Efficacy of Bacteria Encapsulated Self-healing Concrete Exposed to Salt Water and Freeze-Thaw Cycling

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Abstract

The formation of micro-cracks in concrete is unavoidable. Researchers have been investigating techniques which can repair cracks autogenously. One technique uses a specific type of bacteria within the concrete to produce minerals and effectively seal cracks. Concrete is a highly versatile material, which leads to its use under various environmental conditions that can affect the viability of bacteria, and therefore inhibit the self-healing process. Two environments to which concrete is commonly exposed that may affect the viability of bacteria are a marine environment and freeze-thaw cycles. This paper reports the efficacy of self-healing concrete using bacteria encapsulation under these two exposure conditions.

Concrete was mixed with a self-healing agent. After 28 days of curing, samples were cut into 25 mm thick disks that were cracked. Multiple disks were exposed to different environmental conditions. A marine environment was simulated by placing disks in a 3.5% saltwater solution. Freeze-thaw cycles were simulated by freezing disks submerged in water for 6 hours and then thawing them for 18 hours. The widths of the cracks were monitored under a microscope for 18 weeks. The results suggest that the bacteria remain effective in salt-water environments whereas cold temperatures limit their healing properties.

Key Words: Self-healing concrete, bacteria encapsulation, marine environment, freeze-thaw cycles

Introduction

Approximately 70% of the world's population lives in concrete structures. Concrete is the most utilized construction material worldwide, and twice as much concrete is produced as steel, aluminum, plastic and wood combined. Concrete can be recycled at the end of its service life, by being crushed and then used as aggregate in new concrete or fill. Annually, 140 million tons of concrete are recycled in the United States (Fast Fact). Concrete is used widely because it has relatively low cost, high accessibility, and most of all it is versatile.

The basic constituents of concrete are cement, water, and aggregates—a combination of crushed rock and sand. Generally the production of concrete is localized; cement is produced within the region, aggregates are available within tens of miles and water is locally available (Fast Fact). The water to cement ratio and type of aggregate can be altered to produce concrete with different strengths and properties. Admixtures can be used to further modify the properties of concrete. This adaptability of components enables concrete to be designed to meet the needs of a particular project. One such adaptation is self-healing concrete.

Researchers have been evaluating several concepts for a self-healing concrete (Li and Herbert 2012). A self-healing concrete would help produce a more sustainable infrastructure by reducing the cost of maintenance and repair. Li and Herbert analyzed some of the methods for self-healing concrete in the article *Robust Self-healing Concrete for Sustainable Infrastructure* (Li and Herbert 2012). They evaluated five approaches of self-healing concrete against six criteria: "long shelf life, pervasiveness, quality, reliability, versatility, and repeatability." The five broad approaches included: "chemical encapsulation, bacterial encapsulation, mineral admixtures, chemicals in glass tubing, and intrinsic healing with self-controlled tight crack widths." Each approach utilizes the versatility of concrete by incorporating additives that result in crack repair. However, each approach contains limitations in at least one criteria or requires additional research to be done.

This paper focuses on the bacterial encapsulation approach of producing self-heling concrete, and the criteria of versatility. For each of the previously mentioned methods, there is insufficient data for a proper assessment of reliability and versatility. Reliability relates to the consistency of the results. Versatility relates to the different environments to which the concrete will be exposed. The efficacy of the bacteria encapsulation self-healing method was evaluated only when it was exposed to fresh water at room temperature. However, concrete can often be exposed to salt water in coastal environments. Also in cold environments, concrete will be exposed to freeze-thaw cycles. Both of these environments may affect the bacteria's ability to adequately repair cracks. Two separate tests were conducted to evaluate the selected environments.

Methods and Materials

a) Bacteria Selection

Bacterial spores have the ability to withstand methods commonly used to kill viable bacteria. The endospores protect the genetic makeup of the bacteria, which can become active when conditions are favorable for growth (Piggot and Coote 1976). Two bacteria of *Bacillus Pseudofirmus* DSM 8715 and *Bacillus Cohnii* DSM 6307 were selected based on a literature review. The selected spore-forming bacteria are naturally occurring, and can resist the high alkalinity of the concrete matrix (Jonkers 2011). Research has demonstrated the healing capabilities of the selected bacteria. The objective of the research was to evaluate the viability of the same bacteria under exposure to salt water and freeze-thaw cycles.

The bacteria were purchased initially as freeze dried samples from the German Collection of Microorganisms and Cell Cultures (DSMZ) in Braunschweig, Germany. The bacteria samples were stored at 4°C, until cultivation. The supplier has a list of recommended media for microorganisms. Each bacteria was cultured according to specification recommended by the supplier which was defined on the list of media as DSMZ 31, an Alkaline Nutrient Agar. The intended medium requires a base medium (DSMZ 1) consisting of 5.0 g peptone and 3.0 g meat extract per 1 liter of distilled water. The media was prepared from Difco™ Nutrient Broth, which matches the desired media. An addition of 10.0 mg of $MnSO_4 \times H_2O$ was implemented as recommended when bacillus strains are cultured to increase sporulation. The base medium was altered to achieve a pH of 9.7, by the incorporation of sterile 1 M Na-sesquicarbonate solution (4.2 g $NaHCO_3$, 5.3 g Na_2CO_3 anhydrous, 100 ml distilled water). The media were sterilized with an autoclave at 121°C for 30 minutes, in Erlenmeyer flasks, before inoculation of the freeze dried bacteria. To make agar plates, the same medium was used with 15 g of Difco™ Nutrient Agar (DSMZ 2007). The bacteria were cultured separately overnight, in an incubated shaker table at 30°C. After 24 hours, the media was streaked on agar plates for short term use, and other samples were frozen at -80°C for future use.

b) Encapsulation Method

Encapsulating the bacteria within aggregate has resulted in extend viability and functionality (Jonkers 2011). For the research being conducted, the bacteria were encapsulated in a lightweight aggregate (LWA) ranging from 5 to 10 mm in diameter.

The encapsulation method was adopted from Mors, and Jonkers (2006). The selected process could be scaled easily for mixes of different proportions. The process begins with heating water to 80°C, with constant stirring with a magnetic stir-bar. During the heating process, calcium lactate is dissolved at a concentration of 350 g/L (Figure 1). After the calcium lactate is completely dissolved and the water maintains a temperature of 80°C, the bacteria are added and stirred for approximately 2 minutes. The elevated temperature of the water inactivates the bacteria and drives spore production. After the bacteria are uniformly distributed, the LWA is then added and automatically stirred at the elevated temperature for 30 minutes (Figure 2). The encapsulation process occurs directly before mixing. The process is completed by draining excess water to leave the aggregate in a saturated surface dry condition.

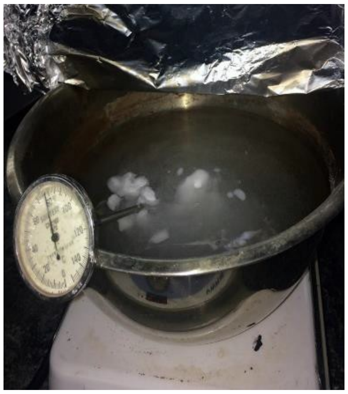

Figure 1: Calcium Lactate solution

Figure 2: LWA soaking at 80°C

c) Mix Design

A simple mix design was used to conduct the experiments. The constituents include Type II cement, water, sand, and the LWA impregnated with the healing agents. The concrete is then mixed with a mortar hand mixer and a 100 mm diameter x 200 mm high cylinder was cast. The concrete mix design proportion is presented in Table 1. The mix design was based on even parts of sand and LWA by volume with a water to cement ratio of 0.5. Samples were cured for at least 28 days.

Table 1: Concrete Mix Constituents for a Single 100 mm x 200 mm Cylinder

MATERIAL	**CONCRETE COMPOSITION**
Type II cement	706.8 g
Water	353.4 ml
Sand	1627.85 g
Lightweight aggregate*	460 g

*Healing agent was encapsulated in aggregate prior to mixing

d) Tests and Data Collection

Each concrete cylinder was cut into seven disks having a thickness of about 25 mm. A wet concrete polisher was used to clean the surfaces of each disk. Each disk was cracked using displacement controlled loading as shown in Figure 3. Disks were not removed from the plastic mold in which they were cast so that crack widths would be easier to maintain after the disks cracked.

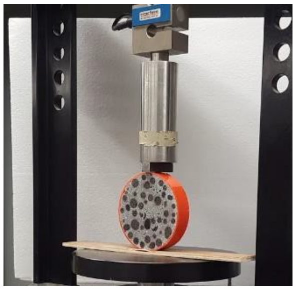

Figure 3: Cracking of disks

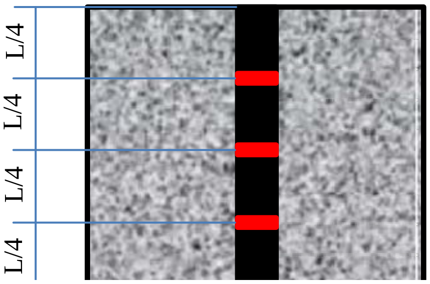

Figure 4: Crack width measurements

The healing progress was monitored under a microscope. Photographs were taken weekly using a Nikon SMZ18 microscope and a Nikon DXM12C camera over 18 weeks. The NIS Element software was used to measure crack widths and lengths. A single location on each disk was selected to be evaluated under the microscope. This was done by marking a square area to ensuring that the photographs could be lined up from one week to another. Each selected section of the disks was captured under three different magnifications, which allowed different levels of detail to be observed. The lower magnifications recorded a larger area and had the most potential to capture overall changes. The larger magnifications helped to identify subtle crystallization and captured better defined images. Additional weekly photographs were taken to capture both sides of the entire disks. This helped to determine if other sections of the disks experienced the same results as the location captured under the microscope.

To quantify the percent of healing, the crack widths were measured at 3 locations under the lowest microscope magnification. Figure 4 shows the locations of the measurements. The results reported in this paper are the average of the readings at the three locations for a given week. A percent change in crack widths was calculated by comparing each week's average to the average at week zero, and the healing index was calculated according to (Wiktor and Jonkers 2011):

$$\mathrm{H.I} = \frac{(W_0 - W_C)}{W_0} \times 100 \tag{1}$$

where *H.I.* is the healing index, W_0 is the crack width at week zero, and W_c is the current crack width.

In the research conducted by Jonkers, et.al (2010) the healing process was confirmed only when concrete was exposed to fresh water. Therefore, all the disks were initially stored fully submerged in fresh water for 4 weeks until initial healing occurred, which indicated that the bacteria was active. Subsequently, disks were exposed to salt water and freeze-thaw cycles. The marine environment was simulated by placing samples in a 3.5% saltwater solution. Freeze-thaw cycles were simulated by having disks submerged in water and placed in a freezer for 6 hours followed by 18 hours of thawing, and then repeating these cycles.

Results and Discussion

a) Salt Water

The disks used for the salt-water experiment were allowed to cure for more than 28 days before being cracked and submerged. For the first 4 weeks of the experiment, all disks were placed in fresh tap water. After initial healing was observed, four of the seven disks were moved into a salt-water environment. Three of the seven disks were kept in fresh water as control specimens. The initial crack widths varied for each disk. Each week, three measurements were taken as previously discussed in the Data Collection section. Disk 7 had the largest average initial crack width of 0.35 mm and disk 6 had the smallest average initial crack width of 0.14 mm. The overall average of the initial crack widths was 0.25 mm. Crack widths were recorded on a weekly basis, and the healing index was calculated. Figure 6 and 7 shows the calculated crack width for the salt water and control disks, respectively.

The test results show that healing can still occur while disks are exposed to salt water. Disk 5-A fully healed after ten weeks of which 6 weeks was in salt water. In the same conditions, disk 7-A experienced almost no healing. It is possible that since disk 7-A had the largest overall crack widths it did not heal. Disk 5-A had an initial crack width of 0.2 mm and Figure 5 shows 3 images of it.

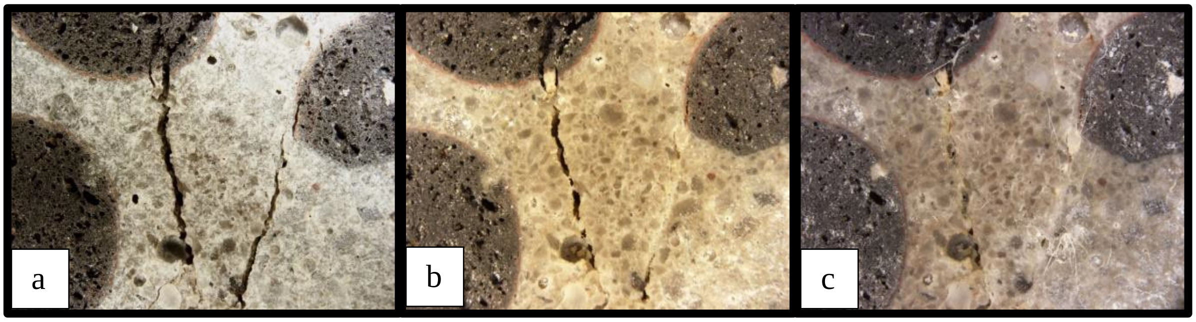

Figure 5: Microscope photos collected for disk 5-A.

a) Initial photo captured: 6/04/15 **b)** Healing on right side completed, captured: 7/09/15 **c)** Healing on left side is significant, captured: 9/24/15

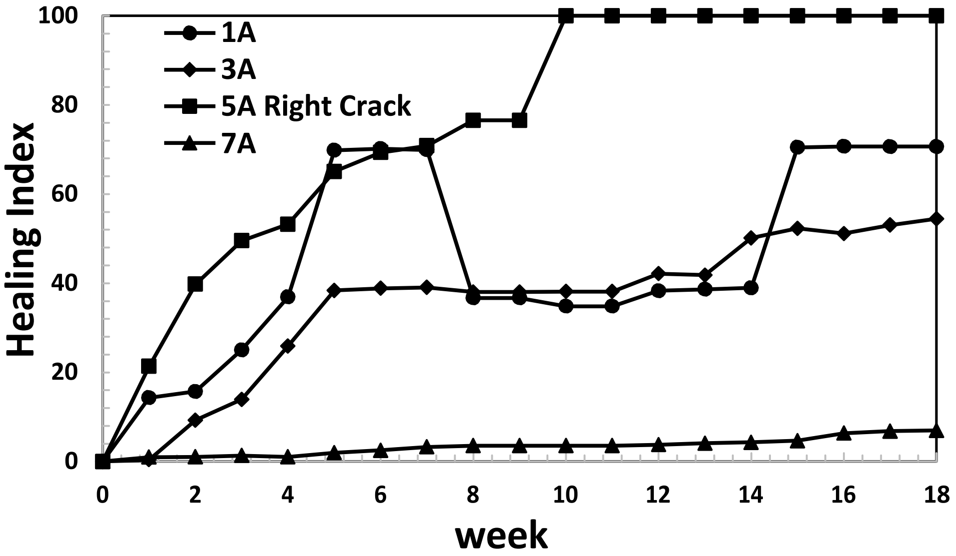

Figure 6: Healing index for disks exposed to salt water

The results in Figure 6, salt water exposure, and Figure 7, fresh water exposure, show that in both environmental conditions a disk was able to fully heal, while another disk experienced nearly no healing. In the fresh water condition, disk 2-A had an average crack width of 0.33 mm, which was similar to disk 7-A, and the lack of healing in disk 2-A may be due to the large crack width rather than the environmental condition. Disk 6-A, which had the smallest average crack width, was able to fully heal after 9 weeks, similar to disk 5-A in the salt-water environment.

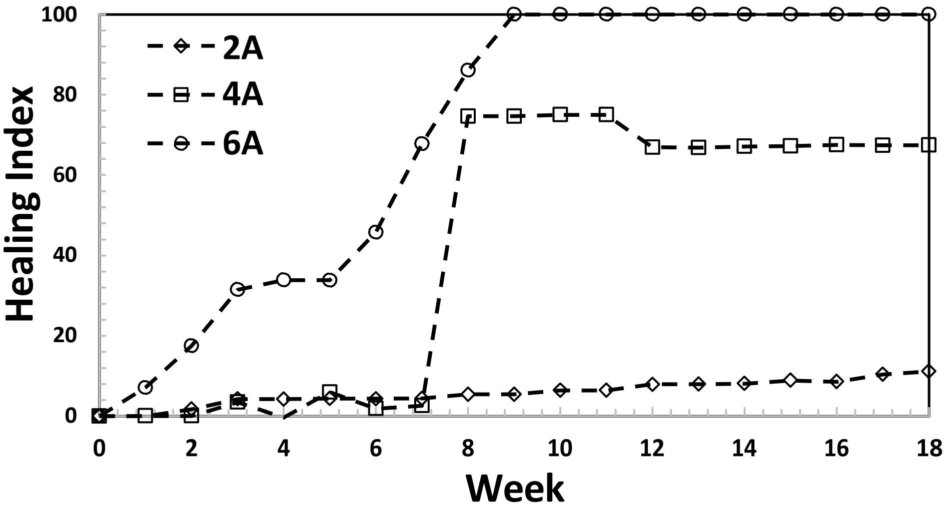

Figure 7: Healing index for disks exposed to fresh water

Figure 8 shows the healing index of the exposed and control disks over an 18-week period. There is no significant differences between the healing rates of exposed and control disks. This suggests that salt-water exposure does not significantly impact the healing rates of bacteria based self-healing concrete. The crack size appears to be the controlling factor, with healing occurring when the maximum crack width was below 0.25 mm.

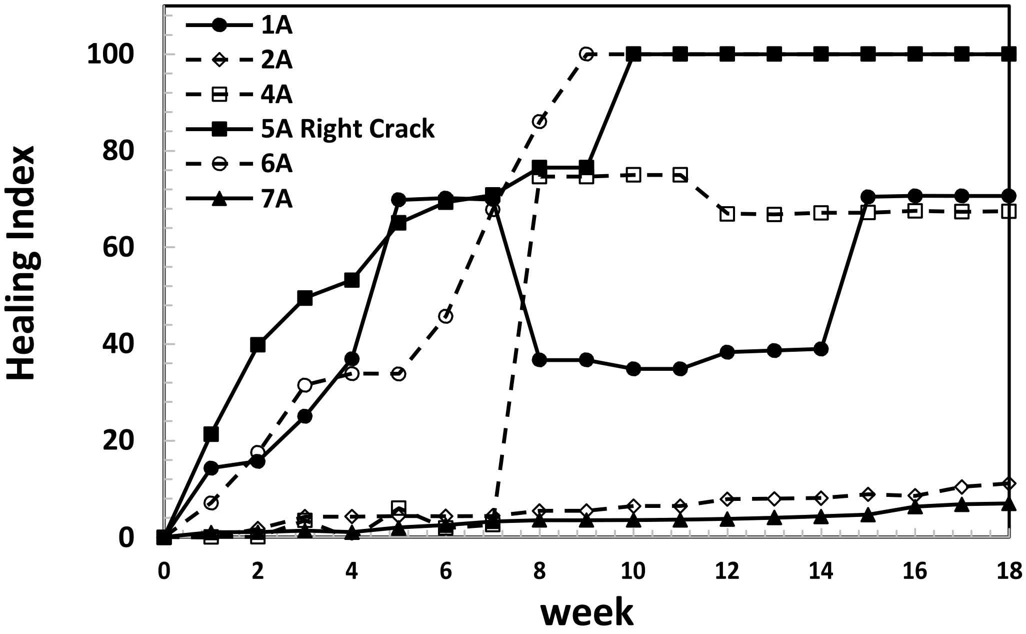

Figure 8: Healing index for disks exposed to salt water and fresh water

b) Freeze-Thaw

The disks used for the freeze-thaw experiment were allowed to cure for 28 days before being cracked and submerged. For the first 4 weeks of the experiment, all disks were placed in fresh tap water. After initial healing was observed, four of the seven disks were moved into the freezer and subjected to 6 hours of freezing and 18 hours of thawing. Three of the seven disks were kept at room temperature as control specimens.

The initial average crack widths of the disks at week 0 was 0.19 mm. The individual crack widths ranged from 0.09 mm to 0.3 mm. The healing index for each disk was calculated over 18 weeks. Figures 9 and 10 show the healing index for the freeze-thaw and control disks, respectively. The healing progress continued only for one disk, Disk 1-1, during the freeze-thaw cycles. For disks 1-3 and 1-7 the healing partially continued during the freeze-thaw cycles, but the concrete spalled at the location of the crack and the healing stopped. Disk 1-3 resumed the healing process after week 14 when it was removed from freezer and kept at room temperature again for the final four weeks. Disk 1-3 fully healed after 18 weeks. Unfortunately, disk 1-7 spalled too severely and the healing index could not be calculated after week 16. The healing resuming for one disk after it was removed from the freezer at week 14 suggests that the bacteria could be affected by the temperature conditions. The freeze-thaw cycles reduce the activity of the bacteria, and therefore the potential for healing.

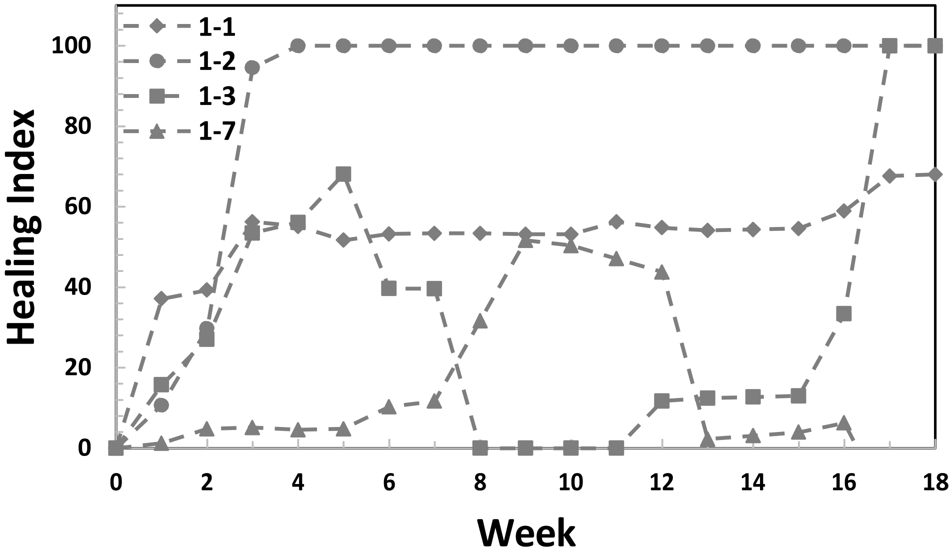

Figure 9: Healing index for disks exposed to freeze thaw cycles

None of the three disks that were kept at room temperature as control disks fully healed. Figure 10 shows the healing index for the freeze-thaw control disks. The maximum healing was 50% after 18 weeks for disks 1-4 and 1-5. It is not clear why two of the salt-water exposure control disks healed above 70% while the freeze-thaw control disks did not healed above 60% although they were subjected to the same condition. It is possible that the selected location of the measurements, shown in Figure 4, was located on aggregate where the least healing was observed. Also, the initial crack width was larger in the freeze-thaw control disks, which may have contributed to the reduced healing.

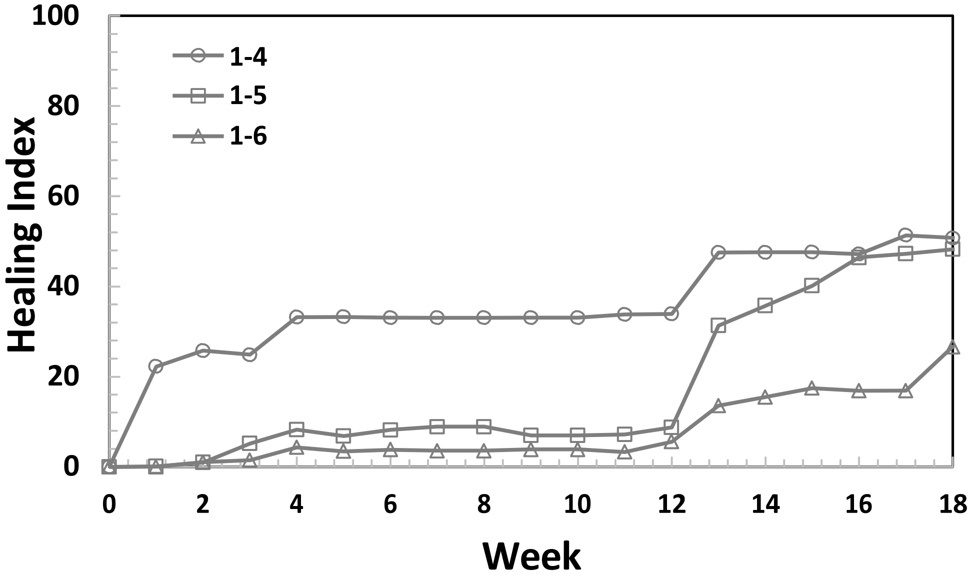

Figure 10: Healing index for samples exposed to Room Temp

Conclusions

A self-healing concrete would help produce a more sustainable infrastructure by reducing the cost of maintenance and repair. Concrete is used in various environmental conditions. To investigate the efficacy of bacteria-based self-healing concrete, the effects of common environmental conditions must be evaluated. Two common environments that may affect the bacteria's viability are salt water (e.g., a marine environment) and exposure to freeze-thaw cycles in cold climates.

An experiment was conducted to evaluate the efficacy of the bacteria-based self-healing concrete in a marine environment. Salt water is not a limiting factor in the bacteria's ability to repair cracks. Crack widths exceeding 0.25 mm did not show significant signs of healing. Also the lightweight aggregates in which the bacteria were encapsulated did not show significant signs of healing compared to locations outside of the lightweight aggregates. The lack of healing on the surface does not mean that healing did not occur beneath the surface.

The freeze thaw experiment affected the efficacy of the bacteria. However, with the selected bacteria's ability to produce endospores, bacteria activity levels elevated when the disks were reintroduced to higher temperatures. Nevertheless, healing of the control disks did not exceed 50%, because large sections of the evaluated area contained cracks through sections of lightweight aggregates.

Acknowledgment

This research is funded by University Transportation Research Center (UTRC).

References

DSMZ. (2007) "List of Recommended Media for Microorganisms." *DSMZ: List of Media for Microorganisms*. Ed. Jörg Overmann. N.p

"Fast Facts – Behind the Numbers." Fast Facts – Behind the Numbers | Concrete Joint Sustainability Initiative. N.p., n.d. Web. 1 Dec. 2015.
http://www.sustainableconcrete.org/?q=node/42

Jonkers, H. M (2011). "Bacteria-based Self-healing Concrete." *Heron*, v. 56, 5-16.

Jonkers, H. M., Arjan T., Gerard M., Oguzhan C., and Schlangen E. (2010) "Application of Bacteria as Self-healing Agent for the Development of Sustainable Concrete." *Ecological Engineering*, v. 36, Issue 2, 230-35.

Li, V. C., and Herbert E. (2012) "Robust Self-healing Concrete for Sustainable Infrastructure." *Journal of Advanced Concrete Technology,* v.10, 207-18.

Mors, R. M., and H. M. Jonkers. (2006) "Practical Approach for Production of Bacteria-Based Agent-Contained Light Weight Aggregates to Make Concrete Self-Healing." *Magnel Laboratory for Concrete Research.*

Piggot, P. J., and J. G. Coote. (1976) "Genetic Aspects of Bacterial Endospore Formation." Bacteriological Reviews. *U.S. National Library of Medicine.*

Wiktor V. , Jonkers H. M. (2011) "Quantification of crack-healing in novel bacteria-based self-healing concrete." *Cement and Concrete Composites*, V.33, Issue 7, 763–770.