Evaluation of test methods for self-healing concrete with macrocapsules by inter-laboratory testing

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Tim Van Mullem[1], Giovanni Anglani[2], Hanne Vanoutrive[3], Girts Bumanis[4], Chrysoula Litina[5], Marta Dudek[6], Arkadiusz Kwiecien[6], Abir Al-Tabbaa[5], Diana Bajare[4], Teresa Stryszewska[6], Robby Caspeele[1], Kim Van Tittelboom[1], Jean Marc Tulliani[2], Elke Gruyaert[3], Paola Antonaci[2], Nele De Belie[1]

1: Magnel-Vandepitte Laboratory, Department of Structural Engineering and Building Materials, Ghent University, Belgium

2: Politecnico di Torino, Italy

3: KU Leuven - Ghent Technology Campus, Belgium

4: Riga Technical University, Latvia

5: University of Cambridge, United Kingdom

6: Cracow University of Technology, Poland

Abstract

Self-healing of concrete is a promising way to increase the service life of structures. Innovative research is being performed, yet it is difficult to compare results due to a lack of standardised test methods. In the framework of the COST action SARCOS (CA15202) [1] six different inter-laboratory tests are being executed, in which different test methods are being evaluated for six self-healing approaches. Here, the results of the inter-laboratory test concerning mortar and concrete with macrocapsules filled with a polyurethane healing agent will be discussed. The specimens were manufactured in one laboratory and then shipped to the other five participating laboratories. All six laboratories evaluated two test methods: a water permeability test and a capillary water absorption test. For the water permeability test, mortar specimens were cracked and afterwards their crack width was controlled using an active control technique. Due to the active crack control, the crack width of 90% of the samples deviated by less than 10 µm from the target of 300 µm. This made it more straightforward to compare the permeability test results, which indicated a similar sealing efficiency for several of the laboratories. For the capillary water absorption test, concrete specimens were cracked in a crack-width-controlled three-point bending test setup without active control after unloading. Compared to the water permeability specimens, there was a lot more variation on the crack width of the capillary water absorption specimens. The variability on the crack width and differences in quality of waterproofing resulted in diverging findings in the capillary water absorption test.

Introduction

Self-healing concrete is a promising material which can repair its own defects, thereby overcoming the need for expensive manual repair interventions. Although a lot of tests have already been carried out on different types of self-healing concrete, it is often difficult to compare results from different studies as no standard methods are yet available to test the efficiency and the consequent enhancement of the self-healing concrete properties. Additionally, there are a lot of factors (age of cracking, type of crack, conditions of exposure and healing duration, through-crack stress state, etc.) which can influence the healing behaviour [2]. In the framework of the EU COST Action CA 15202 SARCOS six different inter-laboratory tests have been running to evaluate test methods to assess the efficiency of self-healing concrete. Each test focusses on a different healing mechanism. The inter-laboratory test which is described here focusses on self-healing mortar and concrete with macrocapsules. Six labs participated in this inter-laboratory test: Ghent University, Politecnico di Torino, Riga

Technical University, Cracow University of Technology, Cambridge University and KU Leuven - Ghent Technology Campus.
For the inter-laboratory test programme two different tests were evaluated: a water permeability test and a capillary water absorption test. To execute the water permeability test, mortar specimens were cast. Concrete specimens were cast to perform the water absorption test. Since the crack width has a large influence on the water permeability test results, an 'active' crack width control technique was used after cracking of the mortar specimens to limit the variation on the crack width [3]. A 'passive' crack width control technique [4], by means of crack width control during loading, was used for the concrete specimens. All specimens were cast by Ghent University and were then shipped to the participating labs. The test protocol and any deviations by a specific lab have been recorded [5].

Materials and methods

<u>Healing agent</u>

As healing agent a single-component polyurethane (PU) was used. The used agent has a low viscosity and polymerises when it comes in contact with moisture in the air or in the concrete. In order to prevent the polymerisation up until the moment of crack creation, the PU was encapsulated in well-sealed tubular glass capsules with an internal diameter of 3 mm. The length of the capsules was 55 mm for the mortar specimens and 60 mm for the concrete specimens. Samples containing glass capsules are referred to as CAPS, reference samples are referred to as REF.

<u>Water permeability test on mortar specimens</u>

To execute the water permeability test, unreinforced mortar specimens (40x40x160 mm) were made with a cast-in hole (ø 5 mm) positioned with its centre 15 mm from the bottom side [3]. The mortar consisted of: 519 kg/m³ CEM I 42.5 N, 258 kg/m³ water, 1313 kg/m³ sand (0-2 mm), 89 kg/m³ limestone filler, and 0.79 l/m³ superplasticizer. For the CAPS specimens two capsules were placed at a height of 5 mm above the bottom side of the specimen so that the distance between the cast-in hole and the capsules, and the distance between the capsules and the bottom side of the specimen would be approximately equal. A Carbon Fibre Reinforced Polymer (CFRP) laminate was glued on the top side of the specimens using epoxy resin. One side of the cast-in hole of the specimens was connected to the water flow test setup. The other side was sealed. At an age of 14 or 15 days the specimens were cracked until failure in a three-point bending test setup with a span of 10 cm. Both halves of the mortar specimen stayed together due to the CFRP. Immediately after cracking the specimens were placed with their crack face upwards and the crack width was restrained under the microscope using screw jacks until the target crack width of 300 μm was obtained. The crack width of a specimen was determined from five measurements at three different locations (15 measurements in total) along the crack mouth. The cracks at the side faces were sealed. Prior to executing the water permeability test, specimens were submerged in water for 24 h. The tube of the specimens was connected to a water reservoir at a height of 50±2 cm with respect to the cast-in hole. Water could only leak out of the bottom of the crack. The amount of leaked water was recorded over time for a minimum of 6 minutes to determine the water flow in g/min.

<u>Capillary water absorption test on concrete specimens</u>

To execute the capillary water absorption test, concrete specimens (60x60x220 mm) were made with 2 reinforcement bars (ø 3 mm) positioned at a height of 12 mm from the bottom. The concrete consisted of: 378 kg/m³ CEM I 42.5 N, 185.2 kg/m³ water, 743 kg/m³ sand (0-5 mm), 1013 kg/m³ gravel (2-8 mm), 58 kg/m³ limestone filler, and 1.33 l/m³ superplasticizer. For the CAPS specimens, 4 capsules with a length of 60 mm were placed just above the reinforcement, except for Lab 6 for which the CAPS specimens contained 5 capsules with a length of 49 mm, instead of 60 mm. A day before cracking, the specimens were provided with a notch (depth of 3-5 mm). At an age of 14-16 days the specimens were cracked in a crack-width-controlled three-point bending test setup, to obtain a target crack width of 300 µm. The crack width of a specimen was determined from five measurements at four different locations (20 measurements in total) along the crack mouth. After measuring the crack width, the specimens were submerged in water for 24 h, to allow for a complete polymerisation of the PU. Subsequently, the specimens were oven dried (40°C) for 2 weeks. The bottom and the sides of the specimens were waterproofed by using aluminium tape, except for a zone with a width of 14 mm centred on the crack. The dry weight of the specimens was recorded and they were then placed in a container with water so that the water level was maintained 2-3 mm above the notch. The water uptake was recorded over a period of 24 h. By plotting the water uptake (in g) versus the square root of time (in $h^{0.5}$), the sorption coefficient (SC) was determined as the slope of the trend line of the data.

Results and discussion

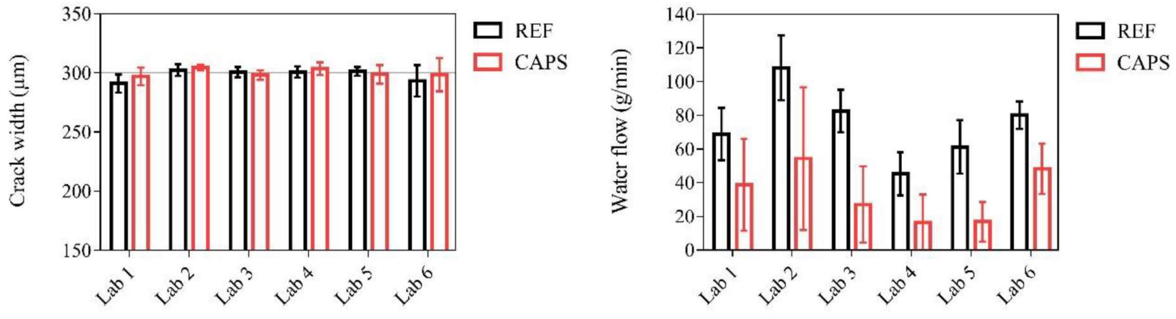
Water permeability test on mortar specimens

The mean crack width of the mortar specimens for the different labs is given in Figure 1 (a). Within each lab, the crack width of the REF specimens was comparable to the crack width of the CAPS specimens. Therefore, the crack width of the REF and CAPS specimens were considered together when comparing the results of the different labs in a Brow-Forsythe test (equal variances could not be assumed ($p < 0.1\%$)). This test indicated a significant result (level of significance = 5%, $p = 4.1\%$). A subsequent Tamhane's T2 post hoc test showed only a difference between Lab 1 and 2 (level of significance = 5 %, $p = 2.8\%$). It is noted that these statistical tests only gave a significant result due to the small variation on the crack width (for all labs the standard deviation of the REF, respectively CAPS series was lower than 8 µm, except for Lab 6 for which it was equal to 14 µm). Considering that for 90% of all samples the crack width of an individual specimen was within 10 µm from the target crack width of 300 µm (for 95% it was within 15 µm), it can be considered that the crack width of all labs is comparable. The low variability on the crack width is the result of the active crack width control technique which allowed to regulate the crack width precisely to the target crack width.

After measuring the crack width of the specimens and submerging them, a water permeability test was executed. The results of this test are given in Figure 1 (b). These results show more variation, as could be expected from previous research [3]. The mean water flow measured from the REF specimens is comparable for Labs 1, 3, 5, and 6. The variability on the CAPS specimens is higher than the variability on the REF specimens. This can be explained by the fact that the outflow of PU is not the same in all specimens. Table 1 shows the sealing efficiency calculated by dividing the difference between the mean water flow of the REF and CAPS specimens by the mean water flow of the REF specimens. Labs 1, 2 and 6 obtain a similar sealing efficiency of approximately 40-50%, and also Labs 3, 4 and 5 obtain a similar sealing efficiency of approximately 64-73%. Overall all labs were able to come to the same conclusion: the addition of the macrocapsules was beneficial, yet did not result in a perfect healing.

Capillary water absorption test on concrete specimens

The mean crack width of the concrete specimens for the different labs is given in Figure 2. The REF series of Lab 2 and 4, as well as the REF and CAPS series of Lab 3 and 5, did not obtain a target crack width of 300 µm (as analysed with a t-test, level of significance = 5 %).



(a) (b)

Figure 1. (a) Mean crack width of the mortar samples (error bars represent standard deviation, scale of the vertical axis is identical as for the concrete specimens); (b) Mean water flow of the mortar samples (error bars represent standard deviation)

Table 1. Sealing efficiency calculated from mean water flow

	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
Sealing efficiency	44%	50%	67%	64%	73%	40%

The statistical difference of the REF series of Lab 2 is related to a low standard deviation 10 µm, as the mean was equal to 290 µm, which can be considered equal to the target for practical purposes. Comparing these results with the results of the mortar specimens clearly shows that there is a much larger variation on the crack width of the concrete specimens (the standard deviation of the REF and CAPS concrete series varied from 10 µm (CAPS series Lab 2) to 76 µm (CAPS series Lab 3), while the maximum standard deviation for the mortar specimens was 14 µm (CAPS series Lab 6)). This can partly be explained by the fact that the mortar specimens have a more uniform matrix compared to the concrete specimens and thus also a different texture and crack geometry. Additionally, the larger variation is also partly a consequence of the 'passive' crack control. During loading in the three-point bending setup the crack width is precisely controlled by measurements of an LVDT or a CMOD. Yet, at the moment that the specimen is unloaded, after reaching a maximum crack width, the crack partially closes as a result of an elastic regain in the reinforcement bars. This elastic closure is not the same for all specimens, explaining the high degree of variation. A previous round robin study also used a comparable 'passive' crack control to crack reinforced mortar prisms (40x40x160 mm) [6]. The variability reported there was also much larger than the one reported in the current paper for the mortar specimens controlled in an 'active' way.

It is noted that while the target crack width after unloading was defined (300 µm), the maximum crack width under loading was left at the discrepancy of the participating labs as different displacement-controlled loading systems were used. Labs 1, 2, and 6 opened their specimens the furthest (485 µm) and they obtained results equal to the target.

The results of the water absorption tests are summarised in Table 2, where the sorption coefficients are given for the cracked REF and CAPS specimens, as well as uncracked control specimens (UNCR). There is quite a lot of variation on the results, e.g. Lab 4 obtained a sorption coefficient for the REF specimens which is more than double from what is obtained by Lab 1, 2 and 6. The variation on the crack width between the different labs does not seem to be the sole cause of these variations; the sorption coefficient of the uncracked specimens also shows a lot of variation. The quality of the waterproofing has been investigated in a follow-up study [5] and it was found that this had a significant influence.

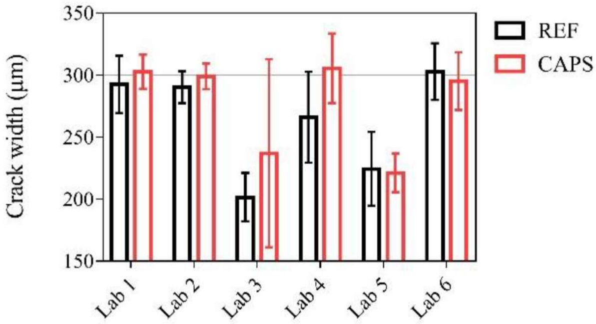


Figure 2. Mean crack width of the concrete samples (error bars represent standard deviation)

Table 2. Sorption coefficient for both uncracked specimens, reference specimens, and specimens with capsules

	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
SC_{REF} (g/√h)	4.13	4.49	8.80	10.82	7.79	3.92
SC_{CAPS} (g/√h)	4.11	4.33	6.37	9.11	7.13	3.97
SC_{UNCR} (g/√h)	1.78	2.44	6.73	7.76	6.27	2.09

Conclusion

Results on the executed inter-laboratory test on concrete with macrocapsules showed that the target crack width of 300 µm could be obtained with great accuracy in mortar specimens as a result of the applied 'active' crack width control technique. The crack width has a major influence on the water permeability. Due to the equal crack widths, all labs could come to the same conclusion with regards to the sealing efficiency: the macrocapsules were beneficial, yet did not result in a perfect crack sealing.

Only three out of the six participating labs were able to obtain the target crack width in the concrete specimens used for capillary water absorption testing (after inducing cracks via a 'passive' crack control). Upon unloading there is an elastic regain in the reinforcement which is not controlled and which contributes to the variability of the crack width. This could have influenced the results of the capillary water absorption tests, although the quality of the waterproofing is also an important factor.

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References

[1] COST Action SARCOS homepage. https://www.sarcos.eng.cam.ac.uk/. Accessed June 24 2021

[2] Ferrara L, Van Mullem T, Alonso MC, Antonaci P, Borg RP, Cuenca E, …, De Belie N. Experimental characterization of the self-healing capacity of cement based materials and its effects on the material performance: A state of the art report by COST Action SARCOS WG2. Construction and Building Materials. 208 Vol 167 pp. 115-142.

[3] Van Mullem T, Gruyaert E, Debbaut B, Caspeele R, De Belie N. Novel active crack width control technique to reduce the variation on water permeability results for self-healing concrete. Construction and Building Materials. 2019 Vol 203 pp. 541-551.

[4] Van Tittelboom K, De Belie N, Van Loo D, Jacobs P Self-healing efficiency of cementitious materials containing tubular capsules filled with healing agent. Cement and Concrete Composites. 2011. Vol 33 (4) pp. 497-505.

[5] Van Mullem T, Anglani G, Dudek M, Vanoutrive H, Bumanis G, Litina C, …, De Belie N. Addressing the need for standardization of test methods for self-healing concrete: an inter-laboratory study on concrete with macrocapsules. Science and Technology of Advanced Materials. 2021. Vol 21 (1) pp. 661-682.

[6] Tziviloglou E, Wiktor V, Wang J, Paine K, Alazhari M, Richardson A, …., Jonkers H. Evaluation of experimental methodology to assess the sealing efficiency of bacteria-based self-healing mortar: round robin test. RILEM Conference on Microorganisms-Cementitious Materials Interactions (Delft, the Netherlands). 2016. pp 156-170.