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Oct 29th, 5:00 PM - 5:15 PM

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Visar Krelani
University for Business and Technology, visar.krelani@ubt-uni.net

Liberato Ferrara
Politecnico di Milano

Recommended Citation

Krelani, Visar and Ferrara, Liberato, "Reducing the Porosity and Sealing Cracks by Using Crystalline Admixture in Conventional Concrete" (2017). *UBT International Conference*. 49.
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Reducing the Porosity and Sealing Cracks by Using Crystalline Admixture in Conventional Concrete

Visar Krelani[1], Liberato Ferrara[2]

UBT – Higher Education Institution, Kalabria, 10000, Prishtinë, Kosova
[2]Politecnico di Milano, P.za L. Da Vinci, 20133, Milan, Italy
visar.krelani@ubt-uni.net

Abstract. There is a continuous increase of quality on civil engineering materials in developed countries and parallel increase of need for new constructions in developing countries. Professional community should propose solutions for the durability that can resist in different severe environments. The most important factor that can affect concrete durability is represented by the pore distribution. Transport properties can take place through the porous network inside the cementitious composites and the aggregates interface, permitting the ingress of aggressive agents damaging concrete function intrinsically as a material and the well-functioning of the entire structure. The use of a crystalline admixture during the mixing procedure can fill the pores and capillarity of the cement composites, while in case of the appearance of the cracks, can perform as sealing agent, representing a secondary innovative benefit. Concrete structure, in this case will be more durable and there will be no need for un-planned intervention.

Keywords: durability, self-sealing, concrete, crystalline additive

Introduction

Concrete as known; represent intrinsically porous material, which are represented from the pores at nano, micro and millimetric-scale. Porosity depends from the project of mix design that can be modified while mixing procedure or even by the wrong casting and low attention during curing. This is due to water presence based on mix design or due to entrained air and capillarity of the material itself, where water than could penetrate into concrete structure and consequently create physical and chemical processes that can seriously damage concrete durability.

A better control of porosity is possible by using "supplementary cementitious materials" (fly ash, silica fume, slag etc.) that are characterized with a finer distribution on the granulometric curve, when compared with cement, which will densify the matrix and reduce the general porosity or reduce the pore diameter. This effect then generates the reduction of general permeability and the ingress of potentially aggressive ions giving to the matrix the possibility to control the water and humidity movements, giving even some positive effect on concrete durability.

Last decades, beside "supplementary cementitious materials" mentioned before, in the civil engineering market, other effective contributors at the density of the matrix as densifiers or refiner of the pores, are shown such as specific admixtures known as "admixtures for the reduction of the permeability". These admixtures can be hydrophobic or crystalline admixtures.

The constitution of the first is represented from chemical compounds, similar constituents as soap or based fatty acids in petrol, which doesn't react into the porosity of the matrix itself but they contribute to create a hydro-repellent layer up to the pores. Crystalline admixtures, in the other side are powder that are added usually to the dry components of the concrete and mixed together and represent something as one percent of the cement mass. Based to its constituents these admixtures represent a strongly hydrophilic nature, which reacting with humidity of the atmosphere, creates a crystalline structure, densifying the matrix while during the calcium hydro silicate (CSH) phase reducing the porosity, consequently the permeability opposing the ingress of the water and aggressive agents.

For a better understanding of this admixture, different exposure conditioning has been performed such as normal or increased water pressure and there was shown an important effectiveness as a porosity reducer. Otherwise, the same admixtures showed good results ion reducing the hydraulic shrinkage which can be seen better when concrete is limited by different constrains, which delayed the appearance of the cracks and reducing their width. When the last happen, another advantage is shown by improving the freeze-thaw cycles.

An important testing of this admixture has been done into the structures of Shanghai Airport, Terminal 3, which is totally constructed into the seawater; structures that are exposed into sever conditioning. Concrete samples have been extracted for further chemical studies and results have shown that; crystalline products are formed, such as calcium, oxygen and silica with scent of zolfo and aluminum (ettringite), plus calcium carbonate CaCO3. Increasing the number of crystals some of the studied cracks have shown reduction of their width, result that pushed us for a deeper study on literature about the self-healing capacity of cementitious composites.

One of the first reports about self-healing capacity was found by the French Academy of Science, dates back to 1836: it was reported that the conversion of the calcium hydroxide leaching from the hydrated cement into calcium carbonate closes the cracks on atmospheric exposure.

Abrams, in 1913, was among the first researchers who explained the autogenic self-healing in concrete. He suggested that that the healed strength of concrete is caused by the retarded or the interrupted hydraulicity of the cement.

Gilkey in 1930 studying a concrete about six months old, found that the recovered strength is inversely proportional to the age of concrete. In the same report Bogue concluded that the healing action is represented by the continued hydration, supplemented by physical stresses, helping the formation of the precipitated bonds between severed grains. Another idea was reported by Loving 1936, who found that cracks in the concrete tubes were filled by the calcium carbonate. Whitehurst (1951), in a soniscope testing of cracked concrete structures subjected to wet spring, following a freezing and thawing season reported an increasing of the dynamic modulus in the healed concrete.

An important study on the strength recovery and on the explanation of the possible healing mechanisms of the healed cracks surfaces was performed by Lauer and Slate. They showed that the strength gain from autogenous healing in the water is not linear with time but follows of a parabolic trend with time whereas in a 95% relative humidity environment this healing activity is more nearly linear, though the recovery is slower but in a greater length in time. Dhir et al. 5 performed an extended experimental campaign investigating the autogenous healing potential of nine different mortars, varying the aggregate/cement ratio and comparing virgin with fractured specimens. Investigation showed that all types of tested mortars had the ability to self-heal. This ability is highlighted in percentage of recovery due to higher content of cement in front of other mortars with the higher water/cement ratio that showed a higher initial tendency of healing but lowers in time.

Several studies performed by Van Tittelbom et al., Li et al. and Edvardsen et al. [8] showed a reduction in water permeability of concretes between the un-cracked and cracked state, lead to the conclusions that this reduction was performed by the self-healing of the cracks.

The potential of this kind of admixture on reducing the porosity [9] and the advantages as permeability reducers doesn’t represent last advantage and the last but not the last, the possibility to reduce or potentially heal the cracks pushed us to perform and extend experimental campaign and a detailed study in the self-healing capacity of cementitious composites with and without crystalline admixture.

Experimental Activities

The mix composition, detailed in Table 1, has been designed for a target cube compressive strength at 28 days equal to 30 MPa. Because of the interest to evaluate the effects of crystalline additives on the permeability and at the self-healing capacity of concrete, a companion mix has been also produced with a 1% additive addition, by weight of cement.

Table 1. Mix-design of the investigated mortar

Constituent (kg/m3)	Without Additive	With Additive
Cement type II-42.5 R	300	300
Water	190	190
Superplasticizier (lt/m3)	3	3
Fine Aggregate 0-4mm	1078	180
Coarse Aggregate 4-16mm	880	880
Crystalline Additive	-	3

The additive was dry mixed with the raw aggregates at the very beginning of the mixing sequence, which was then followed by the addition of cement and, upon further mixing, by the incorporation of water and superplasticizer.

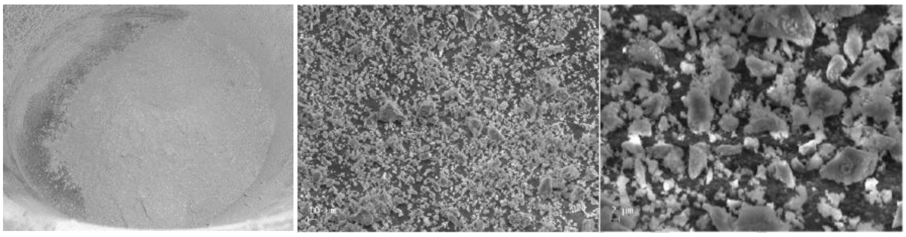


Figure 1. Observations on the crystalline additive; Visual observation on different scale of additive particles of in a scanning; (left) original visualization of the additive; (middle, right) scanning electron microscope in different magnifications

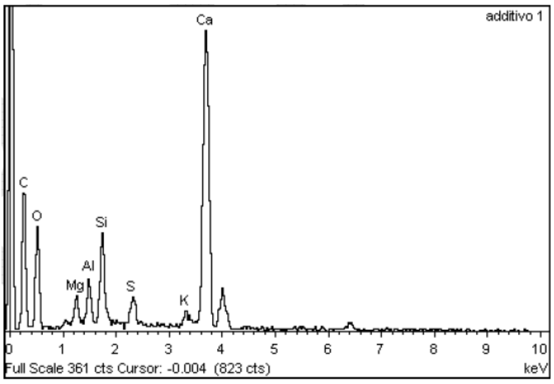


Figure 2. Chemical characterization of the crystalline additive showed in (Figure1) by the Energy dispersive spectroscopy (EDS) test.

In Figure 1 it is possible to observe the particles of the crystalline admixture. They have irregular shape and size in the range of about 1-20 μm (Figure 1, middle-right); their morphology is similar to that of cement grains; as a matter of fact, also according to the manufacturer, cement is present in the admixture and this is confirmed by the presence of calcium, oxygen, silica, magnesium, aluminum and potassium in the EDS microanalysis shown in Figure 2. This spectrum is comparable with that of an Ordinary Portland Cement (OPC), except for the peak of Sulphur which is slightly higher.

Figure 3. Manufacturing of the specimens; (left) casted slab; (middle) reference cube specimens; (right) slabs under wet towels.

Slabs 1m long x 0.5 m wide and 50 mm thick were casted (Figure 3, left) with both mixes; after three days curing in laboratory environment under wet towels (Figure3,right). Slabs were cut into prismatic “beam-like” specimens, each 500 mm long and about 100 mm wide (Figure 4) and cured in a moist room.

Comparison cube specimens (Figure 4, middle) were also cast for compressive strength measurements.

Figure 4. (left) Slabs stored on a chamber room; (middle) cutting machine; (right) specimens like “beam” after cutting

At the end of the curing period detailed above, the beam specimens were pre-cracked up to different levels of crack opening, equal to 150 and 300 μm. Un-notched specimens were pre-cracked employing the three - point bending (3pb) test set-up shown in Figure 5, where the clip-gauge measuring the Crack Opening Displacement (COD) at mid-span (used as test control variable) is also shown. Some specimens were kept un-cracked for reference as well.

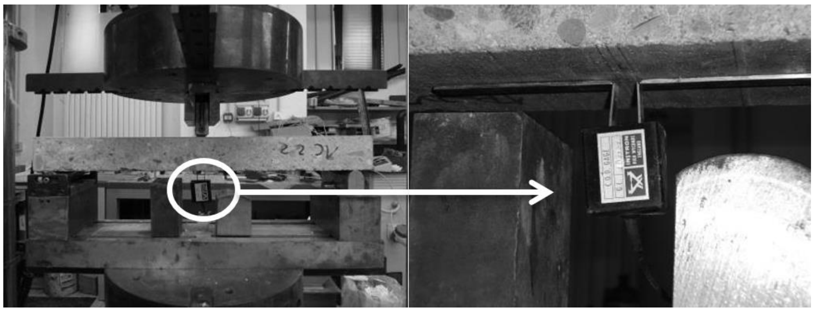


Figure 5. (left) Three point bending test set-up for pre-cracking procedure; (right) clip gauge used for measuring the crack opening mouth displacement

Besides these “natural” exposure conditioning, accelerated conditioning in a climate chamber, also to assess the reliability of accelerated haling procedures, were performed (Table 2) [10]. The performed cycles, each lasting 6 hours and meant as representative of exposure to either a winter or summer Northern Italy climate, are shown in (Figure 7, left). Exposure up to 1, 2 and 4 weeks in climate chamber for both types of accelerated cycles was performed (Figure 7, right). At the end of the scheduled exposure times, the specimens were first of all analyzed with an optical microscope to visually check the presence of the healing products in the cracks.

Table 2. Exposure conditioning and the duration for each of them

Type of the Conditioning	Duration of the Conditioning
H – Water Immersion	1, 2, 3, 6, 12 months
D – (Dry) Natural air exposure	1, 2, 3, 6, 12 months
HD – Wet/Dry	1, 3 months
C – Climatic Chamber	1, 2, 4 weeks

Figure 6 (left) Climatic chamber; (middle) Water immersion; (right) air exposure

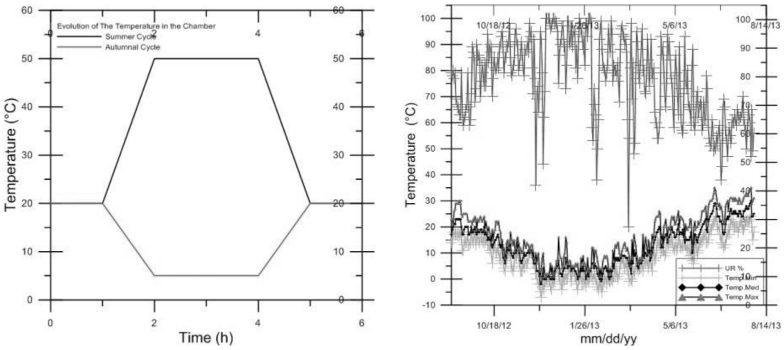


Figure 7. (left) Temperature and relative humidity simulated by the climate chamber; (right) T and RH recorded along the specimen exposure period.

Table 3. Synopsis of experimental programme (n° of specimens per each test condition)

	Exposure condition and duration															
	Water immersion					**Open air exposure**					**Climate chamber**					
											winter			**summer**		
	1m	**2m**	**3m**	**6m**	**12m**	**1m**	**2m**	**3m**	**6m**	**12m**	**1w**	**2w**	**4w**	**1w**	**2w**	**4w**
	w/out additive															
Un-cracked	1	2	2	2	2	1	2	2	2	2	2	2	2	3	3	3
Pre-cracked 100 μm											2	2	3	3	3	3
Pre-cracked 200 μm	1	2	2	2	2	1	2	2	2	2	3	3	3	3	3	3
	with additive															
Un-cracked	1	2	2	2	2	1	2	2	2	2	2	2	2	6	6	6
Pre-cracked 100 μm											2	2	3	6	6	6
Pre-cracked 200 μm	1	2	2	2	2	1	2	2	2	2	3	3	3	6	6	6

m = months
w = weeks

Then the specimens were tested up to failure according to the same set - up employed for the pre-cracking. A typical response, in terms load vs COD in the case of healing, is shown in the Figure 9 that corresponds with the response exhibited by the same specimen in the pre-cracking stage.

Discussions of Experimental Results

The two investigated concretes, with and without the crystalline admixture, were first of all characterized by meaning the development of their compressive strength all along the 28 days curing period before the pre-cracking.

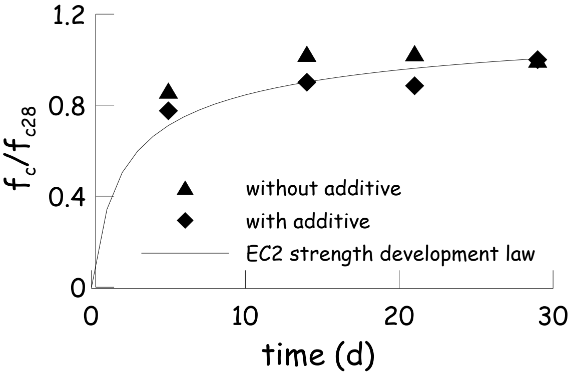


Figure 8. Strength development of concrete with and without crystalline additives vs. EC2 previsions

Based on the EC2, cubic specimens were tested by comparison between the concrete with and without admixture, to evaluate the effect of admixture in the strength development of concrete. Then, EC2 predictive law [11], has been plotted on the same graph and compared with the strength development of both concretes.

By observing the graph in the Figure 8 it can be stated that: the crystalline admixture alone, in a sound concrete specimen, does not affect the strength of the material nor its development within time.

Based in the porosimetry test named as mercury porosimetry test, the obtained results shown differences between two types of concrete where the total pore area equal to 6.461 m²/g of the concrete without admixture is higher compared to the same concrete with the mentioned admixture where same parameter shows a total pore area equal to 5.083 m²/. This result has been obtained after first month of water exposure.

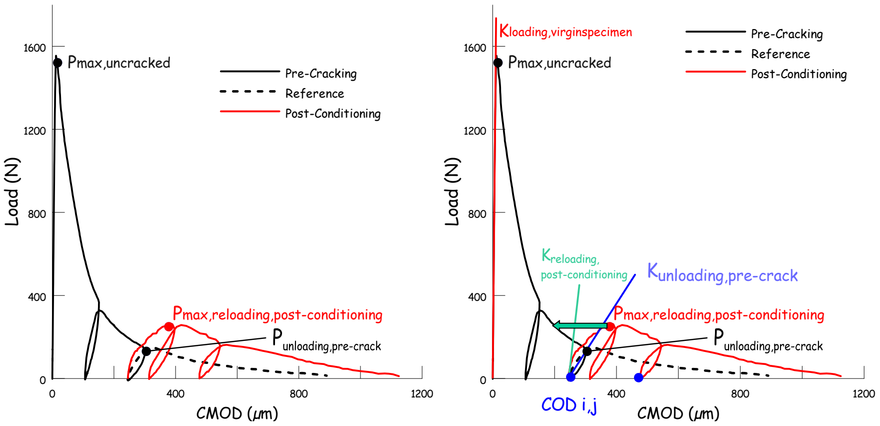


Figure 9. Example of load vs. COD curves for specimens submitted to pre-cracking and post-conditioning 3pb tests; definition of quantities for calculation of self-healing indices

$$\mathrm{ILR} = \frac{\sigma_{\mathrm{N,max\ reloading,\ post\text{-}conditioning}} - \sigma_{\mathrm{unloading,pre-crack}}}{f_{ctf} - \sigma_{\mathrm{unloading,pre-crack}}} \qquad 1$$

$$\mathrm{ICH}_{\mathrm{stress\text{-}crackopening}} = \frac{\mathrm{COD}_{\mathrm{pre-cracking}} - \mathrm{COD}_{\mathrm{post-conditioning}}}{\mathrm{COD}_{\mathrm{pre-cracking}}} \qquad 2$$

In Figurer 9 the results of a typical test, in terms of load vs. COD curves, are shown: it is worth remarking that the graphs are built up in a way that the curves pertaining respectively to the pre-cracking test and to the post-conditioning up-to-failure test for the same specimens are compared.

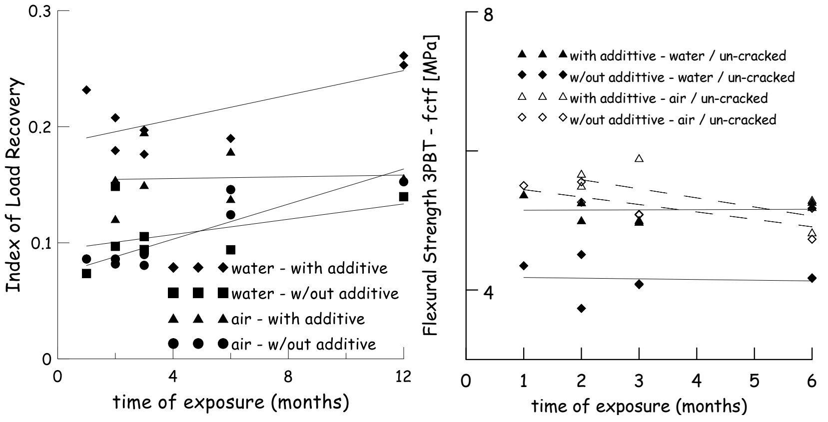


Figure 10. (a) Index of Load Recovery (as evaluated from 3pb test results) vs. exposure time for water immersion/air exposure (b) and flexural response un-cracked specimens.

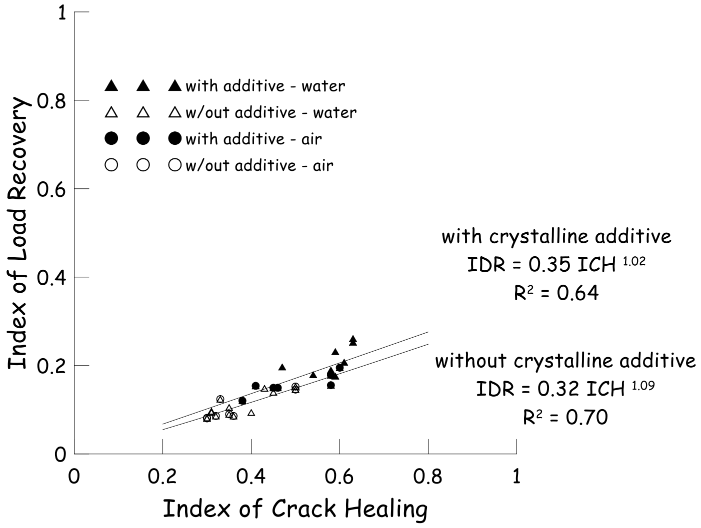
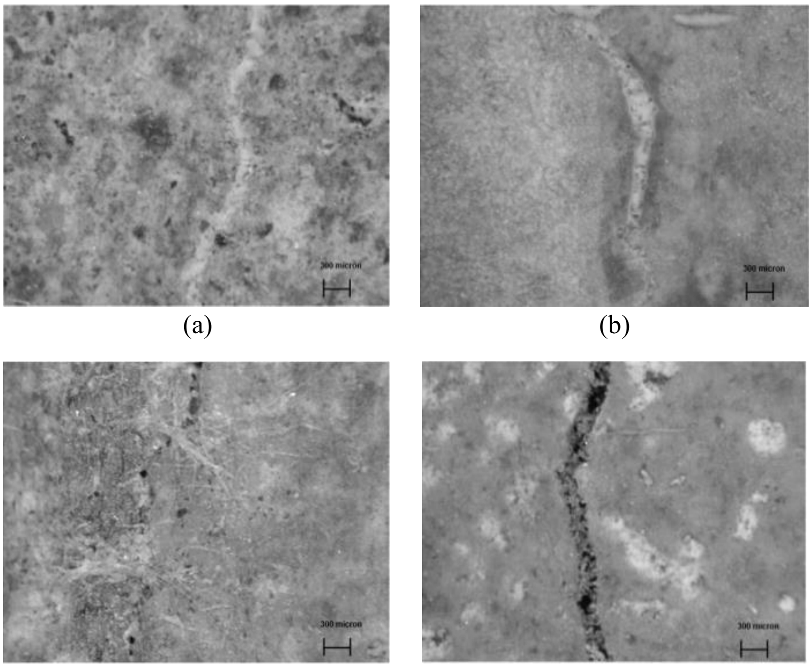


Figure 11. Index of Damage Recovery as evaluated from 3pb (left) vs. Index of Crack Healing as estimated from fitted damage evolution laws and (right) Index of Load Recovery vs. Index of Crack Healing as evaluated from stress vs. COD curves obtained from 3pb tests.



(a) (b)

(c) (d)

Figure 12. Healed cracks for specimens with (a) and without (b) crystalline additive after six months of immersion in water; specimens with (c) and without (d) crystalline additive after six months of exposure to open air.

Based in the equation 1 has been possible to be calculated the load recovery and the results has been plotted in the Figure 10. From the observed results (Figure 10, a), recovery of load bearing capacity, with respect to the loss of load bearing capacity (softening) experienced upon cracking, as also affected by presence/absence of crystalline additive and different exposure conditions, is absolutely coherent with observed trends of recovery of other mechanical properties. Whereas, most important recovery of bearing capacity, could be observed in the most favorable examined cases, specimens with the additive immersed in water.While, un-cracked specimens showed a stabled strength in time of exposuremeasured and identified from stress-crack opening flexural response (Figure 10, b). These results confirm the idea that hydration products have been produced in higher level when cracks are formed, where the same were not produced in un-cracked specimens.

Load recovery versus estimated crack healing (Figure 11) shows that some load bearing capacity appears to be recovered since for very low values of estimated crack healing while for higher recovery of bearing capacity higher closure of the crack is needed.

Pictures obtained by stereo-microscope in Figure 12 confirm the aforementioned statements, where immersion in water triggers the self-healing also for specimens without any additive, but at a much slower pace: only after 2 to 3 months effects start being visible and after 6 months a performance comparable to specimens with the additive was achieved; specimens without any additive exposed to air hardly shows recovery and only after prolonged (6 months) exposure period the crack closure can be appeared.

Conclusions

In this papers just a part of the results has been shown, there is still undergoing the experimental campaign, which can complete the here showed experimental campaign. Whatever, it has been showed that concrete owes intrinsically the capacity to heal the cracks, even this can be scant, which directly depends in the exposure conditions.

When crystalline admixture is added into the mix design, the capacity to heal the cracks increases, and there is shown more systematic and reliable performance, obtaining recovery of the load capacity up to 80%, thanks to the contribution of the admixture into the porosity and not just the reducing of the total volume of pores was obtained but consequently the healing of the crack and the continuation of the hydration process, which is another process that was promoted by the admixture.

The methodology showed here has been confirmed and validated due to the different exposure conditioning, duration or the presence of the crystalline admixture comparing with the same mix design without the mentioned admixture. The formed products contribute directly to one of the most fundamental phenomena of the design of reinforced concrete, such as presence of the cracks. So, by reducing the porosity and engineering the healing capacity, it is possible to introduce a new valuable sustainable concept to the concrete structures. In this sense the effect of the admixture shows the possibility to create more durable concretes and increase the lifecycle of the whole structure due to the steel defense in front of the aggressive agent, reducing or stopping the corrosion into concrete reinforced structures.

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