Northumbria Research Link

EVALUATION OF EXPERIMENTAL METHODOLOGY TO ASSESS THE SEALING EFFICIENCY OF BACTERIA-BASED SELF-HEALING MORTAR : ROUND ROBIN TEST

Eirini Tziviloglou (1), Virginie Wiktor (1), Jianyun Wang (2), Kevin Paine (3), Mohamed Alazhari (3), Alan Richardson (4), Marielle Gueguen (5), Nele De Belie (2), Erik Schlangen (1) and Henk Jonkers (1)

(1) Delft University of Technology, The Netherlands

(2) Ghent University, Belgium

(3) University of Bath, United Kingdom

(4) Northumbria University, United Kingdom

(5) University Paris – Est, MAST, CPDM, IFSTTAR, France

Abstract

Self-healing concrete has created a lot of public interest in recent years. Several research groups worldwide are currently working on creating durable and sustainable self-healing concrete structures. HEALCON (the concrete which repairs itself) is a European Union funded project, which focuses on developing cementitious materials with different self-healing mechanisms. The self-healing mechanisms can either repair the cracks and regain liquid-tightness, bridge the cracks and recover structural performance, or do both. One of the promising materials that have been studied within the project is the bacteria-based self-healing mortar, which is able to regain liquid tightness after cracking and healing. Within HEALCON an experimental methodology, which comprises of tests for evaluating the ability of the cementitious material to regain liquid-tightness and mechanical properties, has been developed.

This study focuses on evaluating the suggested experimental methodology through a round robin test (RRT) among five laboratories within the framework of RILEM/TC 253 MCI (Micro-organisms-Cementitious Materials Interactions), WG4 (Engineered bacteria-based protective systems for cementitious materials) and it concerns only the part that examines the sealing efficiency. The testing sequence includes:

- tests for material characterization,
- crack introduction on mortar prisms,
- healing treatment and
- water tightness examination.

Specimens with and without bacteria-based self-healing agent were tested. After the completion of the tests the results of the different laboratories were gathered for purposes of comparison. The comparison revealed high scatter in the results of the suggested methodology. Therefore, the current paper gives some recommendations, for improving the tests procedures, which will later be adapted to the second RRT that will follow.

1. INTRODUCTION

Self-healing concrete is a concept that has recently received a lot of attention; therefore, there are many research groups that are currently focusing on developing cementitious materials that can repair micro-cracks by themselves. In the beginning of 2013 the HEALCON project started and it will be completed in the end of 2016. HEALCON is a European Union funded project consisting of twelve academic and industrial partners. The project focuses on developing cementitious materials with different self-healing mechanisms that can seal the occurring micro-cracks which would otherwise lead to durability problems in the new structures.

One of the objectives of the project was to create a test methodology in order to evaluate self-healing at lab scale, since there is a lack of standard testing procedures in the literature. Hence, a test sequence was developed comprising of tests that could evaluate the ability of the cementitious material to regain liquid-tightness and mechanical properties such as flexural strength and stiffness. The ultimate target is to create a normative document, which would include an experimental methodology that would be scientifically sound and relatively easy to be implemented by other scientists. As a part of the effort to fulfil this target, a round robin test (RRT) was carried out concerning only the part that examines the sealing efficiency (SE) through crack permeability via water flow and water absorption. The included tests were :

- Flexural and compressive strength tests (material characterization),
- Crack introduction on mortar prisms using three-point-bending,
- Crack permeability tests via water flow and
- Water absorption test.

2. EXPERIMENTAL APPROACH

Three types of mortar specimens were considered; i.e. one with bacteria based self-healing agent incorporated in lightweight aggregates (BAC), one with lightweight aggregates without the healing agent (CTRL) and one with normal weight aggregates without the healing agent (REF). The focus of the current study was to investigate the robustness of the suggested test methodology rather than drawing conclusions for the healing ability of the bio-based mortar.

The RRT was held within the framework of RILEM/TC 253 MCI (Micro-organisms-Cementitious Materials Interactions), WG4 (Engineered bacteria-based protective systems for cementitious materials) among five different testing laboratories with Delft University of Technology as the task coordinator. The laboratories in alphabetical order were:

- University of Bath (BU)
- Delft University of Technology (TUD)
- French institute of science and technology for transport, spatial planning, development and networks (IFSTTAR)
- Ghent University (GU) and
- Northumbria University (NU)

Due to some technical problems IFSTTAR was only able to contribute to material characterization. Before starting the RRT, all the participants received, from the coordinator, pre-cast mortar prisms plus detailed instructions regarding the testing procedures. After the completion of the tests the participants sent the data to TUD for further processing.

A schematic representation of the experimental procedure that was followed during this RRT in every laboratory for each set of mortar prisms received is shown in Figure 1.

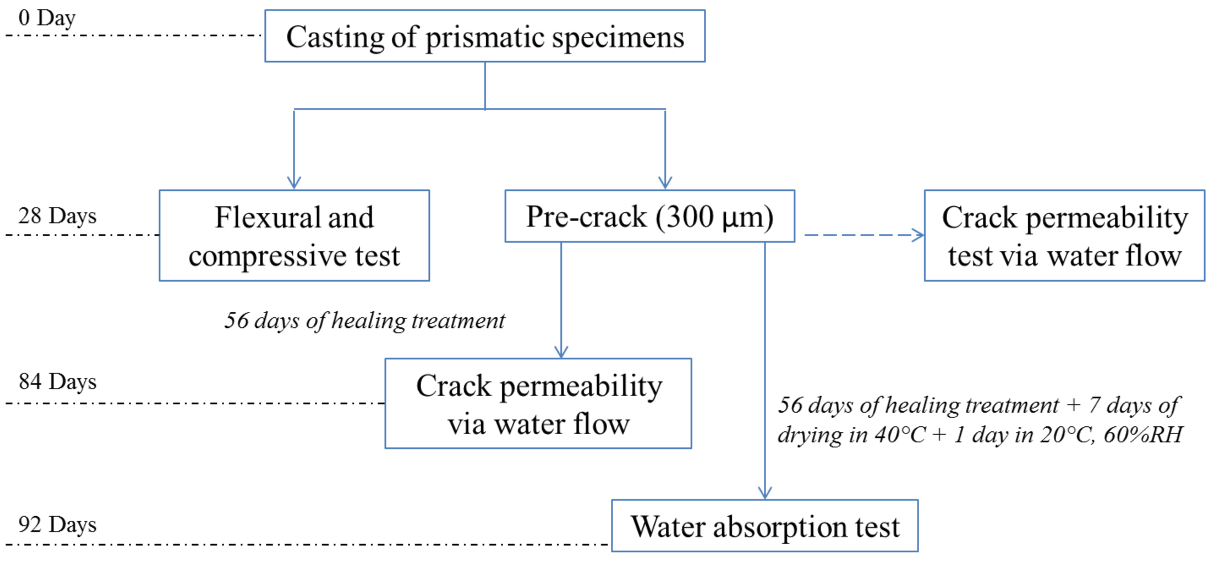

Figure 1. Schematic representation of experimental procedure that was followed in every laboratory starting at t=28 days for each set of prisms received from TUD.

3. MATERIALS AND METHODS

3.1 Preparation of the mortar prisms

The mortar prisms were prepared at TUD and were shipped to the other four laboratories at the age of 7 days. As mentioned above, three types of mortar mixtures were investigated: one reference mixture (REF) with normal weight aggregates, one control mixture (CTRL) with non-impregnated lightweight aggregates (LWA) and one mixture (BAC) with impregnated LWA with the bacteria based self-healing agent. For the three mixtures were used: ordinary Portland cement (CEM I 42.5N, ENCI, The Netherlands), 0/1 mm and ¼ mm sand or 0 /1 mm sand and 1/4 mm LWA (expanded clay particles, Liapor 1/4 mm, Liapor GmbH Germany). The detailed mixture proportions are presented in Table 1. The preparation of the healing agent is described in [1]. All specimens were cast in polystyrene moulds and were kept in sealed plastic bags until they were tested.

Table 1. Mixing proportions of mortar specimens.

Mixture	CEM I (kg/m^3)	Water (kg/m^3)	0/1mm Sand (kg/m^3)	1/4 mm Sand (kg/m^3)	LWA 1/4 mm (kg/m^3)
REF	463	231.5	810	810	0
CTRL	463	231.5	810	0	257
BAC	463	231.5	810	0	283*

*weight of LWA after the impregnation with the healing agent (assuming weight increase of 10%).

For material characterization of the hardened mortar, prisms (40 mm x 40 mm x 160 mm) were cast in accordance with EN 196-1 [2]. For the water absorption test, reinforced (two steel wires ø1 mm) mortar prisms (40 mm x 40 mm x 160 mm) were cast (Figure 2a). For the water flow test, reinforced (two steel wires ø1 mm) mortar prisms (40 mm x 40 mm x 160 mm) with a 5-mm diameter cylindrical hole in the centre were used (Figure 2b). A notch of approximately 2-3 mm deep was made in the middle of every reinforced prism at the age of 28 days.

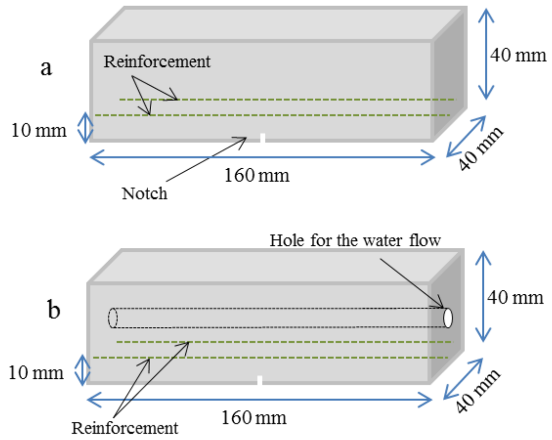

Figure 2. Typical prism: (a) for water absorption test and (b) for water flow test.

Table 2 presents the number of specimens that were used per test in each laboratory.

Table 2. Specimens used in each laboratory for each test.

Mixture	Material characterization tests	Water flow test	Water absorption test
REF	3	6	9
CTRL	3	6	9
BAC	3	6	9

3.2 Material characterization, crack introduction and healing regime

The material characterization tests included flexural and compressive tests on prisms as it is described in EN 196-1 [2]. The loading speed was 30 N/s and 250 N/s for flexural and compressive strength respectively.

Cracks were introduced on the reinforced mortar specimens (with and without the cylindrical hole) at the age of 28 days by means of 3-point-bending. In this procedure the reinforced prisms were placed on the testing machine, where a vertical load was applied at their middle span, so that the crack opening increased constantly by 0.5 μm/s. When a crack opening of approximately 350 μm was reached, the samples were slowly unloaded. For TUD, GU and BU laboratories the development of the crack width was monitored through LVDT (Linear Variable Differential Transducer), while for NU the vertical displacement reading was converted to horizontal deformation value. Following the crack creation, the samples were submerged horizontally (on spacers of 5 to 10 mm) in a plastic bucket filled with5 litres of tap water per three specimens in order to heal the cracks, for 56 days.

3.3 Assessment of water tightness through crack permeability tests

A crack permeability test via water flow was used to evaluate the ability of a crack to resist a flow after the healing treatment. The test was performed before water submersion on three specimens and after water submersion on another three. The reason for testing different specimens before and after healing was to avoid any loss of healing agent or mortar components during the first permeability test that could possibly affect the healing process. During the test, water coming from a water column with a head of 0.5 m passed through the cylindrical hole in the middle of the specimen. The water that leaked from the crack, which was located in the mid-section of the prism, was collected in a container placed on an electronic scale. The test data (mass of water and time) was recorded by a computer connected to the electronic scale. In BU the mass was recorded once, at the end of the test. Each test lasted for 5 minutes. The graphs that were obtained by this test showed a linear relation between the mass of water leaked from the crack and the time. The sealing efficiency (SE) of a specimen after cracking and healing was calculated according to Equation 1.

$$SE = 100\% \left[1 - \left(M_{healed} / M_{non\text{-}healed}\right)\right] \quad (1)$$

Where M_{healed} : the average (out of three specimens) mass derived from test data of the healed specimens; and $M_{non\text{-}healed}$: the average (out of three specimens) mass derived from test data of the non-healed specimens.

Furthermore, another test was developed to investigate the ability of a cracked and healed specimen to absorb water. The procedure was based on the test described in EN 13057 [3]. During the test, the bottom of the specimens (the side with the notch) came into contact with water. The method used reinforced prismatic mortar specimens; non-cracked, cracked non-healed and cracked healed. Before testing, the specimens were kept at 40 °C for minimum 7 days, until a stable weight change was achieved. Then, the specimens were stored for 24h at 20 °C with 60 % relative humidity for one day. The specimens were weighed prior to the test. The test was carried out in a container and the specimens were standing on spacers, so that there was a gap between them and the bottom of the container. The water level in the container exceeded the bottom of the specimens by approximately 2-3 mm. Before the test, the specimens were partially waterproofed (with aluminium adhesive foil), as seen in Figure 3.

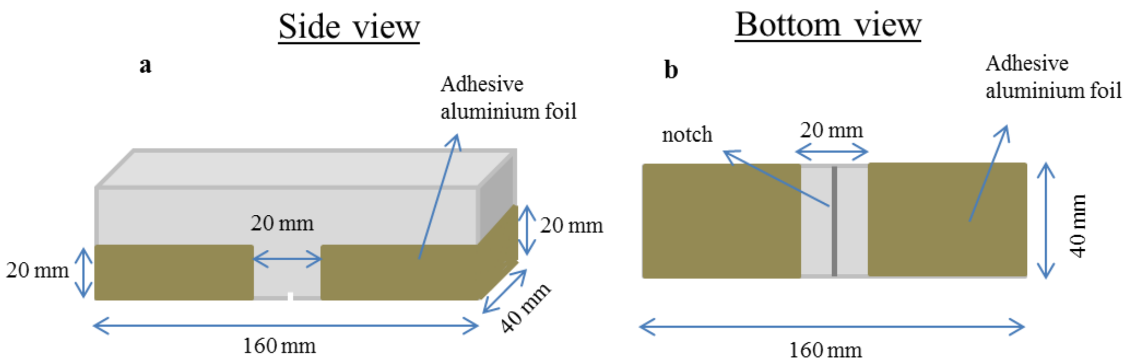

Figure 3. Water proofed specimen prepared for water absorption test: a) side view, b)bottom view.

During the test the specimens were weighed frequently for a period of 8 h (after 0.25 h, 0.5 h, 1 h, 2 h, 4 h, 6 h, 8 h), after removing the excess of water on their surfaces with a cloth. This data was used to plot the graph of the water uptake with the square root of time. By plotting this graph it was possible to calculate the sorption coefficient (SC) of a specimen, as it is presented by Hall [4] for materials with coarse pore structure with little suction. In such cases, the experimental data are fitted to Equation 2:

$$i = A + SCt^{1/2} - Bt \quad (2)$$

Where i: the water uptake in g; t: time in h; SC: the sorption coefficient in $gh^{-1/2}$ and A and B are constant. A typical curve that can be obtained during water absorption test is shown in Figure 4. After the completion of the test, the average SC (out of three specimens) of each specimens category (non-cracked, cracked non-healed and cracked healed) was calculated in order to compare the absorption capacity of each category.

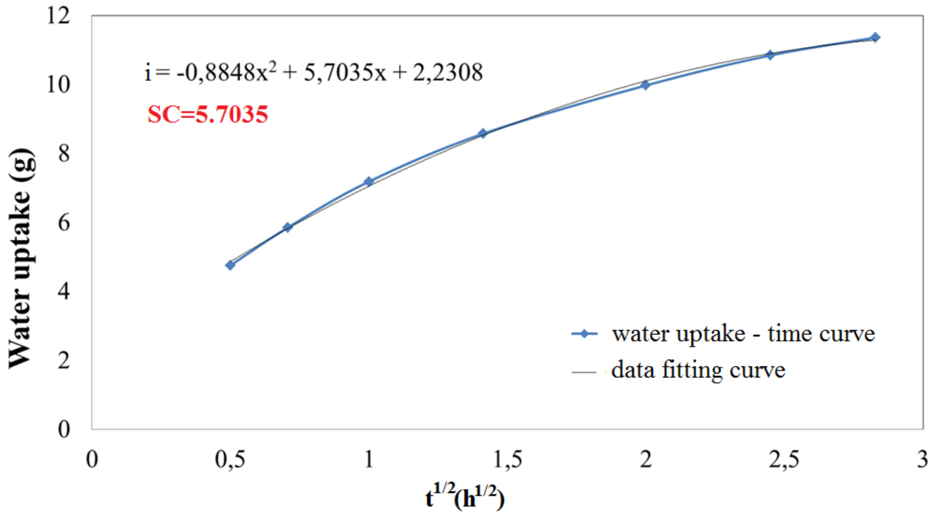

Figure 4. Typical graph obtained during water absorption test and polynomial data fitting curve.

4. RESULTS

4.1 Flexural and compressive strength

The results obtained from flexural and compressive strength tests from each mix category are presented in Figure 5 and 6 respectively.

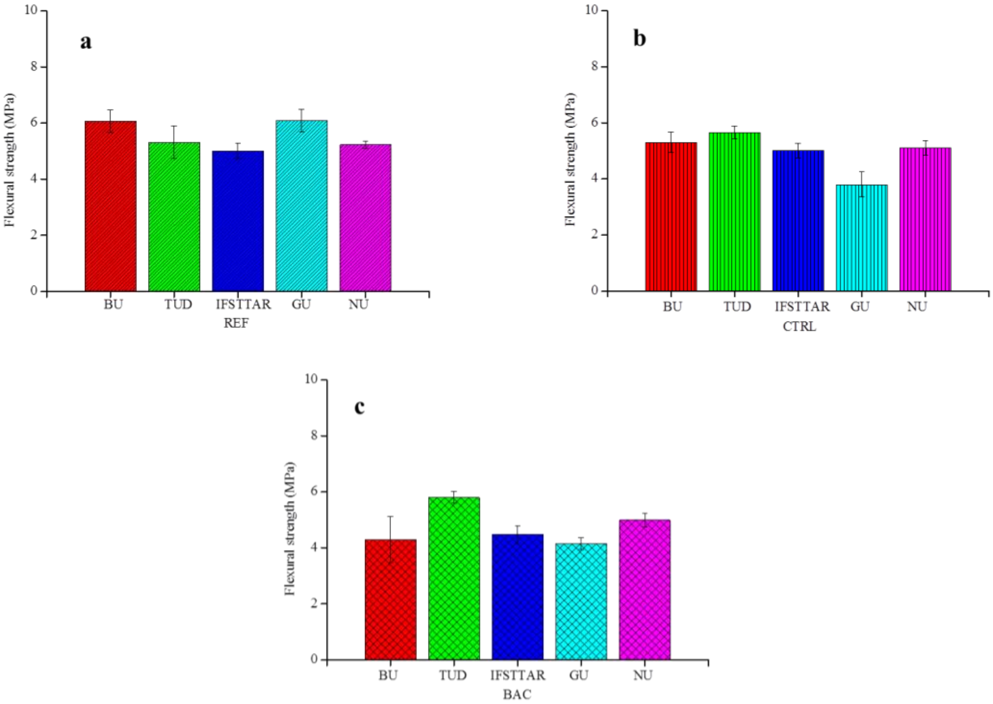

Figure 5. Flexural strength test results: a) REF specimens, b) CTRL specimens and c) BAC specimens.

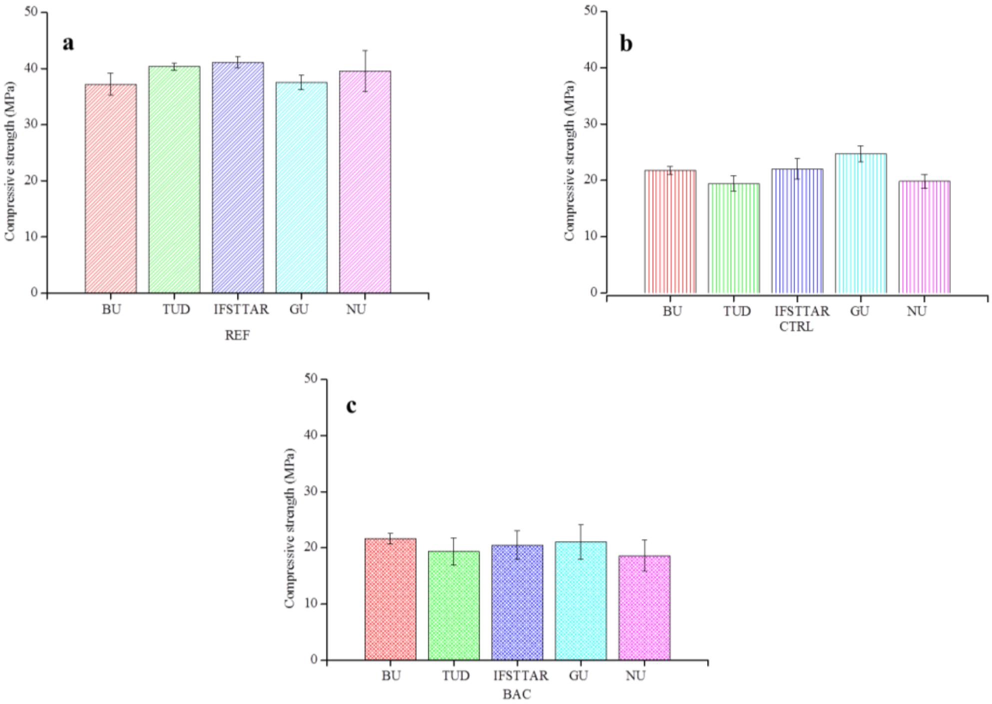

Figure 6. Compressive strength test results: a) REF specimens, b) CTRL specimens and c) BAC specimens.

The flexural strength results revealed that there was no significant difference among the three mixtures. In fact, most of the flexural strength values obtained varied between 4.5 and 6 MPa. In addition, not only the values obtained within each laboratory showed relatively low scatter but also the inter-lab variations were insignificant in view of observed standard deviations.

The compressive strength results show the effect of the replacement of normal weight sand with LWA. Actually, the compressive strength of REF mixture on average was approximately 39 MPa, while CTRL and BAC specimens showed a reduction of almost 50%. However, the intra- as well as the inter-lab scatter was also relatively low.

The results from both strength tests showed that the prisms of each mortar batch exhibited comparable behaviour. Thus, one can conclude that the quality of the distributed material was similar and therefore all participants started the RRT with the same material.

4.2 Crack introduction

The crack introduction on the mortar prisms, as it is mentioned above, was made via 3-point-bending. The final crack width after unloading of the specimens was measured either via microscopy on the bottom of the cracks or from the readings of the LVDTs. There was no designated method for the crack width measurements. The values displayed in the diagrams of Figure 7 were obtained via microscopy for TUD, GU and NU. For TUD and GU the crack values are the mean from a range of values measured on the bottom of the specimens and for BU the crack values were obtained from the LVDT readings. Therefore, the real crack width obtained in different laboratories varied.

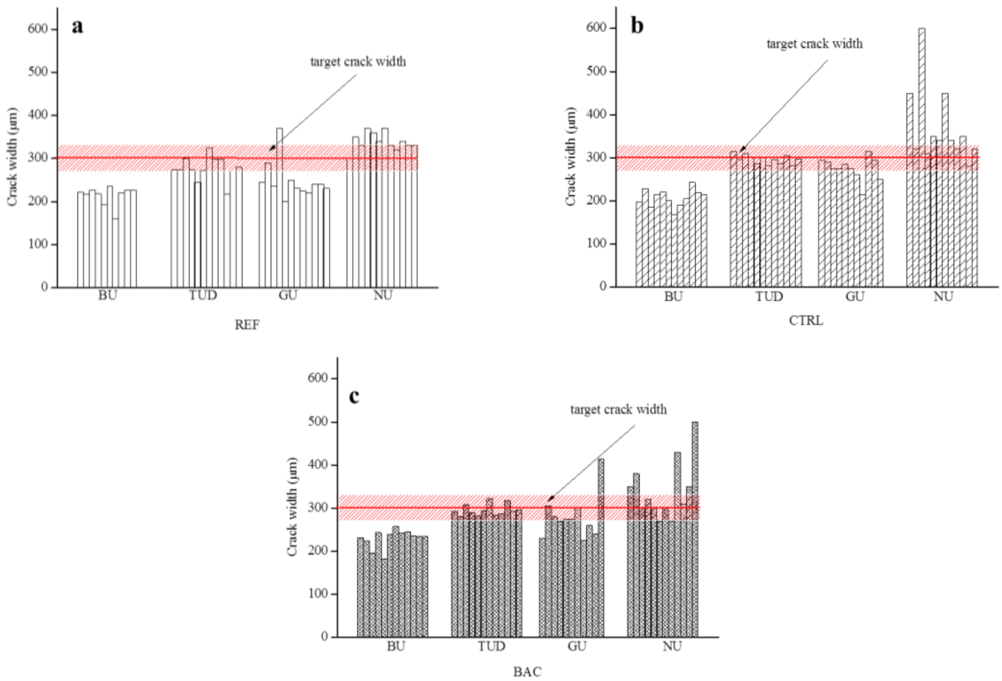

Figure 7. Crack width values after unloading: a) REF specimens, b) CTRL specimens and c) BAC specimens.

The intra-lab variation regarding the crack width values was relatively low, regardless the proximity to the target crack width value (300 μm). However, there was a high scatter of the results of the different participants, even within the same mixture. The differences in crack width values among the laboratories can be attributed to several reasons that are listed below:

- The microscopic measuring method of the crack width differed for each laboratory.
- The crack width was controlled for TUD, GU and BU via LVDT sensors. However, the number of sensors and the position on the specimen varied. For example:
    a. BU used one sensor on the front of the specimen.
    b. TUD used two sensors on the front and back of the specimen,
    c. GU used one sensor on the bottom of the specimen and
    d. NU monitored indirectly the crack width; by conversion of the vertical displacement of the machine to the horizontal crack opening.

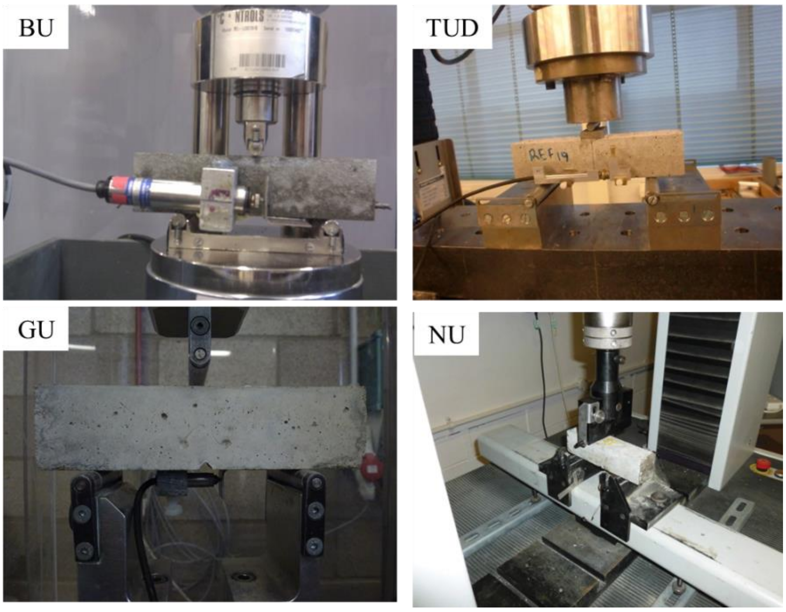

Figure 8. Set-ups of the different laboratories for crack introduction via 3-point-bending.

Even though every laboratory was targeting the same crack width, either the measuring position or the crack monitoring method were different. As a result, the average microscopic measurements among the laboratories was expected to show a high scatter.

4.3 Crack permeability test via water flow

Tables 3, 4 and 5 show the results obtained by the crack permeability tests via water flow before and after healing treatment, as well as the calculated SE for REF, CTRL and BAC specimens respectively. In cases where some test data was missing by one participant the calculations were made on basis of the values actually available.

Table 3. Results obtained by crack permeability test via water flow- REF specimens.

REF						
Lab.	Repl.	Mass of water before healing (g)	Average mass before healing (g)	Mass of water after healing (g)	Average mass after healing (g)	SE (%)
BU	i	20.0	102.3	179.0	146.0	- 42.7
	ii	45.0		61.0		
	iii	242.0		198.0		
TUD	i	385.6	264.5	13.2	73.1	72.4
	ii	114.9		155.7		
	iii	293.1		50.3		
GU	i	1.5	1.7	Missing data	3.0	76.4
	ii	2.7		4.5		
	iii	0.7		1.5		
NU	i	375.6	502.9	171.5	224.3	55.4
	ii	906.5		146.1		
	iii	226.5		355.4		

Table 4. Results obtained by crack permeability test via water flow- CTRL specimens.

CTRL						
Lab.	Repl.	Mass of water before healing (g)	Average mass before healing (g)	Mass of water after healing (g)	Average mass after healing (g)	SE (%)
BU	i	137.0	146.3	8.0	55.0	62.4
	ii	191.0		60.0		
	iii	111.0		97.0		
TUD	i	260.4	297.1	167.3	199.4	32.9
	ii	332.0		234.7		
	iii	298.8		196.1		
GU	i	182.7	208.5	75.1	58.5	71.9
	ii	234.2		71.9		
	iii	Missing data		28.7		
NU	i	1836.7	1959.8	175.8	388.1	80.2
	ii	1136.5		472.8		
	iii	2906.0		515.8		

Table 5. Results obtained by crack permeability test via water flow-BAC specimens.

BAC						
Lab.	Repl.	Mass of water before healing (g)	Average mass before healing (g)	Mass of water after healing (g)	Average mass after healing (g)	SE (%)
BU	i	1.7	245.56	354.0	221.00	10.0
	ii	691.0		89.0		
	iii	44.0		220.0		
TUD	i	319.2	313.40	109.0	144.77	53.8
	ii	261.7		222.0		
	iii	359.3		103.3		
GU	i	249.9	240.99	39.1	104.27	56.7
	ii	232.1		169.5		
	iii	Missing data		Missing data		
NU	i	257.3	232.51	290.6	122.63	47.2
	ii	322.7		25.4		
	iii	117.5		51.9		

The intra- as well as the inter-lab variations were significant. Particularly, the inter-lab variations for REF and CTRL specimens regarding the initial as well as the final flow were very pronounced. Subsequently, the SE values showed a great scatter. In REF mixture, negative values of SE were observed as well. For the BAC specimens, the differences in average values among the laboratories were slightly decreased. However, no obvious trend was revealed within the same mixture or from one mixture to another.

The high scatter of the intra-lab results within one mixture and for the same set of experiment (before or after healing) can be explained by the different crack geometries/widths that were obtained in each specimen. It was assumed that the most governing factor for this test was the crack width at the point of the intersection between the crack and the cylindrical hole in the middle of the specimen. Furthermore, in some cases higher flow values were observed after healing than before healing. This can be explained by the fact that the test was performed on different sets of specimens before and after healing. Hence, if the (average) crack width of the specimens tested before healing was smaller than the crack width of the ones tested after healing it was very likely to acquire higher flow values after healing.

4.4 Crack permeability test via water absorption

The figures that follow (Figures 9,10 and 11) present the average sorption coefficient of each mix category (out of three replicates) calculated as seen in 3.3.

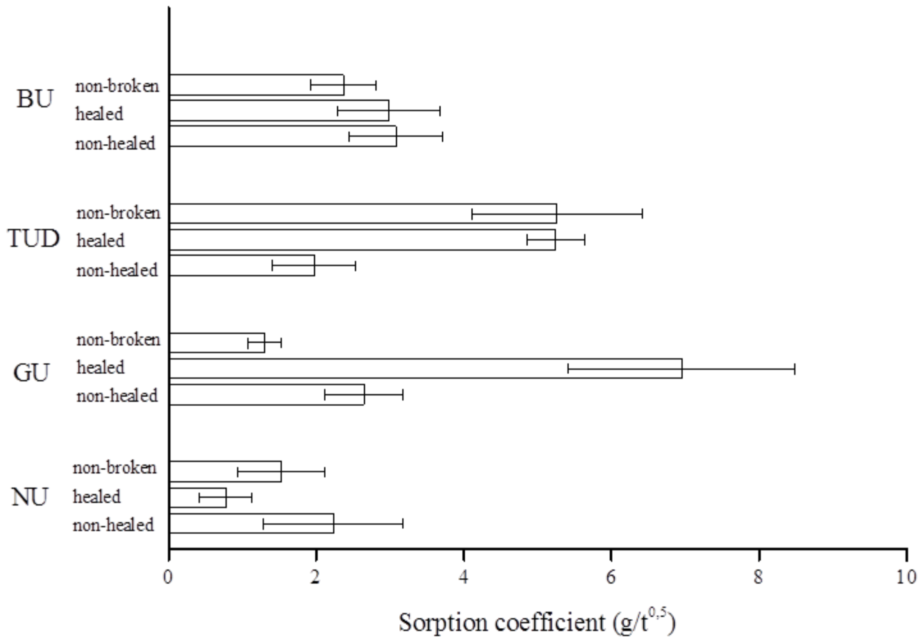

Figure 9. Average SC calculated from water absorption test - REF specimens.

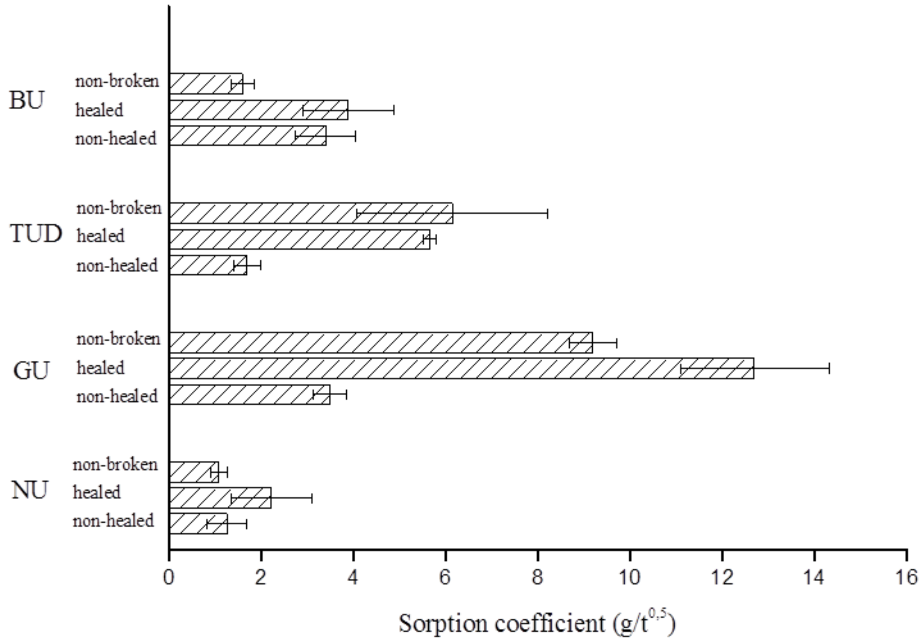

Figure 10. Average SC calculated from water absorption test - CTRL specimens.

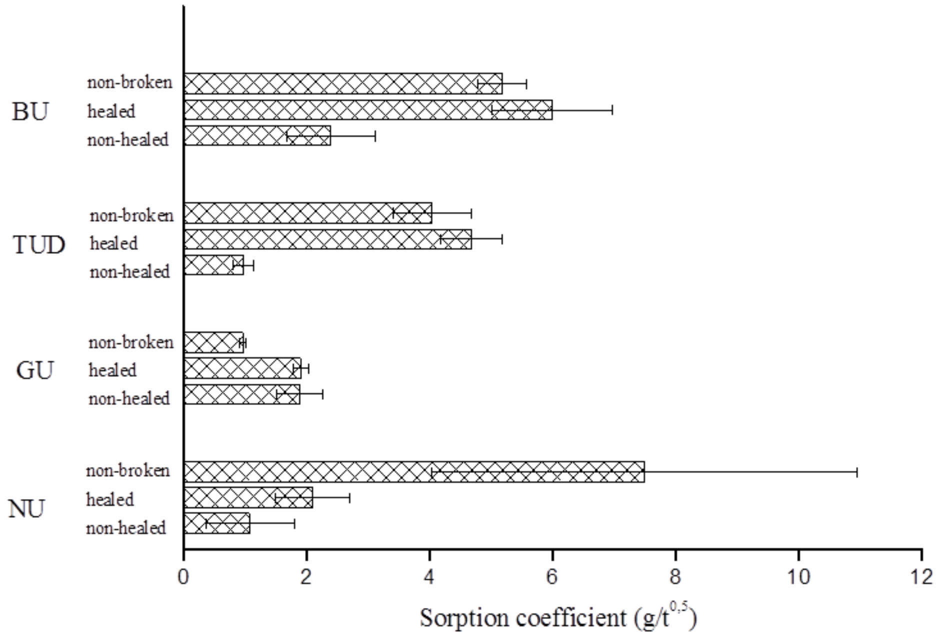

Figure 11. Average SC calculated from water absorption test - BAC specimens.

The results of the water absorption test revealed a very random behaviour for all categories of specimens (REF, CTRL and BAC). In fact, the results showed:

- a relatively high standard deviation within the measurements of each individual laboratory,
- no clear trend in the values of sorption coefficient; for example $SC_{non-healed} > SC_{healed} > SC_{non-broken}$

5. DISCUSSION AND RECOMMENDATIONS

Even though every laboratory targeted the same crack width, large scatter in the measured values was observed. This is partly due to the position of the LVDT as mentioned earlier.
Moreover, it was observed that the crack shapes/geometries were very often different even in intra-lab level. In some cases, there was also crack branching (Figure 12) at a height of some millimetres over the notches. The differences in the cracks can be explained by :

- The presence of the notch in every specimen. The notches were manually introduced and were supposed to have a 2-3 mm depth. However, it was hard to achieve the exact constant depth along the width of the sample. Furthermore, the method of introducing the notch as well as its width and shape varied among the laboratories.
- The type of the reinforcement. The two steel reinforcing wires that were used were quite flexible. Thus, the reinforcement resembled more to a curved/corrugated wire rather than a straight steel bar. As a consequence, the shape of the reinforcement varied for every sample that was cast.

All of the above can play a role to the variations in crack widths and geometries. Thus, even though each mortar batch was supposed to exhibit similar cracking patterns and behaviour, judging by the material characterization, the results proved otherwise.

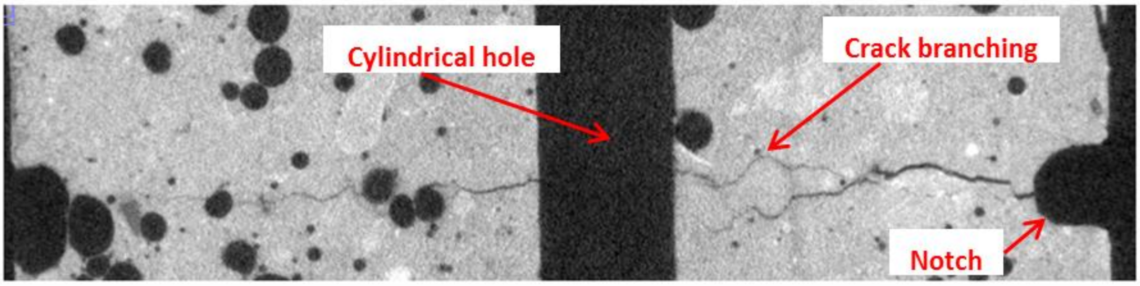

Figure 12. Computed Tomography (CT) image on a cracked mortar specimen with cylindrical hole showing crack branching

Large scatter in crack width values and geometry translated in large scatter in the results of the water tightness tests. The factors that could have affected the scatter of the results are summarized below. First, the fact that the cracks differ substantially in shape and width can alter significantly the type and the amount of the flow. Furthermore, the possible leaks of the waterproofing aluminium foil at different points and with varying opening sizes can also create a large scatter in the absorption results. Finally, the variability in width and depth of the notches of each specimen could have definitely affected the amount of the absorbed water in the different specimens.

It is therefore recommended by the authors to:

- cast the prisms with the notch rather than creating it afterwards, since is not always easy to control its depth and width and
- use straight steel bars with larger diameter in order to avoid arbitrary bending of the reinforcement that can lead to varying shapes of cracks.

In addition, in order to restrict the variations in the crack permeability test via water flow it is suggested:

- to perform the initial (before healing) and final tests (after healing) on the same set of specimens.

Finally, for the water absorption test it is recommended :

- to determine whether the partial sealing of the bottom face can significantly affect the results. Therefore, two configurations, i.e. bottom side non-sealed and bottom side-partially sealed, should be investigated.

6. CONCLUSIONS

The current paper presented the results of a preliminary study from a round robin test that tested a set of suggested methods to evaluate the efficiency of self-healing in cementitious materials. The results of this study revealed a relatively large scatter for the suggested crack permeability tests. Thus, further improvement and tuning is needed in order to obtain more uniform and reproducible results.

The most significant factor that influences the outcome of the tests is the shape and the width of the crack. Consequently, there is need to minimize the crack variations by improving cracking method or by excluding the factors that affect the crack development.

A second round robin test will follow including the recommendations given on this paper, so as to further evaluate the quality and reproducibility of the results obtained by the proposed tests.

ACKNOWLEDGEMENTS

The authors acknowledge the financial support of European Union Seventh Framework Programme (FP7/2007-2013) under grant agreement no 309451 (HEALCON).

REFERENCES

[1] Tziviloglou E., Wiktor V., Jonkers, H.M. , Schlangen, E. , 'Development of bio-based self-healing concrete to increase durability of structures', Ahmed, SM, Xenidis, Y, Azhar, S, Smith, NA, Yans, CE (Eds.) Proceedings of the 8th international conference on construction in the 21st century, Greenville: CITC, (2015) 330-337.

[2] EN 196-1:1995en, Methods of testing cement-Part 1: Determination of strength.

[3] EN 13057:2002en, Products and systems for the protection and repair of concrete structures - Test methods - Determination of resistance of capillary absorption.

[4] Hall C., 'Water sorptivity of mortars and concretes: a review', *Magazine of Concrete Research*, **147** (1989), 51-61.