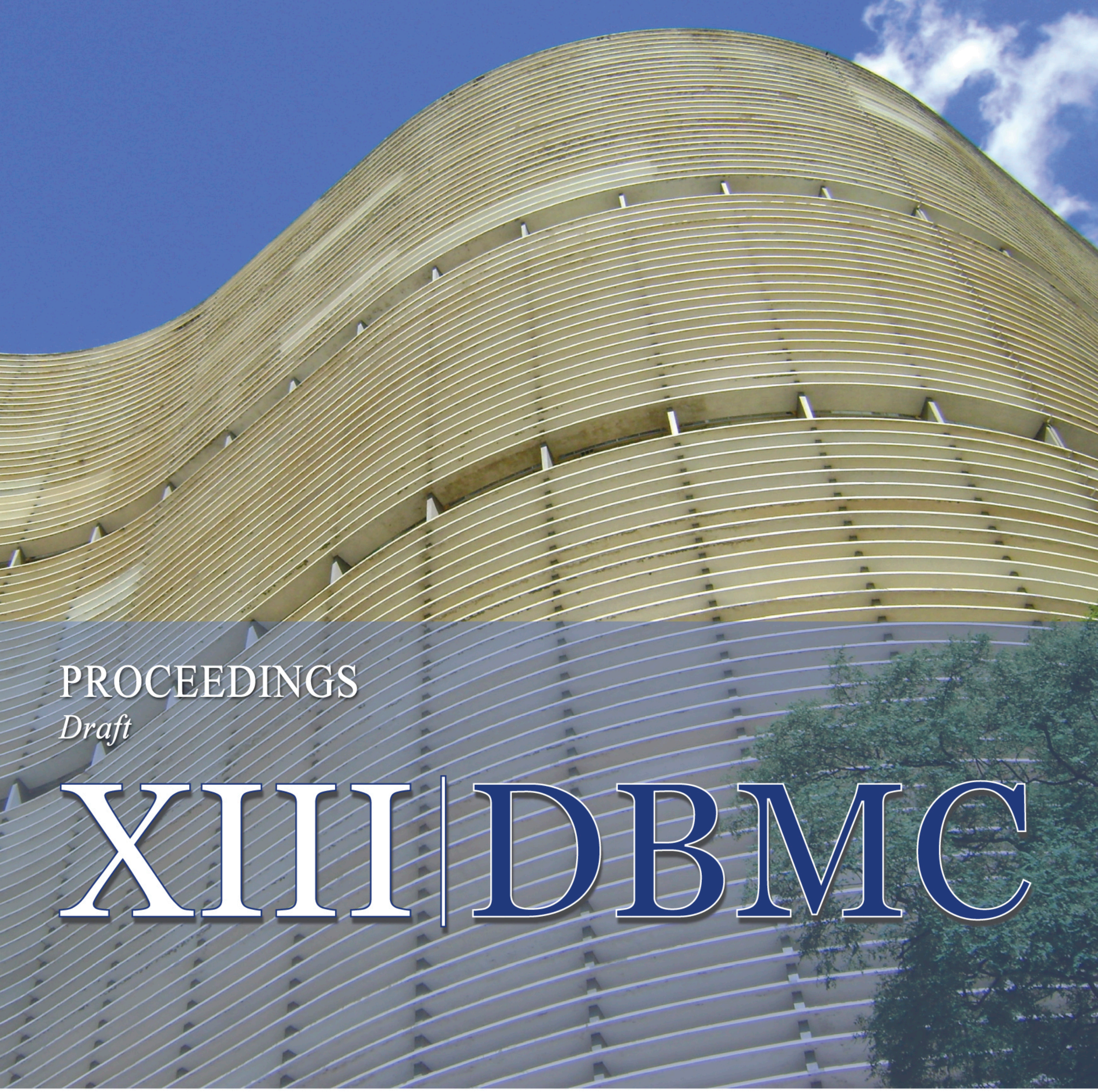

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XIII | DBMC

XIII International Conference on Durability of Building Materials and Components

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Preface

> *The ability to predict the service life—the durability—of a new building material is being ardently sought after in many parts of the world. Widespread efforts are correspondingly being made to increase the durability of materials already in wide use. Any estimate of the economic losses resulting from failures in durability lead inevitably to dollar figures so astronomically large that they are usually regarded with incredulity.*
>
> R. F. Legget
> Proc. of the 1st DBMC
> Ottawa, 21-23 Aug. 1978.

Some 35 years after the 1st DBMC (Ottawa, 1978), sponsored by ASTM, NIST and NRC, durability is an even more important subject. At that time, an accurate service life estimate was considered a condition to life cycle cost (LCC) analysis. Nowadays, service life is also crucial information for the environmental life cycle analysis (LCA). The relevance of durability became widely recognized among scientists: in 1992 the World Business Council for Sustainable Development coined the term "eco-efficiency", referring to a higher production with fewer environmental and economic resources and less environmental impact. The increase of durability (or service life) of products is one of the critical tools to improve eco-efficiency.

In that period the research community, which was organized into CIB and RILEM working groups, developed a methodology to plan and predict the service life of buildings and constructed assets at the design stage, which was consolidated in the ISO 15686-x standards. As a result of these scientific advances, the degradation factors and mechanisms of most materials and their assemblies are now much better comprehended. Information Technology advances facilitate to mapping the relevant environmental variables by using GIS platforms. Dose-response functions are available, making possible the estimation of degradation rates by combining environmental variables with material characteristics. Even effects of climate change on service life have been a matter of discussion. Altogether, it is an impressive achievement, recorded in the previous 12 DBMC conference. However, despite this scientific progress, the use of these tools in day-to-day production processes, including materials and components R&D, architecture and civil engineering design, and environmental and economic life cycle analysis is still very limited in most countries. Today's research challenge is not only to increase the knowledge in the field, but also to make this knowledge readily available to the society, by developing more user friendly tools and better educate engineers and architects on the use of these tools.

The 13th edition of the DBMC conference was sponsored by ASTM, NIST, Rilem, CIB and University of Porto. It was joint organized by Polytechnic School of University of São Paulo and Secovi-SP, the most important Brazilian real estate industry's association, an union that simbolizes our commitment to transfer the knowledge to the society. The support of Saint Gobain Group, Grace Construction Products, Votorantim Cimentos, Gerdau, Fapesp, CNPq and Capes made the conference possible.

We expect that these proceedings will help professionals and academy to incorporate service life planning concepts in their day-to-day decision-making processes.

São Paulo, 25-Aug-2014
Marco Quattrone
Vanderley M. John
Editors

SELF-HEALING OF SELF-COMPACTING CONCRETES MADE WITH BRAZILIAN BLAST FURNACE SLAG CEMENTS ACTIVATED BY CRYSTALLINE CATALYST

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Abstract

Different approaches have been developed to study new types of concrete that have the ability to repair cracks. Autogenic self-healing improves the natural healing mechanism of concrete through incorporation of a catalyst that activates material components already present in the concrete. The occurrence of the crack is the trigger mechanism because it allows the penetration of water to activate the crystalline catalyst (Cat-X) and newly fractured sub-hydrated cement and non-activated blast furnace slag (BFS) particles. Test samples were examined using three types of Brazilian commercial cements: blast furnace slag cement (CP III RS), slag modified Portland cement (CP II-E) and Portland cement (CP V ARI) with percentages in the range of 55%, 35% and 0%, respectively. Test specimens were loaded in compression to 90% of failure load, in order to generate a network of micro-cracks. Samples were immersed in water to trigger the self-healing mechanism, followed by various tests after 28, 56 and 84 days. Recovery of mechanical properties and watertightness indicated greater self-healing in the CP III, CP V and CP II cement samples, respectively. As the BFS content ratio was increased to 55%, there was a noticeable increase in the mechanical recovery and permeation reduction properties due to the addition of Cat-X. Therefore, it appears that the specimens with added BFS and Cat-X are less affected by mechanical loads.

Keywords*: self-healing concrete; self-compacting concrete; blast furnace slag; crystalline catalyst.*

1 INTRODUCTION

Concrete is a material with a long history of use as a building material; its importance will continually require more research to solve the almost unavoidable issue of cracking in reinforced concrete. Research has focused on a scientific approach to the casual observations of self-healing cracks in concrete—a phenomenon that has been well-known since ancient times. The goal is the development of a self-compacting concrete with self-healing capabilities that enables the design of more durable concrete structures subjected to water exposure.

Self-compacting concrete (SCC) is one of the latest achievements in concrete technology. However, in comparison to conventional concretes, SCCs show much higher levels of thermal and autogenous shrinkage; these high strength SCCs are far more likely to crack at an early age than normal strength concretes. Investigations into the self-healing properties of SCCs subjected to mechanical loading led to the development of further hydration processes for unhydrated cementitious particles when exposed to water [1]. In subsequent investigations of the self-healing capabilities of cementitious composites incorporating different supplementary cementitious materials (SCM), a more evident self-healing product was observed from mixtures incorporating blast furnace slag (BFS) [2, 3]. Moreover, the high surface area of BFS provides more nucleating sites, as well as OH^- ions and alkalis for the pore fluid when BFS is hydrated [4]. The hydration products of Portland-slag cements are the same as Portland cement, except that smaller quantities of calcium hydroxide ($Ca(OH)_2$) are found at higher slag contents. The lower $Ca(OH)_2$ content and the finer pore structure of BFS concrete could therefore contribute to its greater chemical resistance [5].

Crystalline catalyst (Cat-X) is a synthetic cementitious material classified as a hydrophilic waterproofing and as a permeability reducing admixture hydrostatic according to ACI 212.3R-10. Its reactive components react with $Ca(OH)_2$ to form crystalline products that disconnect pores and fill cracks in the concrete. The crystalline products can only occur when sufficient moisture is present [6]. By means of diffusion, the reactive chemicals in Cat-X use water as a migrating medium to enter and travel down the capillaries of the concrete. This process precipitates a chemical reaction between Cat-X, moisture and the by-products of cement hydration, forming a new non-soluble crystalline structure, including apatite and enstatite crystals. This integral structure fills the capillary tracts rendering the concrete waterproof. Cat-X has many physical and chemical properties that are surprisingly similar to those of clay minerals. The high cation exchange capacity and the intercalation of carboxylic acids (-COOH) into the Cat-X structure makes their interlayer sodium ion (Na^+) exchange through calcium ions (Ca^{2+}) versatile and easily achieved. Cat-X has been extensively studied for the suppression and control of alkali-aggregate reactions (AAR) in concrete through the catalysed alkali discharge resulting from its chelating function [7-9]. This water stopping technique was used to improve the impermeability property of the concrete and decrease the migration of radionuclides (^{137}Cesium) during the treatment, storage and disposal of low level radio-active waste in Japan and was also used for waterproofing the bridges and tunnels of the Japan Railways Shinkansen bullet train lines [10].

The effect of water exposure conditions plays an important role in self-healing efficiency and the types of healing products formed. The exposure conditions of wet/dry cycles show that, in an optimal mechanical recovery, a mixture of calcium carbonate ($CaCO_3$), calcium silicate hydrate (C–S–H) and ettringite are found as major internal crack healing products.

Continuous water exposure leads to the formation of $CaCO_3$ on the crack mouth, which is preferable in terms of water tightness; however, this formation decreases the recovery of mechanical properties [11].

Therefore, the following mechanisms must be considered for a robust autogenic self-healing concrete: (a) A mechanism for continuous hydration with the use of cement composed of large amounts of BFS; (b) A mechanism of chemical healing with a dosage of Cat-X to cause a cementitious recrystallization effect in the concrete cracks; and (c) A mechanism of crack restriction with the addition of ductile type alkali-resistant (AR) glass fibers to enhance the cementitious matrix [12]. Multiple cracking with relatively small crack widths (in comparison to ordinary cracked concrete) generally results in the increased resistance of this material to the ingress of fluids and gases [13]. The proposed self-healing mechanisms have the added advantage that a second healing action may occur and will be easily implemented into concrete structures that are not easily accessible for maintenance and repair, such as underground structures, bridges, and dams. Although the initial costs will be higher, the maintenance costs can be reduced, and the service life of the structures may be extended, as damage is immediately repaired [14].

2 EXPERIMENTAL PROGRAM

In this program, the experiments performed on hardened concrete can be grouped into tests to determine the mechanical properties and tests to determine the permeation properties. Concrete specimens for self-healing tests were loaded to 90% of their compressive strength at 28 days age. Mechanical properties,including compressive strength and ultrasonic pulse velocity (UPV) were determined at 28 days, and then after 28 and 56 days of lime water curing following pre-loading by using three specimens at each age. The permeation properties were also determined at the same intervals by using water sorptivity and rapid chloride permeability (RCPT), in accordance with the ASTM C1585 and ASTM C1202 standards, respectively.

2.1 Material properties

Three types of Brazilian commercial cements with distinct percentages of blast furnace slag (BFS) ranging from 0 to 55%, were used in all mixtures: The CP III 40 RS cement (slag content < 55%) is equivalent to ASTM Type IS (MS) blast-furnace slag cement. The CP II-E 40 cement (slag content < 34%) is equivalent to ASTM Type I (SM) slag-modified Portland cement.The CP V 42.5 ARI cement is equivalent to ASTM type III high early strength Portland cement. A specific crystalline catalyst (Cat-X) was used in this study at the rate of 2.5% by weight of cement content.

2.2 SCC mix design, preparation and casting

Six SCC concrete mixtures were prepared with the proportions summarized in Table 1 for all three types of cements, with and without the addition of the crystalline catalyst. The total cement content and the water/cement ratio (w/c) of 0.5 (by mass) were kept constant. The crystalline catalyst was not considered as a binder. SP was added to achieve an initial slump of 80mm ; then PCE was added to get a slump flow of more than 700 mm for all mixtures. The SP content was not kept constant, though the PCE content remained constant.

Table 1 - SCC mix design (kg perm^3)

Ingredients	Quantities	Note
Water	196 kg	w/c ratio = 0.5
Cement	392 kg	CPIII / CPII-E / CPV
Crystalline catalyst	10 kg	2.5% of cement content
Quartz sand	573 kg	70% of fine aggregate
Crushed stone sand	246 kg	30% of fine aggregate
Crushed stone 9.5 mm	651 kg	70% of coarse aggregate
Crushed stone 25 mm	279 kg	30% of coarse aggregate
SP superplasticizer	2.4 kg	0.6% of cement content
PCE superplasticizer	4.0 kg	1.0% of cement content
Viscosity modifier	2,0 kg	0.5% of cement content
AR glass fiber	0,9 kg	Alkali-resistant 12 mm

Visual inspection of fresh concrete did not detect any segregation or bleeding in any of the mixtures during the tests. From each concrete mixture, Ø100 x 200 mm cylinder specimens were prepared for the mechanical and permeation tests. All specimens were cast in a single layer without any compaction. At 24 hr, the specimens were removed from the molds and stored in lime-saturated water at 23 ± 2° C for 28 days. To test the permeation properties of the pre-cracked concrete specimens, the cylinders were sawed into 50 mm thick slices, and the two middle slices from each specimen were used for testing.

2.3 Compressive strength recovery

After 28 days of moist curing, the compressive strength of each mixture was determined, and the remaining specimens were pre-loaded to 90% of their corresponding compressive strength. The pre-loaded specimens were then further stored in lime-saturated water at 23 ± 2° C for an additional 28 days and 56 days, respectively. The mechanical properties of the compressive strength of the concrete specimens before and after preloading load them to 90%, stop, then reload them to failure at 28 days.

2.4 Sorptivity test

The sorptivity test method is used to determine the rate of absorption of water in the concrete by measuring the increase in the mass of a pre-dried specimen resulting as a function of time, when only one surface of the specimen is exposed to water. The exposed surface of the specimen is immersed in water and the water ingress of the unsaturated concrete is dominated by capillary suction during the initial contact with the water.

2.5 Rapid chloride permeability test

The rapid chloride permeability test (RCPT) is a measure of the concrete’s resistivity which is an indirect measure of chloride penetrability. In this test, a 50 mm thick, 100 mm diameter, water saturated concrete specimen was subjected to a 60 V applied DC voltage for 6 h. One end of the specimen is in contact with a 0.3 M NaOH solution, while the other end is in contact with a 3.0% NaCl solution. This amount, determined in coulombs, is related to the concrete’s ability to resist chloride ion penetration through the fissured (F) specimens at 28 days and the fissured and cicatrized (FC) specimens (28+28 days) for all mixtures.

3 RESULTS AND DISCUSSION

3.1 Unhealed fissured specimens

The undamaged control specimens had a higher compressive strength comparing the properties of the fissured specimens. For this reason, the extent of any self-healing is best detected by comparing the results of fissured and undamaged specimens' properties at a particular age.

Because these fissured specimens were tested immediately after the release of the pre-loading, they had no time to undergo any crack healing. The results in Table 6 reveal that, for the unhealed specimens, the compressive strength, ultrasonic pulse velocity (UPV), water absorption rate and rapid chloride permeability test (RCPT) were affected by the crystalline catalyst (Cat-X) and blast furnace slag (BFS) content in the commercial cement.

Table 2 - Properties of unhealed fissured specimens

	Strength	**UPV**	**Absorption**	**RCPT**
Mix 1	50,6 MPa	4514 m/s	0.0243 mm	1764 C
Mix 2	44.2 Mpa	4413 m/s	0.0309 mm	1860 C
Mix 3	43.3 Mpa	4566 m/s	0.0326 mm	3927 C
Mix 4	40.7 Mpa	4247 m/s	0.0407 mm	4023 C
Mix 5	53,7 Mpa	4565 m/s	0.0327 mm	5139 C
Mix 6	52,6 Mpa	4679 m/s	0.0406 mm	5262 C

The Cat-X enhanced the compressive strength of the unhealed fissured specimens by 14% on CPIII, 6% on CPII and 2% on CPV. Therefore, it appears that the specimens with the addition of BFS and Cat-X are less affected by mechanical loads The water absorption rate was reduced by 21.4% on CPIII, 19.9% on CPII and 19.5% on CPV. In addition, the RCPT values reduced by 5.2% on CPIII, 2.4% on CPII and 2.3% on CPV. However, an increase in BFS content from 34% to 55% showed no significant effect on the permeation reduction effect. The measurable damage reduction was least clear in the ultrasonic pulse velocity (UPV).

The use of supplementary cementitious materials (SCM) such as BFS may have a significant effect on the permeability of concrete as measured by the RCPT. While the active component in cement is a hydraulic binder, blast furnace slag also has latent hydraulic properties, this means that the hydration reaction with water must be activated by substances such as $Ca(OH)_2$, e.g., from clinker hydration. However, it is known that currently used SCMs such as BFS react more slowly compared to cement [15].

3.2 Effects of self-healing

Repairing cracks in mechanically loaded reinforced concrete elements has been a long-time goal. The conventional repair methods either physically block off water by applying a waterproofing material with crack-bridging abilities or inject low viscosity resins into the cracks. By contrast, self-healing concretes (SHC) can waterproof the entire concrete structure—as well as the cracks—with the application of a crystalline waterproofing catalyst [16]. Table 3 presents the results of compressive strength, UPV, sorptivity and RCPT tests from specimens that were subjected to preloading and later stored for up to 56 days in water to evaluate the extent of self-healing. Autogenous self-healing can reduce the water permeability of cracked specimens.

Table 3 - Properties of fissured and cicatrized specimens

	Strength	**UPV**	**Absorption**	**RCPT**
Mix 1	58.8 MPa	4540 m/s	0.0182 mm	1086 C
Mix 2	55.5 Mpa	4646 m/s	0.0204 mm	1170 C
Mix 3	47.7 Mpa	4566 m/s	0.0267 mm	3108 C
Mix 4	45.1 Mpa	4665 m/s	0.0273 mm	3225 C
Mix 5	55.7 Mpa	4492 m/s	0.0274 mm	2451 C
Mix 6	53.7 Mpa	4494 m/s	0.0265 mm	2658 C

Cat-X enhanced the compressive strength of the fissured and cicatrized specimens by 5.9% on CPIII, 5.8% on CPII and 3.7% on CPV. the water absorption rate reduced by 10.8% on CPIII and 2.2% on CPII, but increased by 3.4% on CPV. In the RCPT, the values reduced by 7.2% on CPIII, 3.6% on CPII and 7.8% on CPV. These values show that the self-healing effect depends on the type of cementitious materials used, the BFS content and the addition of Cat-X.

By comparing the plot of the water absorption of the healed specimens stored in water as the BFS incorporation level increased up to 55% with Cat-X (as shown in Figure 2), it can be observed that a significant reduction of cumulative water absorption with the square root of time has occurred. However, the healed specimens without Cat-X show a higher recovery percentage. This suggests that the self-healing effect also depends on the exposure conditions to water and air. One can hypothesize that, during the drying phase, the excess water has evaporated; therefore, the ion concentrations in the water present in the cracks have increased. In this situation, the amounts of reactants available for further reactions are considerably concentrated, while the amount of water was also sufficient for through-solution reactions. This phenomenon should enhance chemical reactions, precipitation and further hydration. Moreover, the penetration of CO_2 into the crack during the drying period would lead to the additional formation of carbonates that are helpful for sealing the cracks, as observed by others [11].

As the BFS incorporation level increased up to 55%, the compressive strength reductions were more visible in the 56 day samples. At 90% pre-loading, the recovery amounts reduced from 29.6% to 18.4% in CPIII, 14.3% to 13.6% in CPII and 4.0% to 3.9% in CPV. Based on the previous discussions, it was observed that, in the SCC mixtures with BFS, the unhydrated BFS material available for further hydration was also greater. Therefore, it appears that the BFS significantly influences the self-healing of the mechanically pre-loaded specimens; even after 56 days, moist curing is critical to long term self-healing. The C–S–H gels formed through the activation of the BFS reactions developed a good bond within the micro-cracks. The measurable damage and healing was the least clear with the ultrasonic pulse velocity (UPV).

Similar conclusions can also be made for the RCPT. However, it should be noted that, among the effects of self-healing, the reductions were more visible in the 28-day healing window. The reduction amounts were 52% to 49% for the CPV, 38% to 37% for the CPIII and 21% to 20% for the CPII-E. The effects of self-healing are more visible on the permeation properties when compared to the mechanical properties. Therefore, in looking at the RCPT reductions presented in Table 11, it can be observed that the results of tests such as the recovery of the mechanical properties and watertightness show greater self-healing in the samples with CP III, CP V and CP II cement, respectively.

Pre-loading the concrete caused an increase in its total porosity and a loss in its ultimate compressive strength. As micro-cracks developed inside the concrete structure, the pore structure was modified and the continuity of the cracks was increased. Internal cracking due to the mechanical loading initially reduced the compressive strength from 5.1 ~ 18.9% when pre-loaded up to 90% of ultimate compressive strength; after 56 days, recovery was 3.9 ~ 29.6%. This was because the high volume of BFS, a hydraulic latent material, had a significant amount of unhydrated particles available in its microstructures; this contributed to the self-healing effect of the pre- existing cracks activated by the crystalline catalysts. The recovery of the compressive strength and permeation properties can be related to the progressive filling of cracks by the newly formed C-S-H gels due to their self-healing effects [17]. Slag cement and slag-modified concretes with addition of Cat-X benefitted from prolonged limewater curing and show more significant reductions in water absorption rates than the control mixes. Ongoing chemical reactions between the crystalline catalyst and the cement by-products could be promoted by these curing conditions resulting in reduced volumes of permeable voids.

4 CONCLUSIONS

- As the BFS content ratio was increased to 55%, there was a noticeable increase in the mechanical recovery and permeation reduction properties due to the addition of Cat-X. Therefore, it appears that the specimens with added BFS and Cat-X are less affected by mechanical loads.
- Slag cement and slag-modified concretes with the addition of crystalline catalyst benefitted from prolonged limewater curing and show more significant reductions in water absorption rates than the control mixes.
- The recovery of the mechanical properties and watertightness indicate greater self-healing in the samples with CP III, CP V and CP II cement, respectively.
- If there is a tendency for a slower self-healing in samples with BFS (CP III and CP II) at an early age than compared with the samples with 100% Portland cement (CP V), the latent hydraulic behavior of the BFS is critical to its long-term self-healing.
- It was clear that the crystalline catalyst was effective in improving the mechanical and permeation properties of the SCC stressed by mechanical load. It is necessary a minimum dosage of 6 kg to 10 kg/m^3 (or 3 kg to 5 kg/m^3 in concentrated form) for a self-healing effect.

Exposure conditions play an important role in the self-healing process; in general, wet/dry conditions is recommended for the optimal mechanical recovery. For developing techniques that can assure long-term durability for concrete structures when subjected to continuous water exposure, is recommended improving with alternative binders such as blast furnace slag (BFS), silica fume and crystalline catalyst (Cat-X[*]).

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[*] Xypex Admix C-500

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