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Influence of crystallizing type chemical admixture on precast micro concretes: a statistical analysis and holistic engineering overview

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ABSTRACT: The aim of this paper is to evaluate the influence of a crystallizing chemical admixture on precast micro concretes with two water contents (7% and 11%, by dried mass) and two different conditions of exposure. Thus, precast micro concretes with a composition of 1:3 (cement: fines) with and without crystalline chemical admixture were evaluated on compressive strength (at the age of 28 and 154 days) and water absorption by immersion (at the age of 154 days). Statistical analysis showed that the only significant factor was the effect of the water content on the compressive strength. Besides that, the most significant factors for the water absorption and voids index properties were the water content, followed by the exposure conditions, and the interaction between the water content and the presence of the chemical admixture. The crystalline admixture was insignificant in the conditions of this research.

KEY WORDS: Concrete; Crystallizer; Chemical admixture; Precast concrete; Micro concrete.

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RESUMEN: *Influencia de un aditivo químico tipo cristalizador en el microhormigón prefabricado: un análisis estadístico y una visión holística de la ingeniería.* El objetivo del trabajo es evaluar la influencia de un aditivo químico cristalizador sobre micro hormigones prefabricados con dos contenidos de agua (7% y 11%, en masa seca) y dos condiciones de exposición diferentes. Así, microhormigones prefabricados con una composición 1:3 (cemento: finos) con y sin aditivo químico cristalino fueron evaluados, analizando su resistencia a compresión (a la edad de 28 y 154 días) y la absorción de agua por inmersión (a la edad de 154 días). El análisis estadístico mostró que el único factor significativo fue el efecto del contenido de agua sobre la resistencia a la compresión. Además de eso, los factores más significativos para las propiedades de absorción de agua e índice de vacíos fueron el contenido de agua, seguido de las condiciones de exposición y la interacción entre el contenido de agua y la presencia de la mezcla química. La mezcla cristalina fue insignificante en las condiciones de esta investigación.

PALABRAS CLAVE: Hormigón; Aditivo cristalizador; Aditivo químico; Hormigón prefabricado; Microhormigón.

1. INTRODUCTION

Concrete structures are exposed to several durability problems associated with water content penetration through their pores and capillary networks (1) and the consequent reduction of their service life. In general, in order to prevent this from happening, solutions have been developed for existing and new concrete structures.

For existing concrete structures, researchers have developed solutions such as different products, chemical bases and commercial systems to apply on concrete surfaces as waterproofing barrier systems. These products and systems include emulsions, acrylic membranes, asphalt blankets, polymeric mortars (2), liquid-applied films or sheets made of acrylic resins, bitumen, styrene-butadiene-styrene copolymer, ethylene propylene diene monomer (EPDM), acrylics, polyethylene; silane, siloxane (3) and silicone (4) and, more recently, crystallizing hydrophobic minerals (5), dual-crystallization (DCE) (6, 1) and multi-crystallization technology (7, 8) with hygroscopic and hydrophilic characteristics. On the other hand, in new concrete structures, solutions involve a concrete mix design using supplementary cementitious materials such as metakaolin, silica fume, nano-silica, fly ash (9, 10), nano metakaolin (11), rice husk ash and waste glass (12) or super absorbent polymers (SAPs) (13, 14). Also, they could involve the use of chemical admixtures (CAs) such as shrinkage-reducing admixtures (15, 16) and crystallizing or crystalline chemical admixtures (17), which can also be useful to prevent moisture damage and to decrease the permeability and the capillary absorption of hardened concrete (18).

In particular, the crystallizing or crystalline chemical admixtures, also called crystalline catalyst in the literature (19), can be classified as PRAH (permeability-reducing admixture in hydrostatic conditions), admixtures that increase the resistance to the penetration of water under pressure (20). These crystalline admixtures have a hydrophilic nature, and their active components react with calcium hydroxide to form crystalline products that fill cracks and disconnect pores in the concrete only in the presence of sufficient moisture (21). As a result, crystalline products can be formed, depending on the size of the pores and the pH concentration of the pore solution (19, 22), resulting in a reduction of water content penetration through the pores.

Furthermore, while the crystalline admixture, consisting of Portland cement, quartz sand and active chemicals, has its compositions kept confidential by manufacturers (22, 23), it can be defined generically as a composition similar to cement (24) with a greater number of finer particles (25). This type of admixture has been studied in concrete, including self-healing or autogenous crystallization of concrete, with good results (26-28).

In general, the main results obtained by some of these researches were that the crystalline admixture reduced the concrete permeability to water (23), especially with a more evident effect for a higher w/c ratio and depending on the type of binder (25). Also, it observed that it was a tendency to decrease the capillarity absorption (29), the total absorption of water (by immersion) and the void index at each wetting and drying cycle (30). At the same time, the formation of needle-shaped crystalline compounds (24), an increase in resistance to chloride penetration (31) and a self-healing ability (32-36) were noted. However, some research has also shown that the use of the crystalline admixture did not have great effects. For example, for high-performance concrete (HPC) with fibers, the addition of this admixture did not significantly change the mechanical properties and water permeability when subjected to constant loading (37). In this HPC, the main product found in the cracks was aragonite, a crystalline form of calcium carbonate, probably formed due to the presence of magnesium in the admixture.

Another study (35) tested four different types of exposure (water immersion, water contact, a humidity chamber, and air exposure in laboratory conditions) for the self-healing of concretes with and without crystalline admixture for 42 days. It was concluded that, among the types of exposure tested, water immersion was the best condition to promote self-healing and that the presence of water was essential for the healing phenomenon in concrete with and without crystalline admixtures.

In some circumstances, the self-healing ability of the concretes cannot be confirmed (37). In this case, it was found that under air curing, the admixture did not make any difference and under wet curing, there was a small, but insignificant improvement, confirming the need for water for its actuation. On the other hand, another study (38) demonstrated a good self-healing effect of the crystalline admixture after intentional cracking and 7 weeks of water flow under constant pressure (1.5 MPa). As a result, the water percolation reduced significantly (99%) and stabilized at a low value. Thus, there is still not a full consensus on the beneficial effects of crystallizing admixtures in concrete, or related to the effect of using this type of admixture in concrete with a low water binder ratio. However, concrete with a low water/binder ratio, usually molded by compaction, vibration or compression, is widely used in Brazil for the manufacturing of precast concrete, such as blocks, pipes, and columns, among others. This type of concrete can demonstrate good cohesion, low porosity and low permeability (39).

Due to the need for water to act on the crystallization admixture, its performance in concrete with a lower water/binder ratio can be decreased in the early ages. However, with the supply of external water, after the cement has hydrated and reached its maturity, there is the possibility that this admixture will reduce the permeability in precast concrete for which the maximum value of water absorption is 8% (40). This paper moves forward in this direction and, in this perspective, addresses a knowledge gap by investigating the effect of the crystalline admixture in precast micro concrete (concrete with small aggregates). Thus, the aim is to compare the performance of precast micro concrete, in terms of its compression strength and water absorption, analyzing the water contents (7% and 11% of water over the total dried mass) and the crystalline admixture in two types of environmental conditions. A statistical analysis was conducted for better analysis of the results.

The connection between the study conditions and differences corresponding to the microstructural changes induced in the concretes (including changes in pore systems and their properties) will be produced in terms of technical, technological and engineering arguments on the different behaviors and performances of the studied micro concrete and their predicted durability. Besides that, general and substantial changes in the studied micro concretes are discussed, in relation to both the microstructure and the pore system, as well as in relation to mechanical changes, based on statistical analysis and an engineering context. The effect of the chemical admixture is intimately associated with the mechanical, micro structure and porosity (fundamental aspects of durability), and this part constitutes the main novelty of this paper.

2. MATERIALS AND METHODS

Precast micro concrete was produced with unitary binder: aggregates proportions of 1:3.0, by mass. The binder: filler: micro filler: artificial sand proportions were 1:0.24:0.16:2.6, respectively. This mix composition was adopted and recommended by a local company that manufactures precast concrete.

The crystalline chemical admixture was used in a content of 2% in relation to the cement mass (as the maximum content indicated by the manufacturer) as a partial replacement for the micro filler. Besides that, the water contents (percentages of water over the total dry mass) were determined according to the literature (41), in which the optimum water content was 9% (determined in a compaction test with normal energy). Thus, the values adopted in this research were 11% and 7% over the total dry mass of mixture, corresponding to the water: cement ratio of 0.44 and 0.28, respectively. These water content values allowed the handling of the precast concrete after molding without damage and with good cohesion.

2.1. Materials used

Brazilian Portland cement type CP II F-40 was used (composed of 11–25% in mass of limestone filler and 75–89% of clinker, with a small amount of calcium sulfate), whose mechanical strength requirement is a minimum of 40 MPa of compressive strength at the age of 28 days (similar to the European CEM II/A-L), together with micro filler, filler, fine aggregate, crystalline chemical admixture and water. The chemical and physical properties of the cement used are given in Table 1.

The filler and micro filler used were waste from crushed granitic rocks. Figure 1 shows the granulometric distribution curves of these two materials. D50 particles of 26 µm and 62 µm were observed for the micro filler and filler, respectively. The granite sand was used as fine aggregate (Table 2 and Figure 2). It was observed that the sand used was almost entirely within the “usable zone”, according to Brazilian Standard NBR 7211: 2009 (63). The crystalline admixture utilized was a crystallizing powder admixture. This was a gray powder with a pH value of 12 ± 1.0, equivalent sodium oxide less than or equal to 3%, and a volume density of approximately 750 kg/m^3. The recommended content is between 1% and 2% of the weight of the binder. Besides that, this crystalline chemical admixture is usually composed of a mixture of cements, amino alcohols, and other binding materials (64).

2.2. Preparation of samples for tests

To perform the tests, it was decided to mold cylindrical samples, adapting the method proposed by NBR 7182: 2016 (65) for soil compaction testing. The cylinder used in molding was a small cylinder (Proctor) 10 cm in diameter and 12.7 cm in height, with a volume of 997.5 cm^3.

The purpose of this adaptation was to simulate press molding, because the press was unable to mold the cylindrical samples required for the tests. A previous study (41) showed that the density obtained through molding in the press for the manufacture of modular blocks (the same used to obtain the water content adopted in the present work) approached the density obtained manually, using the cylinders described with normal energy. In this research, the density of each mixture was also measured.

After that, the components of the micro concrete were placed in a receptacle in the following order: sand, filler, micro filler, cement and admixture. They were then pre-mixed manually until well homogenized and placed in a planetary mixer with a capacity of 20 liters. The mixing was carried out by adding water with a mixer rotation of 125 rpm for 5 minutes.

The molding of the samples was performed in 3 layers with 26 strokes per layer with a proctor socket of 2.5 kg, falling from a height of 30.5 cm, equivalent to normal compaction energy (65). The specimens used in the tests were molded, adapting the proposed method for soil compaction with normal energy. After compacting the three layers, the specimens were

Table 1. Physical characteristics and chemical composition of Brazilian Portland cement type CP II F-40, similar to European CEM II/A-L.

Property		Test method	Equivalent European test	Result	Requirement according to Brazilian Standard NBR 16,697 (42)
Specific mass (kg / dm³)		NBR 16605 (43)	UNE 80103 (44)	3.1	-
Standard paste consistency (%)		NBR 16606 (45)	EN 196-3 (46)	29.1	-
Chemical composition (%)	Loss on Ignition (%)	NBR NM 18 (47)	EN 196-2 (48)	5.0	≤ 12.5
	Insoluble residue	NBR NM 15 (49)	-	1.3	≤ 7.5
	MgO	NBR NM 11-2 (50)	EN 196-2 (48)	2.5	-
	SO_3	NBR NM 16 (51)		2.8	≤ 4.5
Fineness	Fineness index by means of 75 µm sieve (nº 200)	NBR 11579 (52)	EN 196-6 (55)	0.3	≤ 10.0
	Fineness index by means of aerodynamic sieving	NBR 12826 (53)		1.9	-
	Specific surface (m²/kg)	NBR 16372 (54)		4,516.0	-
Setting times (minutes)	Initial	NBR 16607 (56)	EN 196-3 (46)	158.0	≥ 60
	Final			205.0	≤ 600
Compressive strength (MPa)	7 days	NBR 7215 (57)	EN 196-1 (58)	28.0	≥ 25.0
	28 days			40.0	≥ 40.0
	90 days			41.0	-

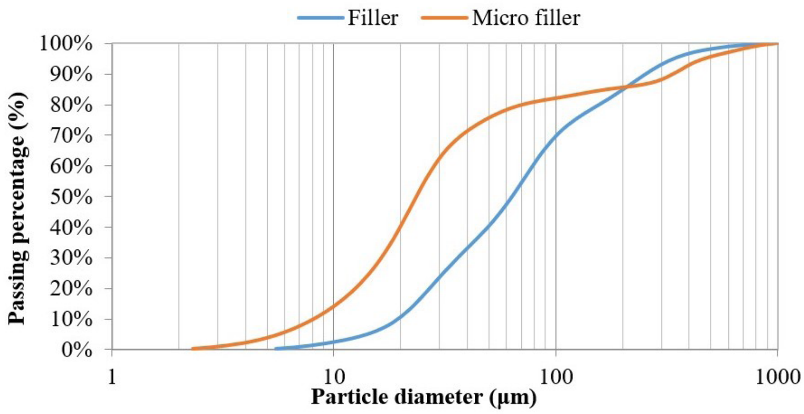

Figure 1. Filler and micro filler particle size distribution curve.

Table 2. Characteristics of the granite sand (fine aggregate) used.

Property	Brazilian Standard	Equivalent European test	Result
Maximum diameter (mm)	NBR NM 248 (59)	EN 933-1 (60)	4.75
Fineness module			3.16
Specific mass (kg / dm³)	NBR NM 52 (61)	BS 812-2 (62)	2.65
Unit mass (kg /dm³)			1.60

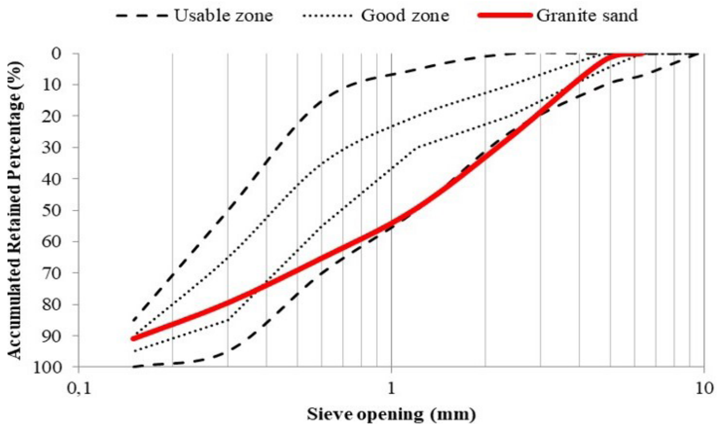

Figure 2. Grain size distribution curve of the granite sand.

leveled, removing excess concrete using a metal ruler. After extraction, the specimens were labeled and wrapped in four layers of plastic film.

2.3. Curing procedures and conditions of exposure

After the demolding, because of technical problems in the moist room at the laboratory, the curing was performed for 12 weeks (84 days) in the following way: all the specimens were labeled and wrapped in four layers of plastic film in the laboratory at 25±2 °C and 60± 5% of relative humidity. After 84 days of ageing, two exposure conditions were conducted: firstly, immersed in lime-saturated water and then kept for an additional 67 days until the age of 151 days, which was named the "saturated" condition. Secondly, they were kept dry (wrapped in four layers of plastic film). This last condition was named "sealed". These procedures were adopted to evaluate the action of the crystalline admixture in the presence of water.

2.4. Test methods

The compressive strength tests and water absorption tests were performed on cylindrical specimens with dimensions of 10 cm x 12.7 cm (diameter x height). The former test was performed according to the Brazilian standard NBR 5739: 2018 (66) at the age of 28 days and 154 days on the sealed specimens and saturated specimens.

The absorption of water and void index in the hardened micro concrete were investigated at the age of 154 days, following Brazilian standard NBR 9778: 2009 (67). Three specimens were tested for each condition of the study.

2.5. Statistical analysis of data

For a good interpretation of the results, the values obtained for the compressive strength, water absorption and voids index were submitted to the analysis of variance (ANOVA) test, using Statistica software version 10.0 from StatSoft®.

In addition, factorial experiments can allow general conclusions, which are more interesting than the isolated analysis of each variable. Dixon tests were therefore performed to analyze outliers and it was possible to observe the influence of the variables: crystalline chemical admixture, water content, exposure conditions (sealed or saturated) and age of the tests, as well as the interaction of these variables.

2.6. Scanning electron microscopy (SEM)

To analyze the microstructure of these different materials, images were taken with a scanning electron microscope Jeol JSM-6610 from LABMIC laboratory (High Resolution Microscopy Multiuser Laboratory). Amplifications from 100x to 5000x were used for comparing the cement matrices, assessing their homogeneity, the presence of fissures, and hydrated products, among other aspects.

The micro concrete samples were reduced in a concrete cutter and manually fractured using tools. They were dried in a laboratory oven until they were ready to be preconditioned. After drying, the fractured surface was coated with gold. These tests were run on samples of more than 1170 days and comparisons between the micro concretes studied were made.

3. RESULTS AND DISCUSSION

3.1. Compression strength

The averages of the results obtained for density and compressive strength are shown in Table 3. Figure 3 presents the average and the standard deviation of compressive strength at the age of 28 days (orange color) and 154 days with sealed (blue color) and saturated (grey color) micro concrete specimens, with (A)

Table 3. Density (g/cm³) and compressive strength (MPa) at the ages of 28 days and 154 days in the two different conditions of exposure (sealed and saturated).

Type of concrete/ water content	Age test	Exposure condition	Density (g/cm³)*	Compressive strength (MPa)*	CV**
Without crystalline admixture - 11% water content (N11)	28	Sealed	2.17	32.7	10
	154	Sealed	2.15	32.0	17
	154	Saturated	2.20	31.6	13
With crystalline admixture - 11% water content (A11)	28	Sealed	2.19	28,9	6
	154	Sealed	2.15	30.0	23
	154	Saturated	2.20	29.8	18
Without crystalline admixture - 7% water content (N7)	28	Sealed	2.04	19.4	13
	154	Sealed	2.01	17.7	10
	154	Saturated	2.14	21.9	7
With crystalline admixture - 7% water content (A7)	28	Sealed	2.05	21.6	10
	154	Sealed	2.05	19.8	6
	154	Saturated	2.15	23.3	4

*Average value. **CV – Coefficient of Variation (%)

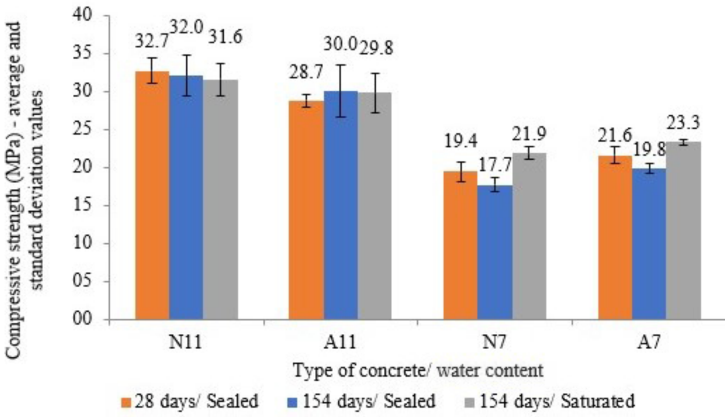

Figure 3. Compressive strength results for the micro concretes studied: average results and standard deviation.

and without (N) crystalline chemical admixtures for 7% and 11% of water content.

From Table 3 and Figure 3, it is observed that the densities of the concretes with 11% water content were considerably higher than those with 7%, as expected. Comparing the densities with and without crystalline admixtures for the same water content, the values were the same or the differences were negligible. This same observation was validated for the densities of the micro concretes at 28 and 154 days with and without crystalline admixture and in the water content studied.

In relation to the compressive strength, the micro concretes with 11% water content had higher values of compression than the micro concretes with 7% water content at all ages and conditions studied (about 33%). Thus, the greater amount of water in the micro concrete with 11% water content caused both greater compaction of the mixtures and greater hydration of the cement. As a result, the compressive strength increased. The effect of the age of these micro concretes is negligible or small.

Analyzing the micro concretes with 11% of water content, there was a tendency to a reduced compressive strength when the crystalline admixture was used. This trend occurred for both ages: 28 days (reduction of 12%) and 154 days, in both conditions of exposure (about 6%). As for the micro concretes with 7% water content, there was an inverse behavior, which meant an increase in compressive strength when the admixture was added (from 6% to 12%, depending on the age and exposure condition). This tendency of behavior with the two water content levels of the micro concretes can be related to the interaction between the filler effect and the influence of the greater surface area of the fines included in the mixtures with crystalline admixture. However, a more accurate analysis can be done through consideration of the statistical results. Table 4 shows the analysis of variance (ANOVA) of the compressive strength results.

Table 4 shows that the model adopted was significant, with a coefficient of determination of the model (R^2) of 0.75. This means that 75% of the total variation of the data is explained by the model. The analysis of variances showed that the only statistically significant individual effect was that of water content at a 95% confidence level. This expresses that this single variable influences the compressive strength of the micro concretes, with the formation of two statistical groups of water contents: one with 11% and other with 7%. The crystalline admixture used did not influence the compressive strength results. Additionally, only the double interaction between the water content and the admixture was significant, with the water content having more influence.

In the literature, there is no agreement relating to the effect of the crystalline admixture on the compressive strength of micro concrete, considering that there was no pre-cracking in self-healing studies. Pazderca and Hájková (23) evaluated the influence of two different crystalline admixtures on the compressive strength at 28 days and concluded that the result was practically the same as for the concrete without admixture. By contrast, in the research developed by Hassani *et al.* (25), the crystalline admixture used significantly influenced the compressive strength at the age of 28 days, with rates of increase of 11% and 21% and a rate of decrease of 24%, depending on the type of cement used. The cement type had a greater influence than the type of admixture studied. The concrete with admixture studied by Reddy and Ravitheja (68) achieved an increase of 11% compared to the reference concrete, which was attributed to the filling of the voids by C-S-H and calcite (one of the morphologies of calcium carbonate). In another study, Al-Rashed and Al-Jabari (8) observed improvements in the compressive strength of different concretes with multi-crystallization, mainly at the ages of 28 and 56 days.

Regarding the effect of exposure at different ages, Weng and Cheng (24) tested different levels of crystalline admixtures (0, 3, 5 and 7%). They observed that the compressive strength of the concretes studied after water exposure was considerably higher than those after air curing exposure at the ages of 7, 28 and 56 days. The increase of crystalline admixture content showed an increase of compressive strength. For example, with 3% of this admixture, there were increases of 14%, 20% and 22% at the ages of 7, 28 and 56 days, respectively, in comparison to those without the admixture.

Table 4. Analysis of variance (ANOVA) of compressive strength results.

Description	SQ	GL	MQ	Fcal	Ftab	p-value	Result
Model	1,375.84	11	125.08	9.660	2.07	0.0000	Significant
Error (residue)	466.2	36	12.95	-	-		-
Total	1,842.04	47	-	-	-		-
Water content	1,199.72	1	1,199.72	92.64	4.11	0.0000	Significant
Crystalline admixture	2.18	1	2.18	0.17	4.11	0.6841	Insignificant
Conditions of exposure (sealed or saturated)	24.66	1	24.66	1.90	4.11	0.1761	Insignificant
Age	0.2	1	0.2	0.015	4.11	0.9020	Insignificant
Crystalline admixture x age	1.93	1	1.93	0.15	4.11	0.7018	Insignificant
Crystalline admixture x conditions of exposure	0.14	1	0.14	0.011	4.11	0.9175	Insignificant
Water content x crystalline admixture	64.28	1	64.28	4.96	4.11	0.0322	Significant
Water content x age	0	1	0	0	4.11	0.9860	Insignificant
Water content x conditions of exposure	34.73	1	34.73	2.68	4.11	0.1102	Insignificant
Water content crystalline admixture x age	3.8	1	3.8	0.29	4.11	0.5913	Insignificant
Water content x conditions of exposure (crystalline admixture x age)	0.47	1	0.47	0.036	4.11	0.8503	Insignificant
Error (residue)	466.2	36	12.95	-	-		-
Coefficient of determination (R^2_{mod}) 0.75							
Correlation coefficient (R_{mod}) 0.86							

3.2. Water absorption and void index

The water absorption and void index results are summarized in Table 5. Dixon's test was performed and no data were excluded. As mentioned before, this test was performed only for the age of 154 days.

Similar to the compressive strength, the effect of the water content of the micro concretes was clear in this test. By increasing the water content from 7% to 11%, the average value of the total water absorption decreased by 56% and the void index by 47%.

Figures 4 and 5 show the average and standard deviation results of the water absorption and void index of all the studied micro concrete mixes.

As concerns the type of exposure conditions, it was observed that the saturated condition promoted a tendency of reducing both the total water absorption and the void index in the mixes studied. However, this trend was greater for N11 (5.4% for both the water absorption and void index). In the A11 micro concrete with the crystalline admixture, these reductions were 4.1% and 4.6%, respectively. For the mixture with 7% water content, the greatest reduction trend was for the mix without admixture (N7), with 3.9% for both indexes. For the micro concrete with admixture (A7), this trend was much lower, with values of 0.9% and 1.5%, respectively.

Related to the effect of the crystalline admixture, for the 11% water content, it was observed that both the water absorption and the void index showed no considerable reduction. However, for the water content of 7%, this admixture caused a tendency to reduce the total water absorption and the void index in both cases (sealed and saturated samples). This behavior was consistent with an increase in compressive strength obtained, although, in that case, such an increase could not be confirmed by the statistical analysis performed.

Table 5. Summary of water absorption (%) and void index (%) average results.

Type of concrete/ water content	Exposure condition	Water absorption (%)	CV* (%)	Void index (%)	CV* (%)
Without crystalline admixture - 11% water content (N11)	Sealed	8.5	3.3	17.6	3.2
	Saturated	8.0	1.2	16.7	0.9
With crystalline admixture - 11% water content (A11)	Sealed	8.4	1.2	17.6	1.0
	Saturated	8.1	0.9	16.8	0.7
Without crystalline admixture - 7% water content (N7)	Sealed	13.1	1.8	26.0	1.4
	Saturated	12.6	1.7	25.0	1.3
With crystalline admixture - 7% water content (A7)	Sealed	12.4	2.1	25.0	1.3
	Saturated	12.3	1.3	24.6	1.0

*CV - Coefficient of Variation

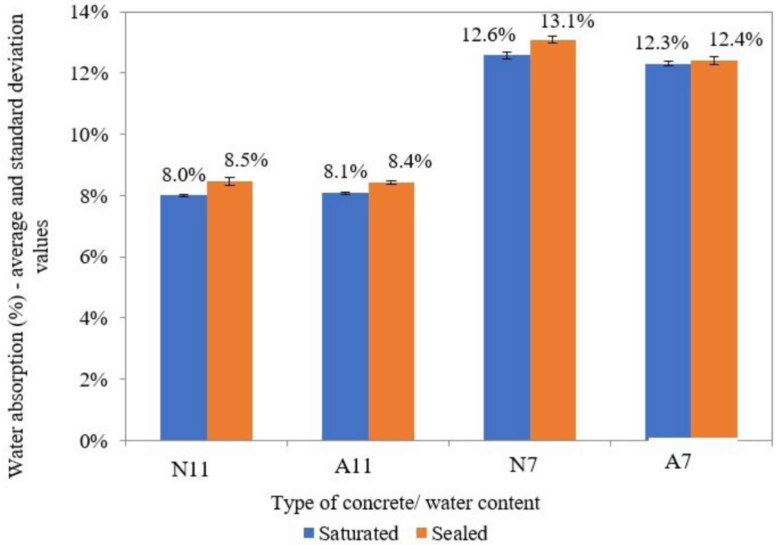

Figure 4. Water absorption (%) – average and standard deviation values of the micro concretes studied.

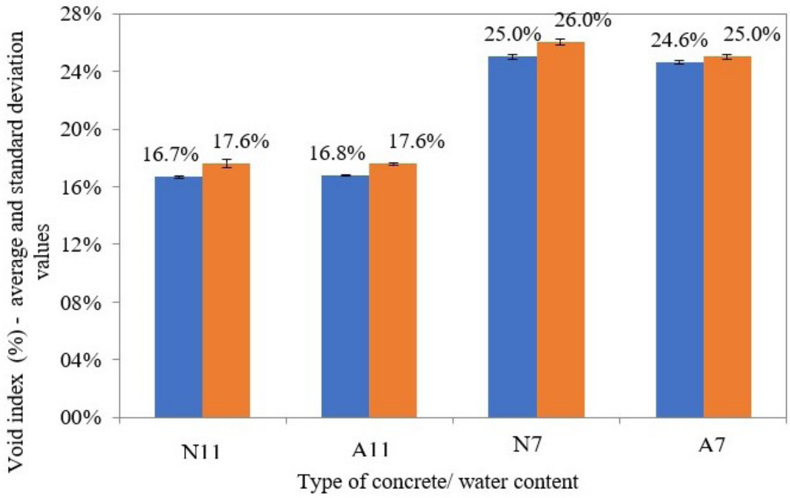

Figure 5. Void index (%) – average and standard deviation values of the micro concretes studied.

Tables 6 and 7 present the ANOVA results for the absorption and void index, which were analyzed together because they were obtained from same test.

The ANOVA results evaluated the influence of three independent variables on the absorption and void index: water content, crystalline admixture and exposure conditions. All three factors were significant, and the interaction between the water content and crystalline admixture was equal in the compressive strength. When comparing the Fcal values, considering that Ftab has the same value, it was observed that the most significant variable, much more than the others, was the water content, followed by the exposure conditions, the interaction between the water content and crystalline admixture, and finally the crystalline admixture.

By the grouping of means, using the Duncan test, the mixes were divided into four distinct groups, shown in Table 8. Across the groups, the water content and conditions of exposure were more predominant than the other variables analyzed. The crystalline admixture, although significant, did not interfere much in the division of groups (see Table 8).

Different from the results obtained in this research, a study by Petrucci and Hastenpflug (30) indicated that 0.8% content of crystalline admixture reduced the total absorption (by immersion) and the void index at each wetting and drying cycle (four in total), from a

Table 6. ANOVA water absorption results of micro concretes.

Description	SQ	GL	MQ	Fcal	Ftab	p-value	Result
Model	0.011526	7	0.0016	445.06	2.66	0.0000	Significant
Error (residue)	0.000059	16	0.000004				-
Total	0.011585	23					
Water content	0.01137	1	0.01137	3,073.13	4.49	0.0000	Significant
Crystalline admixture	0.000029	1	0.000029	7.93	4.49	0.0124	Significant
Conditions of exposure (sealed or saturated)	0.000075	1	0.000075	20.38	4.49	0.0003	Significant
Water content x crystalline admixture	0.000038	1	0.000038	10.31	4.49	0.0055	Significant
Water content x conditions of exposure	0.000001	1	0.000001	0.33	4.49	0.5727	Insignificant
Crystalline admixture x exposure conditions	0.000009	1	0.000009	2.55	4.49	0.1300	Insignificant
Water content x crystalline admixture x exposure conditions	0.000003	1	0.000003	0.80	4.49	0.3844	Insignificant
Error (residue)	0.000059	16	0.000004				
Coefficient of determination (R^2_{mod}) 1.00 and Correlation coefficient (R_{mod}) 1.00							

3.1% water absorption rate to 0.3% for conventional concrete. In comparison with the reference concrete (without admixture), the reduction was 90.5% for the water absorption rate and 90.2% for the void index.

In the research developed by Hassani *et al.* (25), also with conventional concrete, the crystalline admixture used did not influence significantly the void ratio (ANOVA results), calculated by the ASTM C642-13 standard, presenting a maximum reduction of 10% for a given type of concrete.

3.3. SEM analyses

To connect the results obtained with the microstructure of the micro concretes evaluated, the SEM images are shown in Figures 6 and 7. It was thus possible to observe the influence of the water content and the type of exposure condition on the appearance of the micro concretes.

Analyzing Figure 6 showing the micro concretes without admixture, it is noted that the micro concrete

Table 7. Void index ANOVA results.

	SQ	GL	MQ	Fcal	Ftab	p-value	Result
Model	0.039084	7	0.005583	550.82	2.66	0.0000	Significant
Error (residue)	0.000162	16	0.00001				
Total	0.039246	23					
Water content	0.038523	1	0.038523	3,800.40	4.49	0.0000	Significant
Crystalline admixture	0.000068	1	0.000068	6.70	4.49	0.0200	Significant
Conditions of exposure (sealed or saturated)	0.000374	1	0.000374	36.90	4.49	0.0000	Significant
Water content x crystalline admixture	0.000082	1	0.000082	8.10	4.49	0.0118	Significant
Water content x conditions of exposure	0.000005	1	0.000005	0.50	4.49	0.5000	Insignificant
Crystalline admixture x conditions of exposure	0.000023	1	0.000023	2.30	4.49	0.1488	Insignificant
Water content x crystalline admixture x conditions of exposure	0.000009	1	0.000009	0.90	4.49	0.3554	Insignificant
Error (residue)	0.000162	16	0.00001				

Table 8. Grouping of means by Duncan's test.

Type of concrete/ water content	Exposure conditions	Water absorption average value (%)	Average void index (%)	Groups			
				1	2	3	4
N11	Saturated	8.0	16.7		x		
A11	Saturated	8.1	16.8		x		
N11	Sealed	8.5	17.6			x	
A11	Sealed	8.4	17.6			x	
N7	Saturated	12.6	25.0	x			
A7	Saturated	12.3	24.6	x			
A7	Sealed	12.4	25.0	x			
N7	Sealed	13.1	26.0				x

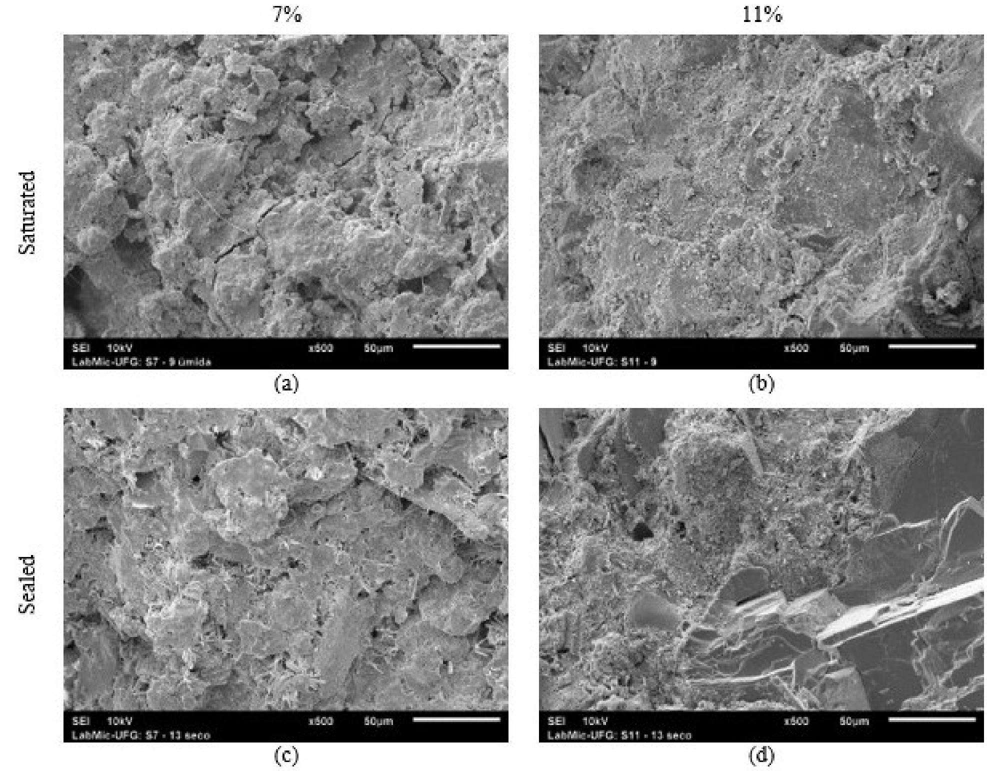

Figure 6. SEM images without the crystalline admixture, with amplification of 500x, showing influence of water content and exposure conditions: (a) saturated with 7% water content; (b) saturated with 11%; (c) sealed with 7% water content and (d) sealed with 11%.

specimens with 7% water content (N7) were more heterogeneous and porous than the mixtures with 11% (N11), including the presence of a crack (in the saturated condition). Considering the exposure conditions (saturated or sealed), the appearance was similar: the concrete surfaces had the same aspects. This corroborates the compressive strength and water absorption results, which indicated a better performance of the micro concretes with 11% water content and no difference between the types of exposure conditions.

Considering the water content, the same behavior seen in Figure 6 was observed in Figure 7, especially for the sealed condition. Comparing the exposure type, for 7% water content with the crystalline admixture the saturated condition promoted a more compact and homogeneous cement matrix, corroborating the small improvement in the compressive strength and water absorption results. This could indicate that water is necessary for adequate performance of the crystalline admixture.

To analyze the effect of the crystalline admixture, Figure 8 shows some SEM images of mixtures with 7% water content in the saturated and sealed conditions.

Figure 8 presents cementitious matrices with no great differences between them. The presence of the crystalline admixture seems not to significantly alter the appearance of these micro concrete samples, but has a tendency to produce slightly more porous micro concretes, especially for the saturated condition. The use of wet curing (saturated) produced, apparently, less homogeneous materials. But, as seen in the macro properties presented before, the exposure condition showed no significant differences in the results. For 11% water content, related trends were observed with similar micro concretes studied, analyzing the same aspects.

4. GLOBAL DISCUSSION

For conventional concretes, the effect of low water/cement ratios on the durability of the concrete is known in the literature, especially in relation to porosity and permeability and from the point of view of the transport and entry mechanisms of aggressive agents and moisture (water content) (9, 10).

However, when referring to very low w/c ratios, there seems to be a minimum limit for the hydration of cementitious compounds (69). Thus, concretes with a 0.44 w/c ratio (11% water content) showed better performance in the measured properties than the concretes with w/c 0.28 (7% water content). These results were true, evident and significant in all the properties evaluated, including from a statistical point of view, even with the degree of significance (Fcal in

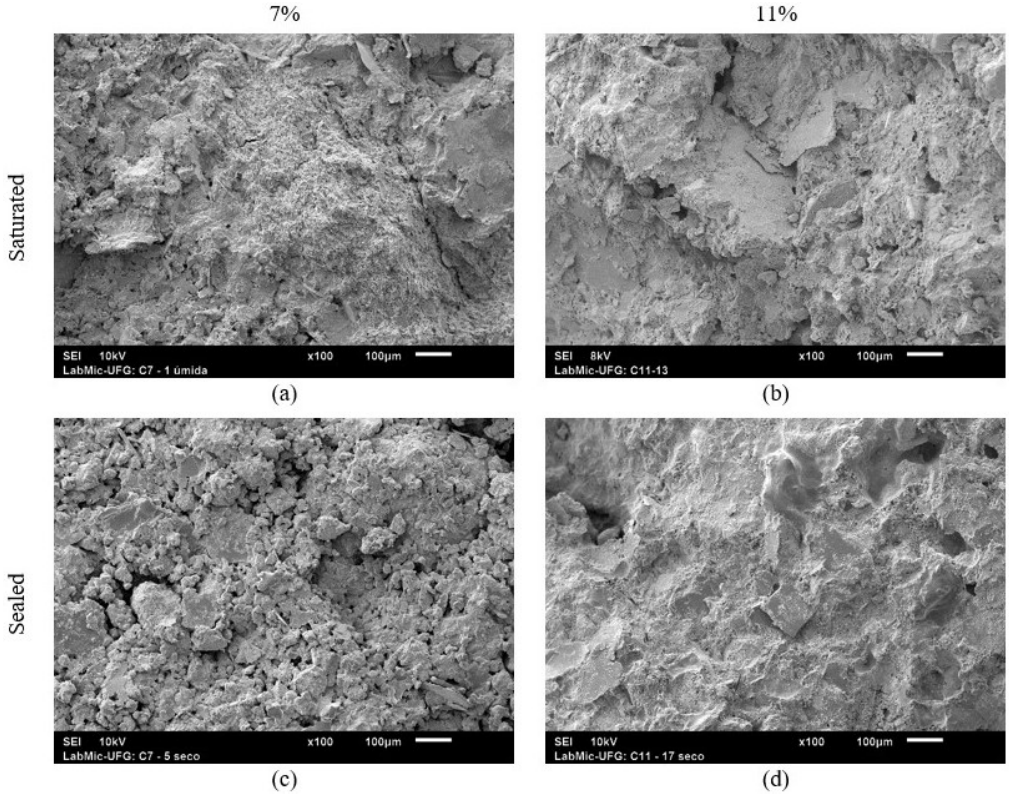

Figure 7. SEM images with the crystalline admixture with amplification of 100x, showing influence of water content and exposure conditions: (a) saturated with 7% water content; (b) saturated with 11%; (c) sealed with 7% water content and (d) sealed with 11%.

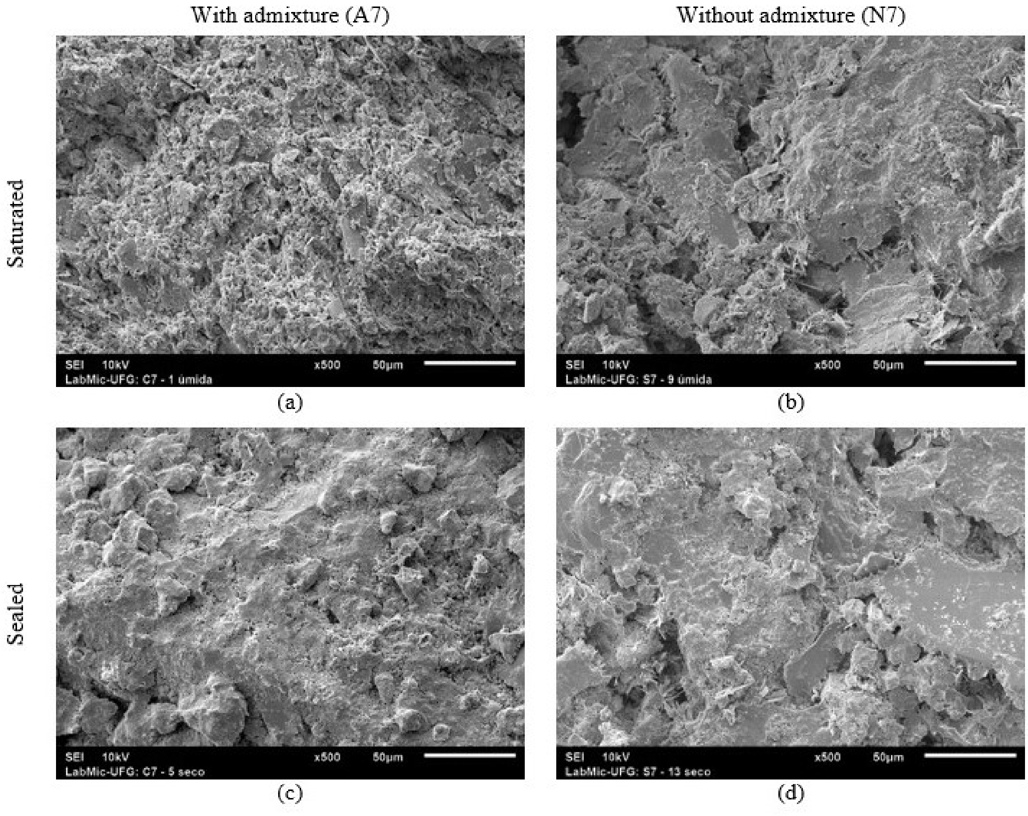

Figure 8. SEM images with 7% water content, with amplification of 500x, showing influence of admixture and exposure conditions: (a) saturated with the crystalline admixture; (b) saturated without the admixture; (c) sealed with the admixture and (d) sealed without.

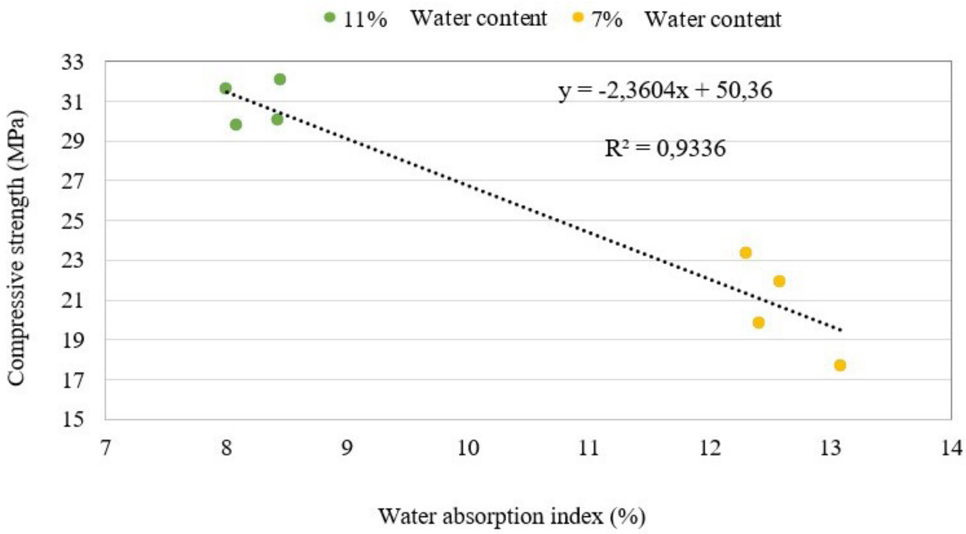

Figure 9. Relation between compressive strength (MPa) and water absorption index (%) values of the studied micro concretes with different water content.

relation to Ftab and the p-value). The water content, taken in isolation, was the most significant variable and, no less important, this variable interacted with the presence of the admixture (taking the variables two by two) and was also significant in all the other properties evaluated.

The effect of the crystalline admixture seems to have been the same for the two water content levels studied, as shown by the properties and analyzed microstructure. This may have been a consequence of the low w/c ratios studied, where the action of the crystalline admixture did not stand out, and this was one of the objectives of the study. This does not mean that for higher water content this effect cannot be more evident and that other types of crystalline admixture cannot be studied in drier precast concretes. The study by Roig-Flores *et al.* (35) showed positive effects of the use of a crystalline admixture in concrete with a water/cement ratio greater than 0.45. And the research by Al-Rashed and Al-Jabari (8) presented a tendency to an improved self-healing capacity with ratios of 0.37 to 0.54. There are also records of insignificant or even the same effects. In a study by Hassani *et al.* (25) these admixtures had less influence on the water penetration in concrete than the water/binder ratio (0.40 and 0.60) and the type of cement. And in the study by Escoffres, Desmettre and Charron (37) on high-performance fiber reinforced concretes with a water/binder ratio of 0.43, the use of these admixtures did not change significantly the mechanical and water permeability properties when submitted to monotonic tensile loading.

Additionally, the exposure conditions expressed significant results among themselves for the properties of the void index and water absorption, with lower values for higher water content. In terms of significance, this is the second most important property considered in this study; that is, Fcal is the second largest in terms of Ftab (or p-value).

As concerns the age in the compressive strength tests, this was Insignificant when taken alone, or with double or triple interaction. This means that the results at 28 days and 154 days were statistically equal to the results of the compressive strength obtained for the studied concretes. From a practical point of view, in this case, the result at 28 days showed a faster response in the investigated property and in the analyzed dataset, and could distinguish the concretes in different compressive strength classes – NBR 8953: 2015 (70).

The relationship between the average results of the compressive strength and water absorption at 154 days is shown in Figure 9.

5. CONCLUSIONS

This study analyzed the influence of crystalline admixture on the compressive strength and water absorption of precast micro concretes. Based on the discussions from a practical and application point of view, it can be concluded that:

- the water content (w/c ratio), taken alone, was the most significant variable and, no less important, this variable interacted with the presence of the admixture (taking the variables two by two) and was also significant in all the other properties evaluated. The effect of this property is known in the literature and had the expected effect in this study.
- the effect of the admixture seems to have been the same in the two water contents studied. This may have been a consequence of the low w/c ra-

tios studied, where the action of the admixture did not stand out, and this was one of the objectives of the study.
- the exposure condition had significant effects on the properties of the void index and water absorption, with lower values for higher w/c ratios.
- the age in the compressive strength tests was Insignificant when taken alone, or with double or triple interaction. This means that the results at 28 days and 154 days were statistically equal to the results for the compressive strength obtained from the studied concretes.

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Authorship contribution statement

Edgar Bacarji: Conceptualization, Investigation, Methodology, Original draft, Review & editing.

Rayane Campos Lopes: Data curation, Investigation, Methodology, Writing, Editing.

Gabiel W. Bacarji: Data curation, Investigation, Methodology.

Andrielli Morais Oliveira: Project administration, Visualization, Writing, review & editing.

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