PUBLISHABLE SUMMARY

1. Executive summary

HEALCON is a project funded by EU-FP7 and coordinated by Prof. Nele De Belie (Ghent University). Its aim is to design smart concrete with self-healing properties to create durable and sustainable concrete structures.

In a first phase of the project, different types of healing agents and encapsulation techniques have been developed and fine-tuned and first steps towards upscaling have been taken. It concerns:

Bacterial healing agents:

- Spores of Bacillus Sphaericus encapsulated in melamine formaldehyde microcapsules
- Bacillus spores, incorporated into zeolite powder, and organic mineral compounds embedded in expanded clay particles
- A mixed ureolytic culture, made from a sub-stream of a vegetable treatment plant, combined with anaerobic granules

Superabsorbent polymers:

- pH sensitive superabsorbent polymers, tuned for use in self-healing concrete (high swelling capacity at neutral pH and reduced swelling capacity at high pH)
- Superabsorbent polymers coated in a Wurster process

Elastic polymers

Depending on the type of damage, another self-healing concept was envisioned in the project. While bacterial healing agents and superabsorbent polymers are suitable for healing of early age cracks in structures which require liquid tightness since they produce a non-elastic material that fills the crack, elastic polymers can be used for healing of bending cracks since they can cope with the opening and closing movement of cracks under dynamic load.

The efficiency of the different self-healing mechanisms with regard to mechanical behaviour, liquid-tightness and durability was firstly quantified at lab-scale. A fair evaluation was done, based on standard test procedures, developed within the project. Besides, computer models were developed to simulate the fracturing and self-healing mechanisms in order to refine lab tests and to ultimately scale the mechanisms to an industrial level. Then, healing agents were incorporated in large scale elements (slabs and beams) to validate experimentally the self-healing methodologies. To characterize healing, non-destructive techniques as ultrasonic transmission and impact generated transmission measurements have been used in the small and large scale tests. Furthermore, a wireless monitoring system, suitable to continuously assess the concrete health and healing success has been developed in applied.

As the technologies developed have to be cost effective, functional and adaptable to engineering design, an end-user board followed the project from the start and helped to define technical and application requirements. Moreover, a life cycle cost analysis, supplemented by a life cycle assessment has been performed in order to demonstrate the impact of the self-healing technologies on environment and economy.

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2. Summary description of project context and objectives

Reinforced concrete is designed to crack, but crack widths are limited to 0.2 to 0.4 mm depending on exposure class and type of concrete (reinforced or pre-stressed). Although these cracks do not impair structural stability, through-going cracks drastically affect liquid tightness. This is a major problem in tunnels and large underground structures, where cement hydration reactions and temperature/shrinkage effects in large concrete segments might result in the formation of early age cracks. Expensive preventive measures are taken or repair works are needed right after construction. Furthermore, even if not through-going, cracks will allow faster penetration of aggressive liquids and gases, compromising the long-term durability of the structure. Current practice requires regular inspection, maintenance and repair, to ensure structural safety over the service life of the structure. These practices involve large direct and indirect costs, such as economic losses from traffic jams. Additionally, not all structures are easy to access for inspection and repair.

In their search to overcome these problems, researchers have been inspired by nature. Biological systems such as bones, skin or plants have the capacity to detect damage very quickly and have moreover the unique feature to repair the damage efficiently. It would be an enormous advantage if this concept could be translated to our engineering materials, such as concrete. The application of so-called "self-healing" concrete, which will in an autonomous way repair cracks, could reduce the maintenance costs drastically. Additionally, indirect costs such as due to traffic congestion can be avoided.

The overall objective of the project is to design, develop, test, apply and evaluate self-healing methods for concrete structures. Depending on the type of damage, another self-healing concept is envisioned. While static cracks (e.g. early age cracks due to shrinkage) can be filled with a non-elastic material, moving cracks (e.g. bending cracks in bridge beams) are preferably filled with elastic healing materials. This means that biogenic healing agents as well as polymeric healing agents (hydrogels and elastic polymers) are considered.

In summary, the main objectives of HEALCON are:

OBJECTIVE 1: Development of efficient self-healing techniques that enable concrete to regain liquid-tightness via bioprecipitation and application of hydrogels

Incorporation of bacteria in concrete can enhance crack healing by production of $CaCO_3$, as a result of their metabolic activity and of subsequent chemical reactions including the metabolic products. While encapsulation of micro-organisms would help to increase their survival in concrete, the key element to have a successful self-healing is the choice of the bacterial strain. Promising results were obtained with micro-encapsulated bacterial spores and bacterial spores impregnated in porous aggregates by project partners UGent and TUDelft. The main challenge within the HEALCON project in this respect is to fine-tune and up-scale the procedures to produce axenic cultures of *Bacillus*, to optimize nutrient media and to search for optimal encapsulation materials and techniques and test the impact on fresh and hardened concrete properties. As the production and encapsulation of pure cultures can be quite expensive for use in self-healing concrete, other processes are designed for the production of a mixed culture (non-axenic) with good ureolytic characteristics and capable to induce calcium carbonate precipitation.

Superabsorbent polymers (SAPs) are three-dimensional, crosslinked polymeric networks that are not soluble, but which can absorb large quantities of water. Due to their swelling properties, the synthesized SAPs incorporated in mortar immediately block the crack and prevent further ingress of water via this crack. Consequently, very high sealing efficiencies are already reached immediately after crack formation. Moreover, after a healing period, the crack can even close due to (mainly) precipitation of $CaCO_3$ or ongoing hydration, leading to an even higher sealing efficiency. At the start of the HEALCON project, research on hydrogels for self-healing concrete was only in its infancy. The challenge for the HEALCON partners in this respect is to optimize this approach, elucidate the interaction between the SAP particles and the cementitious matrix and overcome the disadvantage of swelling at the moment of mixing by (i) coating of the SAPs and (ii) development of synthetic

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superabsorbent polymers with improved swelling and pH sensitiveness. Related to the coating of SAPs, the Wurster process will be applied for the first time on encapsulating active agents.

Another aspects deals with the evaluation of the self-sealing efficiency. Researchers made already some first attempts to evaluate the efficiency by measuring regain in air- or liquid tightness, however, each used their own type of test set-up. Therefore, HEALCON wants to develop the most suitable techniques to measure regain in tightness in order that more standardized methods can be obtained.

OBJECTIVE 2: Development of efficient self-healing techniques for bending cracks in concrete elements under dynamic loading, as solution to prevent future durability problems

The use of encapsulated, polymer precursors as healing agents has shown potential for efficient healing of cracked concrete in proof-of-concept specimens. Also UGent has quite some expertise with these types of healing agents. However, it has to be evaluated whether the healing agents used in proof-of-concept experiments on small samples, can also be used in real size concrete elements and whether they are able to follow the movement of dynamic cracks and guarantee liquid tightness during further use of the construction. In this respect, the importance of using elastic healing agents will be investigated.

Another aspect is related to the encapsulation of the polymer precursors. A multifunctional encapsulation approach has to be developed, which not only protects the content for a long time but also releases it in a predetermined way when cracking occurs. This is a missing factor which is absolutely necessary to make the already developed self-healing approach applicable in practice.

OBJECTIVE 3: Development of computer models to simulate the fracturing and self-healing mechanisms in order to refine lab tests and to ultimately scale the mechanisms to an industrial level

Modelling is an important tool to advance/optimize experimental research. However, at the start of the HEALCON project, there was a lack of published modelling literature on the subject of self-healing concrete. Some analytical models were available to study the distribution of healing agent containing capsules in order to be effective and hydration models were developed which can also simulate self-heling due to ongoing hydration in a crack. HEALCON will build further on the available models to investigate the optimal size, shape, volume percentage and distribution of healing agent containers and to study the effect of ongoing hydration on the closure of the crack. Furthermore, progress beyond the state of the art includes the further development to incorporate healing mechanisms in lattice type models for fracture and transport and the adaptation of finite element models which include mechanical and fracture analyses and transport analyses to assist in the design of large scale lab and field tests.

OBJECTIVE 4: Development of non-destructive testing and monitoring techniques and combine existing ones to characterize the effects of different self-healing mechanisms in small and full-size specimens

Non-destructive techniques have advantages over destructive measuring techniques. In some preliminary studies (executed by TUM and UGent amongst others), some non-destructive test methods (acoustic emission, resonance frequency, …) have shown their usefulness for evaluating self-healing in laboratory tests. Within HEALCON it will be a challenge to select the most suitable candidates to be used for monitoring of damage (e.g. cracking, corrosion of steel reinforcement) and self-healing and to combine existing techniques to increase the reliability of the results. Moreover, taking into account all boundary conditions, HEALCON partners will select/adapt/combine techniques to be able to use them on large scale specimens. The final goal is to assess the condition of the large scale self-healing concrete elements with the selected techniques.

OBJECTIVE 5: Development of a life-cycle assessment methodology to demonstrate the impact of self-healing technologies on the economy, society and environment

As a new technology is developed, it is important to show the impact on economy, society and environment and compare this with the impact of traditional construction methods. Emphasis will be on a life cycle cost analysis, but also life cycle assessment will be performed. This will be done for a specific structural element. The outcome of this task is quite important to persuade contractors and owners to use this technique in the future.

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OBJECTIVE 6: Demonstration of the developed technologies in a real size structure and develop construction specifications for the use of self-healing products developed within the project

Within HEALCON, the newly developed products will first be tested at lab-scale, i.e. by incorporation of the healing agents in mortars/concrete and assessing the sealing/healing efficiency. For the most promising solutions, a verification of the feasibility and efficiency at larger scale and in the field are necessary. These demonstration projects, with birth certificates, are necessary to have a launching point for the newly developed products within the construction industry, overcoming the traditional skepticism of infrastructure owners. A follow-up monitoring should be provided after the HEALCON project has come to an end.

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3. Description of the main S&T results/foregrounds

3.1 Material development and production processes

For many years, researchers were looking for the most suitable healing agents for self-repair of concrete. At the start of HEALCON, self-healing concrete was thus fundamentally explored but reached the stage that these initial concepts must be further developed to ensure practical application in concrete structures. HEALCON partners specifically focused for that on structures which would benefit the most of the use of self-healing concrete. It includes (1) structures that demand liquid-tightness but suffer from early age cracking and (2) structures with a high risk of premature reinforcement corrosion due to bending cracks (Figure 1). For the first application, suitable micro-organisms or hydrogels seemed to be excellent candidates to be incorporated in concrete as they can fill the crack with a non-elastic material. For the second application, elastic polymers seemed to be more suitable as they have to cope with the opening and closing movement of cracks under a dynamic load. In the next sections, an overview is given of the achievements attained within HEALCON related to the further development of the healing agents and their encapsulation.

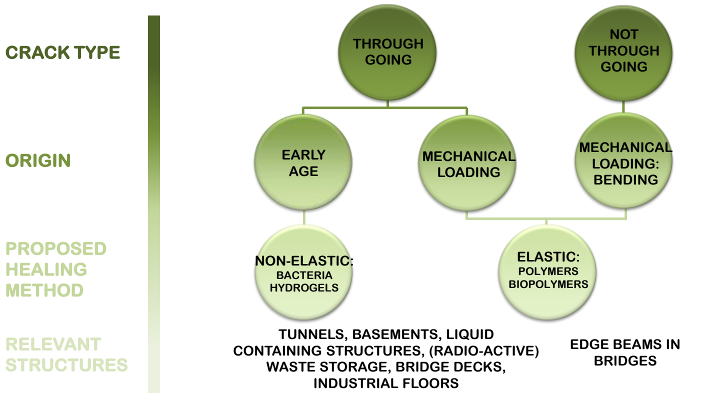

Figure 1 – Overview of the self-healing applications envisioned in HEALCON.

3.1.1 Biogenic healing agents

3.1.1.1 Micro-encapsulated Bacillus Sphaericus (main responsibles: VTT, Devan)

Incorporation of bacteria in concrete can enhance crack healing by production of $CaCO_3$, as a result of their metabolic activity and of subsequent chemical reactions including the metabolic products. While encapsulation of micro-organisms would help to increase their survival in concrete, the key element to have a successful self-healing is the choice of the bacterial strain. Promising results were obtained with micro-encapsulated *Bacillus sphaericus* spores (ureolytic bacteria in dormant state incorporated in concrete) by UGent. Within HEALCON, the biggest challenge was to fine-tune and up-scale the procedure to produce axenic (pure) cultures of *Bacillus sphaericus* and their encapsulation at lower cost. The efforts taken for that are described below.

Production of *B. sphaericus*: At VTT fermentations at 12 L volume (BioFlow fermentor) were initiated with a pure culture of *Bacillus sphaericus* LMG 22257 obtained from LMG culture collection (six fermentations). The strain was deposited in VTT Culture Collection with deposit number VTT E-153472. According to LMG, the current species name is *Lysinibacillus sphaericus* and probably belongs to *Sporosarcina* based on partial 16S rDNA sequencing. Different modifications of the MBS medium were examined in order to increase spore yield. The modified MBS medium (supplemented with thiamine) was used as growth medium and the effect of different carriers (urea, sucrose) was evaluated.

Larger scale fermentations were performed in order to produce spores for encapsulation and field trials. In total, four fermentations - two in 200 L and two in 300 L scale (IF400 fermentor) - in the modified MBS medium have been performed. Spores produced on the modified MBS medium were

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harvested either with centrifugation or with Alfa Laval separator. The concentrates were mixed with cryoprotector and freeze-dried (Christ Epsilon 225 DS lyophiliser). Numbers of viable *B. sphaericus* in the freeze-dried product varied between 10^9-10^{10} spores g^{-1}. In addition, ureolytic and $CaCO_3$ precipitation activity of the samples have been examined. Freeze-dried powders were delivered to Devan for encapsulation studies.

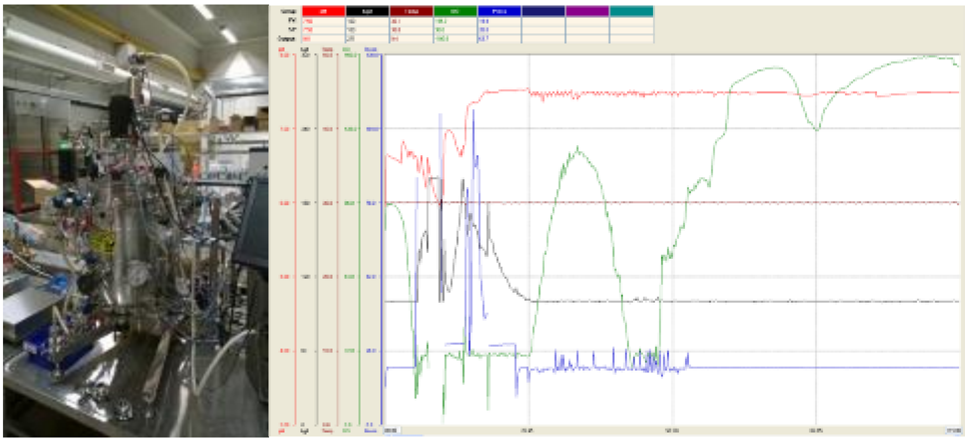

Figure 2 – Fermentor (left) and typical growth curve of *B. sphaericus* during fermentation (right).

For the techniques of master batch, spin-finishing and extrusion-spheronization, one can find all related details or research activities described in a report on co-extrusion of reinforcing fibers with bacteria spores (D3.3). In fact, some of the listed techniques were not fully pursued further throughout the project developments mainly due to related technology risks and high costs for the processing treatment. Here, the costs subject to analysis were mainly calculated from the entrapment process only and thus, not considering the cost of bacterial spores as the active ingredient.

As an important part, research activities related with compatibility of the encapsulation techniques with a few important physic-chemical aspects that shall cover most of the key variables related with the type of biogenic agent's subject to encapsulation were performed, followed by the incorporation of selected protective capsules into cementitious substrates. Parts of these activities were documented in a report on encapsulation process parameters at lab scale for the selected micro-organism (D3.1). It considers how *B. sphaericus* spores were compatible with the materials used in the proposed encapsulation techniques, followed by cementitious substrates' incorporation. For the incorporation in cementitious materials, a few parameters were considered: (1) the mixing stage and its highly abrasive shear forces involved and consequent early release by breakage upon mixing and (2) the modelling of early cracks going through a too flexible shell that may compromise the release concept proposed and fully investigated by University of Ghent prior HEALCON. There are other factors of key importance: high alkaline pH of cementitious media (12 up to 13) which may influence the protective shell chemistry and size distribution of the encapsulated healing agents that affects mechanical properties of concrete and mortars which should be equivalent to the standard concrete and mortars' counterparts.

The selected encapsulation technique was microencapsulation via *in-situ* polycondensation. As the active ingredient is in solid form through a fine mid-coarse powder, a liquid core carrier was proposed (as an excipient) that should be (1) inert to spores but possibly contributing to fast germination upon crack trigger release into cementitious media and (2) fluid enough to spread around through capillary motion aiming to cover most of the early cracks with spores for healing purposes. The core's carrier hydrophobicity property is important in order to achieve a fine spores' dispersion and at the same time, increase encapsulation efficiency. By using hydrophobic types of cores or core's carriers, it will preserve water as continuous phase in the encapsulation process otherwise solvents would be mandatory and incompatibility with the concrete aqueous media is expected. Finally the need to understand the size distribution profile of self-healing bacterial spores, how these spores are normally supplied, which other constituents are in it and how these constituents influenced the

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microencapsulation process were of relevant importance and detailed in a handbook with the constraints regarding the µ-capsules (D2.6) .

For microencapsulation activities done by Devan, UGent and AVECOM partners were focused on the use of *B. Sphaericus* as biogenic healing agent model. *Bacillus Subtilis* has been used as a case model while a substantial amount of *B. sphaericus* were being produced. Although *Bacillus Subtilis* strain is not able to heal concrete through the urease mechanism, it is a strain that is commercially used in textiles for the anti-allergen properties through its microencapsulated form of which Devan has intellectual properties of it (patent WO2010142401).

The argument of using this *B. subtilis* strain model is simple: (1) an implemented industrial encapsulation process with large capacities of production could be adapted and (2) it could be evaluated if these microcapsules would survive the mixing step without significant damage. Furthermore, the dispersion, the adhesion properties of the shell with the concrete matrix and the ability of the encapsulated spores to survive the process conditions adjusted to concrete, were investigated. With partner AVECOM, a steady protocol for assessing bacteria germination after encapsulation was developed specifically for the encapsulated *B. Sphaericus*, considering a breakage step that triggers spores release from the protective shell.

The method for producing *B. subtilis* capsules of micrometre size is by using the *in situ* polycondensation method. This method is suitable for liquid cores for shell materials based on several amino compounds crosslinked with formaldehyde. The microcapsule model proposed to use at HEALCON is a core-shell type using appropriate oil as a carrier, as Figure 3 depicts below.

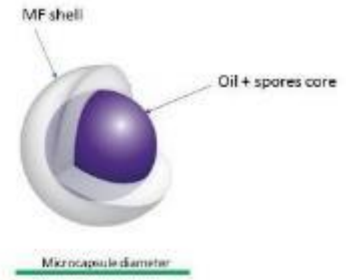

Figure 3 – Schematic representation of the proposed HEALCON core shell type of a microcapsule.

The general *in-situ* polymerization process for encapsulation of bacterial spores is shown in Figure 4. The MF (melamine crosslinked with formaldehyde) shell as expected has shown no evidences of being affected from the alkaline treatment done by immersion tests at pH 12 and 14 buffer solutions during 1 week at 30°C.

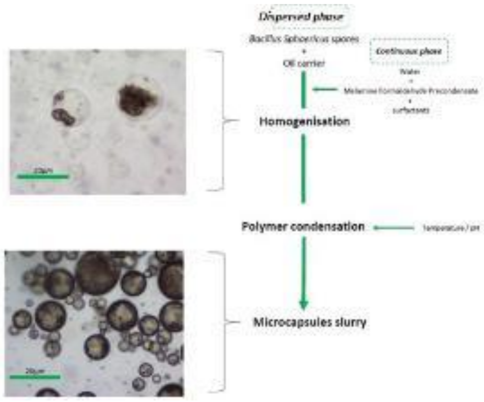

Figure 4 – General in situ polymerization process proposed for the encapsulation of bacteria spores.

Further developments proceeded with the model lab scale batches of microencapsulated cores such as phase change material (PCM) (= most resistant type of shell in Devan's portfolio) or fragrances (= most weak shells of MF) due to the fact that these are the most extreme cases in terms of shell

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resistance. The two materials were delivered to partners UGent, ACCIONA and FESCON as dummy models for concrete application assessment. It was concluded that despite some fragrances were released during the mixing stage, there are evidences of intact round shape microcapsules from images taken.
From the concrete mixing tests with dummy microencapsulated fragrances and PCM, one was able to notice the importance of smaller size distribution of particles, as, despite their shell's weakness, they were able to survive the highly abrasive mixing stage and could homogenously applied into the concrete matrix. Another important aspect is that the cationic character of concrete did not affect the non-ionic character of the microcapsules. This was proved by the non-existence of agglomerates of microcapsules into concrete as depicted in the scanning electron microscopy (SEM) micrographs (lower left and right) presented in Figure 5.

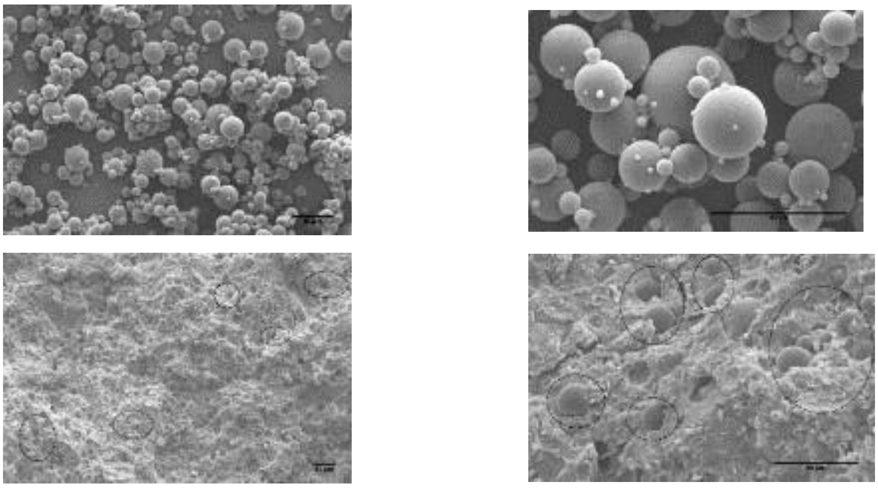

Figure 5 – Upper left and right: SEM micrographs of PCM microcapsules model at low and high magnification / Lower left and right: micrographs of cut pieces of casted mortar substrates containing PCM micrographs.

After preliminary tests using "dummy" models with UGent, VTT, FESCON and ACCIONA partners, it was already possible to conclude:

- MF shells were able to be finely mixed into concrete and mortar's substrates;
- No evidences of MF shell degradation at high alkaline pH;
- Small size distribution with percentile 50 lower than 8 µm are possible to overcome shell weakness factor;
- Adhesion of MF shell onto concrete and mortar's substrate seemed adjusted for the type of release proposed at HEALCON as fragrances microcapsules break upon treated specimens fractures (with a strong release of fragrance upon fracture) whereas PCM microcapsules (the ones designed not to break), like observed in Figure 5 (lower left and right), were strongly adhered onto the surface of fractured specimens.

From the above assumptions, refined developments started with a few grams of *B. sphaericus* and *B. subtilis* with calcium carbonate samples. Calcium carbonate was proposed by partners as an ingredient to promote germination – herein called prebiotics. However, calcium carbonate that was supplied in significant quantities has affected the microencapsulation process by turning the ionic stability very poor. This has resulted into larger quantities of free core. As a consequence, we were unable to proceed with concrete and mortar's tests. Yeast has been also proposed for encapsulation with little evidences of success due to its hydrophilic character.
Several samples of *B. sphaericus* have been received for microencapsulation trials. Those samples were delivered with different types of cryoprotector for compatibility trials and sucrose carrier has been chosen as the final candidate. The amount of sucrose in the spores' powder also differed taking in

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consideration the freeze drying throughput amount with direct impact in terms of overall process costs and hence techno-economic assessment of this healing agent solution. In consultation with the project partners, it was concluded that the best process, from a technical and economical point of view, is the method of encapsulation through the in situ polymerization process. Healing agent is released upon crack formation and it is the one already adapted for larger production and thus with a controlled cost in comparison with other approaches such as master batch or extrusion spheronization.
Therefore, the subsequent tasks for producing larger amounts of microencapsulated *B. sphaericus* spores were taken driven to the up-scaled tests while monitoring of ureolytic activity of given products. All technical solutions have been taken into consideration and the only major limitation still difficult to overcome it is the related high costs of this solution, although the main costs are coming from the active ingredient *B. sphaericus* spores powder and not from the microencapsulation process. Different reports about the techno-economical assessment and life cycle cost assessment describe in detail the cost impact of different healing agent's solutions on concrete material.

3.1.1.2 Spores impregnated in lightweight aggregates (main responsible: TUDelft)

The biogenic healing agent system developed in TUDelft targets the sealing of the micro-cracks by calcium carbonate ($CaCO_3$) precipitation induced by bacterial activity. The healing agent consists of dormant bacteria (spores incorporated into zeolite powder, Figure 6 - a) and organic mineral compounds embedded in expanded clay particles (lightweight aggregates). The bacteria are added in the healing system as spores and they are derived from alkaliphilic bacteria of the genus *Bacillus.* The spores are able to withstand the mixing and the hydration of cement and remain viable until crack occurrence. In addition, their alkaliphilic nature enables them to survive and activate in the high alkaline concrete environment. Further, the organic mineral compounds, i.e. calcium lactate (Figure 6 - b) and yeast extract (Figure 6 - c), act as feed and vitamins for the bacteria respectively. The lightweight aggregates (Liapor 1/4 mm, Liapor GmbH Germany) are used to protect and immobilize the healing agent. In the presence of oxygen and water the dormant bacterial spores are activated. Later, the active bacteria cells convert the calcium lactate, present in the healing agent into $CaCO_3$ by using oxygen. By this means limestone precipitates inside cracks and ultimately protects the concrete material from the ingress of harmful substances that can endanger its durability.

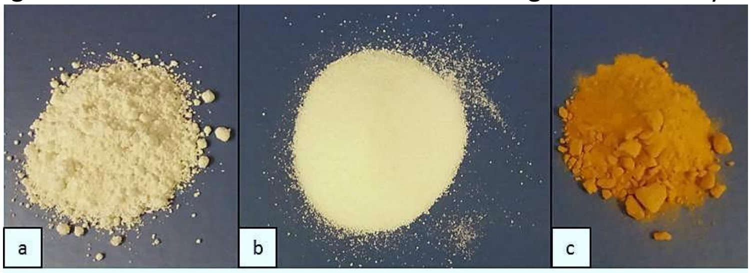

Figure 6 – Compounds used in the biogenic healing agent with lightweight aggregates: a. bacteria spores incorporated into zeolite powder; b. calcium lactate; c. yeast extract.

Calcium lactate (200 g/L) and yeast extract (4 g/L) are dissolved in water (at 75-80 °C). Subsequently, the zeolite powder is added into the solution. The solution with the three healing agent compounds is then incorporated into the lightweight aggregates via vacuum impregnation. After the impregnation, the clay particles are left to dry in order to discard the water. Figure 7 shows a loaded (with the biogenic healing agent) lightweight aggregate cut-into-two. A study (Tziviloglou et al. 2015) on the optimization of the impregnation and drying procedure revealed that the lightweight aggregates increased their initial dry weight after incorporation of the healing agent by approximately 11.3 %. After drying the loaded lightweight particles are mixed with the fresh mortar paste.

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Figure 7 – Impregnated lightweight aggregate. The white spots are the healing agent inside the pores of the particle.

In the presence of oxygen and water the dormant bacterial spores are activated. Later, the active bacteria cells convert the calcium lactate ($CaC_6H_{10}O_6$), present in the healing agent into $CaCO_3$ (see Equation 1 (Wiktor and Jonkers 2011)) by using oxygen.

$CaC_6H_{10}O_6 + 6O_2 \rightarrow CaCO_3 + 5CO_2 + 5H_2O$ (Equation 1)

Therefore, by conducting oxygen concentration measurements on loaded lightweight aggregates submerged in carbonate-bicarbonate buffer (0.1 M, pH=10.5), it was possible to evaluate the effectiveness of the biogenic healing agent system. The oxygen concentration was monitored in sealed glass flasks via optical oxygen sensors (Fibox 4 & Fibox 4 trace, Germany). Two different samples were tested: a. non-loaded (without any healing agent), b. impregnated with healing agent.

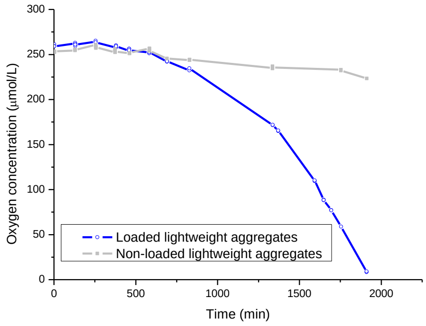

Figure 8 – Oxygen concentration measurements on unloaded and loaded Liapor particles.

The oxygen concentration measurements (Figure 8) revealed that only the samples containing the bacteria spores showed bacterial activity, i.e. oxygen consumption. Thus, the bacteria are active in relatively high pH conditions, and therefore the healing agent can be functional when incorporated in the alkaline concrete environment.

3.1.1.3 Self-protected mixed cultures (main responsible: Avecom)

Although the use of microencapsulated *B. sphaericus* gave notable self-healing properties (Wang J., 2013, De Muynck W., 2008), their production is less competitive in the concrete industry due the expensive production costs. A new production process was developed to obtain a product that contains mainly ureolytic bacteria with the capability to induce $CaCO_3$ precipitation and that is economically competitive. This product was named MUC (Mixed Ureolytic Culture).

To produce MUC (Figure 9 – left), Avecom has developed a thermal stress process for the selection and sporulation of the bacterial strains. High dosages of urea were imposed as another selective pressure condition to allow the selection of the ureolytic bacteria. A sub-stream of a vegetable treatment plant was used as inoculum. The aerobic reactor was kept at a nutrient ratio of COD (Chemical Oxygen Demand) to nitrogen (N) and phosphorus (P), i.e., COD/N/P, of about 100/110/4.5. The production cost of MUC at lab-scale was estimated as 19 Eur/Kg, which was significantly lower than the costs to produce the axenic cultures. This production process was up-scaled within the project and the products still showed capability of precipitating calcium carbonate and good ureolytic activity. Several harvest methods were tested on the produced microbial culture (gravity settling, polymer flocculation, centrifugation, heating, VSEP (Vibration Shear Enhanced Process) microfiltration) taking

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into account cost effectiveness and stability of the harvested cultures. This is in most cases a two-step process that includes harvesting and dewatering. It was concluded that using the drying tray at 60 °C was the most suitable way to obtain the final MUC powder with little time and effort requirements. With all the other methods tested it was still needed to dry the remaining paste. However, for a production larger than 50 L scale, it is advised to use a VSEP microfiltration in order to reduce the amount of product that needs to be heated in the drying tray.

The activity of different samples of biogenic healing agents was evaluated by means of germination, ureolytic and calcium carbonate precipitation activity tests. In comparison with previous samples of MUC, immediately tested for their activity after grinding, the ureolytic activity of samples tested after a storage of 2 weeks up to 3 months significantly decreased. The bacteria stored for 1 and 2 months were still able to hydrolyze > 95 % of the urea dosed, but with a delay of 1 day in comparison with the reference. When applying low temperature and/or salt conditions, the ureolytic activity is slightly lower than in the case of testing in room temperature. However, with these tests it can be concluded that this biogenic self-healing agent can also work at temperatures as low as 10 °C and also in the presence of 10 g NaCl/L. Regarding the capability of precipitating calcium carbonate, there was also a decrease in terms of activity when increasing the storage time. This means that, or there is a significant loss of activity in the first days after grinding or; the up-scaling had an influence on the activity.

The costs of producing MUC were also estimated taking into consideration an industrial scale production. This resulted in a cost of about 11 Eur/kg of MUC.

Given the still quite high cost of the MUC for the concrete market, and given the results obtained in the large scale tests in concrete, it was decided to continue the development and optimization of MUC in order to increase the quality and decrease the final production cost. Different formulations were developed and evaluated. MUC^+ was the best formulation developed and this consists of a mixture of MUC and anaerobic granules (Figure 9 – right). By incorporating this additive, more CO_2 can be produced. To have self-healing concrete, CO_2 production is needed to react with the calcium present in the concrete and in this way form calcium carbonate that can fill small cracks (self-healing concept). In the presence of urea, CO_2 and ammonia (NH_3) are produced by urease activity (hydrolysis of urea). This way, CO_2 producers and ureolytic bacteria are brought together in a mixture able to enhance self-healing capacity in concrete. The final formulation has a production cost of about 4 Eur/Kg.

Figure 9 – MUC (left) and MUC^+ (right)

3.1.2 Superabsorbent polymers

Superabsorbent polymers (SAP) have the property of absorbing large amount of liquid water. The swelling capacity of dry crosslinked SAP particles in pure water is typically several hundreds of percent. The swelling is dependent on the pH and ionic concentration of the absorbed water. In high pH of the cement slurry the swelling is significantly less. The hydrated SAP gel reversibly shrinks to the original dimension upon drying.

The large swelling capacity can be potentially utilized in sealing and self-healing of cracks in concrete. The feasible way of applying the SAP hydrogels is dispersing them in concrete during mixing. Then, as evolving cracks open channels for liquid water to access the SAP particles, the hydrogel swells and sealing of cracks takes place. Subsequently, the SAPs prevent water flow in the cracks, preferably during repeated wet and dry cycles, which facilitates self-healing by precipitation of $CaCO_3$ or by

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cement hydration products on the crack faces. A major drawback in using SAPs is that they will readily take in mixing water and swell during the concrete mixing and casting, hence generating macro pores to the concrete. HEALCON partners proposed to develop synthetic superabsorbent polymers with improved swelling and pH sensitiveness (high swelling capacity at neutral pH and lower water uptake at high pH (~ 13)). Another strategy alleviating this drawback is the encapsulation of the SAPs into a water impermeable shell. Preferably, the shell should also be fragile enough to break when the crack propagates in the vicinity of the SAP particle. Furthermore, the shell should also facilitate good mixing of the coated SAP particles in aqueous concrete mixes and mortars and promote adhesion to the cement matrix.

pH-responsive superabsorbent polymers (main responsible: CEINNMAT)

Several new formulations were developed by CEINNMAT during the project. The specification of the super absorbent polymers (SAPs) included several requirements, mainly an improved swelling, consistence and, mainly a pH sensitiveness in the concrete.

New formulation of CEINNMAT SAP-D and SAP-G showed high performance during characterization tests. A study of the commercial hydrogels with better response was made selecting some for comparison purposes (called here SAP-B).

The swelling capacity of the powdered smart SAP has been characterized by means of the tea-bag method, which allows obtaining the kinetic curves at different pH values easily, though it leads to a small overestimation of the results. This method consists of using a sealed bag permeable to water where a certain amount of dry SAP is placed before immersing it into distilled water for a period of 24h. This special bag allows maximum absorbance of water. The swelling behaviour (SW) is then calculated by the weight difference among the dry and wet samples referred to weight of dry SAP, expressed as gram of absorbed water per gram of dry product (g/g). Using this method, the swelling behaviour in different environments (distilled water, NaOH/HCl and cement slurry) has been determined for SAP-G, which is the most optimized formulation within the HEALCON project framework, and has been compared to both a previous own formulation (SAP-D) and the commercial hydrogel used as reference (Figure 10).

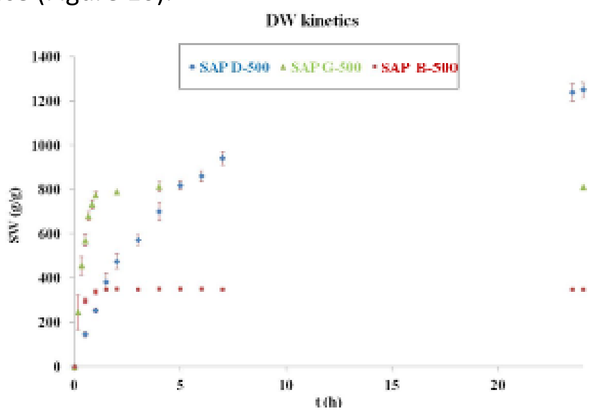

Figure 10 – SAP swelling kinetics in pure distilled water conditions.

The effect of pH on the swelling behaviour of SAPs is influenced by the ionic strength. Although different experimental approaches can be used, the addition of acid/base stocks solutions to distilled water is the methodology that allows more accurate control over the ionic strength. The duration of the immersion in each solution (acid or base) was selected considering the swelling kinetics curves obtained in pure distilled water conditions. Figure 11 shows the results of the tests for the three hydrogels. The working conditions inside the concrete matrix show a range from pH 9 to pH 13 depending on its maturity and degradation stage. The most convenient hydrogel should be the one displaying the highest difference among these pH values.

To complete these swelling tests, ON-OFF kinetics experiments were also conducted consisting of alternatively immersing the hydrogels into two solutions of different pH at which the three SAPs showed significant differences in the swelling behaviour (Figure 11). The ON value corresponded to an

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acid pH ca. 5 and the OFF value to a basic pH ca. 12. This kind of experiment was also used to detect the hysteresis of the products as a continuous decrease on their top swelling values. The hysteresis phenomena are usually associated with the durability of the response of SAPs with time (fatigue).

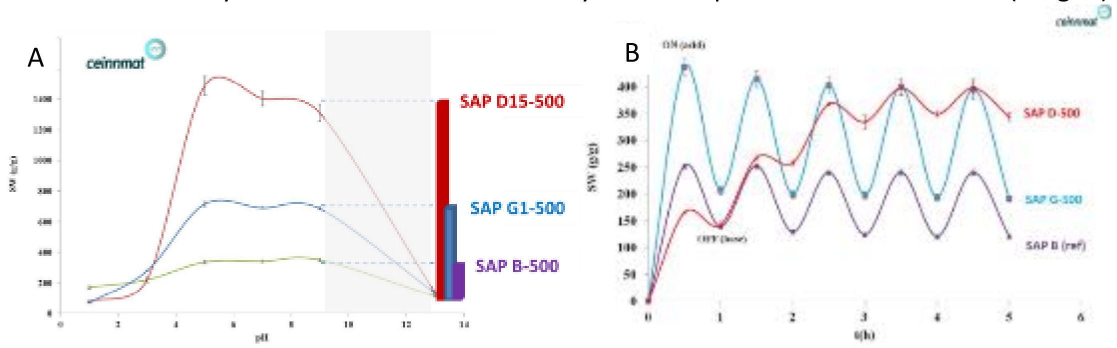

Figure 11 - A. SAP swelling at different pH solutions of grain size of 500 microns. B. Overview of the pH-sensitiveness of new formulation SAP-D and SAP-G compared with a reference (SAP-B) by means of ON (acid) – OFF (base) experiments.

The organic-based smart synthetic SAPs (D and G) have shown better swelling response in the conducted experiments. SAP-G showed a faster kinetics under pH stimuli and higher swelling/shrinkage values which resulted into a high potential capacity for concrete self-healing.
An additional treatment during SAP-G particles production, was able to inhibit the swelling effect during mixing period improving the direct application.
The synthesis of the SAPs consisted of several steps, including a reactor and purification process. A treatment of drying and milling was done later. A new synthesis method was introduced using microwave to make the process more reliable, fast assuring a good quality with a more eco-efficient process.
Scaling-up of the production has been possible following the new process that uses large reactors, in batch and the new development of low temperature microwave drying in continuous flow. The cost at industrial level can be reduced more than ten times using this process.

3.1.2.1 Coated superabsorbent polymers (main responsible: VTT)

In the HEALCON project, dehydrated commercially available SAP hydrogel particles of 400-500 µm in diameter were coated with a 15-20 µm thick water barrier layers. The coating was done in laboratory scale bottom spray fluidized bed processor available at VTT (Figure 12, Aeromatic Fielder MP-1). A bottom spray fluid bed coating process was optimized for 1.5-2 kg batch sizes. The best achieved barrier coatings enabled SAP particles being mixed in mortars and concrete without significant hydration and hence with moderate swelling of the hydrogel during the concrete mixing. Optical microscopy of fracture surface of cast concrete prisms showed the slightly swollen SAP particles were adhered to the cement matrix, forming small cavities. The SAP particles were swollen to their fully hydrated gel volume and extruded from their pores after fracturing the samples and upon exposure to pure water. Such volume expansion of the hydrogel was utilized in self-healing of cracks in concrete.

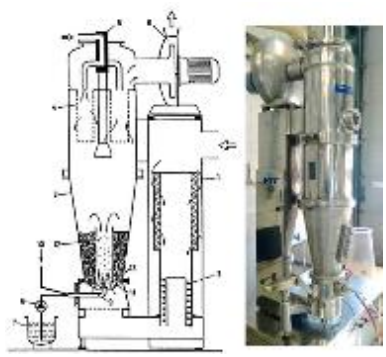

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Figure 12 - Left: principle of operation for the bottom spray fluid bed coating (Würster) process. Right: Photograph of the Aeromatic Fielder Multiprocessor (MP-1) system at VTT. The product container, filters and liquid feed pumping system have been modified to enable solvent borne processing.

Several commercially available and/or developmental primer and barrier polymers were investigated, and successfully applied on SAP particles, such as polystyrene, cyclo-olefin copolymers, and aqueous dispersions of polyurethanes, ethyl celluloses, sodium alginate etc. The polymers were chosen based on their suitability to the coating process, e.g. their solvent solubility, feasibility in high volume application (unit price), water vapor barrier and mechanical properties.

The most effective barrier coatings were based on a primer/wetting polymer layer, a hydrophobic barrier layer applied onto the primer, and an outermost inorganic sol-gel derived ceramic layer promoting the adhesion to the cement matrix. In the fluid bed coating process the polymers and precursors were sequentially sprayed onto the SAP particles from aqueous and organic solvents. Although it was not demonstrated during the laboratory scale experiments, it will be technically possible in the future to apply an up-scaled version of the developed coating process in industrial scale, with full solvent recovery system and hence (nearly) zero VOC emissions. The future up-scaling can be realized on a large bottom spray coating processor, which is commercially available up to one ton scale for the pharmaceutical and food processing industries, or by applying continuous type multi-ton scale fluid bed coating processes, which are more typically utilized in mineral processing industries. In the latter option, the desired coating performance might be potentially more difficult to achieve.

During the HEALCON project, it turned out that the most effective water barrier polymer coating formulation was a three layer coating based on cyclo-olefin copolymer (COC) barrier, polyvinyl butyral (PVB) primer (wetting) layer and an inorganic sol-gel derived zirconium-silicon dioxide adhesion promoting layer covering the COC layer. The barrier COC layers were applied in an up-scalable coating process form tetrahydrofurane (THF) solvent. The primer PVB was successfully applied directly onto the fluidized SAP particles from ethanol solvent. Moreover, dense polyurethane (PU), ethyl cellulose (EC) and sodium alginate (SA) aqueous dispersion could also be successfully deposited in the fluid bed process. Of the water-borne coatings, the PUD on SAPs showed some positive results in aqueous swelling test.

Scanning electron microscopy of the COC barrier coatings, performing the best, showed that the prismatic shaped SAP particles were homogeneously covered by the dense primer and barrier polymer layers (Figure 13). Swelling test data recorded for the barrier coated SAP particles in alkaline saturated $Ca(OH)_2$ solution indicated that the coating gave only a limited workability time in wet mixing. This was also confirmed with concrete and mortar tests. Swelling in pure water was significantly faster, typically leading to the breakage of the coating within two minutes. The performance of the barrier coating was found consistent on the theoretically predicted water vapor transmission rates of the barrier polymer layers. Performance of the coating was highly variable with the studied coating materials and the quality of the coating layer was dependent on the fluid bed coating process variables, such as the use of wetting layer for the barrier polymer, spray rate of the coating solution and the fluidizing air temperature.

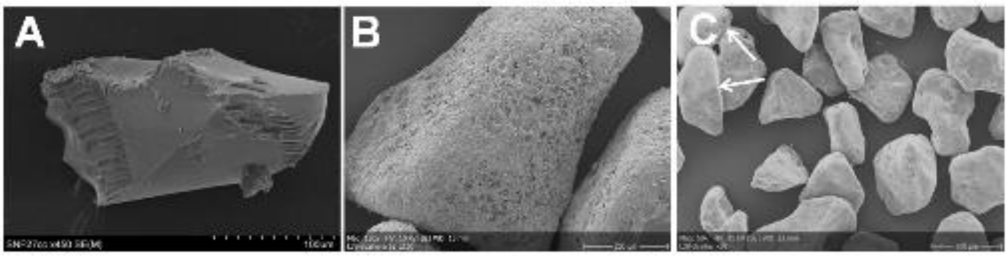

Figure 13 - Representative SEM images of (A) Floset 27cc SAP hydrogel particle. (B) and (C); COC coated Floset 27cc (magnifications 150X and 50X)

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During the HEALCON project, VTT has explored several alternative strategies to find the best barrier coating material and coating process parameters. It turned out that the performance was easily quantified by the 6 minute swelling test which correlated well with the workability in concrete mixtures. The coating performance was found to be highly dependent on the control of the fluid bed coating process. Improvements were made throughout the HEALCON project for the solvent borne and aqueous based coating processes. This is shown in Figure 14, presenting the fast swelling test results for the coated SAP batches generated between June 2014 and March 2016. The best performance was obtained in the very last coating experiments (batch no. 38), the coated SAP material was generated for the HEALCON large scale beams cast at DTI in Denmark during Spring 2016. The yellow circle indicates a PS coated SAP, white circles indicate COC coated SAPs and the crosses indicate various versions of aqueous based coatings (PUD and ECD based), respectively. The red square indicates the uncoated reference SAP. The batch number 38 (March 2016), which is indicated by asterisk had the lowest swelling (8 g/g) in aqueous saturated $Ca(OH)_2$.

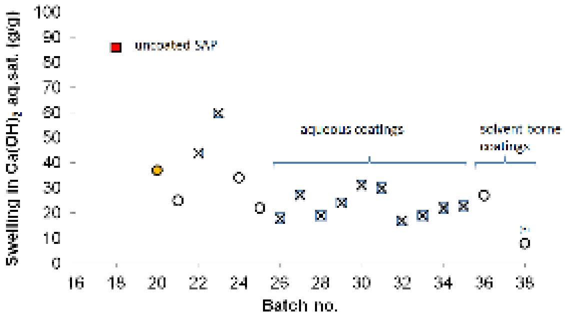

Figure 14 - Examples of swelling test results during the development work with commercially available Floset 27cc SAP. The swelling during 6 minutes was measured in aqueous saturated $Ca(OH)_2$ solution at VTT. The circles and the crosses indicate the solvent borne (PS and COC) and the aqueous based coatings (PUD and ECD), respectively. The square indicates the uncoated reference SAP. The batch number 38, indicated by an asterisk, was the batch generated for the large scale testing in Denmark (March 2016).

3.1.3 Polymeric healing agents

3.1.3.1 Selection/development of the most suitable polymer (main responsible: UGent)

A thorough set of test methods able to assess the performance of polymeric healing agents was first developed and then used to test 3 different commercial precursors of elastic polymers supplied by De Neef (Grace Chemicals). This led to testing of 8 different series, which combined the precursors with water or accelerator. Changes in reaction time, foaming capacity and mechanical properties of the resulting crosslinked polymer were then achieved.

Significant differences were found between all series in terms of healing efficiency, as assessed by capillary water absorption tests. For the series that showed good efficiency, the highest strain capacity observed was 50%, which may not fulfill the long-term requirements for dynamic cracks, assumed to be 100% (Feiteira et al. 2016). The best performing polymer precursor (Flex SLV), which lead to the widest dispersion inside the cracks and the highest healing efficiency, was selected for further assessment of its performance in terms of permeability of the healed cracks under hydrostatic pressure and resistance to fatigue caused by cyclic crack movement.

Permeability tests were performed on mortar prisms using the setup developed by TUDelft (section 2.1) and it showed that the Flex SLV precursor led to practically full recovery of water tightness after healing at all hydrostatic pressures tested, up to 2 bar (20 m water column), which would make it compatible with large water retaining structures.

Another study used mortar prisms and the ultrasound (US) analysis equipment FreshCon supplied by partner SmartMote to monitor the evolution of healing (i.e. curing of the polymer inside a crack) in a continuous way, showing that complete curing of the Flex SLV polymer precursor takes at least 48 h.

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The same setup was used to monitor failure due to fatigue caused by cyclic movement of healed cracks. For Flex SLV, over several hundreds of cycles at a 35% strain (crack increased by 35% of its original size), the amplitude of ultrasound waves transmitted through a healed crack is progressively reduced, suggesting partial detachment of the polymer from the crack faces. At a strain level of 20% however, the amplitude is stable, suggesting that the crack is still successfully bridged by the polymer. This was confirmed by microscopic analysis after the end of the tests. When combined with 5 wt% of accelerator, which induces foaming, microscopic analysis showed that for both 20% and 35% strain levels, partial detachment of the Flex SLV polymer took place. Despite this, the amplitude of the US waves transmitted through the healed cracks showed no major features other than an increase of amplitude due to new precursor being released from the capsules during the tests, which was allowed by the detachment of the polymer originally bridging the crack.
To assess the effect of the precursors' stiffness and brittle behaviour (as opposed to flexible behaviour) on the failure of healed specimens due to excessive crack movement, a small study was performed at VUB (Brussels). The study used digital image correlation, to monitor displacements and strains, and analysis of acoustic emissions to monitor any acoustic events related to damage and failure during loading of healed specimens. The major outcome of these tests was the confirmation that the use of stiff, brittle polymers results in catastrophic failure after only a small crack movement and damage is introduced in the cementitious matrix in the form of new branches of the original crack (Figure 15).

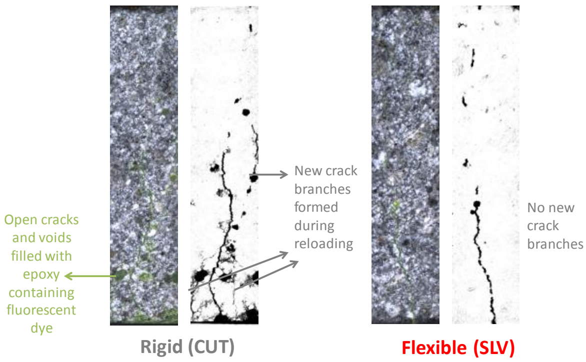

Figure 15 – Behaviour of rigid and flexible polymers upon reloading of cracked and healed specimens.

The study assessing the effect of the mechanical properties of the polymers on the failure of healed specimens also involved tests conducted at École des Mines de Douai using an SEM with a special stage that allows tensile testing of small specimens with parallel SEM observation. The continuous observation of small cement paste specimens that had been cracked and bonded with the commercial PUs allowed very accurate determination of the onset of failure due to detachment of the polymer from the cement paste matrix or rupturing of the polymer matrix. Analysis of the crack's faces also showed that the cell structure of foaming polymer precursors can have an irregular shape and potentially form open paths for water ingress (Feiteira et al. 2017).
The synthesis of a PEG-based polyurethane as an alternative to the commercially available compounds allowed fine tuning of its properties, so that they better match the requirements of the final application, i.e. healing of concrete cracks. The parameters for the reaction of the different components were optimized in collaboration with the Polymer Chemistry and Biomaterials Group (PBM), UGent and a stable compound was finally achieved after testing of different inhibitors. Through variation of the molecular weight of the compound and amount of crosslinker, different mechanical properties were achieved for the crosslinked polymer. When manual healing was applied to cracks, the PEG-based precursors showed a strain capability similar to that of the best performing commercial precursor tested, i.e. ~50% strain. However, this approach had to be abandoned, as it was found during water uptake tests through healed cracks that the PEG-based polyurethanes were suffering degradation from prolonged exposure to water. Different types of precursors (polyester, epoxy, polydimethylsiloxane and polyphenylene oxide) were further tested. All precursors were stable when exposed to water or

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cement slurry (to simulate the environment inside a concrete crack), except for polyester. The epoxy-based healing agent was found to allow high crack movement, in excess of 50% of the initial crack width. Ideally, in the near future this precursor can be combined with the required crosslinker in the same capsule to achieve a single-component product.

3.1.3.2 Encapsulation of precursors of polyurethane (main responsibles: VTT, Devan, UGent)

Partner Devan supplied to UGent batches of microcapsules, obtained through interfacial polymerization of the commercial compound which showed the best healing performance (Flex SLV). The batches contained spherical microcapsules in a wide range of sizes, with diameters below 100 µm. However, through DSC tests, it was found that the microcapsules in these first batches were unstable and the available core content (the moisture-reacting polyurethane used as healing agent) decreased at a fast rate due to the permeability of the shell. Due to this, no healing action was observed in cracked mortar samples containing these capsules. The commercial compound Flex SLV was optimized for the encapsulation process and stability was achieved. Devan supplied new batches of stable capsules with different sizes, amount of core content and levels of hydrophobicity and a batch post-functionalized with an extra shell. These changes were made to improve rupturing of the microcapsules during cracking of concrete, with effects also on the dispersion of the capsules inside the concrete matrix and the stability of their core content. The batches were incorporated in small cement paste specimens that were cracked and analysed using a scanning electron microscope (SEM). However, none of the batches resulted in improved rupturing, which continued to occur only for a small portion of the capsules that were much higher than the average size (>100 µm) or were aggregated in large clusters with an irregular shape.

Besides the microcapsules, partner VTT also tried to encapsulate the precursors of PU in polymer fibres. Several polymers with processing temperatures below +100°C were tested in VTT's laboratory scale polymer-liquid co-extrusion extrusion line. This extrusion line was equipped with a VTT in-house designed tubular nozzle (O.D. 1.0 mm). First the extrusion of potential polymers was tested without liquid injection, then with water injection and finally with liquid PU injection. In total 16 different extrusion tests were performed. A principal challenge in the extrusion process was that the melt strength of liquid filled polymer tube was not sufficient, and the pressurized liquid (water, PUR) punctured a hole on the polymer wall. In a typical experiment, stable extrusion process could be achieved with tube outer diameter of about 1 mm and polymer shell wall thickness of about 0.3 mm. The extrusion line contained also an automated sealing device, generating the closed ends to the liquid filled polymer tube. The cooling and sealing operation occurred in a water cooling path.

First, the co-extrusion of water and liquid PU was demonstrated with polycaprolactone (PCL). However, it turned out that the moisture curing PU was hardened inside the tube within a few days. Hence, it was anticipated that a more hydrophobic shell polymer would be more useful, instead of PCL. The most potential polymer grade was a hot melt polyalfaolefin glue (Evonik Vestoplast 520). However, the elongation at break of this polymer was too high for practical application in self-healing cementitious materials. Therefore, the polymer was filled with talc filler. The compound was added with a twin screw extrusion to ensure the good dispersion of filler.

The encapsulation of liquid PU was done quite well in the last extrusion trial by using the polyalphaolefin-talc compound.

The sealing of tube was done by pressing the tube with a blade when the polymer is still hot. Afterwards the formed tube was cut in separate fibres. Example of the fibres is presented in Figure 16.

Figure 16 – Example of hollow fibre capsules. Length of these fibres was about 20 mm.

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These fibres were mixed with standard mortar (EN 196-1) and prismatic samples were cast 40 x 40 x 160 mm³. After hardening, prisms were bent to rupture and the behaviour of the fibres was observed. It was found out that most of the fibres were not broken. They were almost all pulled out and only a few were broken. Adhesion between fibres and cement stone was inadequate. This adhesion had to be increased to achieve satisfactory behaviour. This was done by plasma treatment and also with polymer adhesive. Plasma treatment was made with open air plasma (Plasmatreat F6 1002S). The surface activity of hollow fibres of linear polyester derived from caprolactone monomer was improved with this treatment. It was noticed that some fibres were pulled out from the matrix. Some were broken at the crack surface. With untreated fibres almost all of the fibres were pulled out. This achieved performance was however not enough for our application. Polymer modification was done using Wacker Vinnapas 5044N to improve bond properties between polymeric material and cement stone. It is a polymer powder based on vinyl acetate and ethylene with very good tensile adhesion strength particularly on organic surfaces combined with good crack-bridging properties and good workability. In the tests it was shown that it was possible to improve polymer fibre - cement stone bond using these kinds of polymer.
Cement based dry plasters are often modified with polymers, which typically improve adhesion and flexural strength properties of hardened product. Fescon made tests with commercial dry products, how polymer modification rate influenced the adhesion between plaster and VTT hollow fibre capsules. The higher the polymer content (Vinnapas) of plaster was, the better the adhesion. The needed dosage for proper adhesion was high, up to 5% (of total dry product). Adding such amounts of polymers in standard plasters and especially concretes is not economically possible. In practise targeting adhesion powder more accurately on the fiber surface is needed.
Due to problems related with the barrier properties of the fibre wall (necessary to prevent any moisture penetrating to PU precursors), it was decided to terminate this research line .
Given the failure to develop stable polymeric capsules within the project, either tubular or in the form of microcapsules, an alternative for upscaling the technique was investigated by UGent. The fitness of glass capsules, used initially as proof of concept with a wall thickness of 0.18 mm, was assessed. Tubular glass capsules with thicker walls were used and it was found that with 0.80 mm walls the capsules had a high survival ratio (9 out of 10 for a capsule length of 30 mm) when added during the aggressive mixing process of concrete, using a conventional 50 l vertical shaft mixer. It was also confirmed that the capsules would still be able to rupture and release the healing agent if crossed by very small cracks of ~30 µm in concrete.
It was also noticed that the sealing of tubular glass capsules with a PMMA glue resulted in progressive reaction of the moisture-curing polyurethane (PU) healing agent inside the capsule, since the thin layer of PMMA allows diffusion of water molecules over time. In the short term this had a positive effect on healing, since the reaction of the PU releases CO_2 and thus creates pressure inside the capsule and improves the release of the PU into the crack once the capsules are ruptured. However, in the long term all the PU inside the capsules would be hardened and not available for healing. Thus, a similar effect was intentionally created by dissolving a small amount of Benzoyl Peroxide (BPO) in the PU healing agent. When capsules are exposed to high temperature 60-80 °C, BPO decomposes and releases CO_2, thus creating pressure. In well-sealed capsules, an addition of 1 wt% of BPO increased the area healed by the PU from ~1 cm^2 to ~4 cm^2.
Finally to achieve the desired upscaling of self-healing concrete, capsules with an external diameter of 5 mm, a wall thickness of 0.80 mm and a length of 30 mm were selected to be used in larger self-healing concrete elements, with randomly distributed capsules added during mixing. Concrete beams (55x15x15 cm^3) were moulded and, after hardening, multiple cracks were created with a 4-point bending load. It was found that a dosage of 13 capsules per litre of concrete would not result in adequate healing, with only about 4 capsules on average being crossed by each cut section. With this dosage of capsules, a small reduction of the compressive strength of ~5% was observed. New specimens with a higher dosage of ~36 capsules per litre were moulded, which led to about 8 capsules intersected by the cracked plane and a reduction in compressive strength of ~8%. Even with this dosage

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of capsules it was not possible to achieve a consistent self-sealing effect, with water still being able to flow across the cracked section for most cracks. Only 1 out of 3 large cracks, with crack mouth sizes of ~500 µm, showed no signs of water leaking. After splitting of the concrete specimen at that specific crack plane, it was confirmed that the polyurethane precursor dispersed inside the crack in such a way that it created an effective barrier against permeation of water across the cracked plane.

3.2 Evaluation of the self-healing performance of mortars

3.2.1 Testing procedures to determine the healing/sealing performance *(main responsibles: TUDelft, UGent)*

3.2.1.1 Recovery of water tightness (sealing efficiency)

The sealing efficiency of a healing agent is investigated by its ability to block the existing crack on a specimen, so that the passage of water through the crack is prevented. For this investigation, two different crack permeability tests have been developed; namely, the water absorption and the water flow tests.

The crack permeability test via water absorption uses three different types of reinforced prismatic mortar specimens; i.e. uncracked, cracked unhealed and cracked healed specimens. The test procedure is based on the method described in EN 13057 and consists of bringing the cracked face of the specimens into contact with water and monitoring the mass of absorbed water. The specimens are partially waterproofed, to maximize the influence of the crack on the total amount of water absorbed (Figure 17).

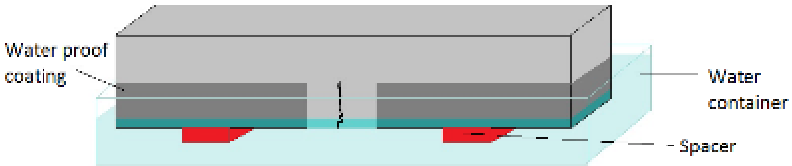

Figure 17 – Specimen specifications for capillary water absorption test.

The specimens are weighed frequently for a period of 8 h. Then the sorption coefficient of each specimen can be calculated. Using the results of the test, we can calculate the recovery of sealing as described in Equation 2.

$$SE = \frac{SC_{unhealed}-SC_{healed}}{SC_{unhealed}-SC_{Sound}} x\ 100\% \qquad \text{(Equation 2)}$$

Where,

- SE: Sealing efficiency
- $SC_{unhealed}$: Sorption coefficient for the cracked and unhealed specimen
- SC_{healed} : Sorption coefficient for the cracked and healed specimen
- SC_{sound} : Sorption coefficient for the uncracked specimen

The crack permeability test via water flow uses two types of reinforced prismatic specimens which are cracked; i.e. unhealed and healed, containing a 5 mm-hole in the middle along their length (Figure 18). Via a water-column (0.5 m), water passes through the plastic tube in the 5 mm-hole and leaks out of the crack. The mass of the dripping water is monitored through a scale placed under the crack. The set-up is depicted in Figure 18.

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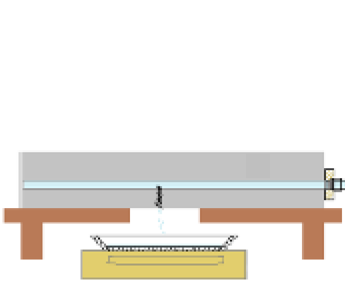

Figure 18 – Set-up of crack permeability test via water flow (Tziviloglou et al. 2014).

Using the results of the test performed on unhealed, as well as, on healed specimens we can calculate the recovery of sealing as described in Equation 3.

$$SE = \frac{W_{unhealed}(t) - W_{healed}(t)}{W_{unhealed}(t)} x\ 100\% \qquad \text{(Equation 3)}$$

Where,

- SE: Sealing efficiency calculated at test time (t)
- $\mathrm{W}_{\mathrm{unhealed}}(\mathrm{t})$: Amount of water that has passed through the specimen's unhealed crack at test time (t)
- $\mathrm{W}_{\mathrm{healed}}(\mathrm{t})$: Amount of water that has passed through the specimen's healed crack at test time (t)Within HEALCON, also some adaptations were made to the set-up in order to determine the water flow under high pressure (up to 2 bar).

3.2.1.2 Recovery of mechanical properties (healing efficiency)

The investigation of mechanical properties recovery is carried out by comparison of the values for flexural strength and stiffness obtained by crack-width-controlled 3-point-bending test, before and after healing treatment. The load is applied in the centre of the specimen and the rate of loading is such, that elongation/crack width is increased by 0.5 µm/s.

For the calculation of the strength recovery 3 types of specimens need to be tested: uncracked, cracked unhealed and cracked healed specimens. All specimens should be of the same age. The recovery of strength is calculated by using the peak load values obtained from loading curve of the abovementioned specimens, as shown in Equation 4.

$$R_{Strength} = \frac{F_{healed} - F_{unhealed}}{F_{uncracked} - F_{unhealed}} x\ 100\% \quad \text{(Equation 4)}$$

Where,

- $R_{Strength}$: Recovery of strength
- F_{healed}: Peak load of healed specimen
- $F_{unhealed}$: Peak load of unhealed specimen
- $F_{uncracked}$: Peak load of uncracked specimen

For the calculation of stiffness recovery, a method similar to the calculation of strength recovery is followed. The recovery of stiffness is calculated by using the slope value (Load/Crack opening) obtained from the loading curve of the specimens, as shown in Equation 5.

$$R_{Stiffness} = \frac{S_{healed} - S_{unhealed}}{S_{uncracked} - S_{unhealed}} x\ 100\% \quad \text{(Equation 5)}$$

Where,

- $R_{Stiffness}$: Recovery of stiffness
- S_{healed}: Slope of healed specimen
- $S_{unhealed}$: Slope of unhealed specimen
- $S_{uncracked}$: Slope of uncracked specimen

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3.2.2 Healing/sealing performance of the developed products (main responsibles: TUDelft, UGent)

Crack permeability test via water flow, as described in 2.1.1 was used to quantify the sealing performance of mortar prisms with biogenic healing agent (B) embedded in lightweight aggregates by TUDelft. Two types of mixtures were investigated. One control mixture (CTRL) with non-impregnated lightweight aggregates and one mixture (B) with impregnated lightweight aggregates. Damage introduction was performed on 28-days-old reinforced prismatic specimens via 3-point-bending test. The specimens were loaded until the formation of a 350-µm-wide crack. Following the crack creation, 6 specimens of each mixture were placed horizontally in a plastic container filled with tap water for crack healing. The container was kept open to the atmosphere at standard room temperature (20 ± 2 °C) with (60 ± 10) % RH for 28 or 56 days. Another 6 specimens of each mixture were subjected to wet and dry cycles for 28 or 56 days. Each cycle lasted 12 hours. The container was kept open to the atmosphere at standard room temperature (20 ± 2) °C with (60 ± 10) % RH.
Figure 19 shows the average (out of three specimens) performance of the two different mortar mixtures before and after healing treatment.

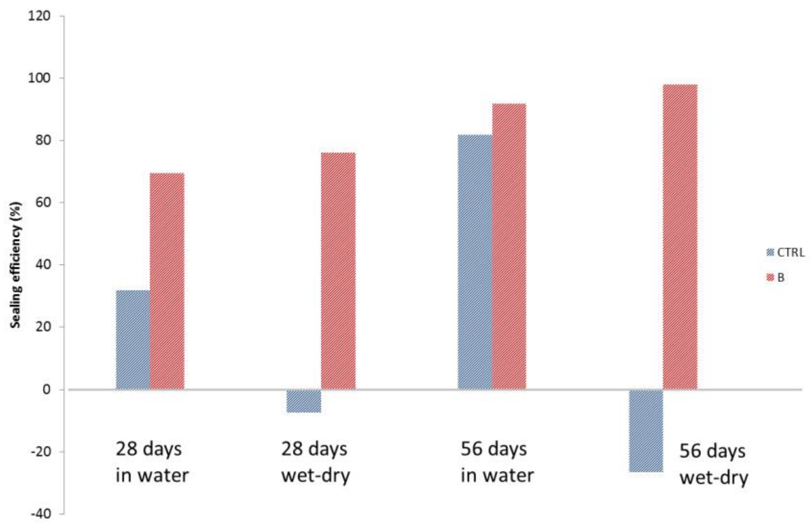

Figure 19 – Sealing efficiency (in %) of biogenic healing agents. The calculations are based on Eq. 3. (Tziviloglou et al. 2014).

The results revealed that the biogenic healing agent exhibits higher sealing efficiency, when compared to control samples, taking into account that the two types of mortar had similar initial crack width on average. The sealing efficiency of the specimens submerged under water for 28 days was different for CTRL and B specimens. In fact, B specimens recovered 69 % of the initial water tightness, while CTRL specimens recovered only 32 %. On the other hand, the sealing efficiency after 56 days of water immersion was very similar for specimens with or without the healing agent. However, the results differed for the specimens that were subjected to wet-dry cycles. The specimens without healing agent exhibited considerably lower recovery of water tightness when compared to B specimens. Particularly, for the CTRL specimens subjected to wet-dry cycles the flow of water out of the crack was even higher than the flow before the healing treatment (negative values). A possible explanation could be that the wet/dry cycles promoted the corrosion of the steel bars existing in the specimens. Therefore, the expansive corrosion products caused the dilation of the crack and consequently a higher water flow. In contrast, the availability of oxygen during dry cycles seems to help the bacterial activity in the B specimens. The increased oxygen concentration led to an improved $CaCO_3$ precipitation. Therefore, the B specimens exhibited an enhanced recovery of water tightness compared with CTRL specimens during wet/dry cycles.

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The specimens were subjected to 3-point-bending after healing, in order to examine the strength and stiffness recovery. However, it was observed that there was no sign of recovery of strength or of stiffness. The fact was expected, since the healing product ($CaCO_3$) is quite brittle and cannot provide the specimen with enhanced strength and stiffness properties.

Similar tests as described above were also performed on the other healing agents by UGent. Water flow tests revealed that cracks with a width less than 0.140 mm could be completely healed autogenously after 28 wet-dry cycles. However, for crack widths which were slightly higher [0.140 mm – 0.160 mm], the beneficial effect of the incorporation of healing agents became clear. As can be seen in Table 1, the SAP G, developed by CEINNMAT, performed slightly better than the commercial SAPs tested here and the sealing efficiency for the SAP G specimens attained almost 100% (Gruyaert et al. 2016). Also for the coated SAP, the sealing was almost perfect. By the presence of SAPs at the crack faces, an immediate sealing effect is also expected and this could also be noticed during the initial water flow tests (by comparing the water flow through the reference specimens and the SAP specimens). It has however to be remarked that for crack widths reaching 0.2 mm and more the healing efficiency drastically decreased for mortar with SAPs: e.g. for the coated SAPs a sealing efficiency of only 49% was recorded for crack widths of ~ 0.190 mm.

Water flow tests also showed that the biogenic healing agents (e.g. MUC^+ in Table 1) enhance crack sealing. For the micro-encapsulated spores, cracks of ~ 0.280 mm could almost completely be healed (SE of 96%) within 28 wet-dry cycles. This shows the potential of the biogenic healing agents to heal large cracks (Gruyaert et al. 2016).

Table 1 – Sealing efficiency (in %) of cracks [0.14 – 0.16 mm] in mortar specimens after 28 wet-dry cycles (12h wet – 12h dry). SAP was dosed at 1 m% relative to the cement content, while MUC^+ was dosed at 3m% relative to the cement content. Also urea and $Ca(NO_3)_2.4H_2O$ was added to the mixes with MUC^+.

Crack width	REF	SAP G	Coated SAP	Commercial SAP	MUC^+
[0.14-0.16 mm]	50	98	97	87	97

The performance of the polymeric healing agents (SLV) was also evaluated by means of the capillary water absorption test and water flow test (Gruyaert et al. 2015). An example is given in Figure 20, showing that perfect sealing is obtained for crack widths of 0.2-0.25 mm by self-healing with PU according to the water absorption test. The water flow test revealed that, at a pressure of 0.05 bar, the water flow through the cracked reference specimens was at average 5 g/min, while no water flow was detected for both the manually and self-healed specimens with PU. This showed the good sealing capacity of the polymeric healing agent which was thus 100%. All manually healed specimens could also withstand a water pressure up to 2 bar (no water flow, even at that high pressure). This was also the case for two out of the three specimens which were self-healed and subjected to the water flow test with high pressure. This means that for one self-healed specimen, a water flow was detected (but was very limited in comparison to the reference) and due to the higher pressure the sealing was partly damaged.

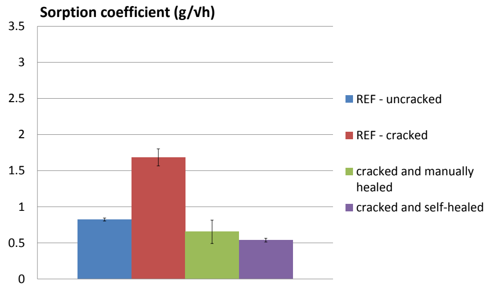

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Figure 20 – Capillary water absorption test on uncracked and cracked reference specimens (without healing agent) and cracked and healed specimens (with PU). The crack width was ~ 0.250 mm. Due to the leakage of healing agent at the mortar surface, the sorption coefficient of the cracked and healed specimens is slightly lower than that of uncracked reference specimens.

3.2.3 Influence on the fresh and hardened mortar properties (main responsible: TUDelft, UGent)

The effect that the biogenic healing agent with lightweight aggregates has on mortar was investigated through the comparison of several properties of the mixtures with and without the healing agent. The fresh properties that were tested; i.e. consistency, air content and bulk density were according to EN 1015-3, EN 1015-7 and EN 1015-10, respectively (Table 2). The tests revealed that the replacement of normal weight sand with lightweight aggregates resulted in a major decrease of the bulk density and tended to increase the air content, while it hardly affected the consistency of the fresh mixture. Further, the presence of the healing agent influenced all of the abovementioned characteristics leading in a lighter and more flowable mortar (Tziviloglou et al. 2016).

Table 2 - Fresh state properties of mortar mixtures (Tziviloglou et al. 2016)

Property	**REF**	**CTRL**	**B**
Flow (mm)	145	155	185
Density (kg/m³)	2192	1652	1546
Air content (%)	5	8	14

Regarding mortar specimens with SAPs, there was still a need to compensate for the uptake of mixing water by the SAPs, although SAP G is pH sensitive and was developed in order to diminish this effect. In comparison to the commercial SAPs, the extra amount of water needed (18 g/g SAP vs. 20 g/g SAP) was only slightly lower. For the coated SAPs, no extra water was added to the mortar mixes tested in the laboratory, but these mixes lost however partly their consistency by the time it was measured (flow value of 145 mm instead of 190 mm for the reference mixes), indicating that the coating is not completely effective within the casting time.
Besides the effect of the incorporation of healing agents on the fresh properties also the effect on the mechanical properties was tested. Figure 21 shows the results from flexural and compressive tests on hardened prisms (REF, CTRL and B (bacteria impregnated in Liapor) as described in EN 1015-11 and determined by TUDelft. According to the tests results, the biogenic healing agent affected considerably the hardened properties of the mixture at the age of 3 days, leading in a weaker material. However, after 7 days the flexural strength of all three types of mixtures exhibited similar values. Additionally, the compressive strength of specimens with normal weight aggregates was constantly higher at all ages compared to the other two mixtures with the lightweight aggregates. Finally, both CTRL and B mixtures showed similar compressive strength after the age of 7 days.

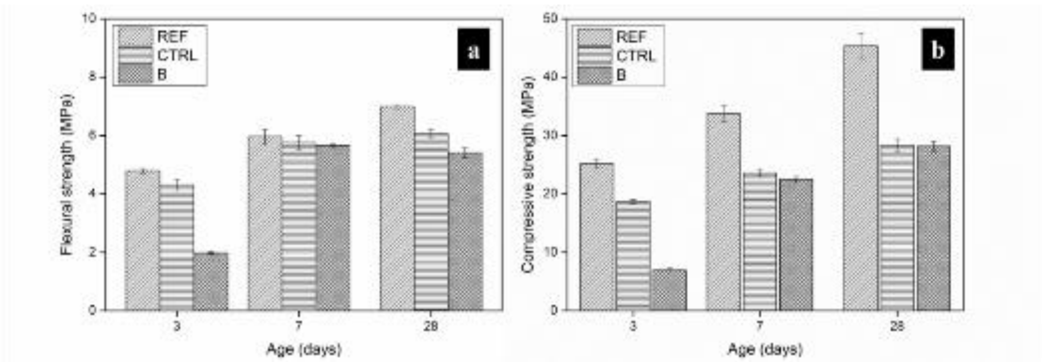

Figure 21 – a. Average flexural strength of prismatic specimens at 3, 7 and 28 days and b. Average compressive strength of prismatic specimens at 3, 7 and 28 days (Tziviloglou et al. 2016).

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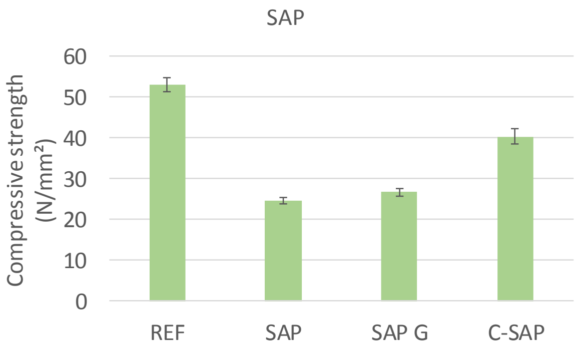
Fescon tested the application of the healing agents in ready mixed mortar. From dry-plaster point of view, there are a couple of factors, which must be taken into account. Besides wet-mixing, the capsules must survive also the dry-mixing phase and application stresses, which differ considerably from the conventional casting process. Fescon tested unmodified Liapor capsules in two different dry-mixer types. Mixer type and mixing time did affect how Liapor particles survived the process. The longer the mixing time, the more particles were broken. Mixers differ from each other by many factors, for example by number of axles, by speed of axles and by space between mixer-wall and spindle. All those factors affect differently to Liapor. A trial was made to make mortar with more expanded clay particles. By adding 30-90 kg/ton fly ash, pumping was possible of mortar with 14 weight-% expanded clay with size up to 4 mm. Also SF and $CaCO_3$ were tried. 28 d strength was 20-25 MPa, while 33 MPa if only with expanded clay and 40 MPa for the normal reference. After both dry (mixing) and wet (mixing, pumping and spraying) process the expanded clay was not ruptured.

Fescon sees potential of self-healing especially in dry-mixed (wet-method) shotcretes. There are extra forces caused by pressured air. There are possibilities to choose between pumping-equipments, for example the size of motor, hoses and nozzles. Nevertheless, the mix must be designed by technical and also by application demands. That design must be done (dry-)product by (dry-) product and by capsule type. An application in shotcrete was tested. Wet shotcrete (grain size 4 mm) with expanded clay particles and bacteria was tested (5 kg per 1000 kg of dry product). Higher dosages gave workability problems. At the beginning of spraying, there was some rebound of the expanded clay particles. Layer thickness was about 20 mm. Samples were then exposed to low temperatures (-28°C outside).

To use healing agents in dry concrete and plasters, adjustments in the recipe will be needed for technical and workability reasons. Pre-testing is needed.

At UGent, additional tests were done in order to investigate the effect of the other healing agents, developed within HEALCON, on the mechanical properties of mortar. Also other influencing parameters were considered (dosage, addition of nutrients for the bacteria, etc.). In Figure 22, an example is given. Here, healing agents (+ urea for the MUC and MUC^+; + urea, yeast and calcium nitrate for the micro-encapsulated spores) are just added 'on top' of a standard mortar mixture, except for SAP and SAP G where additional water was added in order to compensate for the uptake of mixing water. As can be seen, all healing agents have a negative effect on the compressive strength of mortar, which depends on the used dosage. For each healing agent, a compromise has to be sought in order to obtain a good self-healing efficiency with minimum reduction of the mechanical properties and tailor-made design of the mixes will be imposed.



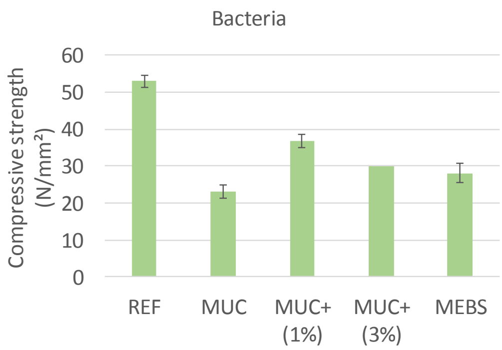


Figure 22- Infuence of the addition of healing agent on the compressive strength of mortar. Dosages: all types of SAP: 1 m% relative to the cement content; MUC and MUC^+ (1%): 1 m%; MUC^+ (3%) and Micro-encapsulated bacteria spores: 3m%.

Fescon tested also encapsulated SAPs in wet shotcrete product. The application was made by hand (not shotcreting). Workability-time was reduced down to 5-10 minutes if both dry- and wet mixing was done. In case of just wet mixing, workability time was approximately 10-20 min. Wihout SAP,

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workability time was approximately 1 hour. Regarding self- healing, SAP clearly filled cracks in still water. Healing was to be seen up to 0.6 mm, but only locally. This means the dosage of SAP needs some optimizing.

Furhermore also some tests were done with jointing grouts containing Devan's microcapsules. Only wet mixing process was included. Here also plasticity reduced at higher dosages. This time (hand application), the effect on compressive strength was not that dramatical as earlier with Devan capsules after wet shotcreting (with wet shotcrete). Minor healing process could be seen in still water.

3.2.4 Influence on the fresh and hardened concrete properties (main responsible: DTI, Acciona)

In connection with the mix design development of concrete for both large scale lab testing and field testing, DTI has tested the influence of a number of different healing agents on fresh and hardened concrete properties. The healing agents tested were MUC, MUC^{++} (contained straw fibers), impregnated Liapor, coated SAP and SAP-G.

The MUC (5 kg/m^3) did not have any negative effect on the fresh concrete properties, but for MUC^{++} (4 kg/m^3 plus 2 kg/m^3 urea), it was necessary to add twice the reference amount of superplasticizer and the concrete was relatively tough (probably due to the straw fibers in the MUC^{++}). Impregnated Liapor (167 kg/m^3) was added as replacement of aggregate and this had no negative effect on the fresh concrete properties. The coated SAP (5.2 kg/m^3) influenced the consistency of the concrete, due to the water uptake process (extra water was added in the mix design to account for this) that started 5-10 min after mixing. Within 30 min from mixing, the concrete went from being a SCC (slump flow of 550 mm) to having no workability. For SAP-G (3 kg/m^3), extra water was also added to account for the water uptake, but furthermore, it was necessary to add 3.5 times the reference amount of superplasticizer. The concrete did however not lose consistency within one hour.

The 7 and 28 day compressive strength values of the concretes together with the 28 day chloride migration coefficients are compared in the Table 3. It should be noted that the SAP concretes are not air entrained.

Table 3 – 7 and 28 days compressive strength and 28 days chloride migration coefficients for the reference concrete and different types of self-healing concrete

	7-days strength [MPa]	28-days strength [MPa]	28-days CMC [x 10^{-12} m^2/s]
Reference	30.7	51.8	15.5
MUC	30.5	50.4	16.3
MUC++	23.2	42.2	16.3
Impregnated Liapor	23.9	38.2	14.7
Coated SAP	31.3	51.1	-
SAP-G	19.5	41.4	22.5

Particles of liapor without self-healing agents were incorporated into concrete by Acciona. Liapor particles decrease the workability of concrete and a higher amount of superplasticizer is needed in order to obtain the original workability of concrete without liapor maintaining the water/cement ratio. The workability decreases even more when bacteria were incorporated into lightweight aggregates. Even so, the amount of superplasticizer is lower than needed in for concrete with SAP G and ME (micro-encapsulated) spores. Liapor with impregnated bacteria was added into the concrete mix replacing 20 vol% of aggregates, micro-encapsulated (ME) spores slurry contains 40 wt% of dried microcapsules

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and was added in a dosage of 6 wt% relative to cement weight and SAP-G was added in a dosage of 0.5 wt% relative to the cement weight.
Density of liapor is lower than the other aggregates and this physical property is important in casting process since the vibrating time has to be lower than for conventional concrete in order to avoid floating of particles on the surface. In addition, liapor particles with self-healing agents increase the setting time of concrete.
Influence of fragrance capsules provided by Devan in fresh concrete was also tested to expect the influence of micro-encapsulated spores on the fresh properties of concrete. The behaviour of this kind of capsules is similar than the capsules with bacterial spores. In this case, the workability of concrete was slightly affected and a slight increase of superplasticizer amount was needed. In self-healing concrete with micro-encapsulated spores, not only spores have to be included in the mix-design, but alsonutrients for bacteria have to be included. In this case, the amount of superplasticizer has to be even higher than for concrete with fragrance capsules.
SAP G was incorporated into the concrete and the workability decreased due to the absorption of water. Higher amount of superplasticizer was necessary to get the same workability than the concrete used in other specimens (with self-healing agents and reference).
The inclusion of self-healing agents into concrete decreases thus the workability and higher amount of superplasticizer is needed.
Compatibility of Sika Viscocrete 5970 superplasticizer regarding yeast extract has been tested using the Marsh Cone. The flow time gives an indication on the viscosity, which depends upon cement-superpasticizer compatibility. In this case, as the flowability increased with the amount of superplasticizer (Figure 23), it could be concluded that the superplasticizer was compatible within the mix.

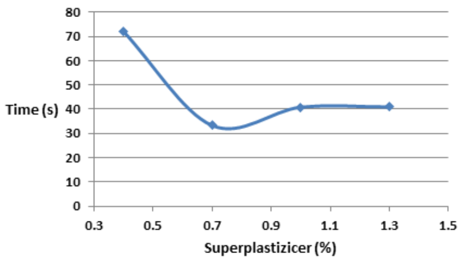

Figure 23 – Compatibility test between cement, yeast extract and superplasticizer – Marsh cone test

Regarding mechanical properties, the compressive strength decreased when self-healing agents were incorporated to concrete as shown in Table 4. Liapor with impregnated bacteria was added into the concrete mix replacing 20 vol% of aggregates, micro-encapsulated (ME) spores slurry contains 40 wt% of dried microcapsules and was added in a dosage of 6 wt% relative to cement weight and SAP-G was added in a dosage of 0.5 wt% relative to the cement weight.

Table 4 – Compressive strength values of concrete with and without healing agent.

Compressive Strength	**REF**	**Liapor**	**ME spores**	**SAP G**
7-days strength (MPa)	36.4	31.1	25.3	34.7
28-days strength (MPa)	47.1	38.7	33,0	41.2

3.3 Non-destructive testing

3.3.1 Non-destructive techniques (main responsible: TUM)

The main objective of the application of non-destructive testing (NDT) techniques was to study the healing efficiency of the different self-healing methods investigated in this project. Therefore several different techniques, utilizing mainly the elastic wave propagation in cementitious material, have been used to characterize the material properties. Based on preliminary work, potential NDT applications as

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for instance acoustic emission (AE) analysis already demonstrated its suitability for this application. The first task was to enhance the variety of NDT techniques and to deliver information about e.g. the elastic moduli, wave velocities and permittivity. In cooperation with UGent and TUDelft, first small-scale test specimens with a size of 55x15x15 cm³ have been prepared. Predetermined breaking points and additional reinforcement bars (diameter of 6 mm) enabled to achieve crack width controlled cracks by 3-point-bending tests. Subsequently, test specimens with different healing agents were investigated:

1) Glass capillaries filled with precursors of PU:
   - Flex SLV
   - MEYCO MP 355 1K
2) SAPs: FLOSET CC 27 (commercial product)
3) Biogenic healing agents (bacteria impregnated liapor particles)

Since every method has an individual healing methodology, the monitoring procedure has to be adjusted individually. Thus, new approaches are necessary to perform measurements which allow determining their potential of self-healing. In the first part, laboratory tests demonstrated that selected NDT techniques are able to define the material characteristics in the initial, cracked and healed state. The developed testing procedure for the evaluation of the healing efficiency is shown in Figure 24. Three main states of the individual test specimens are decisive:

- Initial: Specimens are in a sound and undamaged state.
- Cracked: Crack inducement has been applied. Different crack widths for each segment by a 3-point loading operation.
- Healed (bacteria-based): Cyclic water exposure for 8 or 6 weeks has been applied. Additionally, the beams were dried for two weeks before execution of NDT tests.
- Healed (PU-based): Beams were stored in a climate chamber (20°C/65% humidity) between 3 and 7 days. The curing process was monitored in-situ.

Figure 24 – Non-destructive testing procedure for the evaluation of self-healing efficiency.

Besides single measurements at a certain point in time, ultrasonic monitoring techniques for real-time studies were applied. Based on investigations of the setting and hardening process of fresh concrete, the analysis method was adapted to monitor the continuous healing process of polymeric healing agents. As a result, distinct types of injection materials can be evaluated separately in terms of hardening time and even in its real-time hardening progress. After the healing period a localization of recorded acoustic emissions, emerged while reloading the beam, confirmed the ultrasonic monitoring results. Furthermore, elastic moduli determined by vibration analysis revealed sufficiently sensitive data to compare different states of healing efficiency. Subjected to the existence of corresponding reference specimens, this concept enables to quantify the individual healing efficiency of the investigated healing agents. For specimens with polymeric healing agents, it was shown that cracking as well as healing can be clearly monitored. With acoustic emission, concrete and capsule rupture and failure of the polymeric healing agent could be detected and localized. The ultrasonic transmission method has proved essentially effective for the determination of the crack depth and its propagation during all different states (initial, cracked, healed). Real-time monitoring of the evolution of ultrasonic waves allowed to follow continuously the cracking and healing process.

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For test specimens with SAP and impregnated or encapsulated bacteria, modified curing procedures were prepared. Wet-dry cycles between 6 and 8 weeks should cause optimized autonomous (bacteria-based) or enhanced autogenous healing conditions. Due to water immersion (specimens completely under water) not only the activation of the healing agents is triggered, but also microstructural modifications of the concrete or mortar are provoked. These material changes could be detected and exhibited significant impacts in the NDT analysis results. This hampered the evaluation of the self-healing efficiency to some extent. Finally, for instance, impregnated bacteria-based concrete beams revealed analogous analysis parameter as concrete with non-impregnated clay particles. For the test specimens with implemented hydrogels no additional $CaCO_3$ precipitation could be examined in a long-term curing period, similar to bacteria-based specimens.

3.3.2 Monitoring techniques (main responsible: TTI)

TTIs main contribution to the HEALCON project was the development of a wireless monitoring system, suitable to continuously assess the concrete health and healing success in the field. Prototypes of this wireless systems were installed as part of the demonstrator installations (section 4), and are available for use beyond the project. TTI supported the work of other project partners by providing real time measurement data and provided support in access and interpretation of that data whenever this was requested. The assessment of available monitoring techniques at the beginning of the project suggested that impedance and potential measurements would likely provide most insight into the processes within the concrete, supplemented by environmental data such as temperature and humidity, both within the concrete and outside. TTI developed both hard and software to measure these parameters with wireless, battery powered sensors. A lot of work went into the design of a database framework and web based interface application to store and process the multi-dimensional data provided by the active impedance sensors which conduct a frequency sweep from 10 Hz to 100 kHz during each measurement to characterise the electrical properties of the concrete. In house lab scale tests were done using 15x5x5 cm concrete prisms, with and without healing agents. These were cracked and subsequently immersed in chloride solution, while constantly being monitored. Data from these experiments, were used to adapt TTIs upscaling strategy for the large scale laboratory tests. Wireless Sensor networks for monitoring were installed both at the large scale tests in Spainand Denmark. Monitoring data from both installations is being collected on a server in Germany and made available to project partners through a web interface in real time. While the impedance measurements provided interesting insight into the electrochemistry in small scale samples under well controlled conditions, the technique is not well suited for upscaling for large scale tests or field use. Although still included in the large scale test in Spain, far more satisfying results are achieved using the potential measurements. These measure the electrochemical potential between reinforcement and a segmented Nickel multi-reference electrode embedded in the concrete. The potential distribution is very sensitive to reinforcement corrosion processes, which occur in combination with cracks if corrosives enter the concrete and reach the reinforcement steel. As such, the monitoring does not only indicate damage to the concrete, it can also locate the damage within longer structures within the accuracy of the individual electrode segments length. As such this wireless monitoring data can give a continuous, real time health assessment of the monitored concrete structure. The monitoring sensor results were cross-checked with traditional in situ measurements and verified by project partners. Monitoring works reliably and provides the desired results with all types of tested self-healing concrete, including traditional concrete.

3.4 Modelling of self-healing concrete

3.4.1 Simulation aided design of tubular polymeric capsules for self-healing concrete

At TU Delft, in collaboration with UGent, numerical models have been used to help design tubular polymeric capsules for their use in self-healing concrete. Encapsulation protects the healing agents

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from undesired or premature reactions and degradation, to guarantee their availability at the onset of damage in the host concrete matrix. The liquid agents are then released from the capsules typically due to mechanical triggers, i.e. once a certain damage level in concrete is achieved, after which a crack crosses the capsule and eventually causes its rupture.

Other than being able to effectively release the healing agent after the onset of damage, capsules need to meet other more basic, but challenging requirements. They have to resist the mechanical stresses experienced during placing of concrete, in case of pre-placement of the capsules in the formwork, or during the mixing process, if added to fresh concrete during mixing. The material used for the capsules also has to be compatible with both the healing agent on the inside and the aggressive, high pH environment of the concrete matrix on the outside. Furthermore, the capsule’s wall has to have adequate barrier properties, with low permeability and diffusivity, to be able to retain its content but also to avoid any undesired chemical interaction between the healing agent and the concrete matrix. Finally, capsules have to rupture for very low imposed deformations, so that they release their content when crossed by a crack in concrete.

In general it is thought that polymeric capsules can fulfil the first requirement. The second requirement is more problematic: most polymers are very ductile, so the capsules do not break when concrete does. The model proposed is used to numerically test whether different geometries (diameters and wall thicknesses) and different polymeric materials are suitable for the purpose.

In this work, the Delft lattice model is used to simulate rupture of tubular capsules subjected to mechanical loading. In these models, material is discretized as a set of small truss or beam elements that can transfer forces. In the Delft lattice model as used herein, all individual elements exhibit linear elastic behaviour. The fracture simulation is achieved by performing a linear elastic analysis of the lattice under loading, and removing an element which exceeds a prescribed fracture criterion (e.g. strength, strain, or energy) from the mesh. This analysis is then repeated in a step-wise manner, removing a single element in each-step. Thus, a non-linear analysis is performed by actually performing a number of linear analyses. Using this method, realistic crack patterns are found.

In the simulations so far, a 30x30x30 mm^3 mortar block with a single tubular capsule is simulated. It is schematically shown in Figure 25. It is subjected to uniaxial tension along the axis of the capsule, and the breakage of the capsule is monitored. The mortar is simulated as having a Young’s modulus of 20 GPa and a tensile strength of 3.5 MPa.

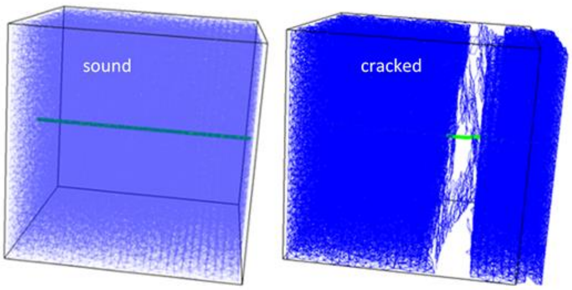

Figure 25 - Lattice with an embedded tubular capsule.

Different polymeric materials have been tested, with their mechanical properties shown in Figure 26.

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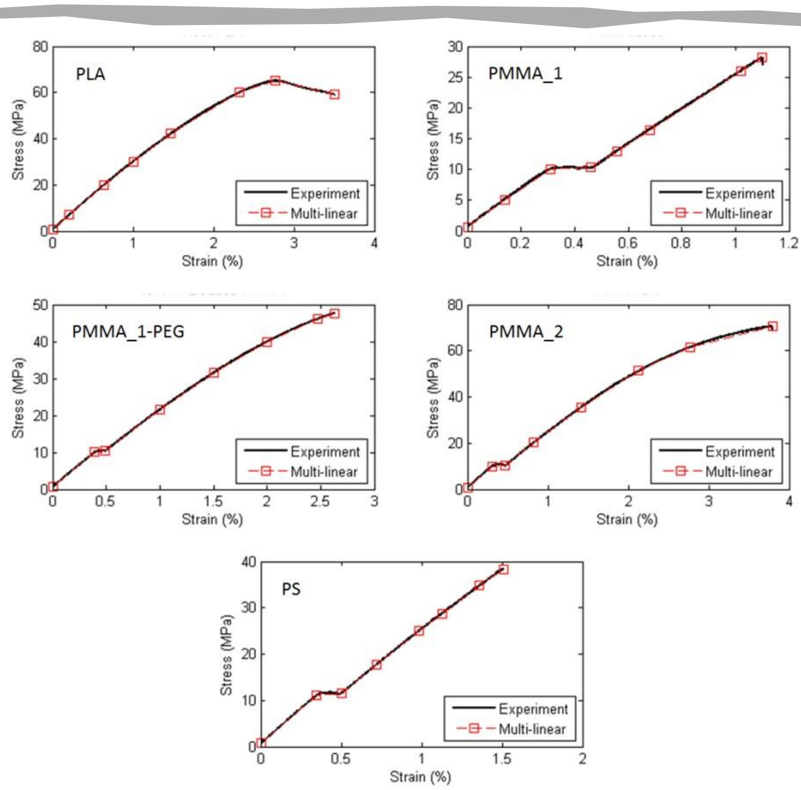

Figure 26 - Experimental and schematized stress/strain relationships of encapsulation materials

An example of the simulation output is given in Figure 27.

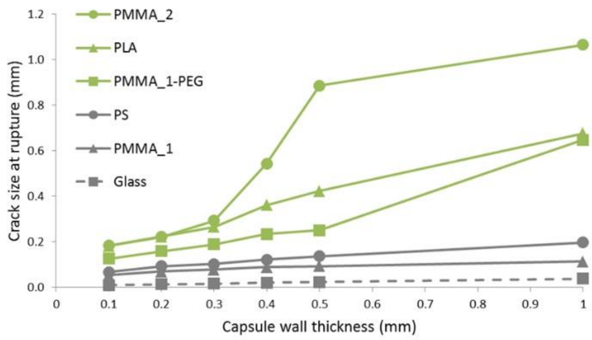

Figure 27- Model output for tubular capsules with an external diameter of 5 mm embedded in a mortar matrix under tensile stress

The model is available for the future and will be used in design of tubular carriers.

3.4.2 Modelling of self-healing in Liapor and hydrogel based systems

TU Delft proposed a self-healing system by using the Liapor, which is a kind of Light Weight Aggregate (LWA), as the capsule to hold the biological bacterium and other healing agents, mainly calcium lactate (Figure 28). A numerical model was developed to evaluate the self-healing efficiency based on a given volume fraction of Liapor used in this self-healing system, as well as optimize the mix proportion of the mortar. The results show that a satisfying self-healing efficiency can be expected with the volume fraction of Liapor in the range of 20% ~ 30%, even when the crack width is as large as 0.2 mm. Also, it is found that the self-healing efficiency is dependent on the crack width, but independent on the crack depth.

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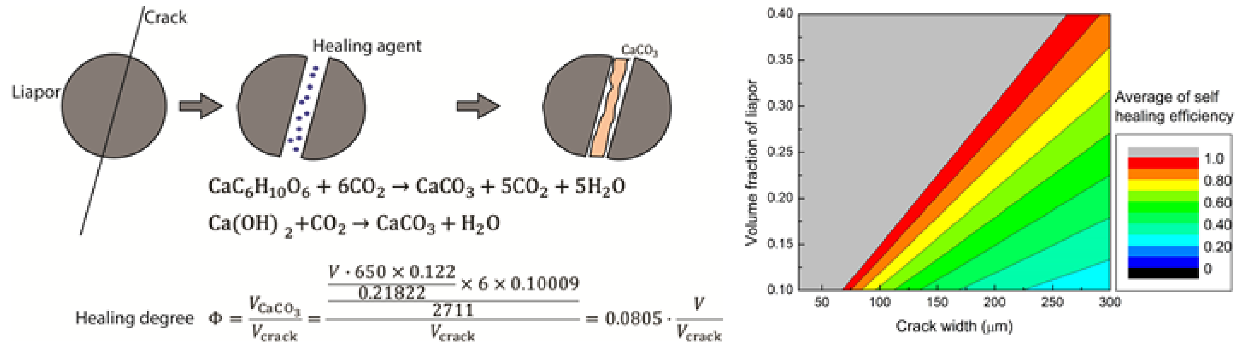

Figure 28 - Mechanism and ultimate efficiency of self-healing system proposed by TU Delft

UGent proposed a self-healing system by using the further cement hydration. To ensure a good water supply, the Super Absorbent Polymer (SAP) is introduced in this system (Figure 29). A numerical multi-scale (meso and micro scales) model was developed to evaluate the self-healing efficiency of this system. The results show that, (1) the efficiency of the entire system is mainly controlled by the type, usage and curing of the cement in the mix proportion of the mortar, instead of SAP; (2) only the crack with a very small width can be fully physically healed by this self-healing system.

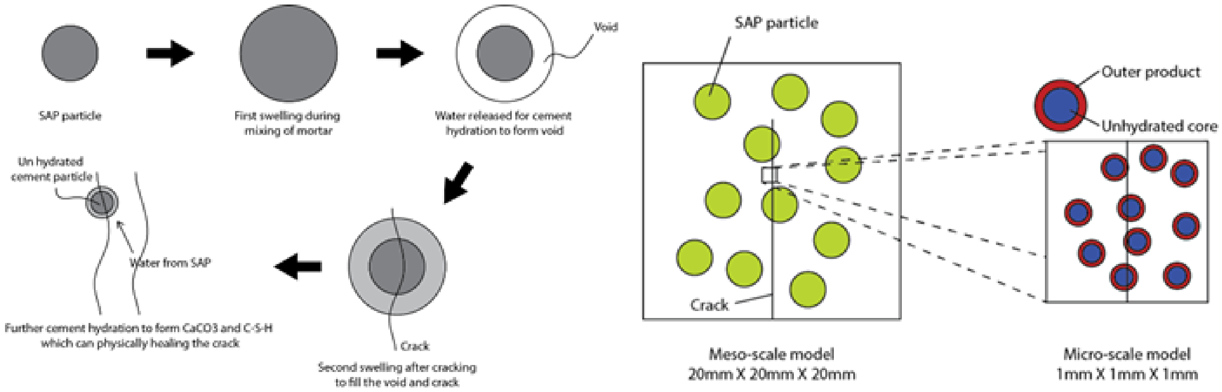

Figure 29 - A schematic of mechanism and mode of self-healing system proposed by UGent

This project has received funding from the European Union's Seventh Framework Programme for research, technological development and demonstration under grant agreement no 309451.

3.5 Large scale/field testing

3.5.1 Beams (main responsibles: DTI, TUM, TTI)

3 concrete beams of dimensions 250x40x20 cm were cast for large scale lab testing:

- Reference beam
- Beam with MUC (5 kg/m^3)
- Beam with coated SAP (5.2 kg/m^3)

The beams were cast in plywood formwork containing four 10 mm steel rebars. Along one rebar, a MuRE sensor was installed for potential/corrosion measurements. 28 days after casting, five cracks with widths between 0.1 and 0.6 mm were formed in each beam using a 3-point bending setup (Figure 30). Notches were cut and filled with fast hardening repair mortar to hold the desired crack width after unloading.

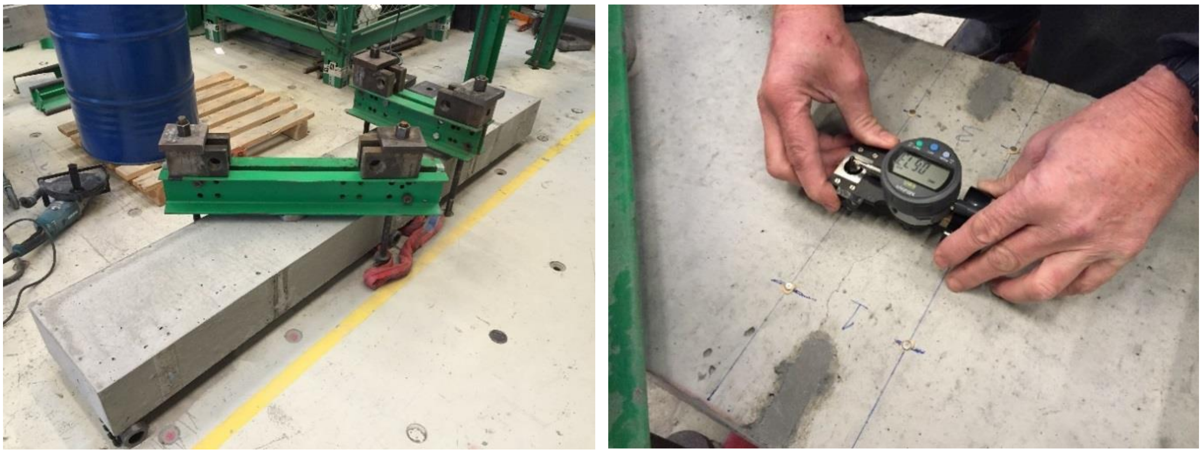

Figure 30 – Crack creation via 3-point bending tests

The beams, installed in the lab, were then subjected to cyclic water exposure for six weeks with one hour water exposure every six hours (Figure 31). After the cyclic water exposure period, cores were drilled around cracks and impregnated with fluorescent epoxy. Plane and thin sections were prepared for macroscopic and microscopic examination. The macroscopic and microscopic examination indicated that the cracks were empty with only a thin dense calcite crust formed along the crack faces. For the SAP concrete, the smaller cracks (0.1 and 0.2 mm) seemed to be at least partially closed at the surface.The beams were finally exposed to a 3 wt% NaCl solution.
Embedded MuRE's were used in combination with TTI's wireless electrometers and monitoring system to monitor reinforcement corrosion processes within the beams. This method, which also works on traditional concrete, has been demonstrated as a valuable and reliable tool to assess the health of the concrete in real-time. Reinforcement corrosion has been detected after chloride exposure within some of the cracks across all beams. For the REF and MUC beams, the MuRE sensor registered a 100 mV jump in potential shortly after the chloride exposure was initiated.

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Figure 31 – Set-up for exposure of the beams to water/NaCl solution.

For large-scale concrete beams, produced by DTI, NDT techniques as ultrasonic transmission as well as impact generated transmission measurements were conducted to evaluate the healing efficiency. Therefore, according to the NDT testing procedure, measurements had to be repeated three times (initial, cracked, healed). Known from previous studies, e.g. small-scale tests, the ultrasonic transmission method is useful for crack depth determination by picking the onset of the longitudinal wave. To obtain an accurate scientific statement about the fracture process due to the crack initiation, a reliable reproducibility of the measurement signals is required. By using contact ultrasonic transducers which have to be mounted at the specimen surface, frequent detachments will cause damages. By means of a pretreatment of the concrete surface (e.g. epoxy layer) material degradations can be reduced. According to the crack inducement, determined crack depths by ultrasound measurements corresponded to the different crack widths. After a certain curing period by means of water exposure, no significant $CaCO_3$ participation at the crack face could be verified by visual inspection and exemplary drilling cores. By analyzing the obtained NDT data, no clear distinction regarding crack closure could be recognized.

Furthermore, also at UGent, two beams, similar to the design adopted by DTI, were cast. The self-healing beam included 3250 glass capillaries (diameter 5 mm, wall thickness 0.8 mm, length 50 mm) filled with Flex SLV, accelerator and BPO. Six cracks were created via three-point bending tests. After exposure to 3wt% NaCl solution (24 hours, once a week, for 5 weeks), evaluation of the healing will be done by measuring the delayed ingress of chlorides (after exposure to 3wt% NaCl solution), visual and inspection. At the moment of reporting, tests are still running and will be followed up beyond HEALCON.

For the field testing, 4 concrete beams of dimensions 250x40x20 cm were cast:

- Reference beam
- Beam with MUC^{++} (4 kg/m^3) and urea (2 kg/m^3)
- Beam with impregnated Liapor (167 kg/m^3)
- Beam with SAP-G (3 kg/m^3)

Similar design and cracking procedure as for the large scale lab test was used, but the MuRE sensor was replaced by three ERE20 reference electrodes for potential/corrosion monitoring.

To demonstrate the applicability of the developed self-healing technologies in a practical context, the four manufactured demonstrator beams have been installed at a field exposure site in Taastrup, Denmark on December 13, 2016. The exposure site is situated in a typical Danish road environment, i.e. an environment, which is regularly exposed to de-icing salts during winter seasons.

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Figure 32 – Exposure site for the field tests in Taastrup (Denmark).

3.5.2 Slabs (main responsibles: Acciona, TUM, TTI)

Slabs with dimensions of 1200x800x200 mm were cast with each mix design, and the concrete was reinforced with 12 mm ribbed bars placed in the formwork. The healing agents that have been used in slab elements were superabsorbent polymers (SAP G), bacteria impregnated in a lightweight aggregate, liapor, and microencapsulated spores. SAP G and bacteria impregnated liapor were added with aggregates while ME spores suspension plus calcium nitrate solution (Ca source) and yeast (nutrient) were added with mixing water.

The concrete mix design has been developed using CEM I 52.5 N SR, quartzite sand, and granitic aggregates. Slabs with a volume of 192L have been manufactured in several batches sufficiently close in time in order to avoid cold joints and using a poker vibrator to homogenize the concrete.

Fresh and hardened properties of the different types of concrete were determined. The slump of different specimens was similar by means of modifying the amount of superplasticizer.

Strategically placed holes can be used to insert sleeves in order to generate cracks in concrete. These holes were formed using metallic pipes covered with demoulding agent located in selected places sited along the width of the slab before casting concrete. Before setting time, to make their extraction easier, these pipes have been removed from the concrete. Cracks were made 28 days after casting. Expansion sleeves were located in holes and wedges were sequentially hammered into the sleeves with the purpose to generate cracks and the required crack widths were obtained and retained by expansive screws.

Storage conditions were indoor with a room temperature of 20 °C, and slabs were demoulded after one week and were covered with a plastic in order to maintain the humidity of concrete until 28 days. In order to speed up the healing process, the surface of slabs was covered with water 1 hour per day during 6 weeks. To confine the water on the surface of the slabs, stainless steel basins with graduated test tube were attached on their surface, and the side of the cracks located in the sides of the slabs were sealed by means of butyl tape.

A scale loupe was used to study the evolution of cracks after the healing process. In this case, cracks go through the slab and in the upper side the width of the cracks of all slabs remained the same. However, in the case of Liapor and ME spores slabs partial closure of cracks has been seen at the bottom side of the slabs.

Two test methods were used to evaluate self-healing capability. The first test evaluates the amount of water that flows out from the bottom of slabs in a given time, tested before and after healing process. The second test evaluates the amount of water that flows out from the bottom of slabs in a given time when a pressure is applied (1.5 bar). In this case, the test was only performed after the healing process. The setup used for both methods was the same.

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The results obtained with these methods indicate that the amount of water that flows out from the bottom in all slabs has decreased after healing process. We have to take into account that the water flow of the reference slab also decreases with time due to the autogenous healing. Reference and SAPG slab performed the same, while the water outflow from the other two slabs was slightly lowerFor SAP G slab, the cracks were quite wide (> 200 µm) and this can be the reason why healing was limited. The amount of water that flowed out in liapor and ME spores slabs was lower due to the fact that partial closure of cracks appeared.

Non-destructive measurements were also performed on the slabs. The testing procedure was identical to the beam study. Due to a different crack initiation multiple cracks were formed along the proposed main cracks. These undesired micro-cracks led to inconsistent results. First approaches in terms of Rayleigh-wave studies showed appropriate outcomes. In order to deduce further steps and to proof the suitability of this analyzing tool, more examinations are essential.

The slabs, too had been instrumented with a monitoring system by TTI, which monitored corrosion potential, electrical impedance, as well as environmental parameters (temperature/humidity) in specific locations. Due to the design of the slabs and their reinforcement, locating corrosion accurately was not possible, but the system was still capable of detecting corrosion or its absence.

3.6 Life cycle cost assessment and life cycle analysis

Within HEALCON, COWI and DTI have studied the economic and environmental impacts of the technologies for self-healing studied in the HEALCON project.

The study was carried out as a case-study considering the construction of a water-tight tunnel. Two options were considered in the case-study, i.e. ensuring the water tightness using 1) traditional methods, i.e. the application of an external membrane (reference scenario) or 2) using self-healing agents (alternative scenario). Figure 33 illustrates the cross section of the tunnel (left: reference scenario, right: alternative scenario).

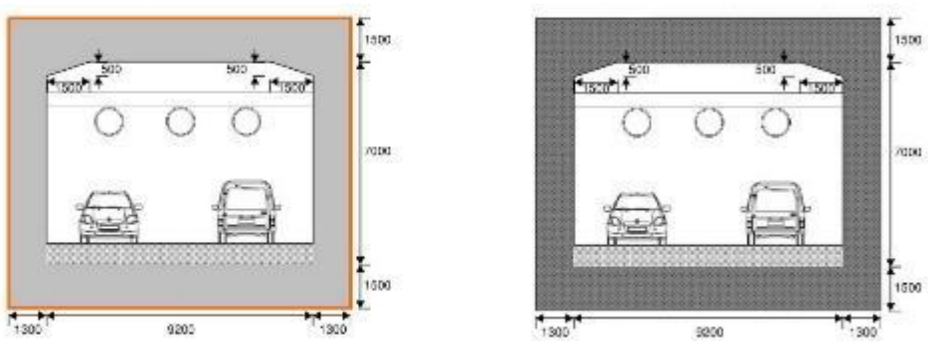

Figure 33 – Case study: Water tight tunnel with membrane (left) or self-healing concrete (right)

Five different self-healing agents were considered for the alternative scenario: MUC, Liapor with bacteria, bacteria in microcapsules, SAP-G, and coated SAP. The study covered evaluations regarding life cycle assessment (LCA), i.e. environmental, as well as evaluations on the life cycle cost (LCC), i.e. economical. As the information about the long-term efficiency of the healing-agents is limited, the study focused on the initial cost (environmental and economic) of the reference scenario and the five alternative scenarios with self-healing agents. Hence the operation-phase of the tunnel, including repairs, such as crack injections, was not considered.

In order to link this study to the research carried out in the consortium, information from consortium-partners regarding e.g. the amount of self-healing agents per m³ concrete, and the production cost of the self-healing agents per kg was used for the LCC analyses. Furthermore, information from the consortium-partners regarding e.g. energy-consumption and composition of constituents for the various healing-agents have been used together with tabulated values from internationally recognized software on the emission from these constituents for the LCA analyses. The LCA analyses revealed that

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there is no significant difference between the reference scenario and the alternative solutions with regard to global warming potential (GWP) and Primary energy non-renewable, total (PENRT). The LCC analyses showed that two of the alternative scenarios (bacteria with encapsulation and SAP-G) were approx. 2 times as expensive as the reference scenario, one scenario (Liapor with bacteria) was approx. 40% more expensive than the reference scenario, and two scenarios (MUC and coated SAP) were comparable in cost with the reference scenario. It is, however, emphasized, that these analyses (both LCC and LCA) solely covers the construction phase, and that costs (environmental as well as economic) during the operation-phase are not covered by the analyses presented in this study. Furthermore, the LCC analyses done here were based on the production-cost of the healing-agents at laboratory-scale mid-2016, and additional efforts spent during the last months of the HEALCON project to upscale the production of the healing agents and reduce the costs were not yet taken into account as this work was carried out after the submission of the deliverable containing the LCC & LCA studies. Moreover, large-scale production of the healing-agents will potentially reduce their cost.

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5. The potential impact (including the socio-economic impact and the wider societal implications of the project so far) and the main dissemination activities and exploitation of results

Within HEALCON, further steps have been taken to bring the concept of self-healing closer to the Market. The achievements go far beyond the state-of-the-art at the start of the HEALCON project. Suitable healing agents, including their encapsulation, have been (further) developed and tested with success at lab-scale. However, application of self-healing concrete in large scale structures, showed that some further research with regard to optimization of the mix design (e.g. dosage of healing agent) would be advisable to benefit from maximum self-healing capability. The self-healing concrete, with autonomous crack sealing/healing as a built-in function, is designed to extend the lifetime and to enhance the safety and durability of the material. However, within the timeframe of HEALCON, studies related to the durability of self-healing concrete were initiated but, no evidence could yet be given of the performance of self-healing concrete in the long term.
Although not measurable at the moment, HEALCON contributed to the improvement of the performance of concrete structures for the near future. These substantially improved concrete structures will lead to an enhanced safety in infrastructures, i.e. less failures during service and reduction of the maintenance activities. As these maintenance activities represent a waste of time and money (e.g. traffic jams, hold-ups, work zone operations which affect commercial activities), HEALCON contributed to reduce these societal impacts. Moreover, according to European statistics on worker accidents, the construction sector is one of the most dangerous sectors, representing 20% of the casualties (European Agency for Safety and Health at Work). It is evident that a reduction of maintenance activities will benefit worker protection and overall safety. The purpose of Europe 2020 Strategy where innovation is at the core of it, is to achieve a sustainable and cost-effective future for European citizens, and reducing cumbersome maintenance and its negative side-effects is a solid step towards that goal.

5.1 Regarding the economic and environmental impact

In the original DoW of HEALCON it was mentioned that, for self-healing concrete structures, the initial costs will be higher than with traditional concrete. A very rough estimate showed that the concrete material costs may be doubled (e.g 200 € instead of 100 € per m³) by including 1 vol-% of self-healing additives. This meant that the total cost of a construction element with self-healing concrete (including steel reinforcement, formwork, placement) would be about 20% higher. However, the reduction of maintenance and repair costs, and the increase in service life will result in a financially positive situation. Manual injection of one crack per m³ concrete (which is realistic) in a tunnel will cost 120-140 €/m³, so this is more expensive than application of self-healing material, even without the indirect costs for closing the tunnel etc.
Within HEALCON, COWI and DTI have studied the economic and environmental impacts of the technologies for self-healing studied in the HEALCON project.
The study was carried out as a case-study considering the construction of a water-tight tunnel. Two options were considered in the case-study, i.e. ensuring the water tightness using 1) traditional methods, i.e. the application of an external membrane (reference scenario) or 2) using self-healing agents (alternative scenario). Figure 33illustrates the cross section of the tunnel (left: reference scenario, right: alternative scenario).
Five different self-healing agents were considered for the alternative scenario: MUC, Liapor with bacteria, bacteria in microcapsules, SAP-G, and coated SAP. The study covered evaluations regarding life cycle assessment (LCA), i.e. environmental, as well as evaluations on the life cycle cost (LCC), i.e. economical. As the information about the long-term efficiency of the healing-agents is limited, the study focused on the initial cost (environmental and economic) of the reference scenario and the five

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alternative scenarios with self-healing agents. Hence the operation-phase of the tunnel, including repairs, such as crack injections, was not considered.

In order to link this study to the research carried out in the consortium, information from consortium-partners regarding e.g. the amount of self-healing agents per m³ concrete, and the production cost of the self-healing agents per kg was used for the LCC analyses. Furthermore, information from the consortium-partners regarding e.g. energy-consumption and composition of constituents for the various healing-agents have been used together with tabulated values from internationally recognized software on the emission from these constituents for the LCA analyses. The LCA analyses revealed that there is no significant difference between the reference scenario and the alternative solutions with regard to global warming potential (GWP) and Primary energy non-renewable, total (PENRT). The LCC analyses showed that two of the alternative scenarios (bacteria with encapsulation and SAP-G) were approx. 2 times as expensive as the reference scenario, one scenario (Liapor with bacteria) was approx. 40% more expensive than the reference scenario, and two scenarios (MUC and coated SAP) were comparable in cost with the reference scenario. It is, however, emphasized, that these analyses (both LCC and LCA) solely covers the construction phase, and that costs (environmental as well as economic) during the operation-phase are not covered by the analyses presented in this study. As self-healing concrete is designed to seal/heal cracks inherently, it is expected that maintenance costs will be limited in comparison to traditional concrete. However, based on engineering judgment of the current status of the healing agents, it cannot be excluded that there will be some additional costs during this phase for self-healing concrete. Nevertheless, it is expected that by implementing concrete structures with self-healing properties, costs for maintenance activities and the related indirect costs (e.g. traffic congestion and other delay and cost from repairing early age surface cracks) will be avoided to a large extent. Furthermore, it has to mentioned that the LCC analyses done here were based on the production-cost of the healing-agents at laboratory-scale mid-2016, and additional efforts spent during the last months of the HEALCON project to upscale the production of the healing agents and reduce the costs were not yet taken into account. Moreover, large-scale production of the healing-agents will potentially reduce their cost.

5.2 Direct economic benefits to the proposing Partners

The main outcomes of the project are:

- encapsulated bacterial self-healing agents
- tailored hydrogel-based self-healing agents (especially for healing of early-age cracks)
- encapsulated polymers (for self-healing of dynamic cracks)
- new encapsulation techniques
- concrete with self-healing properties due to encapsulated microbial and hydrogel-based healing agents
- novel (non-destructive) tools for testing of self-healing and for monitoring (such as wireless sensors)
- models to simulate crack formation and self-healing

HEALCON consists of large companies as ACCIONA and COWI working on building and civil infrastructure projects and having experience and market knowledge. Besides HEALCON also has SME partners e.g. Avecom, CEINNMAT and Devan Micropolis working in the area of advanced encapsulated products, TTI in diagnoses tools, and Fescon in innovative concrete solutions. For SME Partners, the commercial exploitation of advanced concrete additives providing self-healing properties will provide a better position in the concrete additives market. This market is traditionally dominated by large companies and now by companies from Oriental countries. For SME Partners it would be expected that the turnover increases after Project completion.

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- By participating in the HEALCON project, Avecom was able to investigate/develop a new product for a different market that is totally new for Avecom – concrete/construction market. To develop this product, a local waste product was used and its valorisation was achieved.
- TTI developed a monitoring system that combines embedded electrodes with wireless sensors and a very powerful data access and analysis portal and is capable to monitor the health of concrete structures and alert operators about location and presence of corrosion processes. Similarly sensitive potential measuring solutions were previously not available in wireless or long term battery powered form, which gives TTI a potential competitive edge in this market.
- For Fescon it was most beneficial to be well informed about current developments in Europe-world-wide. As no commercial healing-additive are available yet, there is, at the moment, no direct commercial benefit for Fescon.
- By participating in the HEALCON project Acciona could benefit in the use of this kind of self-healing materials in order to increase the durability of concrete and reducing the frequency, severity, and cost of repairs as well as number of maintenance tasks.
- By participating in the HEALCON project, Devan-Micropolis had the opportunity to explore alternative means of encapsulation techniques with different healing agents proposed by research partners, and thus further select the most adequate technique suitable for upscaling. Devan was able to design a commercial prototype that may be developed further according with Market needs, price feasibility according with product's offer and its unique selling value for the self-healing goal in cementitious substrates.

5.3 Concerning the environment

Maintenance and rehabilitation works have a notable impact on the environment caused by the use of resources, energy and pollutant emissions, and by the traffic jams that they usually produce, which involve more fuel consumption and more emissions. The White Paper on Transport "European Transport Policy for 2010" takes into account major policy initiatives that may impact on transport, in particular energy and environment, and establish as a priority the need for integration of transport in sustainable development. Self-healing concrete technology is an important link between the conflicting sectors. Self-healing structures will have a longer (maintenance free) service life, resulting in lower CO_2-emissions.

5.4 European approach

HEALCON has shown that connection of highly specialized partners from across Europe has led to a successful progress in the research on self-healing concrete. A coordinated European effort to share the results of single countries, allowed us to advance faster in the technological development. Cooperation among universities, industry, standardization bodies and end-user board members has been necessary for the proper implementation in the sector of civil and materials engineering.

5.5 Main dissemination activities

HEALCON project has been disseminated up to now in the following dissemination activities:

- 34 papers in proceedings of conferences/workshops
- 6 papers in journals of high impact index
- 1 book chapter
- 11 articles published in popular press
- 1 film in YouTube
- flyer
- 7 interviews in several media
- 42 oral presentations to a scientific event

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- 1 workshop
- 5 exhibition posters
- 1 stand in a fair
- 3 press releases
- 2 TV clips
- Video
- Newsletter
- Internal newsletter
- 17 mentions of HEALCON project in websites
- 1 final conference of the HEALCON project
- 3 end-user board meetings

It is worth mentioning the participation in two international self-healing conferences (ICSHM 2013 celebrated in Gent, Belgium and ICSHM 2015 celebrated in Durham, USA) where the HEALCON project has played an important role. Moreover, finally, a HEALCON conference celebrated in Delft presented the results of the whole project.

5.6 Exploitation

HEALCON project aims to explore fundamental science with applied sciences such as civil engineering for the building industry that expects to bring innovation for a semi-traditional field. Therefore it is possible to expect an increase of Market acceptance in the near future when self-healing concept and autonomously repair in cementitious materials would be a common trend, as a result, expecting prices to go down. Thus, the final version of the Exploitation Plan proposes to define a priority sequence of the 6 most promising Key Exploitable Results (KER) selected throughout HEALCON and possibly giving some exploitable channels that may be useful to each KER's leaders and related partners to pursue and therefore benefit in the scope of Foreground to HEALCON. It is herein reported a commercialization strategy key guidelines, starting from the most pre-eminent KER and further complemented with Life-Cycle Cost (LCC) analysis[1]

KER number 1 – Selection of the mixed bacterial culture (Process &
KER number 2: Confidential
KER number 3: Confidential
KER number 4: Confidential
KER number 5: Confidential
KER number 6 – Standard Test Methods

5.7 HEALCON Value Chain Proposal

The projected value chain for HEALCON can be parted in three different range of products, processes and services
1 – Self-healing Agents
2 – Self-healing Concrete
3 – Self-healing Services and Standardization

All of these exploitable results are part of the whole value chain proposed by HEALCON project whereas research and development have come up with specific healing agents that intend to promote healing efficiency, increase life-time of a building structure and save a considerable maintenance. These healing agents may be incorporated individually or in combination in order to enhance healing

[1] HEalcon Deliverable D11.7 - Final version of the Exploitation Plan

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activity without affecting key parameters for self-healing concrete formulations that will be applied in specific Market sectors where technology and Market risks are minimal.

Self-healing concrete products are the result of concrete based materials with specific self-healing properties given by the incorporation of healing agents. Self-healing concrete products shall ideally preserve the main properties of standard concrete formulations in order to broad Market application in the building sector and use their unique selling point which is self-healing or autonomously healing as an important added value to increase Market penetration of such innovative solution. Finally, standard test methods provided by KER number 6 which may drive the self-healing concept to the Market with its proposed methodology and quantification of healing efficiency that would have a push-pull marketing tool effect to this currently little-known concept in the building Market sector.

Tackling Self-healing Concrete (SHC) Market Hurdles for Commercialization

1. Quality of the Product

Possible market misperception: Self-healing agents are meant for durability but SHC strength and workability decreases (and hence might impair durability as well)

Needed approach:

- Implement different SHC classes with proper mix design or the incorporation of additives to increase strength and workability;
- Standardization as a way to assess and classify the quality of fresh mix throughout 28 days curing. Introduce a certification stamp on different classes of SHC.

2. Makeability

Possible market misperception: SHC is more difficult to be casted as conventional concrete (and hence introduces risks of execution)

Needed approach:

- Define clear execution protocols;
- Upscaled SHC mixes which are robust and can be delivered in reasonable time and quantities.

3. Efficiency

Possible market misperception: Laboratory scale investigation of healing-agents performances for specific crack width ranges are not representative for reality

Needed approach:

- Broad investigation on real-scale scenario for crack width healing efficiency for specific range of crack width values;
- Improve know-how concept on SHC performance with crack control additives at real-scale scenario.

4. Value for Money

Possible market misperception: Sales proposition is not evident: "client to start using SHC, we need to tell that our concrete so far was not good enough"

Needed approach:

- Further efforts on durability assessment in real conditions;
- Life-cycle cost analysis for specific use cases, such as for replacement of waterproof membrane, injection and maintenance works.

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5.8 Project website

On the website of HEALCON (www.healcon.eu) more information about the HEALCON project (research, consortium, publications, news and events, contact) can be found.

5.9 Consortium

Coordinator:	
Universiteit Gent	Belgium
Partners:	
Avecom N.V.	Belgium
Technische Universiteit Delft	The Netherlands
Acciona Infraestructure S.A.	Spain
Technische Universitaet Muenchen	Germany
Technologie-Transfer-Initiative GmbH	Germany
Teknologian Tutkimuskesk VTT	Finland
Cowi A/S	Denmark
Teknologisk Institut DTI	Denmark
Comercializad ora Espanola De Innovaciones Y Materiales	Spain
Fescon OY	Finland
Devan Micropolis	Portugal

5.10 Contact details:

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6. Use and dissemination of foreground

6.1 Section A

Intellectual property rights, ownership and access rights: The beneficiary who carries out the work generating that foreground will be the owner of that foreground. In case that several beneficiaries have jointly carried out work generating foreground, an agreement regarding the terms about ownership have to be made.
On the other hand, access rights to background and foreground will be granted to the other partners. Finally, consortium partners will not publish confidential information without permission of the others partners of the project until five years after the end of the project.
More IPR management basic aspects are defined in the Consortium Agreement.

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Dissemination indicators
In order to monitor the dissemination effectiveness several indicators have been used:

- **Website:** The project website (www.healcon.eu) acts as a dissemination platform with the aim to establish an efficient and effective dissemination and communication tool for the HEALCON consortium for the duration of the project. The website is one of the main dissemination tools of the project, which will ensure the successful use of project results and show/transfer non-confidential information to the widest possible audience (including the industrial and academic community). In this sense publications generated in the HEALCON project can be downloaded in the public part of the website. The website has a clear structure with two types of webpage navigation depending on the type of user i.e. visitor (public) or consortium member (member area). The aim of the website is on one hand to inform general public about the HEALCON project and on the other hand to constitute a tool to communicate and to exchange information on the project between partners. The Healcon webpage was visited by 1112 people in the whole project. Project public deliverables provide an important dissemination platform. They are available on the project website.

- **Publications:** To effectively disseminate the project, a three-fold project brochure has been created. The brochure describes the Consortium partners and provides their main contact details as well as the problem statement, the general and technical objectives of the project and the methodology. The brochure is user-friendly, compact and easy to understand. The brochure is presented as part of the Dissemination Plan and it will be distributed at several events.

– Forty-two papers have been published in Proceedings of a Conference/Workshop
– Five papers have been published in high impact index journals, and a chapter has been published in the book "Advances in Polymer Science" by Springer.
– A video with HEALCON project information has been uploaded to YouTube.
– Besides, HEALCON is presented in several press releases, mentions in websites and radio interviews.

- **Events:** HEALCON project has been disseminated in 29 events including conference, fairs, and workshop. A total of forty-two papers have been published in proceedings of conferences or workshops, and Forty-two oral presentations have been presented in the events.

– Fairs events are used to start new networking collaborations with other related R&D projects, taking into account the possibility of organizing thematic stands or speeches with other related funded projects. Scientific events were selected on demand; depending on the subject and the progress of the different technical tasks of the project. These events were of national or international nature. This project has been exhibited in 'Industrial Technologies 2014' in a

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stand dedicated to several European projects. Two posters have been made in order to disseminate HEALCON project in several events.

- **Networking:** Some of the partners involved in the HEALCON consortium are involved in relevant currently ongoing EU and national projects related to the topic such as: TRAINER, SHE, RESCOAT, IOP programme, SHeMat, CAPDESIGN. The networking with these projects will meant an added value to the project development and the possibility to combine the efforts of the different projects.. In this way a workshop within ICSHM2013 (Fourth international conference on self-healing materials) has been organized in order to promote the exchange of knowledge.
  A Symposium on Self-Healing Materials was organized as part of the E-MRS-EUROMAT conference. Different projects related to self-healing materials like HEALCON, SHINE, SAMBA and SHeMat participated in the symposium.
  An overview presentation of the HEALCON project was presented in a Workshop celebrated by SHINE partners in Delft (The Netherlands). This event was used for networking between partners involved in projects related to self-healing materials. A joint A1 publication of HEALCON and CAPDESIGN about 'Simulation aided design of tubular carriers for self-healing concrete' is currently under revision.
- **End-users Board:** Three end user board meetings have been celebrated since beginning of the project. Moreover, end-users were invited to the final HEALCON conference (Delft, 28-29 November 2016). The first end user board meeting was realized on 17 May 2013 in Brussels, the second one has been realized on 7 October 2014 in Munich and the last one was celebrated in Taastrup, Denmark on 10 June 2016.This kind of event is dedicated to implement self-healing concrete in practice taking into account end-user requirements. In this sense, several end-users representing public and private bodies from different European countries attended these workshops. As a result of the first meeting a list of requirements has been prepared in order to ensure the connection between the technologies to be developed and market needs and requirements to be implemented on real structures. During the second meeting, an update on the project status and answers to the questions raised by the end-users were given. During the third meeting, the large scale tests and demonstrators were discussed. The details of the meetings are reported in the EUB minutes.
- **Demonstration site:** Visit of different kinds of stakeholders were organised in the pilot site during the demonstration phase for explaining the concept of the project, the technologies behind, the expected results, the replication potential, etc. This visit was celebrated within a Workshop celebrated in Madrid. The demonstration site has been visited by at least 15 stakeholders. Percentage of attendant people's organization type has shown in Figure 34.

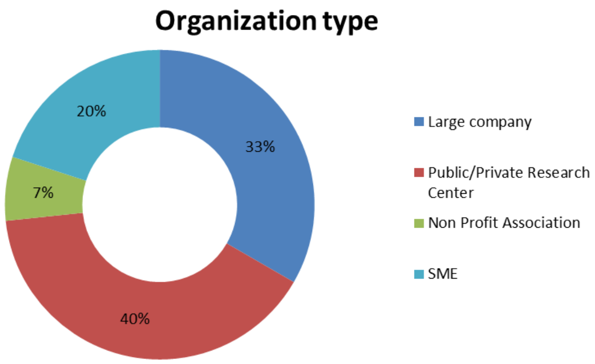

Figure 34 – Workshop Madrid

- **Standardisation:** According to HEALCON proposal, several activities should have been carried out to move towards standardization mainly related to laboratory tests to prove the efficiency

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of self-healing technologies. Therefore, in the beginning of the project it was decided as most appropriate option to organize a CEN Workshop Agreement (CWA), as a first valid step towards future standardization.

- In April 2015, the laboratory tests to evaluate self-healing efficiency were presented to the Dutch and the Belgian Standardization Committees for Concrete. The presentations were well received and both committees agreed to support a New Work Item Proposal (NWIP) to the European standardization technical committee for concrete (CEN/TC 104).
- In May 2015, the meeting of CEN/TC 104 was held. A presentation of the laboratory tests was also given during this meeting. The delegates agreed that the topic is premature to be standardized. It was therefore suggested that the testing methods should be further investigated and evaluated by a RILEM committee, where a recommendation document could be published and eventually evolve into a standard document. At that time, TUDelft and UGent participated already in RILEM/TC 253 MCI (Micro-organisms-Cementitious Materials Interactions). Consequently, it was decided that the testing procedures and the obtained results would be evaluated through round robin tests (RRT).
- In anticipation of the RRT and the RILEM recommendation, steps towards the CWA were taken. During the self-assessment an approval of CEN/TC 104 was needed. Yet, a disapproval was given (March 2016).
- After the decision of March 2016, the options for developing a national document were examined. The Dutch mirror committee and the SBRCURnet committee suggested to make a pre-normative document in English which will include the testing procedures with the adaptations/changes that were made after the completion of the RRT.
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6.2 Section B

An exploitation plan has been produced and updated along the HEALCON project, with the aim of preparing the market take-up of the developed technology, always considering how to minimize exploitation risk and detail the actions for exploiting the main results. The exploitation plan was connected to the dissemination plan and led by the same DET team. Also an Exploitation Strategy Seminar was organized in March 2015.
The exploitable foreground of HEALCON has its purpose through its key results and any IPR that can be linked with it and is typically owned by the partner leader that carried out the research activities from which it resulted. Partners who are willing to exploit foreground generated by HEALCON are described in Table 5 for each exploitable result and IPR intention.

Table 5 - General overview of HEALCON's KERs and main risks, IPR intended and time to market

KER N.	Exploitable Result	Lead Partner	IPR Owners of the ER	Partners planning to exploit	IPR Intended	Time to Market
1	Selection of the mixed bacterial culture (Process & Product)	Avecom	UGent and Avecom	Acciona and Fescon	Licensing or Direct Industrial Use	4 years after the end of project
2	Concrete Precast formulation	Acciona	Acciona	Avecom; CEINNMAT and Fescon	Licensing or Direct Industrial Use	4 years after the end of project
3	Concrete Ready Mix formulation	Acciona	Acciona	Avecom; CEINNMAT and Fescon	Licensing or Direct Industrial Use	4 years after the end of project

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4	pH sensitive Hydrogels	CEINNMAT	CEINNMAT	CEINNMAT; Acciona	Direct industrial or Licensing	3 years after the end of project.
5	Encapsulation of hydrogels	VTT	VTT	VTT, Acciona and Fescon	Patent or Direct Industrial Use	2 - 4 years after the end of project.
6	Standardization methods for self-healing efficiency on mortars.	TUDelft	UGent and TUDelft	UGent and TUDelft	Direct Industrial Use	Ready to Offer Services but not Advertised prior Standard

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