Cementitious Composite Materials with Self-healing Properties Using Integral Waterproofing Admixtures by Mass Crystallization

Tudor Panfil Toader[1,2](✉), Carmen Dico[1], and Călin Mircea[2]

[1] NIRD URBAN-INCERC Cluj-Napoca Branch, 117 Calea Florești, Cluj-Napoca, Romania
tudor.toader@incerc-cluj.ro

[2] Faculty of Civil Engineering, Technical University of Cluj-Napoca, 15 Constantin Daicoviciu Street, 400020 Cluj-Napoca, Romania

Abstract. Cementitious composite materials are the most used material in the realization of transport infrastructures. In order to increase the service life of these structures, it is necessary to design cementitious composite materials with special properties, such as the self-healing capacity, which allows the occurrence of a self-closing phenomenon after cracking. This paper shows the performance recorded for two cementitious composites with self-healing capacity induced by the use of an integral waterproofing admixture by mass crystallization. The experimental results obtained indicate a good self-healing capacity, quantified by a degree of healing of at least 57% after 192 h of conditioning, respectively, total closure of cracks after 336 h of conditioning. This work contributes to the increase of knowledge in the field of cementitious materials with self-healing capacity, indicating a possibility of obtaining this effect through the use of a waterproofing admixture by mass crystallization, simultaneously with the presentation of the possibilities of use of industrial waste such as fly ash and limestone slurry.

Keywords: Self-healing · Waterproofing by mass crystallization · Durability

1 Context

The life of building elements produced using cementitious composite materials is influenced by the external factors to which they are exposed and by current mechanical stresses or accidental overloads in operation, to which they are subjected. As a result of this combination of factors, the building elements made of cementitious compounds end up cracking, thus allowing the corrosive agents to penetrate the reinforcement, which corrodes it. Following the cracking of the cover represented by the cementitious composite, access routes (microcracks/cracks) are created towards the reinforcement. Through these cracks/microcracks, the corrosive agents from the outside come into contact with the reinforcement producing corrosion, which, over time, leads to the loss of the bearing capacity of the reinforcement, respectively of the structural elements because the reinforcement has the role of taking over the bending efforts that occur in the elements.

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The use of self-healing cementitious materials improves the durability performance and extends life cycle of the elements, without requiring costly and time-consuming repair of cracks. Although cracking cannot be avoided over the life of a structure, the ability of certain materials to heal these cracks makes them extremely valuable materials for the construction industry.

This phenomenon is called "self-healing (SH)". Autogenously crack healing phenomena can occur largely within cement-based materials through four mechanisms, including (1) calcite formation (calcium carbonate, $CaCO_3$), (2) continuous hydration of the cement matrix upon contact with moisture, (3) swelling of the cementitious matrix, and (4) particle sedimentation in the crack [1, 2]. SH results in the recovery of certain mechanical or durability properties of cement paste. Over the past two decades, numerous research studies have been conducted to establish the various characteristics of natural healing in cement-based materials [3, 4]. Although the concepts and mechanisms of autogenic and autonomous healing have been defined and are known, from a design and application perspective, it is necessary to evaluate the effectiveness of various self-healing technologies based on the intention of practical application [1, 5–11]. SH intrinsic has limited applications due to its limited crack closure capability (~200–300 μm) and the fact that the phenomenon occurs conditionally [12–14].

From the literature it is known that the self-healing mechanism can be initiated in: (1) concrete with mineral additions, (2) concrete with magnesium oxide addition, (3) improved concrete with crystalline mixtures, (4) reinforced concrete with high performance fibers improved with crystalline mixtures, (5) concrete with pre-placed microcapsules containing polymeric healing agent and (6) concrete with encapsulated bacteria [1, 15].

The general approach to research methodology indicates that methods of quantitative assessment of the self-healing capabilities of various technologies are needed. In particular, since cracks have a direct impact on durability performance rather than mechanical properties, it is very important to evaluate the recovery of durability performance through self-healing. In general, crack healing was evaluated using non-destructive testing (NDT) or microstructure analysis [16]. NDT, which has been performed using radiation testing [17], acoustic emission [18], ultrasonic testing [19, 20] and image analysis [21], is a relatively simple process, but has a low degree of reliability and limited applicability in the direct evaluation of mechanical or durability performance. Alternatively, water permeability tests were also used in the direct evaluation of self-healing durability perforation recovery [22] and, concurrently with the water absorption test [23], accelerated chloride diffusion tests [24, 25].

One of the methods of inducing self-healing ability is the addition of discontinuous and randomly dispersed fibers that can control the formation and limit the growth of cracks inside the matrix due to the bridging effect. They also provide sufficient support and time for any type of SH mechanisms to emerge later.

Among these synthetic fibers, polypropylene (PP) fibers are commonly used for reinforcing cement-based materials and have attracted the attention of researchers due to their relatively low cost and weight, being inert in a high pH environment, resistant to corrosion and cracking from shrinkage, allow easy mass dispersion, have high melting point and chemical stability [1, 26–28]. Studies have shown that the inclusion of fibers results in improved post-cracking behavior as well as less brittle concrete behavior.

Also, for the purpose of inducing self-healing capacity, the researchers investigated the effectiveness of introducing specific crystalline waterproofing admixtures (CWA) into concrete, in varying proportions, depending on the water-binder ratio. Since the durability of concrete is one of the most important properties, both financially and in terms of sustainability, reducing water penetration as much as possible should be a priority. A mix of the concrete, properly engineered, produced, and performed properly, with a low ratio of water-binding agent, resulting in a final product with low permeability and high durability [29, 30], but according to the literature it is recommended to be used in the design and preparation of the concrete has a water-cement ratio greater, because the high content of water in the mixture promotes the self-healing [22]. Azarsa et al. specifies that each type of CWA produces different crystals responsible for reducing the penetration of water and the occurrence of the self-healing phenomenon of concrete [31].

With regard to self-healing, the opening of cracks that can heal completely varies, depending on the mode of induction of this property, from 0.1 mm [31–33] to 0.3 mm or even 0.4 mm [31, 34]. As for other properties, CWA has been reported to improve resistance to freeze-thaw cycles, reduce chloride ion penetration [35], improve sulfate attack resistance and do not significantly affect the compressive strength of concrete [36].

The aim of the research activity carried out and presented in this paper is to evaluate the self-healing capacity of two cementitious composite mixtures produced using a waterproofing admixture of crystallization in mass, compared to a control mixture (produced without using this type of admixture).

2 Materials and Methods

The materials used for the preparation of the cementitious composite mixtures were: Portland cement EN 197-1-CEM I 42.5 R, produced at Aleşd cement factory, Bihor County, Romania; fly ash from the thermal power plant resulting from the burning of coal to obtain electricity at Mintia thermal power plant, Hunedoara, Romania; washed river aggregates (0–4) mm granular class; limestone slurry, resulting from the cutting of marble rock taken as a by-product of a marble processing plant in Cluj-Napoca, Romania, superplasticizing admixture type Master Glenium 51 (BASF), integral waterproofing admixture by crystallization (CWA), PVA fibers, 8 mm and water. The preparation of the mixtures was carried out in the laboratory NIRD URBAN-INCERC Cluj-Napoca Branch.

The research methodology consisted in following the following steps:

1. compositional design (according to Table 1) of two cement compositions with self-curing capacity (T1 and T2) and a control composition (TM);
2. the maturation of the specimens (reaching the age of 28 days after casting) was carried out by keeping in the first 24 h after casting, in metal patterns, at a constant temperature (20 ± 2) °C and relative humidity min. 90%, and subsequently, after

Table 1. Mix-design ratios for TM, T1, T2.

Mixtures	CEM [2]	FA [3]	Sand	Water	PVA	LS [4]	CWA[5]	Superplasticizer
TM	1,00	1,12	0,82	0,56	0,04	0,24	–	0,02
T1	1,00	1,11	0,82	0,56	0,04	0,24	0,01	0,02
T2	1,00	1,10	0,82	0,56	0,04	0,24	0,02	0,02

values reported to cement quantity, [2] CEM-cement; [3] FA-fly-ash, [4] LS-limestone slurry, [5] CWA – crystalize waterproof admixture

demolding, by immersing water with temperature (20 ± 1) °C, until the age of 28 days;

3. upon reaching the age of 28 days after casting, the tests for determination of compressive strength (R_c) and bending strength (R_{ti}) were performed, for which the results indicated in Table 2, expressed in N/mm^2, were obtained.

Table 2. Mix-design ratios for TM, T1, T2.

Mixtures	R_c	R_{ti}
TM	57.6	15.8
T1	58.2	16.5
T2	56.6	16.3

According to the mean value of the bending tensile strength, the cracking force required to induce the controlled cracking was determined, representing (87 ± 1) % of the mean bending tensile strength, respectively, P = 6000 [N].

4. evaluation of self-healing capacity by tracking the degree of closure of cracks. For this purpose, through microscopic evaluation, crack openings were measured, initially and subsequently, during exposure to conditioning. Conditioning in order to induce the self-curing effect consisted in exposure to alternative wet-dry cycles, a cycle consisting of immersion 16 h in water (20 ± 1) °C and 8 h kept in dry environment at (23 ± 2) °C.

For a better evaluation, the surface of the prisms were divided into several evaluation areas, depending on the cracking mode, these areas being kept throughout the evaluation process.

The mean healing degree ($GV^m{}_t$) was calculated as a percentage reduction in the mean crack opening at time t, compared to the mean crack opening initially recorded at the time of the crack, Eq. (1):

$$GV_t^m = (D_t - D_0) * 100/D_0(\%) \quad (1)$$

The degree of healing of the maximum crack opening ($GV^{M}{}_{t}$) was calculated as a percentage reduction of the maximum crack opening at time t, compared to the maximum crack opening initially recorded at the time of the crack, Eq. (2):

$$GV_t^M = (D_t - D_0) * 100/D_0(\%) \tag{2}$$

The mean moment healing degree ($GV^{Mm}{}_{t}$) was calculated as a reduction in the mean opening of the crack at time t, compared to the mean opening of the crack recorded in the previous stage, (t − 1), in relation to the length of time spent on conditioning, Eq. (3):

$$GV_t^{Mm} = (D_t - D_{t-1})/(t - (t - 1))\ (\mu m/h) \tag{3}$$

Where: D_0 - mean crack opening, measured immediately after the crack (mm); D_t - mean crack opening measured after t hours of conditioning (mm); D_{t-1} - the average opening of the crack, measured in the previous step (mm); t – conditioning time (24 h; 96 h; 192 h; 336 h; 480 h).

3 Results and Discussions

Evaluation of the self-healing capacity for the projected mixtures T1, T2 and TM following microscopic crack analysis and to evaluate the opening of cracks at set time intervals, namely: at the time of cracking at 24 h, 92 h, 192 h, 336 h and 480 h, the surface of the prism was divided into 9 zones for the composition T1, 20 zones for the composition T2 and 16 zones for the composition TM.

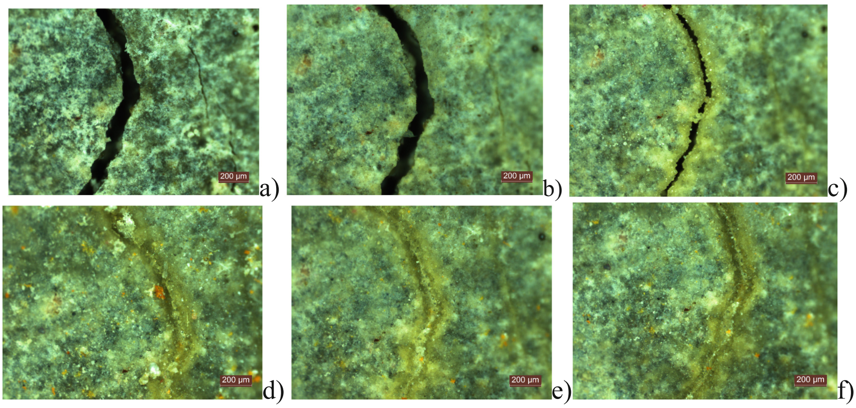

Fig. 1. Example of evolution of self-curing phenomenon for T1 composition: a) induced crack appearance; b) crack appearance after 24 h conditioning for self-curing; c) crack appearance after 96 h self-healing conditioning; d) self-healing crack appearance after 192 h self-healing conditioning; e) self-healing crack appearance after 336 h self-healing conditioning; f) self-healing crack appearance after 480 h self-healing conditioning.

After the initial analysis of the cracks present on the sample composition T1, T2 and TM, they were subjected to wet-dry cycles (16 h wet – 8 h dry), during which the evolution of the cracks was also followed at the predetermined time intervals (24 h, 96 h, 192 h, 336 h, 480 h).

The evolution of the self-curing process of a crack (composition T1) during 480 h of exposure to conditioning in order to induce the self-curing effect is exemplified is shown in Fig. 1.

To induce the cracking state for the mixtures T1, T2 and TM a cracking force of 6000 N was applied, representing (86–88%) of the maximum cracking force.

Experimental results for mixture T1 show that:

- following the cracking, depending on the area assessed, it is observed that cracks with variable openings were obtained;
- the maximum initial opening of the cracks falls within the range (0.139–0.033) mm and decreases as the conditioning period passes, Fig. 2 and Fig. 3;
- the average initial opening of the cracks falls within the range (0.1049–0.031) mm and decreases as the conditioning period passes, Fig. 2 and Fig. 3;
- the healing degree of the maximum opening of the cracks, ($GV^{M}{}_{t}$), increases as the conditioning period passes, reaching 100%, after 96 h in the case of zones 4, 6 and 8, after 192 h in the case of zones 5 and 7, after 336 h in the case of zone 9, after 480 h in the case of zone 1, remaining open with a maximum opening of 0.063 mm, Fig. 4;
- the average degree of healing of the identified cracks, ($GV^{m}{}_{t}$), increases as the conditioning period passes, reaching 100%, after 96 h in the case of Zones 4, 6, 8, after 192 h in the case of Zones 5 and 7, after 336 h in the case of Zone 9, after 480 h in the case of Zone 1, remaining open, with the final average healing degree of 91.42%, Fig. 5;

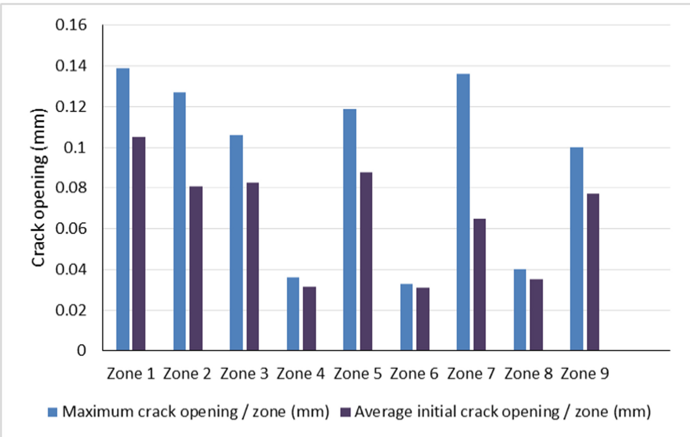

Fig. 2. Maximum opening and average opening of cracks, values measured immediately after cracking.

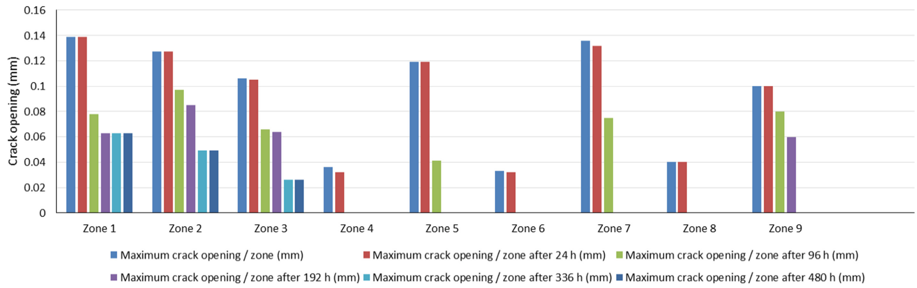

Fig. 3. Evolution of maximum crack opening during conditioning.

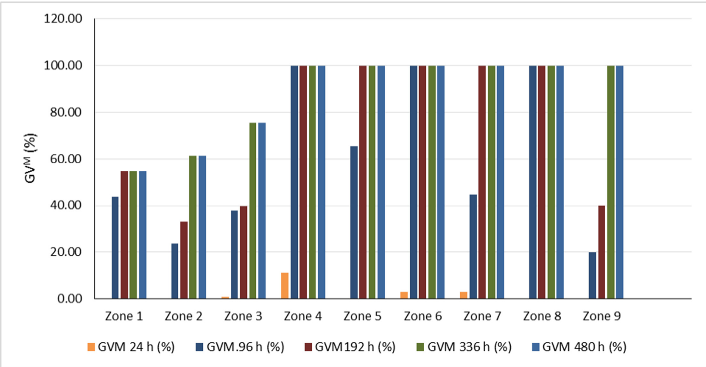

Fig. 4. Evolution of the healing degree calculated for the maximum opening of the cracks, during T1 conditioning

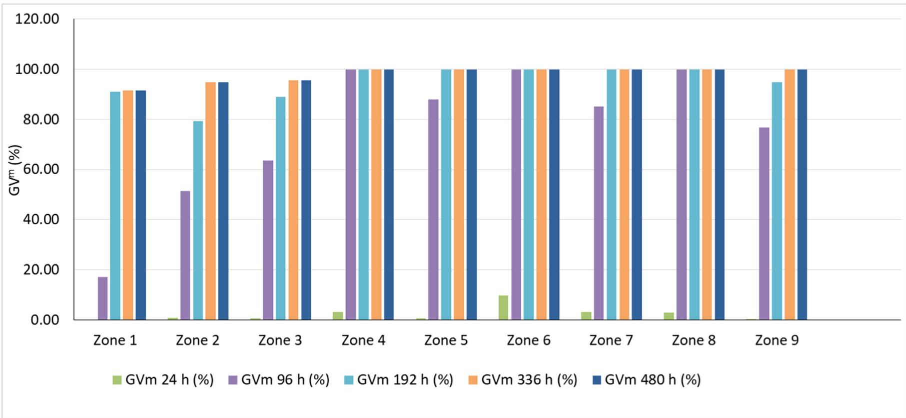

Fig. 5. Evolution of the healing degree calculated for the average opening of the cracks, during T1 conditioning.

– The average moment healing rate ($GV^{Mm}{}_t$) is a measure of the closing speed of the crack between two conditioning time intervals and indicates that in the first hours of conditioning (maximum 96 h) the closing speed is higher, in contrast to the subsequent development of the self-healing phenomenon that occurs at a lower speed, Fig. 6. Exception to this finding shall be made only in the case of the indicator calculated for zone 1. This kinetically delaying behavior of the crack closing process was attributed to the large opening of this crack characterized by an average initial opening of 104.9 μm.

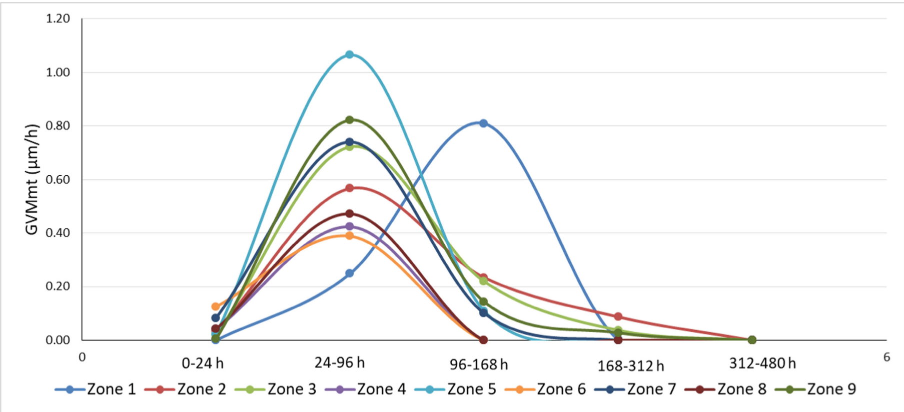

Fig. 6. Evolution of the kinetic indicator of the healing process, average degree of moment healing ($GV^{Mm}{}_t$) T1

Experimental results for T2 mixture show that:

– following the cracking, depending on the area assessed, it is observed that cracks with variable openings were obtained;
– the maximum initial opening of the cracks falls within the range (0.085–0.040) mm and decreases as the conditioning period passes, Fig. 7 and Fig. 8;

– The healing degree of the maximum crack opening, ($GV^{M}{}_t$), increases as the conditioning period passes, up to 100%, after 96 h in the case of Zones 16, 18, 19, 20, after 192 h in the case of Zone 1, 3, 8, 9, 10, 11, 12, 13, 14 17, after 336 h in the case Zone 2, after 480 h in the case of Zone 4. Only cracks in Zone 5 remained open with an average opening of 0.048 mm, cracks in Zone 6 with a maximum opening of 0.060 mm, Zone 7 with a maximum opening of 0.051 mm and Zone 17 with a maximum opening of 0.079 mm, see Fig. 9.

– The average degree of healing of the identified cracks, ($GV^{m}{}_t$), increases as the conditioning period passes, reaching 100%, after 96 h in the case of Zones 16, 18, 19,

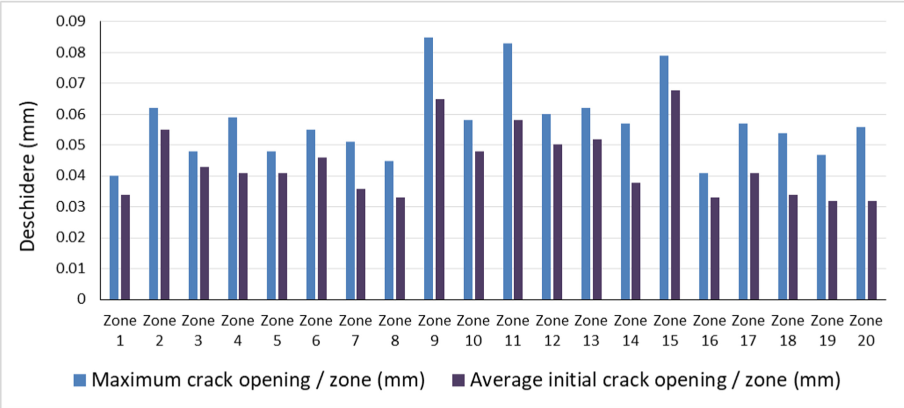

Fig. 7. Maximum opening and average opening of cracks, values measured immediately after cracking.

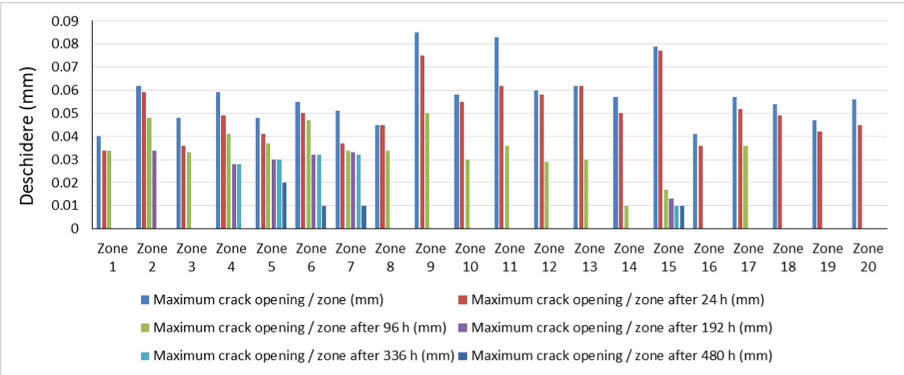

Fig. 8. Evolution of maximum crack opening during conditioning.

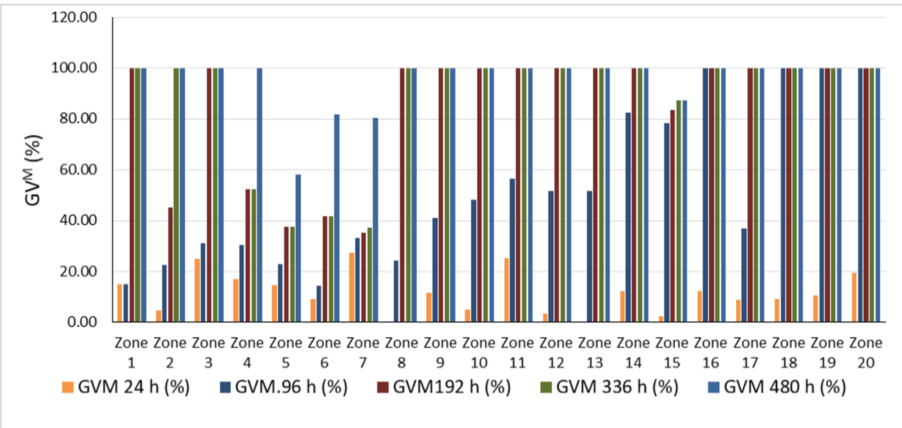

Fig. 9. Evolution of the healing degree calculated for the maximum opening of the cracks, during T2 conditioning.

20, after 192 h in the case of Zones 1, 3, 8, 9, 10, 11, 12, 13, 14 17, after 336 h in the case of Zone 2, after 480 h in the case of Zone 4. Cracks in Zone 5 remained open with an average 97.56%, Zone 6 with 80.43%, Zone 7 with 82.22%, and Zone 17 with 98.52%, Fig. 10.

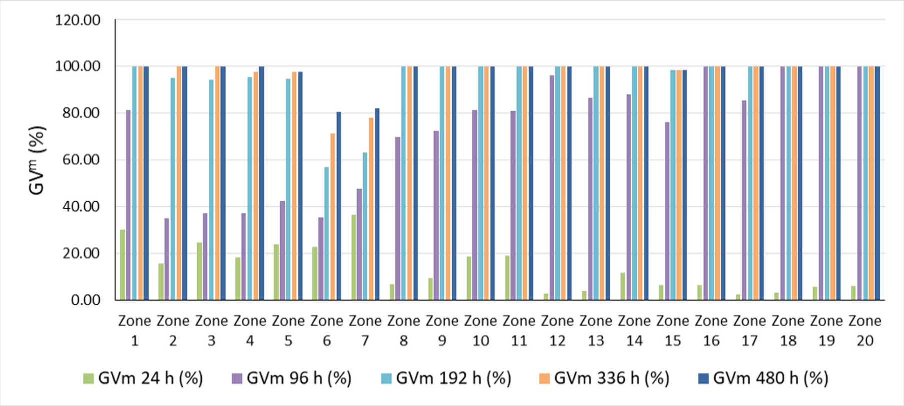

Fig. 10. Evolution of the healing degree calculated for the average opening of the cracks, during T2 conditioning.

– The average moment healing rate ($GV^{Mm}{}_t$) is a measure of the closing speed of the crack between two conditioning time intervals and indicates that in the first hours of conditioning (maximum 96 h) the closing speed is higher, in contrast to the subsequent development of the self-healing phenomenon that occurs at a lower speed, Fig. 11. Exception to this finding shall be made only in the case of the indicator calculated for Zones 5, 6, 7 and 15. This kinetically delaying behavior of the crack closing process was attributed to the large opening of this crack characterized by an average initial opening of between 48 and 79 μm.

Experimental results for TM mixture show that:

– as a result of cracking, according to the assessed area it is observed that cracks with variable openings have been obtained;
– the maximum initial opening of the cracks falls within the range (0.109–0.033) mm and decreases as the conditioning period passes, Fig. 12 and Fig. 13;
– the average initial opening of the cracks falls within the range (0.830–0.029) mm and decreases as the conditioning period passes, Fig. 12 and Fig. 13.

– The healing degree of the maximum crack opening, ($GV^{M}{}_t$), increases as the conditioning period passes, up to 100%, after 192 h in the case of Zones 8, 9, 10, 15, after

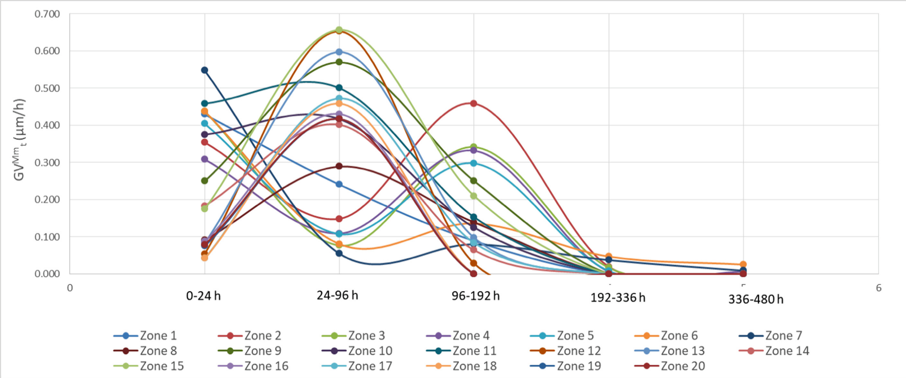

Fig. 11. Evolution of the kinetic indicator of the healing process, average degree of moment healing ($GV^{Mm}{}_{t}$) T2.

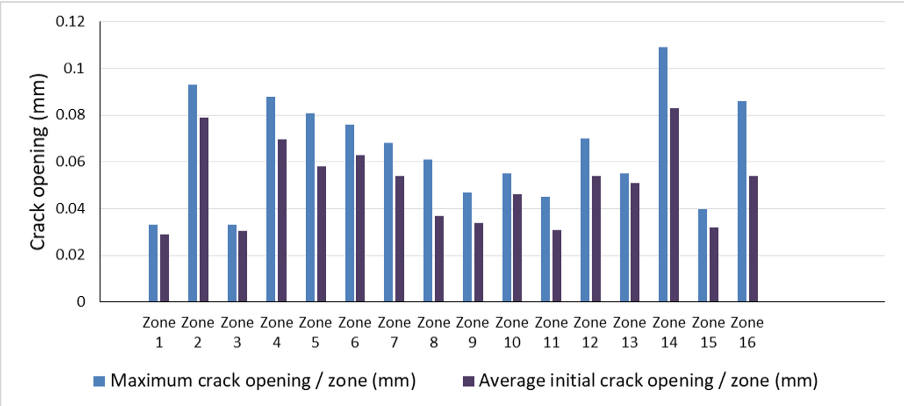

Fig. 12. Maximum opening and average opening of cracks, values measured immediately after cracking.

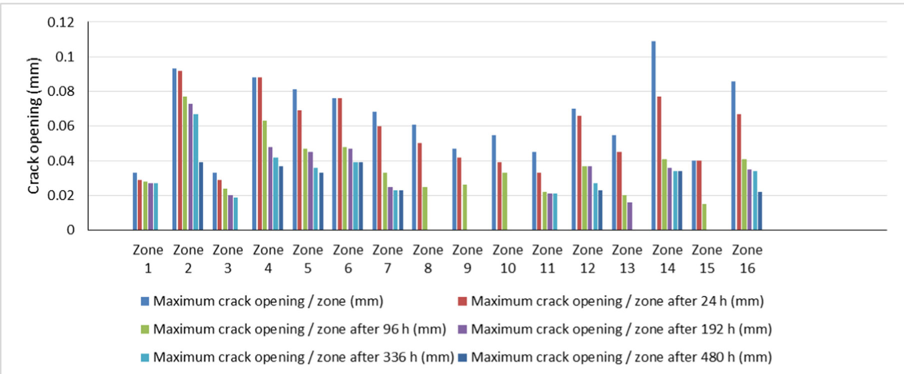

Fig. 13. Evolution of maximum crack opening during conditioning.

336 h in the case Zone 13, after 480 h in the case of Zones 1, 3 and 11. Cracks in Zones 2, 4, 5, 6, 7, 12, 14 and 16 remained open with an average opening between 0.068 and 0.109 mm, see Fig. 14.

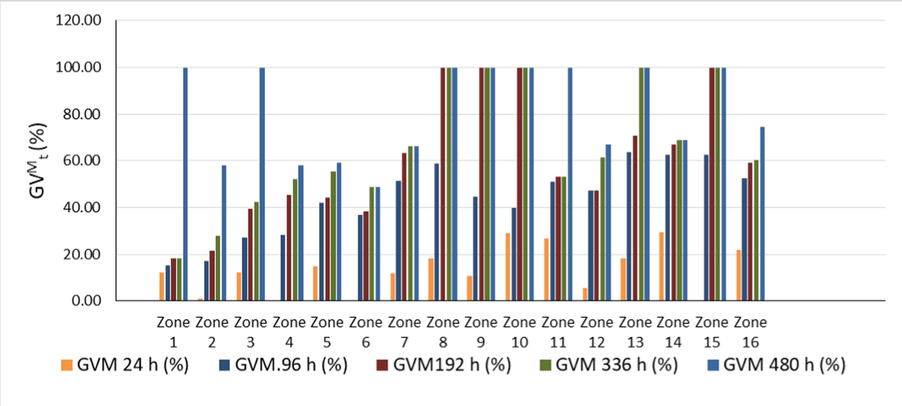

Fig. 14. Evolution of the healing degree calculated for the maximum opening of the cracks, during TM conditioning.

– The average degree of healing of the identified cracks, ($GV^m{}_t$), increases as the conditioning period passes, reaching 100%, after 192 h in the case of Zones 8, 9, 10 and 15, after 336 h in the case of Zone 13, after 480 h in the case of Zones 1, 3 and 11. Cracks that remained open had an average between 77.78% and 98.15%, Fig. 15.

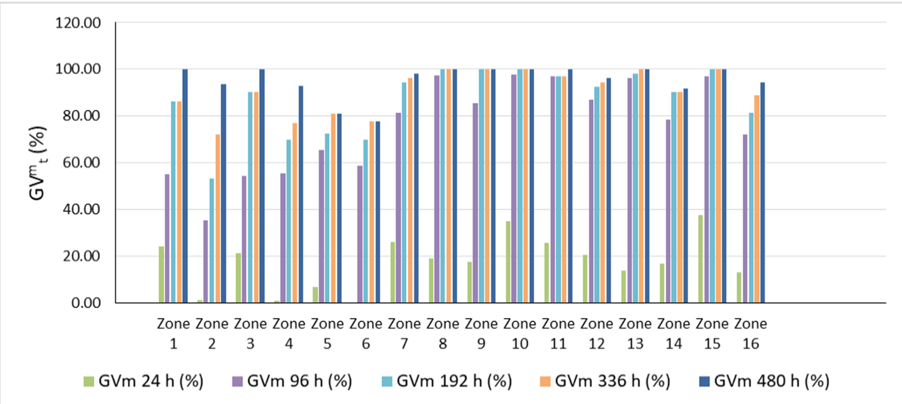

Fig. 15. Evolution of the healing degree calculated for the average opening of the cracks, during TM conditioning.

– The average moment healing rate ($GV^{Mm}{}_t$) is a measure of the closing speed of the crack between two conditioning time intervals and indicates that in the first hours of conditioning (maximum 96 h) the closing speed is higher, in contrast to the subsequent development of the self-healing phenomenon that occurs at a lower speed, Fig. 16. Exception to this finding shall be made only in the case of the indicator calculated for Zones 2, 4, 5, 6, 7, 12,14 and 16. This kinetically delaying behavior of the process of closing the cracks was attributed to the lack in the composition of the cementitious composite material of the integral waterproofing admixture by crystallization.

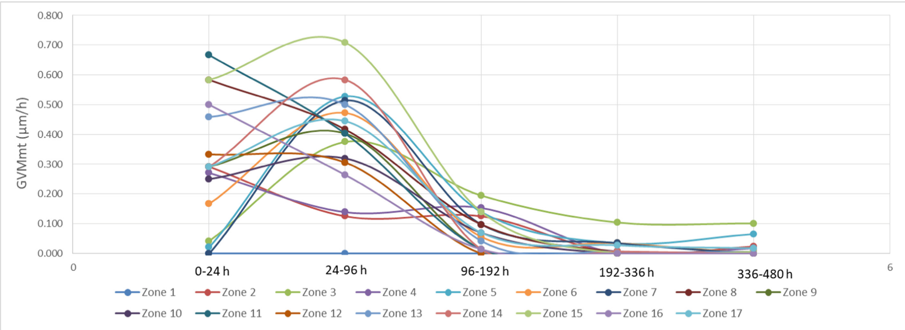

Fig. 16. Evolution of the kinetic indicator of the healing process, average degree of moment healing ($GV^{Mm}{}_t$) TM

4 Conclusions

The experimental results obtained indicate a good self-healing capacity, quantified by the following healing degrees:

1. Control sample mixture (TM):

   a. Maximum crack opening:

      (1) Average of 40.55% after 96 h of conditioning;
      (2) Average of 56.80% after 192 h of conditioning;
      (3) Average of 62.18% after 336 h of conditioning;
      (4) Average of 76.63% after 480 h of conditioning;

   b. Average crack opening:

      (1) Average of 78.85% after 96 h of conditioning;
      (2) Average of 87.20% after 192 h of conditioning;
      (3) Average of 90.69% after 336 h of conditioning;

        (4) Average of 95.63% after 96 h of conditioning;

2. Mixture T1:

    a. Maximum crack opening:

        (1) Average of 59.52% after 96 h of conditioning;
        (2) Average of 74.15% after 192 h of conditioning;
        (3) Average of 87.95% after 336 h of conditioning;
        (4) Average of 87.95% after 480 h of conditioning;

    b. Average crack opening:

        (1) Average of 75.78% after 96 h of conditioning;
        (2) Average of 94.92% after 192 h of conditioning;
        (3) Average of 97.99% after 336 h of conditioning;
        (4) Average of 97.99% after 96 h of conditioning;

3. Mixture T2:

    a. Maximum crack opening:

        (1) Average of 52.09% after 96 h of conditioning;
        (2) Average of 84.79% after 192 h of conditioning;
        (3) Average of 87.82% after 336 h of conditioning;
        (4) Average of 97.94% after 480 h of conditioning;

    b. Average crack opening:

        (1) Average of 72.61% after 96 h of conditioning;
        (2) Average of 94.91% after 192 h of conditioning;
        (3) Average of 97.15% after 336 h of conditioning;
        (4) Average of 97.94% after 96 h of conditioning;

Results obtained on the cementitious composite samples using integral waterproofing admixture by mass crystallization show the effectiveness of using this type of admixture in producing the self-healing effect of the cementitious composites and also speeding the process, thus obtaining very good results.

This work contributes to the increase of knowledge in the field of cement materials with self-healing capacity, indicating a possibility of obtaining this effect through the use of a waterproofing additive by mass crystallization, simultaneously with the presentation of the possibilities of use of industrial waste such as fly ash and limestone slurry.

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