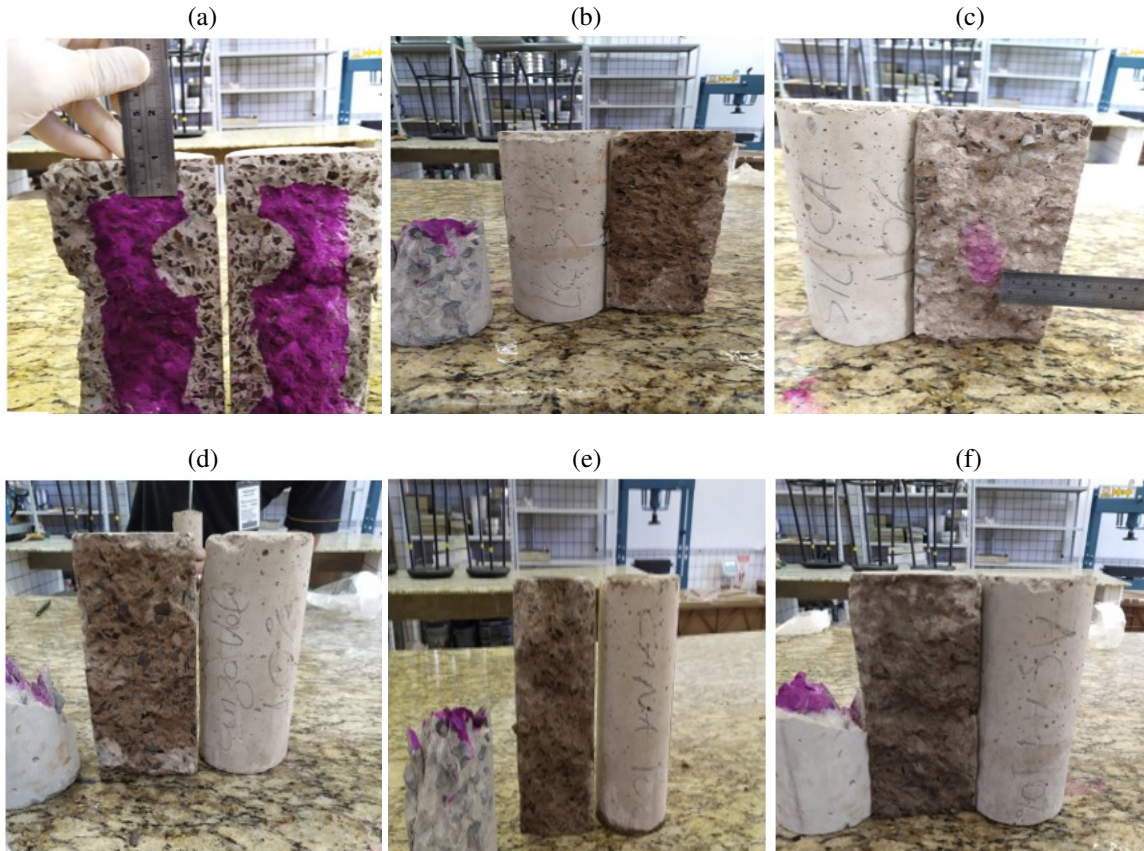
Source: The authors.

In order to investigate this behavior Portlandite $(Ca(OH)_2)$ presence in concretes with LC^3 must be. Other studies had already pointed out higher carbonation fronts in concrete using LC^3 (SCRIVENER *et al.*, 2018; SCRIVENER *et al.*, 2019).
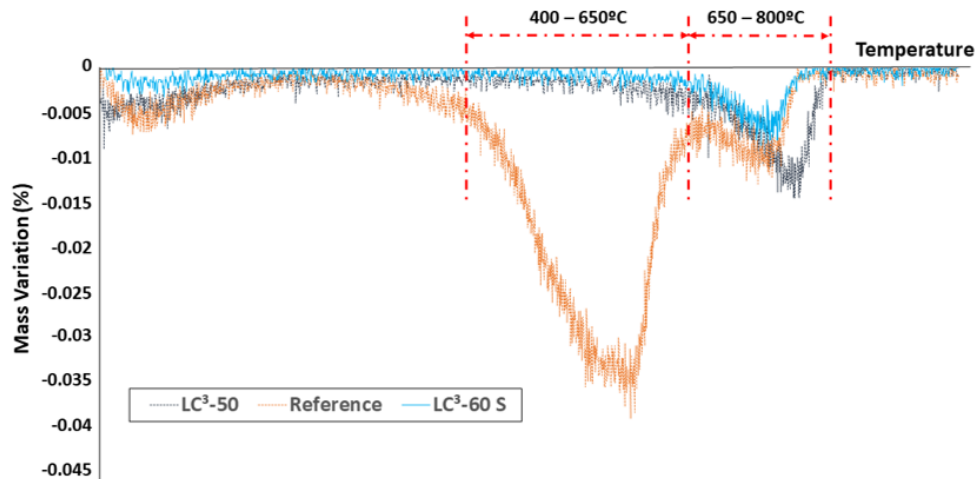
In this work, LC^3 systems have a lower ability to combine with CO_2 due to the low availability of calcium free to react, since calcium is consumed by the pozzolanic reactions with metakaolin and SCM, consequently, greater carbonation fronts are observed values, the presence of $Ca(OH)_2$ is presented in Figure 4 and is analyzed below.

This observation extends to the LC^3-60 concretes in this work. In this case, although the presence of SCM leads to a refinement of the microstructure, these materials increase the consumption of CH by pozzolanic reactions, further reducing the amount of calcium available to capture the CO_2 that penetrates into the concrete. In this sense, analyzing the different SCM, it is possible to notice in Figure 3 that only silica presented a lower carbonation front in relation to the other mixtures, however, still insufficient to provide protection to the reinforce-

ment, since not only the pozzolanic reaction, but the filler effect of silica fume contributes to reduce the concrete permeability.

In order to confirm the affirmative below, calorimetry analyzes were performed in samples from the specimens uncarbonated. The calorimetry results, Figure 4, shown the absence of the peak related to Portlandite in LC^3 mixtures, between 400 and 650 ºC, confirming that the availability of calcium to react with CO_2 that penetrates through concrete pores is limited in LC^3 systems, justifying the carbonation fronts observed, corroborating with literature (DHANDAPANI; SANTHANAM, 2017; SCRIVENER *et al.*, 2018; SCRIVENER *et al.*, 2019).

Finally, it is possible to affirm that concretes containing LC^3 present high carbonation fronts due to the limited availability of calcium to iterate with the penetrating CO_2, consequently, the adoption of curing procedure, 23 ± 2 ºC under saturated conditions for 28 days, and coating layers are recommended to mitigate CO_2 penetration in order to provide durability to field concrete structures.

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Figure 4 – Calorimetry results of Reference (Orange), LC³-50 (Gray) and LC³-60 S (Blue) concretes.

Conclusions

This work analyzed the effects of different supplementary cementitious materials (SCM - silica fume, fly ash, sugarcane bagasse ash and açaí core ash) on concrete carbonation using ternary mixtures containing metakaolin and limestone replacing Portland cement, LC³. The main conclusions are:

- All analyzed concretes (reference, LC³-50 and LC³-60 with different SCM) showed high alkalinity when not exposed to accelerated carbonation tests. However the consumption of all portlandite was an important indicative of low protection against reinforcement corrosion justifying the high carbonation fronts verified, independent of the SCM type present in the LC³ mixture. Consequently, in urban aggressive environments the performance of LC³ concretes may be lower compared to Portland cement concretes.

- Comparing the performance of different SCM in LC³-60, the mixture containing silica fume presented littler improvement compared to others SCM, but still not able to protect the reinforcements.

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