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Experimental investigation on the ability of macro-encapsulated polyurethane to resist cyclic damaging actions in self-repaired cement-based elements

Giovanni Anglani[1], *Jean-Marc* Tulliani[2], and *Paola* Antonaci[1,3*]

[1]Politecnico di Torino, Department of Structural, Geotechnical and Building Engineering, Corso Duca degli Abruzzi, 24, 10129, Turin, Italy
[2]Politecnico di Torino, Department of Applied Science and Technology, INSTM Research Unit PoliTO-LINCE Laboratory, Corso Duca degli Abruzzi, 24, 10129, Turin, Italy
[3]Politecnico di Torino, Responsible Risk Resilience Centre, Viale Mattioli 39, 10125 Torino, Italy

Abstract. The use of polymer precursors as repairing agents in capsule-based self-healing systems has been extensively studied in recent years. In particular, the effectiveness of macro-encapsulated polyurethane in restoring both watertightness and mechanical properties has been demonstrated at the laboratory level, and the experimental methods to test the effectiveness have been validated following pre-standard procedures. However, the use of macro-capsules containing polyurethane precursors for field applications has not been sufficiently implemented yet. For these systems to become appealing to the construction industry, it is essential to further characterize the self-healing effect in terms of stability in time, namely, to investigate the behavior of the self-healing system when subjected to recurring actions that can affect structures in time, after cracking and subsequent self-repairing. The goal of this study was to characterize the ability of commercial polyurethane foams to withstand cyclic flexural actions and repeated temperature variations after release from cementitious macro-capsules embedded in mortar specimens. The specimens were tested immediately after pre-cracking and self-repairing to characterize the initial sealing efficiency through a water-flow test. The same test was repeated at prescribed time intervals to analyze the evolution of the sealing efficiency with the applied mechanical and thermal stresses. The results showed that the proposed system has good stability against the selected damaging actions and confirmed the potential of encapsulated polyurethane for self-healing applications.

1 Introduction

Capsule-based self-healing is receiving increasing attention in the field of R&D for the construction industry because of its great potential to improve the durability and sustainability of reinforced concrete structures [1, 2]. Polymers, and especially polyurethane (PU) precursors, have been widely used in micro- and macro-encapsulated systems due to their relatively fast reaction time upon capsule triggering, and to their consequent ability to seal the crack timely and effectively. The self-sealing effect provided by these polymeric agents has been proved to be successful in blocking the ingress of water through the crack path, in order to prevent the diffusion of water-driven aggressive substances that could attack the concrete matrix and/or the steel reinforcement [1-3]. In addition, their capability to provide also a self-healing effect in terms of restoration of the mechanical properties has been investigated in a few studies, mainly under static conditions [4]. However, the actual loading conditions in real structures can be considerably more complex than just static, such as in the case of low- and high-cycle fatigue (e.g., in industrial buildings), impact and moving loads (e.g., in transport infrastructures), dynamic actions due to high-speed wind and/or oceanic waves (e.g., in tall buildings and offshore structures), etc. Moreover, the environmental conditions along the structures lifetime can also vary significantly, resulting in an additional cause of degradation of the material properties (and self-healing performance) in time. Therefore, it is crucial not only to characterize the self-healing ability upon first appearance of the crack, but also to assess the stability of the self-healed elements in time, under the effects of different time-varying physical and mechanical actions.

Some studies have already been conducted in this direction as reviewed in [5], including: the analysis of the response of pre-cracked engineered cementitious composites to sustained [6] or increasing sustained [7] loads; the study of the stability of different healing products in pre-cracked concrete under freezing/thawing conditions [8]; the investigation on the repeatability of the self-healing action in bacteria-based systems [9] and stimulated autogenous healing systems, with crystalline admixtures [10] and natural fibers [11]. With specific reference to self-healing systems based on encapsulated polymers, only few

* Corresponding author: paola.antonaci@polito.it

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studies have addressed the problem of assessing the robustness and reliability under realistic conditions, hence in the presence of repeated physical and/or mechanical loading. Among these, Feiteira et al. [12] investigated the capacity of flexible polymers to carry increasing strain steps in three-point-bending, in order to characterize their suitability to be used in autonomous healing systems with realistic moving cracks. Anglani et al. [13] examined the capability of self-repaired capsule-based systems (with an expansive polyurethane as a healing agent) to resist cyclic flexural loading steps with increasing intensity.

The research presented in this paper was carried out in continuity with [13], with the aim of exploring the capability of commercial polyurethanes to withstand both cyclic flexural actions and repeated temperature variations after release at the crack site from autonomous capsule-based systems. In fact, both flexural and thermal actions could cause successive opening and closing of the crack, thus potentially damaging the interface between the polyurethane and the cementitious matrix, and impairing the self-healing effect. Two different single-component polyurethane precursors were used (namely a high-viscosity resin with integral catalyst and a low-viscosity resin without catalyst), to study the effect of different polymer characteristics on the stability of the self-healing performance under repeated mechanical and thermal actions. In addition, to simulate the effect of long-term storage of the healing agent inside the capsule (as in the case of late-age cracking), the high-viscosity resin with integral catalyst was encapsulated in two conditions: within six months from the date of delivery from the retailer (i.e., within the product shelf-life as indicated by the producer) and more than three years after delivery and storage in non-controlled indoor conditions. Full details on the materials and methods used are reported in Section 2, while the results achieved are shown and discussed in Section 3. Based on these results, the main conclusions are drawn in Section 4.

2 Materials and methods

2.1 Materials

The macrocapsules used for this study had a tubular shape, with a length of 5 cm and an internal diameter of 6 mm. They had a cementitious shell, end plugs manufactured with an epoxy plaster, and a waterproofing internal coating made of epoxy resin. They were produced by rolling according to the procedure described in [3,13]. As anticipated in Section 1, two different polymeric healing agents were introduced in such capsules, namely, a high-viscosity polyurethane precursor with integral catalyst and a low-viscosity resin without catalyst. They were both commercial products, manufactured by Minova CarboTech GmbH and commercialized under the names of Carbostop U and Carbostop F, respectively. Both being single-component water-reactive resins suitable for concrete crack sealing, they differ for the presence/absence of reaction accelerator, as well as in their viscosity (270–1000 mPa*s vs. 120–200 mPa*s at 25 °C) and foaming factor (up to 60 or 40, respectively, in free rise conditions and for a range of temperatures between 5 °C and 25 °C). The incorporation of a reaction catalyst in Carbostop U makes it more susceptible to premature hardening inside the capsule in case of late-age cracking. For this reason, it was decided to encapsulate it in two conditions: within six months from the date of delivery from the producer and more than three years after delivery. In the first case, it was ensured that the product was used within its shelf-life as indicated by the producer; in the second case, the prolonged storage of the product in non-controlled indoor conditions before use simulated the condition of late age cracking, with potential inactivation of the healing agent due to premature reaction. The specimens containing these capsules were denoted as CAU′ and CAU″, respectively, while those that contained Carbostop F-core capsules were labelled as CAF. Reference specimens without capsules were also produced for the sake of comparison. They were designated as REF.

All the specimens were mortar prisms measuring 4×4×16 cm^3. They were manufactured with a standard mortar mix composition having a water-to-cement ratio of 0.5 and a sand-to-cement ratio of 3, in agreement with EN 196-1. Portland cement (CEM II 42.5 A/LL, Buzzi Unicem S.p.A.), normalized sand (grading 0–2 mm, DIN EN 196-1), and tap water were used. The fresh mix was cast in prismatic steel molds fitted out for the insertion of: 1) a crescent profile placed at the bottom of the mold, that allowed the creation of a transverse U-shaped notch at mid-span of the bottom face of the specimen upon demolding; 2) a removable circular bar with a diameter of 5 mm, placed in longitudinal direction at 25 mm from the bottom face of the specimen: once removed, it allowed the creation of a cast-in hole for the subsequent water-flow test; 3) nylon threads placed at 10 mm from the bottom face of the specimen, that allowed a precise positioning of one capsule per specimen of the CAU′, CAU″, and CAF series.

The specimens were stored in the molds covered in plastic foils for one day, then they were cured in a humidity-saturated environment under plastic sheets up to the age of one week. After curing, to complete the specimen preparation for the subsequent water-flow tests, the cast-in hole was enlarged of 1 mm in diameter over a length of (25 ± 5) mm by drilling, then a short plastic tube was inserted into it and fixed to the specimen using silicone. The other side of the cast-in hole was finally sealed with a silicone plug.

2.2 Methods

Pre-cracking of the specimens was performed at the age of one week by means of three-point-bending tests with a span of 100 mm. A 250 kN closed-loop servo-controlled MTS hydraulic press was used for this purpose. The crack mouth opening displacement (CMOD) was controlled via a clip-on gauge (DD1, HBM GmbH), with a test speed of 3 μm/s. The residual

crack width upon unloading was measured through a stereomicroscope (SMZ18, Nikon Inc.). After pre-cracking, the specimens were stored in lab for one day, to allow the polyurethane foam to completely cure in air. Subsequently, a first water-flow test was performed on all the pre-cracked self-healed and reference specimens, in accordance with the pre-standard procedure described in [1,3]. To do so, first the lateral faces of the crack were sealed with silicon, in order to leave only the crack mouth open; then the specimens were saturated by submersion in demineralized water for 24 hours, and afterwards they were removed from the water and pat-dried on the surface; finally they were connected to an open water reservoir through the plastic tube. The water head was kept constant at (50 ± 2) cm by continuously refilling the reservoir with demineralized water. The water leaked out of the crack mouth during 7 minutes was recorded by a precision balance (P700, Exacta Optech S.r.L.) connected to a PC. The data recorded during the first minute were discarded to remove the transient phase due to the presence of air bubbles, and achieve a regular steady-state flow.

After the first water-flow test, selected specimens were subjected to static re-loading in three-point-bending with the same equipment and testing procedure as for the pre-cracking test. In this way, it was possible to estimate the regain in mechanical strength due to self-healing, and set the test parameters for the subsequent cyclic flexural tests, as described below.

Cyclic flexural tests were conducted on a subset of the CAU′, CAU″, and CAF series, after removal of the silicon sealing applied to the lateral faces of the crack. These tests were performed in force-controlled mode by means of the same 250 kN hydraulic press used for the static pre-cracking and re-loading tests. A sinusoidal law was set for the cyclic loading, with minimum and maximum values defined as 10% and 70% of the expected failure load of the self-healed samples, respectively. This range of min/max flexural loads was selected in order to be sufficiently severe, thus expectedly causing the rupture of the specimen by fatigue for a relatively short number of cycles. The load frequency was set to 3 Hz. During the test, the cyclic variation of the crack width was also monitored through an inductive-bridge displacement transducer (WI5, HBM GmbH) mounted across the crack on one of the lateral faces of the specimen, at the level of the top of the notch. The cyclic flexural testing was repeated in 8 sequences of increasing duration (500; 1,000; 2,000; 5,000; 10,000; 20,000; 40,000; and 71,500 cycles) up to a max. cumulative no. of cycles equal to 150,000 – unless fatigue failure occurred sooner. At the end of each cyclic flexural sequence, the lateral faces of the crack were sealed again with a silicon sealant and the water-flow test was performed again on the same specimens (after 24 hours of water submersion), to monitor the possible degradation of the self-healing-induced watertightness as a consequence of the repeated mechanical stresses.

The remaining specimens were subjected to sequences of thermal cycles by means of a curing cabinet (10-D1429/AT, Controls S.p.A.). Each thermal cycle had a total duration of 24 hours and consisted of an isotherm of 8 hours at -20 °C, a heating ramp of approx. 0.3 °C/min up to 50 °C, another isotherm of 8 hours, and a cooling ramp of approx. 0.3 °C/min down to -20 °C. This range of min/max temperatures was selected in such a way to: a) be representative of real (extreme) conditions, and b) avoid impairing the stability of the polyurethane foam per se, since it is known that the polymer decomposition, involving chain rupture and a decrease of the average molecular weight as well as of the tensile strength, usually happens at much higher temperatures, from 150 °C to 200 °C [14], which are not realistic for most civil engineering applications. At the same time, the selected range of min/max temperatures gives rise to a relatively high thermal gradient, which can accelerate the degradation of the interface bond between the polymeric product and the mortar matrix with respect to the ordinary environmental conditions, due to the higher differential temperature-induced deformation of the two materials. An acceleration factor AF can be calculated following the Coffin-Manson model [15], as:

$$AF = \left(\frac{\Delta T_{test}}{\Delta T_{use}}\right)^{\beta} \quad (1)$$

where ΔT_{test} is the temperature cycling difference in accelerated laboratory conditions, ΔT_{use} is the usual daily temperature range in real service conditions, and β is the Coffin-Manson exponent, here assumed with a typical value of 3 [16].

The same way as in cyclic mechanical testing, also cyclic thermal tests were conducted in steps of increasing duration (from 11 to 32 days), each time with the interposition of a water-flow test aimed at following the evolution of the watertightness under the effect of the repeated thermal stresses.

3 Results and discussion

3.1 Pre-cracking

The pre-cracking test allowed to create a single flexural crack at mid-span of each specimen, with residual widths as reported in Table 1. On average, the specimens of the CAU′ and CAU″ series experienced a higher residual crack width compared to the CAF and REF series. This could be ascribed to the higher foaming factor of their healing agent, which generated a greater expansive action on the crack faces and partially prevented the elastic reclosure of the crack upon unloading.

Table 1. Residual crack width after pre-cracking (average values and coefficients of variation over at least 10 specimens, 5 measurements per specimen)

Series	Residual crack width	
	Average value	CoV
REF	381 µm	4%
CAF	309 µm	7%
CAU′	517 µm	5%
CAU″	465 µm	7%

The capsule breakage was normally identified by a visible drop in the Load vs. Displacement curve. Although the capsule triggering was always correctly obtained through the pre-cracking test, in some cases the system failed in leaking out the healing agent at the crack site. Specifically, this happened in 4 cases out of 16 in the CAU″ series and could be interpreted as an undesirable consequence of the prolonged storage of the healing agent before the execution of the pre-cracking test, which could have prevented the polymer precursor from reacting correctly upon capsule triggering. The specimens of the CAU″ series that showed this behavior were excluded from the subsequent experimental plan, because it was apparent that no self-healing effect could be manifested.

Regarding the influence of the capsules on the intrinsic material strength, a slight decrease was observed in the average value of the flexural peak load for the CAF, CAU′ and CAU″ series, that contained capsules, compared to the REF, that did not. Namely, an average value of 1,201 N was recorded vs. 1,319 N, respectively (i.e., 8.9% reduction). However, such decrement was not considered statistically significant, because an almost identical coefficient of variation (8.6%) characterized the dispersion of the peak load measurements themselves.

3.2 Initial self-healing performance

The initial self-healing performance was assessed with reference to both mechanical and durability properties. Specifically, the regain in mechanical properties was evaluated by means of a static flexural re-loading test as described in Section 2.2. Unlike other studies (see [17] as an extensive review), here it was decided to neglect the residual post-cracking load-bearing capacity, in order to express the mechanical regain with respect to the initial virgin status on average. Hence, the following expression was used for the Mechanical Efficiency indicator (ME) of each self-healing specimens under static flexural conditions:

$$ME = \frac{P''}{P'} \quad (2)$$

In Eq. 2, P'' denotes the max. flexural load of the single specimen in the re-loading test (i.e., after self-repair) and P' the average value of the peak load of all the specimens containing capsules in the pre-cracking test (i.e., the average value related to the intrinsic material strength). According to the data shown in Fig. 1, the values of ME found for the CAF, CAU′ and CAU″ series varied in the ranges of 40–59%, 50–89%, and 75–91%, respectively.

The unexpectedly positive performance of the CAU″ series compared to the CAU′ can be explained by considering that the prolonged storage of the polymeric healing agent in CAU″ might have affected some of its chemical-physical properties, causing in particular an increase of its viscosity, as stated by the producer. The increased viscosity could be responsible for a relatively reduced spreading of the healing agent on the crack faces, compensated by a reduction of the amount of product leaked out of the specimen itself, and most of all by the creation of a denser and more compact polyurethane foam, eventually leading to a higher flexural strength.

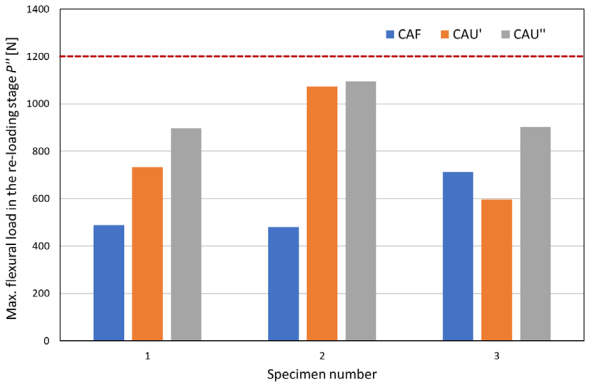

Fig. 1. Results of the static flexural re-loading test: max. flex. load P'' for the CAF, CAU′ and CAU″ series (blue, orange and grey bars) and average peak load P' (dashed red line)

Regarding the durability properties, a water-flow test was used to evaluate the initial self-healing performance in terms of regain of water-tightness, as suggested in [1] (see Section 2.2 for details on the test method). The test was performed immediately after pre-cracking and self-repair, and allowed to obtain the value of the initial Sealing Efficiency indicator (SE) for each of the specimens, according to the following expression:

$$SE = \frac{WF_{ref} - WF_{sh}}{WF_{ref}} \quad (3)$$

with WF_{ref} denoting the average water flow (g/min) of the reference specimens and WF_{sh} the water flow (g/min) of the single self-healing specimen.

The results are reported in Fig. 2. Average SE values of 79%, 74%, and 84% were obtained for the CAF, CAU′ and CAU″ series, respectively. One anomalous value with negative sealing efficiency was discarded for the CAU′ series, as its occurrence could be ascribed to an incomplete crack filling, coupled with a high residual crack width (553 μm), that was not considered representative of the general trend.

The best performance was achieved by the CAU″ series, followed by the CAF and then by the CAU′ series. A possible explanation could lie again in the different spreading of the healing agent on the crack faces, due to the different characteristics of the polymer precursors used.

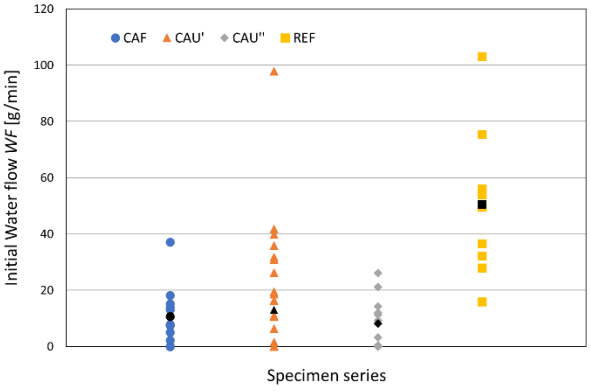

Fig. 2. Results of the initial water-flow test

On one side, the slightly lower foaming factor of Carbostop F (CAF series) could have avoided the partial loss of healing agent due to leakage out of the specimen compared to the CAU′ series (as well as it could have contributed to a achieve a smaller residual crack width). On the other, the same effect could have been obtained in the CAU′′ series because of the increased viscosity of the prolongedly-stored Carbostop U in CAU′′ compared to the CAU′ series, as discussed before.

3.3 Evolution under cyclic flexural tests

The stability of the self-healing performance under the effect of mechanical stresses was evaluated by first subjecting the pre-cracked and self-repaired specimens to up to 8 sequences of cyclic flexural tests (as described in Section 2.2), and then by assessing the sealing efficiency by means of water-flow tests performed at the end of each sequence. The sealing efficiency was calculated in accordance with eq. 3, where WF_{ref} still denotes the average water flow (g/min) of the reference specimens as recorded initially, immediately after pre-cracking, and WF_{sh} indicates the water flow (g/min) of the single self-healing specimen at the end of the given cyclic loading sequence.

A summary of the cumulative number of cycles endured by each specimen is reported in Table 2, together with the values of the SE indicators as a function of the test progression.

A gradual degradation of the sealing efficiency was expected as an effect of the cumulation of cyclic damage, but, on the contrary, quite variable trends were observed. In only one case (CAU′4) the sealing efficiency displayed an actual continuous decrease. In most of the others, an initially decreasing trend was followed by small oscillations and then by a clear increase. In all the cases where the initial sealing efficiency was 100%, it was kept constant throughout the test. Such a behavior could be partially explained by the fact that a part of the healing agent initially contained in the capsule could remain unreacted even after the capsule breakage, because of the relatively large quantity that could be stored in the capsule (up to 1.5 mL) and because of the insulation effect provided by the surrounding reacted polyurethane foam. The cyclic mechanical stress could have triggered a further reaction of this residual amount of healing agent, thus improving the sealing efficiency. Another concurring factor could be given by the clogging action exerted by the small debris and cement particles transported by the water during the repeated water-flow tests themselves. This effect should be significant especially in the cases where the initial sealing efficiency was already high (or the crack width small).

In general, the higher was the initial sealing efficiency, the higher was also the cumulative number of cycles to failure sustained by the specimens. This can reasonably be ascribed to an abundant and effective spreading of the healing agent over the crack faces, that contributed to both the mechanical and durability performance. One exception is represented by CAU′′6, that could not withstand any flexural cycle despite its initial SE value of 100%. In this case, it was observed that the polyurethane foam expanded entirely into the cast-in-hole without bridging the crack faces. Therefore, it was able to block completely the water flow, but could not contribute to the recovery of the load bearing capacity of the fractured sample.

Table 2. Cumulative number of flexural cycles to failure N_f and SE indicator as a function of the cyclic loading sequences

Spec.	N_f	Sealing Efficiency (%)								
		0	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
CAF1	*ongoing*	100	100	100	100	100				
CAF4	5,294	100	100	100	100	-				
CAF5	45	74	-	-	-	-				
CAF6	5,519	85	84	90	92	-				
CAU′′1	1,283	100	100	-	-	-				
CAU′′2	*ongoing*	78	77	88	91	90				
CAU′′4	*ongoing*	100	100	100	100	100				
CAU′′6	0	100	-	-	-	-				
CAU′1	52,701	100	100	100	100	100	100	100	-	-
CAU′3	69	48	-	-	-	-	-	-	-	-
CAU′4	1,716	79	64	59	-	-	-	-	-	-
CAU′5	1,369	61	72	-	-	-	-	-	-	-
CAU′6	21	37	-	-	-	-	-	-	-	-
CAU′8	53,858	68	48	52	53	53	66	73	-	-
CAU′9	45,675	100	100	100	100	100	100	100	-	-
CAU′10	>150k	100	100	100	100	100	100	100	100	100
CAU′14	9,052	100	100	100	100	100	-	-	-	-
CAU′15	>150k	100	100	100	100	100	100	100	100	100
CAU′16	>150k	87	78	69	100	100	100	97	99	100
CAU′17	>150k	79	72	73	77	89	89	88	88	94
CAU′19	>150k	78	82	81	97	100	95	94	96	98

3.4 Evolution under cyclic thermal tests

In analogy with the approach followed in Section 3.3, the stability of the self-healing performance under the effect of thermal stresses was evaluated by first subjecting the pre-cracked and self-repaired specimens to sequences of thermal cycles (as described in Section 2.2) and then by assessing the sealing efficiency by means of water-flow tests performed at the end of each sequence. The duration of each thermal cycling sequence was set to 11, 25, or 32 days (i.e., 11–32 cycles, one cycle per day). Based on the test parameters adopted, Equation 3 allowed to calculate an acceleration factor AF related to the thermal cycling experiment as $AF \cong 100$. Accordingly, the duration of a thermal cycling sequence of 11 days corresponded to 3 years of real (ordinary) thermal variation exposure, 22 days of cumulative thermal cyclic testing corresponded to 6 years, and finally 90 days corresponded to 25 years.

Table 3 shows the evolution of the sealing efficiency as a function of the number of thermal cycles (or else, as a function of the estimated years of service life in ordinary conditions). The same way as for the cyclic flexural loading, also in the case of the thermal cycling the trend of the sealing efficiency is not uniformly decreasing with progression of the test. In fact, most of the specimens of both CAF and CAU′′ series experienced an improvement in their sealing efficiency with increasing number of thermal cycles, (while the CAU′ series generally displayed a decrement).

The explanation for this behavior could lie again in the possible obstruction created by loose sand grains or cement particles that may be displaced during the repeated water-flow tests, and may accumulate between the crack faces or at the mortar-polyurethane interface. To check this, also some specimens of the REF series were subjected to the same testing procedure, and their sealing efficiency at the end of the generic thermal cycling sequence was expressed as a function of their current and initial water flow values. Indeed, an increase in the sealing efficiency (from the initial zero value) was found also in the REF samples, although their permeability remained significantly higher than in the self-healing series.

Table 3. Sealing Efficiency indicator as a function of the thermal cycling sequences

Spec.	Sealing Efficiency (%)			
	0 cycles *0 years*	**11 cycles** *3 years*	**22 cycles** *6 years*	**90 cycles** *25 years*
CAF8	72%	87%		
CAF9	70%	83%		
CAF10	96%	98%		
CAF11	85%	93%		
CAF12	90%	97%		
CAU″8	94%	98%		
CAU″9	99%	91%		
CAU″10	82%	92%		
CAU″11	58%	67%		
CAU″12	72%	86%		
CAU′20	39%	31%	38%	
CAU′21	100%	95%	92%	
CAU′22	100%	99%	99%	
CAU′23	97%	94%	94%	
CAU′24	-94%	-79%	-40%	
REF1	0%	23%	42%	
REF2	0%	15%	39%	
REF3	0%	-5%	35%	
REF4	0%	20%	48%	

4 Conclusions

The ability of macro-encapsulated polyurethane to resist cyclic damaging actions in pre-cracked, self-repaired mortar elements was experimentally investigated in order to characterize the stability of the self-healing performance. Force-controlled cyclic flexural tests ranging between 10% and 70% of the estimated self-repaired strength and cyclic thermal tests with a temperature range between -20 °C and +50 °C were performed in successive sequences. Water-flow tests were executed at the end of each sequence to analyze the evolution of the sealing efficiency. A satisfactory response was observed for both the commercial polyurethane precursors used here as healing agents, highlighting their ability to resist flexural cycling (in several cases up to 150k cycles) without significantly decreasing their sealing efficiency. In many cases, even an improvement was recorded. A similar behavior was experienced also under thermal cycling conditions, although the research is still in progress and cannot allow to draw definitive conclusions.

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