

RECLAMATION

Managing Water in the West

Using Self-Healing Concrete for Concrete Structures, Laboratory Report

Research and Development Office
Science and Technology Program
(Final Report) ST-2019-1791-01



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Using Self-Healing Concrete for Concrete Repairs on Aging Concrete Structures, Laboratory Report

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Abbreviations and Acronyms

CGSL	Concrete, Geotechnical, and Structural Laboratory
CSP	concrete surface profile
OPC	ordinary portland cement
SEM	scanning electron microscope
SHC	self-healing concrete
S&T	Science and Technology
w/c	water to cement ratio

Executive Summary

Reclamation's aging infrastructure, along with the challenge of budget constraints, has created an opportunity to investigate concrete repair methods that will provide positive long-lasting results. Currently, when repairs to critical infrastructure are performed, we find that there are several factors that affect the success of the repair activities. Many concrete repair materials can crack and/or debond after only a short period of time (a few months to a few years), setting the stage for premature repair failure by allowing ingress of water, air, chlorides, and other contaminants into the concrete. These contaminants can lead to further deterioration of the concrete and corrosion of steel reinforcement. Industry-wide, a majority of concrete repairs are estimated to last only about 5 to 7 years.

Increasingly, structures require remedial work or even demolition after only a few years following repair because of deficiencies in repair material performance. Spalling, cracking, rust staining, and corrosion of reinforcing steel are all examples of problems encountered with such repairs. In addition, behind these visible signs of repair problems are more complex issues that are not readily apparent. For example, widespread ignorance or misunderstandings about material compatibility factors and their impact on the design of concrete repair projects are still widespread. The objective of any repair project should be to produce a durable repair at relatively low cost. Equally important are the methods of application, surface preparation, construction practices, and inspection.

Concrete cracking and unsuccessful repair projects are not limited to the Bureau of Reclamation (Reclamation) as the entire concrete industry also faces this challenge. Experimentation and application of mineral-producing bacteria (*Bacillus pasteurii*, and *Escherichia coli*) with the ability of enhancing the autogenous (self) healing properties already present when using Portland cement has shown promise and the benefits include reduction in maintenance costs and increased durability, as well as the elimination of recurring repairs.

In contrast to traditional concrete mixes, Self-Healing Concrete (SHC) contains the addition of bacteria spores and a food source housed within a clay sphere, compressed powder pellet, or polymer pill that are mixed with traditional concrete ingredients. After a crack develops in a concrete placement and water penetrates the opening, the bacteria are activated; insoluble limestone is produced by the bacteria, which fills the crack, and the crack is "healed," thereby protecting the repair area from further damage by the environment.

This report summarizes Reclamation's interest in the SHC technology, efforts to acquire the SHC ingredients, laboratory testing and results produced from the collaboration between Reclamation's Waterways and Concrete Group 1 and the Concrete, Geotechnical, and Structural Laboratory, as well as future outlook.

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Introduction

As Reclamation's infrastructures continue to age, many structures have surpassed their expected service life. The increased demands on these systems further strains their capacity to maintain normal operations. Limited resources for maintenance and repair at all levels also contribute to limiting repairs. Concrete cracks are an important indicator for determining the overall condition of a structure. While not all concrete cracking is detrimental to a structure, the size, location and severity can dictate the importance of repairs. Large cracks can indicate the existence of structural overloading or more severe deterioration. Smaller cracks can also provide evidence of damage, including those related to poor substrate preparation, poor curing, deterioration from a number of causes, and use of poor-quality materials.

Concrete cracks can be hard to control and prevent and can lead to durability issues related to ion ingress. Reclamation and others have published numerous studies analyzing the factors that cause concrete cracks and mitigation strategies with the goal of extending the service life of concrete and concrete repairs. Such publications have examined factors such as poor substrate preparation prior to new concrete placements, concrete shrinkage reducing additives, effects of bond strength between existing and new concrete, and durability issues caused by poor-quality materials. These studies indicate that while some progress has been made by the concrete repair industry there are still durability problems with many repair projects. Obviously, performing repeat repairs after only a few years of service is very costly.

Based on the case studies presented in ST-2016-7064-1 "Using Self-Healing Concrete for Concrete Repairs on Aging Concrete Structures" [1] on premature cracking in concrete placements at Glen Elder Dam and Kirwin Dam additional focus was placed in identifying products with the potential to reduce cracking. Products of this kind can have a positive impact on concrete repair projects by reducing the overall cost since the field conditions can often reflect a larger scope of concrete deterioration than what was anticipated.

Review of Self-Healing Concrete Technologies

Currently, there are several variations of the self-healing technology and literature research has shown that the majority are in research stages. The technologies vary but identify a range of bacteria with the potential to provide self-healing capabilities to concrete structures. A selection of these includes:

- Research by Lund University, Sweden examining the frost resistance potential of self-healing concrete
- Research at Manhattan College, New York investigating the potential of self-healing concrete when exposed to salt water and freeze-thaw cycling
- Development of a concrete material exhibiting self healing properties by the University of Rhode Island

- Exploration of a self-healing concept by Binghamton University and Rutgers University
- Research and Development of Basilisk Self-Healing Concrete from Delft University of Technology

Lund University Frost Resistance Investigation

The potential benefits of self-healing concrete were examined by the Lund University of Sweden to address cracking in dams and hydro power infrastructure [2]. Research was specifically aimed at identifying a solution to concrete cracking and leaking cracks. It has been noted that hydro power is the primary renewable energy source in Sweden, with approximately 1,800 power plants and 10,000 dams of varying size and age [3]. And with no new hydro power projects on the horizon the existing infrastructure is heavily relied upon for power generation. However, the majority of the infrastructure dates from the 1950s to the 1960s with a large portion requiring renovation and refurbishment [3].

The research performed by Lund University investigated the causes of cracks in hydro power stations, and those identified included: cracking from drying shrinkage, thermal cracking, and loads. Consideration was given to concrete damage as a result of internal and external frost damage. Due to Sweden's fluctuating temperatures and the location of most of Vattenfalls hydro power plants in the northern part of the country, the exposure to extreme temperatures is likely. According to the study, temperatures could drop below -25°C at least once every winter and the mean temperature for January is below zero for the whole country [2]. As a result of these extreme temperatures, Sweden's infrastructure places a large importance on the addition of air entrainment during concrete mixing to ensure adequate frost resistance.

Investigation into a new form of crack repair involved the use of a calcium carbonate precipitating bacteria. This bacteria chosen belongs to the genus *Bacillus* and is aerobic. Both oxygen and an external calcium source are required for the bacteria to produce the calcium carbonate that heals concrete cracks. According to researchers, the bacteria was deemed favorable due to its resistance to temperatures of at least -80°C , with activity decreasing in sub-zero temperatures [2]. However, due to its ability to produce calcium carbonate within the concrete matrix the study looked at the impact of this additional byproduct on the porosity and air void content of concrete. Initially there were concerns that the calcium carbonate may fill the air void pockets within the concrete matrix and hinder the concrete's ability to prevent frost damage. According to the study, normal hardened concrete consists of about 12-20 percent of total porosity due to the non-porous aggregates used in Sweden. When the cement paste is fully hydrated the porosity can be as high as 47% for a w/c ratio of 0.6 [2]. Normal air void content is between 4-7% with a limitation to anything above 7% causing a reduction in concrete strength. An air void content of 3.5% is recommended when freezing occurs in contact with fresh water and that value increases to 5% if freezing occurs from contact with salt water.

Lund University's study examined three different concrete batches with the goal of identifying the benefits of the bacteria. The three batches included one without air entraining agent (#C), one with air entraining agent (#CA), and one with both air entraining agent and the bacteria

(#CAB), each mix design is listed in Table 1. For this study the bacteria was added in the form of a solution to the concrete batch #CAB with a content of 18.18 lb/yd³ of fresh concrete. The concentration of the solution was 1.72×10^8 cells/fl. oz with a calcium lactate solution concentration of 2.95×10^{-5} mol/fl. oz, both were of a ratio of 1:1.

Testing was performed on 9 samples that consisted of: four cubes of size 5.91 by 5.91 by 5.91 in.³ and five cylinders with diameter 3.94 in. and height 7.87 in. The samples were evaluated for:

- Compressive strength
- Tensile strength
- Scanning of air voids using scanning electron microscope (SEM)
- Repairing of crack after tensile splitting test
- Freeze-thaw testing

Table 1. Concrete Mixture Proportions for Testing Performed by Lund University

Batch	#C*	#CA*	#CAB***
Water/Cement Ratio	0.5	0.5	0.5
Cement ¹ [lb/yd ³]	638	638	638
Water [lb/yd ³]	319	319	319
Aggregate 0-0.08 in. [lb/yd ³]	1389 ⁵	1289 ⁵	1289 ⁵
Aggregate 0.31-0.47 in. [lb/yd ³]	1698	1576	1576
Air Entraining Agent (AEA) ² [lb/yd ³]	-	0.97	0.98
Super plasticizer ⁴ [lb/yd ³]	3229	-	-
Bacterial preparation [lb/yd ³]	-	-	18.18
Calcium lactate solution CaC ₃ H ₆ O ₃ [lb/yd ³]	-	-	18.18
Air content ³ [%]	1.4	4.6	4.4
Slump test measurement ³ [in.]	2	0.71	0.91
Density ³ [lb/yd ³]	4047	3964	3981

*Concrete without AEA

**Concrete with AEA

***Concrete with AEA, spores and calcium lactate

¹ Anlaggningscement Degerhamn CEM I 42,5 density 5394 lb/yd³

² Sika AirPro

³ Measured after mixing

⁴ Sika Evo-26

⁵ Dry Weight

Results from this study indicated that no changes were evident between samples #CA and #CAB for both compressive and tensile strength. However, sample #C reflected a significant difference due to the absence of air entraining agent, as expected. Results from this study also validated Reclamation's findings that the use of a bacterial agent does not affect the compressive strength of concrete. Lund University's research trials involved the addition of a bacterial solution to concrete ingredients whereas Reclamation's evaluation involved the use of Basilisk Concrete's SHC Agent that houses the bacterial agents within a polymer pill.

Evaluation of the effect of the bacterial solution on the air void content of the concrete matrix was performed by use of scanning electron microscope. SEM pictures were taken of the samples that were used during the compressive strength testing, these samples were 56 days of age and comprised of small pieces taken from the surface and interior of the samples. The samples were placed in a drying cabinet for 3 days and then stored in an airtight jar [2]. Analysis of these specimens treated with the bacterial solution revealed the development of small needle like structures within the air voids. Researchers believed that these structures were ettringite and not related to the addition of the bacterial solution. Lund University's conclusion was that the bacteria do not impact the air voids as theorized, SEM analysis revealed that no calcium carbonate had filled in the cavities, even with the ideal circumstances it is believed that the bacteria would have produced calcium carbonate promptly and that the SEM analysis would have verified this especially after 56 days. Researchers also believe that the use of fresh water during trials created a more optimal synergy with the bacteria, versus the use of salt water, which is more aggressive to concrete.

Investigation into crack healing by the addition of the bacterial solution was only limited to analyzing any healing effects noted from taping the tensile strength specimens back together after failure, and immersing them in tap water for 50 days. The researchers found that of the two specimens from the batch #CAB, only one remained intact after the tape was cut. It was speculated that the other specimen treated with the bacterial solution may not have bonded due to crack widths being too large to heal [2]. The study did not record or analyze the crack healing limitations of the bacteria, neither by inspection of minimum or maximum crack widths having healed via calcium carbonation or SEM analysis.

Freeze-thaw testing was performed on the specimens by exposure to both fresh water and salt water environments. The 56-day trial collected scaling from each specimen, every 7th day the samples were brushed with a stiff brush, and the scaling was then filtered and dried overnight, with the scaling and filter weighed on a scale with 0.035 oz accuracy [2]. For the fresh water trial specimens, the study concluded that both the #CA containing air entrainment and #CAB containing the bacterial solution and air entrainment specimens behaved relatively close to each other when evaluating the accumulated scaling. There was an initial difference between both sets, but it was reported that this variation remained even throughout the entire experiment. The researchers believed that the difference could have been due to an initial difference in the air void content. However, after the 56 day testing trial had ended the difference between the two samples had disappeared. For the salt water specimens, there was a significant difference between the specimens with the #CAB sample having 33% more accumulated scaling than the #CA sample. According to researchers this difference can be due to more than just the initial air content for both samples. Further, the results from this freeze-thaw study were compared to results gathered from other tests performed by the same methods. That comparison revealed that the Lund University trial specimens exhibited a larger degree of scaling than specimens from other tests. In scaling vs. days/cycles graphs, the Lund University specimens #CAS and #CABS produced significantly higher trendlines than specimens from other studies. However, it should be noted that the Lund University specimens were of a w/c ratio of 0.5, and the comparison specimens were of a w/c ratio of 0.4. Researchers indicated that due to the lower w/c ratio used for the specimens in other comparison tests, the denser cement paste along with a higher degree of hydration produced more robust samples that assisted in inhibiting water transport. The

University of Lund specimens with a higher w/c ratio were more responsive to water transport and thus produced higher scaling results.

Conclusions from the Lund University study indicated that the bacterial solution did not change the strength development of the concrete samples, and that frost resistance was also not affected from the addition of the bacteria and calcium lactate when tested in fresh water [2].

Manhattan College Self-Healing Concrete Research

Research at Manhattan College evaluated a bacteria-based agent from Mors & Jonkers (2006) [4] with the capacity to produce minerals within concrete and effectively seal cracks. This type of self-healing concrete or “bioconcrete” had shown promise based on research results from various laboratory scale studies. However, because of the infancy of this technology further research is required to improve the performance, reliability, versatility, and repeatability of the self-healing process [5]. The bacteria selected for the Manhattan College self-healing concrete trials were: *Bacillus Pseudofirmus* DSM 8715 and *Bacillus Cohnii* DSM 6307, and both are naturally occurring, and can resist the high alkalinity of concrete [5]. In order for the bacteria to produce the necessary healing minerals, calcium must be available. However, current limitations on the use of cement warrant the replacement of cement with other mineral admixtures such as fly ash that can decrease the total amount of calcium available for the bacteria. A study was implemented to investigate the effect of different percentages of fly ash as replacement for Portland cement on the healing rate of the self-healing concrete [5]. A separate study also investigated the viability of encapsulated bacteria within concrete and its behavior in marine environments and under freeze-thaw cycles [6].

The investigation related to the replacement of cement by fly ash was due in part to the ACI recommendation that Portland cement content be replaced by a certain percentage of fly ash. Based on the type of fly ash used the content of cement can be replaced by up to 15 to 25% type F fly ash that has a lower calcium content (less than 20%) or up to 20 to 35% by fly ash type C that has a higher calcium content (more than 20%) [5]. For this study concrete cylinders were produced with concrete mixtures ranging from 0% to 15% fly ash. The cylinders were cut into one-inch-thick disks that were cracked and then exposed to fresh water. The cracks were then monitored for a period of 14 weeks. Results from the study indicated that the fly ash content directly affects the healing rate of the cracks. Researchers theorized that it is possible that the healing rate nearly slows down rather than reducing due to the decreased calcium content, but more research was encouraged to gain a definitive result [5].

The second study, examining the effectiveness of concrete mixed with a self-healing bacteria agent researched the performance of the bacterial concrete when exposed to two common environments: marine and freeze-thaw cycles. The marine environment chosen for this study was replicated with a 3.5% saltwater solution, and the freeze-thaw cycles consisted of freezing the bacterial concrete specimens and submerging them in water for 6 hours then following with a 18-hour thawing period [6].

The bacterial concrete specimens for this research study consisted of encapsulating the *Bacillus Pseudofirmus* DSM 8715 and *Bacillus Cohnii* DSM 6307 into lightweight aggregate of

0.196- 0.394 in. diameter in size and mixing them with type II cement, water, and sand with a water-to-cement ratio of 0.5 [6], see Table 2 for content.

Table 2. Mix design for concrete containing bacteria for testing performed by Manhattan College

Ingredient	Content
Type II cement	24.93 oz
Water	11.95 fl. oz
Sand	3.59 lb.
Lightweight aggregate*	16.23 oz

* Healing agent was encapsulated in aggregate prior to mixing

A 3.94 in. diameter by 7.87 in. high cylinder was cast and then cured for 28 days. The cylinder was then cut into seven disks with thicknesses of 1.06 in. each. The disks were then cracked using displacement controlled loading and afterward fully submerged in fresh water for 4 weeks to activate the healing process. After the 4 week healing period the specimens were then tested in the salt water environment and for freeze-thaw resistance. Progress was monitored using a Nikon SMZ18 microscope and a Nikon DXM12C camera, and crack width measurements were recorded each week over the course of 18 weeks.

The salt water trials consisted of monitoring four of the seven specimens in a 3.5% saltwater solution while the other three specimens were left submerged in fresh water as control samples. Initial crack width measurements were taken for the seven specimen and recorded, the cracks ranged from 5.5×10^{-3} in. to 1.4×10^{-2} in. Results from the 18 week trial revealed that there was no significant difference in the healing rates between the specimens in fresh water and the saltwater solution [6]. However, for specimens with an initial crack width of at least 1.4×10^{-2} in. there was no healing noted during the 18 week period. Researchers believe that while there was no significant impact on the specimens from exposure to saltwater there was a crack width limitation for healing. This study indicated that healing of cracks occurred when the crack width falls below 9.84×10^{-3} in.

The freeze-thaw trials also consisted of seven disks prepared in the same manner as those from the saltwater trials. The seven specimens were cracked and submerged in fresh water for 4 weeks to initiate the healing process. After the healing process, four of the specimens were subjected to 6 hours of freezing and 18 hours of thawing [6]. While the other three specimens were used as control specimens and kept at room temperature. Monitoring of the healing process for this trial was evaluated over the course of 18 weeks.

Crack width measurements were taken for each of the specimens at the initial state (after the 4 week healing process) and an average initial crack width was recorded as 7.5×10^{-3} in. After the 18 week trial period the researchers identified the following:

- Only one of the disks subjected to the freeze-thaw experiments continued with the healing process for the full 18 weeks
- Two other disks only partially continued with the healing process, and concrete spalling at the site of the crack may have been the cause of the limited healing.

- Of the two disks that only partially continued with the healing process, one of them was kept at room temperature after 14 weeks and fully healed at the 18 week period.
- The control specimens that were kept at room temperature did not fully heal, only reaching a maximum healing of 50% after 18 weeks.

Researchers theorized that limitation of healing in the control specimens may have been due in part to the selected location of the crack, on aggregate, and also the initial larger size of the crack widths on these specimens. Also, this test indicated that due to the bacteria's ability to produce endospores further activity is evident when the specimens were reintroduced to higher temperatures [6].

University of Rhode Island Microencapsulated Healing Agent

While Reclamation's interest in self-healing concrete is based on the use of bacterial agents to produce calcium carbonation to heal cracks within the concrete matrix, other self-healing methods include the use of non-bacterial solutions that also produce healing effects within the concrete matrix. The University of Rhode Island has developed a self-healing technology that involves the use of polyurethane microcapsules that contain a sodium silicate solution that when released into the concrete matrix reacts with the calcium hydroxide in cement to produce a gel that partially heals cracks [7]. Their study examined the benefits of this technology on the material and mechanical properties of concrete, specifically its effect on compressive strength, flexural strength, and corrosion inhibition. The investigators from this study found that there are successful demonstrations that support the use of polymer composite systems within the concrete matrix, as some have demonstrated up to 80% recovery of toughness after a fracture [7].

The self-healing mechanism for this study was described as follows: the microencapsulated healing agent utilizes stress to rupture the capsules and release the sodium silicate into adjacent cracks, once the sodium silicate reacts with the calcium hydroxide a natural concrete binding material forms (calcium-silica-hydrate (C-S-H) gel). Addition of the polyurethane capsules comprises only 2% of the concrete matrix volume. Ten concrete samples were prepared, five were polyurethane microcapsule specimens and five were control specimens, all were done in accordance with ASTM C-1019. The concrete mix was achieved with 48.50 oz. of Ottawa C-109 sand, 17.64 oz. of Type I/II Portland cement and 8.18 fl. oz. of water. The specimens containing the polyurethane microcapsules were obtained by the addition of the capsules to the mix water at 2% volume [7].

For flexural strength testing the specimen samples were 6.3 in. by 1.58 in. by 0.79 in and for compressive strength testing the specimen samples were 19.69 in. by 19.69 in. 19.69 in. Curing of the specimens was achieved by submerging them in water for two days and maintaining them in a 95% constant humidity environment for 28 days.

To achieve the internal cracking for flexural strength testing, each specimen was subjected to an applied load of 0.25 mm/min to induce microcracking and reach incipient failure. According to researchers, this microcracking closely simulates the cracking and deformations that occur within concrete after applied or natural stress [7]. The specimens were then retested after one week to evaluate the strength recovery due to the calcium-silica-hydrate gel binding reaction. Results

from this study indicate that the five control specimens without the microencapsulated healing agent only recovered about 10-14% of their initial strength. The specimens with the microencapsulated healing agent reflected up to a 26% recovery of their initial strength. Researchers concluded that the specimens with the healing agent benefitted from the additional recovery due to the ruptured capsules that partially healed cracking within the concrete matrix.

The compressive strength trials also consisted of ten specimens, five containing the microencapsulated healing agent and the other five taken as control specimens. For this trial the specimens were loaded at a strain rate of 1 mm/min, and this load was stopped when the sample had reached maximum load but the specimens were not allowed to reach failure [7]. The specimens were then allowed an unspecified healing time, and then retested to failure. Results indicated that the encapsulating healing agent does not interfere with the cementitious matrix and there is also no loss to compressive strength when compared to the control specimens.

Analysis of corrosion inhibition due to the microencapsulated healing agent within the concrete matrix examined the benefits of sodium silica deposits on the reinforcement. The intent of this study was to examine the formation of a passive sodium silica film on the reinforcement due to the ruptured capsules, as well as investigate porosity reduction and its effects on chloride intrusion. Six concrete specimens of 6.3 in. by 1.57 in. by 0.79 in. dimension (three microencapsulated specimens and three control specimens) containing an iron wire were subjected to a three point bend test to induce one large crack directly to the wire upon failure. This crack was used to give a sodium chloride solution a path to the iron wire in the sample [7].

For the corrosion inhibition trial, the chloride solution was comprised of a 0.5M sodium chloride solution. The solution was poured into the large crack of each specimen via a small well until it reached the iron wire. In order to monitor and measure corrosion, the open circuit potential of the wire was recorded with a voltmeter. The experiment progressed until each specimen displayed severe corrosion or the potential reached -0.500V [7]. Results indicated that for the three control specimens, each one experienced a rapid decrease of potential once the sodium chloride solution was added. In 40 seconds the potential had dropped to near -0.350V, indicating a high risk of corrosion. The control specimens then reached severe corrosion at an average rate of 142 seconds. The microencapsulated specimens displayed more favorable results, initially the exposure to the sodium chloride solution caused a rapid decrease of potential just like the control specimens reaching a potential of -0.350V at an average rate of 52 seconds. However, researchers indicate that beyond that point the potential slowly decreased to -0.400V at an average rate of 200 seconds for the three specimens. The three specimens then reached severe corrosion at an average rate of 17.7 minutes.

Researchers believe that the microencapsulated specimens benefitted from the formation of the thin passive layer, this may explain the prolonged period where the potential of the microencapsulated specimens remained at -0.400V. It is also believed that along with the thin passive layer, the cracks healed via the ruptured capsules and reduced the porosity of the concrete thereby limiting sodium chloride intrusion to the wire. Results of the study indicated that the primary benefit was the formation of the thin passive layer as severe corrosion was prolonged when compared to the control specimens. It is further theorized that an increased volume of capsules within the matrix would have allowed a larger deposit of silicates to form on the wire thereby extending the protection layer.

Binghamton and Rutgers University Self-Healing Concept

Research collaboration between professors at Binghamton and Rutgers University examined the application of a fungi into concrete with the potential to heal cracks and mitigate corrosion of reinforcement. This fungi with the ability to heal cracks via calcium carbonation has shown both cost-effective and environmentally friendly application [8]. This approach was considered after identifying that concrete cracks eventually lead to steel corrosion as a result of chloride ion or CO₂ intrusion. When cracks develop they can provide channels for chloride ions to reach the reinforcing steel with the effect of damaging the protective passive oxide layer surrounding the steel. As the University of Rhode Island study reported, once the protective passive layer has been damaged the reinforcing steel can exhibit severe corrosion thereby affecting its structural capacity. Likewise, according to the Binghamton and Rutgers University study any intrusion of CO₂ via cracks can reduce the high alkalinity of the concrete and as a result its ability to provide the environment for the reinforcing steel to form the protective passive layer.

To counter these challenges, researchers looked at the potential of calcium precipitating fungi to heal cracks from within the concrete matrix and provide protection against chloride and CO₂ intrusion. However, the previous studies that were referenced by the researchers determined that the high alkalinity of concrete did not provide a suitable environment for various species of calcium precipitating fungi. Results of those studies indicated that the fungi were unable to produce the calcium carbonation required to heal the cracks. For their study the researchers identified *Trichoderma reesei* Species as the fungi with the capacity to survive within a high alkaline environment. Furthermore, the researchers stated that with water and oxygen the dormant fungal spores would germinate, grow, and precipitate calcium carbonate to heal the cracks [8]. Trials indicated that the *Trichoderma reesei* fungi remained dormant once it was added to the concrete, and were activated at the sign of a crack. X-ray diffraction and scanning electron microscopy were used to validate that the fungi activated, and analysis confirmed that the fungi had indeed produced calcite crystals within the concrete matrix as expected. In these trials the researchers noted that due to the leaching of calcium hydroxide from concrete, the pH levels within the concrete matrix increased from the original value of 6.5 to 13.0 [8]. This interesting note validated that the fungi could still activate and precipitate calcium carbonate at much higher pH levels than expected. The researchers theorized that the ability of the fungi to activate at higher levels would also benefit the formation of the protective passive layer on the reinforcing steel.

Although this study produced favorable results, further testing by Binghamton and Rutgers University will include evaluating the fungi in a non-laboratory environment and investigating the effects of pH, temperature, ultraviolet light, growth medium composition, and fungal spore concentration [8].

Basilisk Self-Healing Concrete

In the spring of 2016 Basilisk Concrete began manufacturing and distributing bacterial self-healing concrete products. This bacterial self-healing concrete technology was patented by Dr. Henk Jonkers of the Delft University of Technology. Dr. Jonkers' research began by identifying

that sub-millimeter sized cracks may have an impact on the durability of concrete due to ingress of water and chemicals that can cause matrix degradation and corrosion to embedded steel [9]. This challenge along with recognizing that regular maintenance and repair of concrete can have a significant impact on costs led to consideration of a type of autonomous self-healing repair system. The system involved the use of a mixture of dormant bacteria along with a food source loaded into a carrier that when introduced into the concrete matrix would produce calcium carbonate resulting in crack healing [9]. In 2011, a research publication by Dr. Jonkers discussed this autonomous self-healing repair system. The bacteria selected for a research study was of the genus *Bacillus*, which is alkali-resistant and is activated by the ingress of water. The by-product of the bacteria is a calcium carbonate that acts as a type of bio-concrete that seals cracks.

While the goal of this study was to provide a long-term benefit of the bacteria's self-healing properties to concrete structures, it was found that direct addition of the bacterial spores to a concrete mix reduced the life-time of the spores to one-two months [9]. Dr. Jonker's research indicated that in dry conditions the bacterial spores could survive for several decades. Therefore, in order to preserve the bacteria, and their food source (calcium lactate) during addition to a concrete mixture a clay particle was utilized. The clay particles ranged from 0.078 – 0.157 in. and they replaced similar sized aggregate material [9]. At the time it was not known how the replacement of some of the aggregate material would impact the concrete properties. But a previous study had demonstrated that the addition of calcium lactate had resulted in a 10% increase in compressive strength compared to control specimens [9]. Therefore, it was believed that the presence of the calcium lactate within the clay particles would benefit the overall concrete properties even though it meant replacement of some of the aggregate.

For this study 50% of the light weight aggregate was replaced with the clay particles, this was concerning since replacement of such large quantity would impact the concrete properties. The concern was later verified when the compressive strength of the bacterial concrete specimen was tested. After 28 days the compressive strength of the bacterial concrete reflected a 50% decrease when compared to the control specimens. While the compressive strength was affected it was also found that crack healing by the bacterial spores occurred.

Testing indicated that sub-millimeter sized cracks (0.0059-inch) were efficiently sealed [9]. Dr. Jonker's research indicated that while concrete structures have a capacity for autonomous healing due to delayed hydration of non-hydrated cement, the healing is limited to cracks of widths smaller than 0.007-inch [9]. Additionally, there is some limitation to the autonomous healing due to the location of the non-hydrated cement particles. While the optimum location of these non-hydrated particles should be within the crack, they are found to lie on the crack surface and therefore do not benefit crack healing [9]. In contrast, addition of the clay specimens within the concrete matrix increased self-healing of cracks. Results indicated that there was 100% healing of cracks (6 out of 6 tested specimens) vs. 33% healing of the control specimens (2 out of 6 tested specimens) in their study [9]. Conclusions of this study found that the bacterial spores are suitable to survive within the concrete matrix and provide healing capacities. However, the cost of this healing technology would need to be competitive with the conventional cost of concrete and impacts to compressive strength should be minimized.

Further publications during the year 2011 indicated that improvements to the Basilisk Concrete self-healing technology were made, improvements to the bacteria spore housing specimen and

reduction of impacts to the concrete properties were the most prominent. As discussed above the clay specimens had been the primary form of housing the bacteria spores and their food source. These clay specimens had replaced 50% of light weight aggregates and a significant impact was evident as shown in the compression strength test results. However, the volume of the clay pellets was then reduced to 20% of the volume of the concrete and it was discovered that this change only weakened the concrete specimens by 25% in compressive tests [10]. In 2011 it was further announced that the clay pellets would be replaced by compressed powder pellets that would comprise less than 1% of the concrete volume and have minimal effect to compressive strength [10].

Following improvements to Basilisk Concrete's self-healing technology, several projects followed to determine the performance of the technology in the field. The following projects were undertaken and are discussed further in Reclamation's Self-Healing Concrete assessment report [1]:

1. Canal Repairs in Ecuador (2014)
2. Parking Garage floor repairs in Vissingen, the Netherlands (2014)
3. Construction of a Wasterwater Treatment Tank in Limburg, the Netherlands (2016)
4. Diaphragm Wall Repairs for Groninger Forum, the Netherlands (2017)

Development of Reclamation's Self-Healing Concrete Research

Increasing the performance of a concrete repair and extending the service life of the structure as long as possible by reducing cracking is the goal. An alternative to standard concrete has been developed with the ability for cracks to self-heal. Self Healing Concrete (SHC) contains the addition of bacteria spores and a food source that are mixed with traditional concrete ingredients. After a crack develops in a concrete placement and water penetrates the opening, the bacteria are activated; insoluble limestone is produced by the bacteria, filling the crack. The crack is "healed" thereby protecting the repair area from further damage by the environment. Although there are different types of bacteria and SHC's being developed today, for the purpose of our initial research a product was selected with the following advantages 1) far enough along in the development to be able to obtain sizable samples for testing 2) a supplier with experience with several projects using the SHC technology that had similar characteristics to Reclamation repair projects, including canal repairs, surface concrete repairs, and concrete crack sealing 3) developed by subject matter experts 4) access to technical and on-site laboratory assistance by the supplier during our laboratory testing.

In the spring of 2016, the technology was made available for the first time through the Dutch company Basilisk Concrete. This technology was patented by Dr. Henk Jonkers from the Delft University of Technology. Through funding provided by the Dutch government there are several projects in the Netherlands and South America that have used the bacteria-based SHC. Following the completion of ST-2016-7064-1, whereby the SHC technology was evaluated from research publications and academic journals it was determined that further exploration of this

technology was beneficial to Reclamation. With the support from Reclamation's Science & Technology (S&T) program and participation from Reclamation partners it was our goal to procure the bacterial Self-Healing Agents, perform laboratory testing, and identify ideal applications in Reclamation's concrete structures.

In late 2016 discussions began with the manufacturer Basilisk Concrete, and clarification was provided on various technical questions not covered in ST-2016-7064-1 regarding the material, chemical and performance limitations of the bacterial SHC. This was also the opportunity to arrange the purchase of the self-healing agents, establish a working relationship with Basilisk Concrete, and collaborate on a laboratory plan of testing between Reclamation's Waterways and Concrete Dams Group 1 and the Concrete, Geotechnical, and Structural Laboratory in FY 17. Basilisk Concrete's willingness to provide supplemental data, access to the bacterial SHC materials, and accessibility to Dr. Henk Jonkers who is the inventor of their bacterial SHC progressed into a joint meeting with both parties to discuss collaboration. In December of 2016, a joint meeting was set up between Reclamation and Basilisk Concrete in Delft, Netherlands. The goal of this meeting was to establish a working relationship, investigate the potential of SHC on specific Reclamation concrete repair projects, receive first-hand knowledge of the SHC handling and material preparation, facilitate purchase of the concrete healing agents, and plan visitation by Basilisk Concrete representatives to Denver TSC for onsite assistance during laboratory trials.

During the trip to Delft, Netherlands the Principal Investigator was introduced to the members of the Basilisk Concrete team, those members included Mr. Bart van der Woerd - Managing Director, Renée Mors - Research & Development Engineer, Jordy Kern - Project Engineer, and Dr. Henk Jonkers - Microbiologist and inventor of bacterial SHC. Several presentations followed on the technical, and material properties of the SHC agents as well as the company's line of products. The Basilisk team provided clarification on several questions related to the compatibility of the SHC for a variety of Reclamation infrastructure construction or repairs projects. Specific clarification was given on the variety of bacteria spores available and their compatibility in varying environments (hot and cold temperatures), limitations of bacterial self-healing, application restrictions (potable water installations), and cost/benefit analysis.

The specific inquiries included the origin and safety of the bacteria spores used in Basilisk Concrete's SHC. According to Basilisk Concrete the bacteria spores (Genus *Bacillus*) were collected from a variety of locations including lakes in Russia, and Egypt. Those particular locations exhibited high levels of alkalinity (10 pH) that mirrored the alkalinity of concrete (pH of 12 to 13) [11]. Thereby, providing the bacteria spores with a habitable environment.

According to the Basilisk Concrete team, once the bacteria spores are collected they undergo a pasteurization process, and through natural selection only a select number of bacterial strains are selected. Each bacterial strain is unique in its capacity to survive in different environments that includes high temperature, low temperature, and high salt concentrations. Depending on the specific project the appropriate bacteria strain is selected for best results.

Reclamation's varied concrete repair projects require that concrete mixes include supplemental admixtures to produce a quality product. The addition of the SHC Agent would require compatibility with the concrete ingredients as well as any admixtures added during mixing.

According to Basilisk Concrete the addition of the SHC Agent does not impact or cause a reaction with the concrete ingredients or admixture products. Because the SHC Agent is comprised of a polymer pill, the bacteria spores and their food source (calcium lactate) are protected within the pill during the mixing process and shielded from the presence of water which acts as the activator (Figure 1). Concern was expressed by Reclamation engineers on the use of the bacteria and their impact on both humans and wildlife. According to Basilisk Concrete the bacteria are food grade [12] and are safe to handle, however potable water regulations do not allow the introduction of bacteria in drinking water installations. Otherwise, the best potential for this technology is for water infrastructure.



Figure 1. Basilisk Self-Healing Concrete (SHC) Agent pellets.

The scoping study report ST-2016-7064-1, briefly discussed the advantages of a higher content distribution of the SHC Agent within a concrete mixture. The higher distribution would have the ability to heal cracks extending through the entire depth of the concrete panel. However, information on the actual distribution content was not available during evaluation of the SHC through research publications. During discussions with Basilisk Concrete and supplemental data gathered from their SHC Agent brochure [12] the ideal dosage amounts are as follows: the recommended dosage of SHC Agent in a concrete mix design is 17 lb/cyd with a theoretical maximum healing benefit at 25 lb/cyd. Other topics not covered in ST-2016-7064-1 included more details on the chemical reactions when the bacteria spores are activated. According to Basilisk Concrete, the bacteria spores are aerobic and when activated consume oxygen in the area of the crack. This chemical reaction is a positive trait as the reduction of oxygen within the concrete matrix would lower the potential for corrosion of the reinforcement by exposure.

Basilisk Concrete offers three variations of their SHC technology, report ST- 2016- 7064- 1 focused on the SHC Agent and during the visit to Basilisk Concrete's home office further information was provided on the other variations. The Liquid Repair System ER7, a 2- component low viscosity solution with the first component containing the bacteria spores penetrating deep into cracks and pores, while the second component acts as the activator. The

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reaction between both components produces a gel formation that seals cracks and pores (Figure 2). According to Basilisk Concrete's laboratory trials and field application at Parking Garage Apeldoorn [1], the product permanently seals, and waterproofs cracks up to 0.0315- inches wide after 2-3 treatments. The Self-Healing Repair Mortar MR3 is a 1- component repair mortar modified with the SHC Agent. Typical applications of the mortar are actively leaking cracks, as these are typically difficult to repair with traditional methods (including bitumen). After application, the mortar will start to react within 24 hours and according to Basilisk Concrete is suitable for permanent and durable repair of cracks larger than 0.0394-inches. Information on these two products was collected by the Principal Investigator and provided to Reclamation's Concrete, Geotechnical, and Structural Laboratory engineers for consideration for laboratory testing alongside the SHC Agent.



Figure 2. Liquid Self-Healing Repair System (ER7) prior to mixing.

Following the visit to Delft, Netherlands a conjunct meeting between Basilisk Concrete, the Principal Investigator from Reclamation's Waterways and Concrete Dams Group 1, and the Concrete, Geotechnical, and Structural Laboratory engineers took place to discuss collaboration between both parties, identify appropriate applications of the SHC for Reclamation projects, and establish an initial set of laboratory testing trials. The preliminary laboratory testing plan would include the full line of the Basilisk Concrete products:

1. SHC Agent
2. Liquid Self-Healing Repair System ER7
3. Self-Healing Repair Mortar MR3

The meeting also confirmed the planned visit by Basilisk Concrete to Reclamation TSC for assistance with laboratory trials and a short seminar/presentation for all TSC engineers on the SHC technology.

In March 2017, the visitation by Ms. Renée Mors of Basilisk Concrete/TU DELFT to the Denver TSC produced the following research progress: a short seminar on the development of SHC was held for all Denver TSC engineers, the laboratory plan discussed previously by both Basilisk Concrete and Reclamation was tailored to Reclamation's Concrete, Geotechnical, and Structural Laboratory equipment capacities, Ms. Mors provided technical guidance on handling of the SHC materials and application, and the following laboratory trials commenced:

1. Compressive Strength Testing of SHC Agent (1, 7, 28, and 56 days)
2. Restrained Shrinkage Ring Test
3. Freeze/Thaw Durability
4. Bond of Repair Materials

The full testing results are covered in this report.

Acquisition of Self-Healing Concrete Materials

Based on the data gathered from the scoping study ST-2016-7064-1 [1] completed in 2016, and interest shown by Reclamation peers to investigate the advantages of SHC for new concrete construction and concrete repairs, contact was made with the manufacturer Basilisk Concrete in the fall of 2016. Initial communication with the manufacturer included requests to clarify and expand on research material previously published on SHC, chemical and performance limitations of the technology, and accessibility of the SHC materials. Supplemental data was provided by the manufacturer on ongoing or completed projects with similar characteristics to Reclamation structures, including canals, structural concrete construction and concrete crack repairs.

Currently, Basilisk Concrete produces three different variations of their bacterial SHC products. The SHC Agent that consists of 1/16-inch diameter polymer pill that houses the bacteria spores and their food source. The polymer pill acts as a protective vehicle for the bacteria and food source during the concrete mixing process. After concrete placement, the polymer pill releases the bacterial spores and food source due to the alkalinity of the concrete [12]. The Liquid Self-Healing Repair System ER7 consists of a two-system solution of bacteria spores and activator. Both parts are powder mixtures that are dissolved in water and applied separately, part A (bacteria spores) is applied first and allowed to penetrate the repair surface, it is then followed by part B (activator). Application of part B produces a chemical reaction between both parts and creates a firm gel that covers and seals cracks and pores [13]. The third product is the Self-Healing Repair Mortar MR3 that consists of Portland cement, limestone powder, fly ash, polymer fibres, plasticizer and the self-healing agent in granular form.

During Basilisk Concrete's visit to the Denver TSC in March of 2017, the following shipments were received in preparation for laboratory testing: SHC Agent (comprised of the full spectrum of bacterial strains), and Liquid Repair System ER7. Due to limited availability, the Self-Healing Repair Mortar MR3 would not be included in the testing plan for FY 17. Availability of the Self-Healing Repair Mortar MR3 was forecasted in 2018, therefore testing of the MR3 Mortar would take place in FY 19.

The Liquid Repair System ER7 arrived in good condition in sealed canisters, however one of the two SHC Agent canisters was damaged during shipment and its contents spilled inside the box. Since the SHC Agent was still confined within the shipping box and no water damage was evident the remaining contents was deemed acceptable for laboratory trials.

Laboratory Testing Program and Results

Laboratory trials were scheduled to coincide with Basilisk Concrete's visit to the Denver TSC in March of 2017. Ms. Renée Mors' presence provided Reclamation with technical guidance with handling of the Basilisk Concrete products that included the Self-Healing Agent, and Liquid Self-Healing Repair System ER7. A brief meeting between members of the Reclamation Construction, Geotechnical, and Structural Laboratory and Ms. Renée Mors was conducted to identify the starting sequence for the trials. Due to a one-day delay of arrival of the Basilisk Concrete Self-Healing materials it was decided to begin batching the control specimen concrete

slabs for the bond of materials testing, freeze-thaw control specimens, and compressive strength control specimens.

Testing objectives included evaluating the compressive strength, shrinkage potential, freeze-thaw resistance, and bond enhancement at an overlay repairs' interface to the substrate concrete, as shown in Table 3. The bond testing program was developed specifically to look at the self-healing nature of the products on a mechanically weakened substrate after micro-fractures from concrete repair surface preparation were developed.

Concrete mixtures for the slabs and cylinders for compressive strength were comprised of Type I/II cement, a 0.48 *w/c*, 3/8-inch coarse aggregate, and natural siliceous river sand. An air-entraining admixture was used to obtain an air content between 6 and 7%. The mixtures had a slump around 6-inches. Water was withheld for the concrete used for freeze-thaw testing and shrinkage testing, resulting in a 0.45 *w/c* and slump of 3-inches.

Table 3. Tests performed on samples of OPC and SHC

Test	Method	Samples
Compressive Strength	ASTM C39	11 specimens total 2 @ 1 day, 2 @ 7 days, 3 @ 28 days, 2 @ 56 days
Restrained Shrinkage	ASTM C1581	3 specimens
Freeze-Thaw	ASTM C666	3 specimens
Bond Testing Slabs	ASTM C1583	11 slabs (each includes a base slab topped with a 2-inch thick overlay)

Substrate and Overlay Bond Testing

The test specimens were cast along with the bottom slabs to be used in the bond test program. Six of the bottom slabs were cast using an ordinary Portland cement (OPC) mixture, and two of the slabs were cast incorporating the SHC into the same base concrete mixture. The base slabs were fog cured for approximately two and a half months before application of the topping slabs. To mimic typical field concrete repair situations, the base slabs were chipped, and the surfaces were prepared to a CSP 5 to CSP 7 [14] surface profile as shown in Figure 3 and then powerwashed; no sandblasting of the surface was performed. A variety of surface treatments and overlay situations were fabricated as shown in Table 4.



Figure 3. (a) Surface preparation for interface between substrate and overlay slab and (b) a close-up of substrate profile.

Table 4. Slab configuration, surface treatments and substrate conditions

Slab ID	Substrate	Interface Conditioning	Overlay
SH1	OPC	Saturated Surface Dry	OPC
SH2	OPC	Basilisk Comp A, Dry Condition	OPC
SH3	OPC	Basilisk Comp A + B, Dry Condition	OPC
SH4	OPC	Saturated Surface Dry	Sika Repair Mortar
SH5	OPC	Basilisk Comp A, Dry Condition	Sika Repair Mortar
SH6	OPC	Basilisk Comp A + B, Dry Condition	Sika Repair Mortar
SH7	SHC	Saturated Surface Dry	OPC
SH8	SHC	Saturated Surface Dry	Sika Repair Mortar
SH9	OPC	Saturated Surface Dry	OPC
SH10	OPC	Saturated Surface Dry	Basilisk Repair Mortar
SH11	OPC	Saturated Surface Dry	Sika Repair Mortar

For this study, the effect of the Basilisk Concrete Liquid Self-Healing Repair System ER7 as a surface treatment was evaluated to determine supplemental bonding strength between the substrate and overlay. Specimens SH2, SH3, SH5, and SH6 were chosen to receive pretreatment of the ER7 as either application of only Part A alone or a combination of Part A + Part B. The ER7 treatment technique varied between those specimens (SH2 and SH5) receiving only the Part A treatment and the specimens (SH3 and SH6) receiving the Part A + Part B treatment. For application of Part A, the liquid solution was applied directly to the substrate with the use of a brush until the surface was saturated. No specific time was logged but it was anticipated that the solution had seeped into the substrate when the surface was semi-dry, application of the topping slab then followed. For application of Part A + Part B, the procedure matched the steps discussed above however following verification that Part A had seeped into the substrate application of Part B then followed. For application of Part B a brush was also used to treat the surface, this process was repeated several times until the surface was coated. No specific time was logged but again it was anticipated the substrate was ready for the topping slab once it was semi-dry.

After the topping slabs were cast over the substrate concrete, the slabs were placed in the fog room for continued curing. The original test plan was to core the slabs at ages 3, 28, and 56 days and test the specimens at each age in accordance with ASTM Standard C1583, “Tensile Strength of Concrete Surfaces and the Bond Strength or Tensile Strength of Concrete Repair Overlay Materials by Direct Tension (Pull-off Method)” [15]. However, the strength of the early age bond was weak, and the 3-day testing was abandoned. At 28-days the slabs were cored through the topping slab and into the base slab using a 2-inch nominal coring barrel as shown in Figure 4. There was still difficulty in coring these slabs without disbonding occurring at the interface between the top and bottom slabs. The 28-day tests were also halted, and the slabs were returned to the fog room and allowed to cure until about 56-days.

In August of 2017, the coring configuration was modified to prevent vibration and the core barrel size was increased from a 2-inch OD to a 2-inch ID (approximately a 1/4-inch increase). These modifications were made to improve the chances of having testable specimens, see Figure 5. The change resulted in improvements to the coring operations with results listed in Table 5, however, in no case was bond achieved in the two slabs that attempted to use the combination of the Liquid Self-Healing Repair System (Part A plus B) as a bonding agent. Only the specimen that was pretreated with the Liquid Self-Healing Repair System (Part A) provided a measured bond strength. It is now thought that the Liquid Self-Healing Repair System (Part A plus B) should be allowed to heal over some duration prior to applying the topping concrete.



(a)



(b)

Figure 4. (a) Core set-up for pull-off bond testing and (b) bond test-set up.



Figure 5. Tested specimens; all fractures at overlay/substrate interface

In an effort to increase the bacteria activity, the tested slabs were placed in a 100 °F chamber and kept moist for approximately two months. A second set of cores were taken from these slabs and compared to the previous 56-day test results with observations listed in Table 6. Those results indicated that there was still no bond between the two slabs with the Liquid Self-Healing Repair System.

Table 5. Pull-off force for slabs containing self-healing concrete repair materials at various ages.

Slab ID	Description	Age	Force (lb)	Average Pull-Of Force (lb)
SH1	Substrate: OPC Surface Prep: Normal/SSD Condition Overlay: OPC	78	300	333
			350	
			350	
SH2	Substrate: OPC Surface Prep: Basilik Comp A,Dry Condition Overlay: OPC	82	500	435
			425	
			380	
SH3	Substrate: OPC Surface Prep: Basilik Comp A + B, Dry Condition Overlay: OPC	56	3 cored at 28day - NO BOND; 3 drilled at ~56 days (7/27/17) - NO BOND	
SH4	Substrate: OPC Surface Prep: Normal/SSD Condition Overlay: Sika Repair Mortar	79	475	592
			750	
			550	
SH5	Substrate: OPC Surface Prep: Basilik Comp A,Dry Condition Overlay: Sika Repair Mortar	79	300	267
			300	
			200	
SH6	Substrate: OPC Surface Prep: Basilik Comp A + B, Dry Condition Overlay: Sika Repair Mortar	56	3 drilled at ~56 days (7/27/17) - NO BOND	
SH7	Substrate: SHC Surface Prep: Normal/SSD Condition Overlay: OPC	78	225	442
			475	
			625	
SH8	Substrate: SHC Surface Prep: Normal/SSD Condition Overlay: Sika Repair Mortar	78	200	225
			100	
			375	
SH9	Substrate: OPC Surface Prep: Normal/SSD Condition Overlay: OPC	28	560	454
			500	
			450	
			450	
		56	310	481
			450	
			415	
			460	
SH10	Substrate: OPC Surface Prep: Normal/SSD Condition Overlay: Basilisk SH Mortar	28	470	406
			610	
			300	
			400	
		56	360	506
			590	
			380	
			560	
SH11	Substrate: OPC Surface Prep: Normal/SSD Condition Overlay: Sika Repair Mortar	28	580	370
			580	
			510	
			300	
		56	600	523
			590	
			475	
			500	

Table 6. Pull-off test results for slabs stored in a moist environment at an elevated temperature

Slab ID	Description	Force (lb)	Average Pull-Of Force (lb)
SH1	Substrate: OPC Prep: Normal/SSD Condition Overlay: OPC	520	373
		600	
		0	
SH2	Substrate: OPC Prep: Basilik Comp A,Dry Condition Overlay: OPC	Surface	No Bond
SH3	Substrate: OPC Prep: Basilik Comp A + B, Dry Condition Overlay: OPC	Surface	No Bond
SH4	Substrate: OPC Prep: Normal/SSD Condition Sika Repair Mortar	100	190
		280	
		190	
SH5	Substrate: OPC Surface Prep: Basilik Comp A,Dry Condition Overlay: Sika Repair Mortar	Surface	No Bond
SH6	Substrate: OPC Surface Prep: Basilik Comp A + B, Dry Condition Overlay: Sika Repair Mortar	Surface	No Bond
SH7	Substrate: SHC Surface Prep: Normal/SSD Condition Overlay: OPC	0	75
		125	
		100	
SH8	Substrate: SHC Surface Prep: Normal/SSD Condition Overlay: Sika Repair Mortar	550	280
		290	
		0	

Freeze/Thaw Testing

Freeze/Thaw durability testing would evaluate the performance of the self-healing material when exposed to the degradation mechanisms of frost, see Figure 6. The specimens were comprised of: three specimens of the SHC and three OPC control specimens. Recordings were taken until 50% damage was reached for both sets of specimens, in accordance with ASTM Standard 666, “Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing” [15]. Mass loss measurements were taken for both sets of specimens at 33 cycles (see Figure 8) and 175 cycles (see Figure 7).



Figure 6. Freeze-thaw bars prior to testing

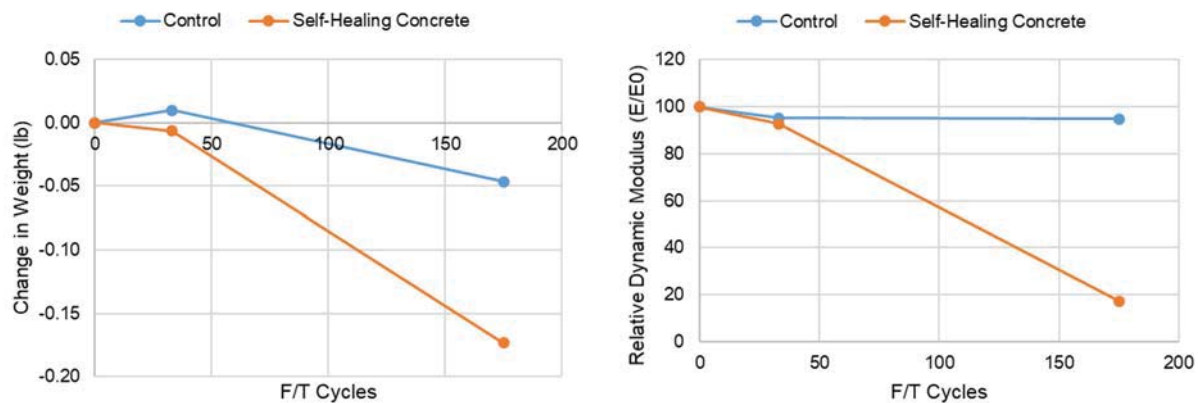


Figure 7. Change in weight and relative dynamic modulus for concrete with and without integral bacteria pellets after 175 freeze-thaw cycles

Based on analysis the SHC specimens exhibited a higher degree of deterioration as compared to the OPC specimens after 175 cycles. The cause was not immediately known but the average degree of mass loss in the SHC specimens indicated that the addition of the self-healing bacteria was a factor. It was theorized that the SHC agent polymer pills may have absorbed water and caused expansion within the concrete matrix leading to accelerated deterioration during the freeze/thaw testing.

A joint meeting with the supplier Basilisk Concrete in September of 2017 resolved that the swelling evident in the laboratory testing trials was due to a chemical reaction when the bacteria metabolized the calcium lactate. Changes to the SHC Agent ingredients had already been made by Basilisk Concrete to address this issue and a new version was available that produced more robust results. It was recommended that the current version of the SHC Agent provided to Reclamation be ground down to powder form as this would improve freeze/thaw testing results.

Further Freeze/Thaw testing of the SHC Agent was not pursued due to budget and time restraints. However, further testing should be considered to determine the full impact of the new version of the SHC Agent when exposed to freeze/thaw effects. During further literature and research paper reviews Reclamation was able to verify that other research studies on freeze/thaw resistance had indicated positive results when using bacteria. Specifically, the study performed by Lund University indicated that there was no significant difference between freeze/thaw deterioration in their control samples and concrete treated with a bacteria agent when tested in de-ionized water [2]. Full results of their findings are provided in the “Other Self-Healing Technologies” section of this report.



Figure 8. SHC specimens after 33 cycles (left), OPC specimens after 33 cycles (right). Surface scaling can be observed in the self-healing concrete specimens

Compressive Strength Testing

Reclamation’s test program next analyzed the compressive strengths of bacteria concrete and conventional concrete. Eleven cylinders were cast and tested: (2 @ 1-day, 2 @ 7-day, 2 @ 28- day, and 2 @ 56-day strength) with results listed in Table 7.

Table 7. Compressive strength for control specimens and SHC Agent specimens

Series ID	Description	1-day (psi)	7-days (psi)	28-days (psi)	56-days (psi)
OPC	Control Specimens	1875	3345	3997	4070
SHC	Self-Healing Agent	1330	3110	3793	3940

Figure 9 reflects the difference in strength development between the conventional concrete control specimens and the SHC Agent specimens. The most notable difference in strength is evident at the 1-day strength testing where the SHC Agent specimens reflect a 30% difference. At 7-day and further out to 56-days the difference between the control specimens and the SHC Agent specimens is far less apparent. It is concluded that although the SHC Agent specimens have a less gradual early strength development it promptly develops strength and maintains close correlation with the control specimens. Therefore, this test program believes that there is no significant impact to compressive strength when using the SHC Agent.

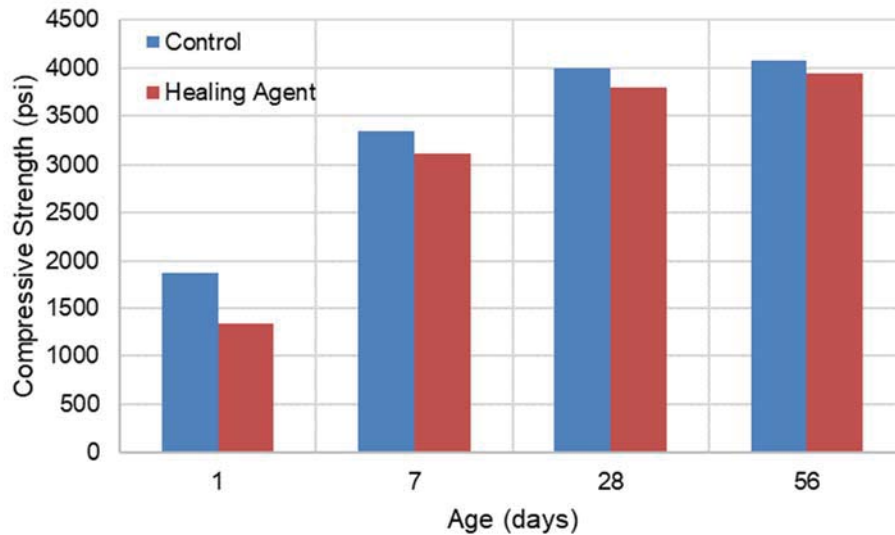


Figure 9. Compressive Strength Development for Control Specimens and SHC Agent Specimens

Restrained Shrinkage Testing

Restrained shrinkage was evaluated in accordance with ASTM C1581. Three specimens were prepared with the same concrete mixture as the freeze-thaw bars. The two mixtures performed similarly, which is expected since they contain the same volume of paste. The average crack width for both mixtures was 0.007-inches, and the average time to cracking was 13 days for the Control and 14.25 days for the Self Healing Concrete. As shown in Figure 10(b), bacteria pellets are exposed on the surface of the sample. From Figure 11, the Control has a maximum strain of $42 \mu\epsilon$ while the SHC sample averaged $50 \mu\epsilon$ before cracking.

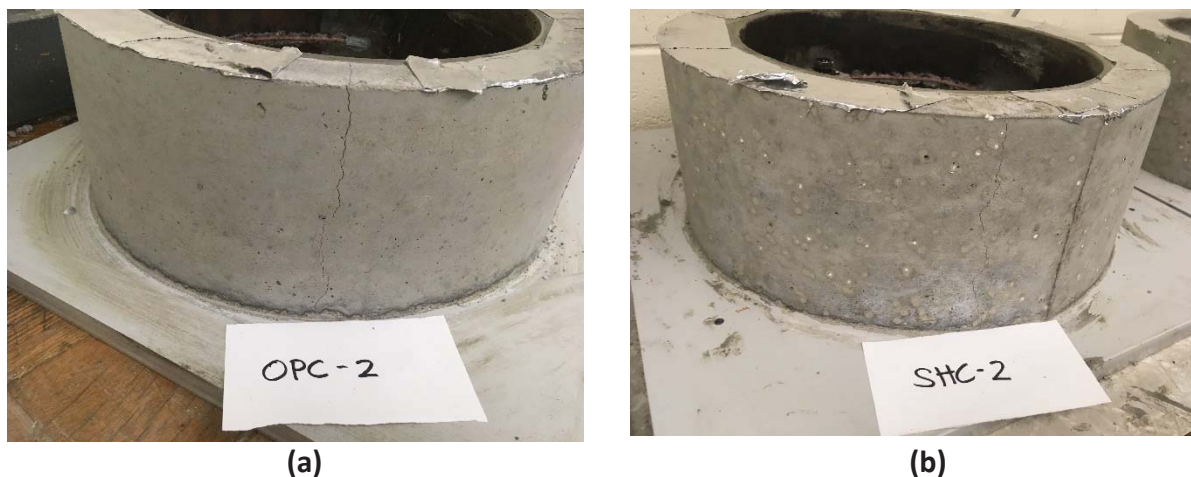


Figure 10. Example of cracked shrinkage specimens of (a) plain concrete (OPC-2) and (b) concrete containing SHC pellets (SHC-2)

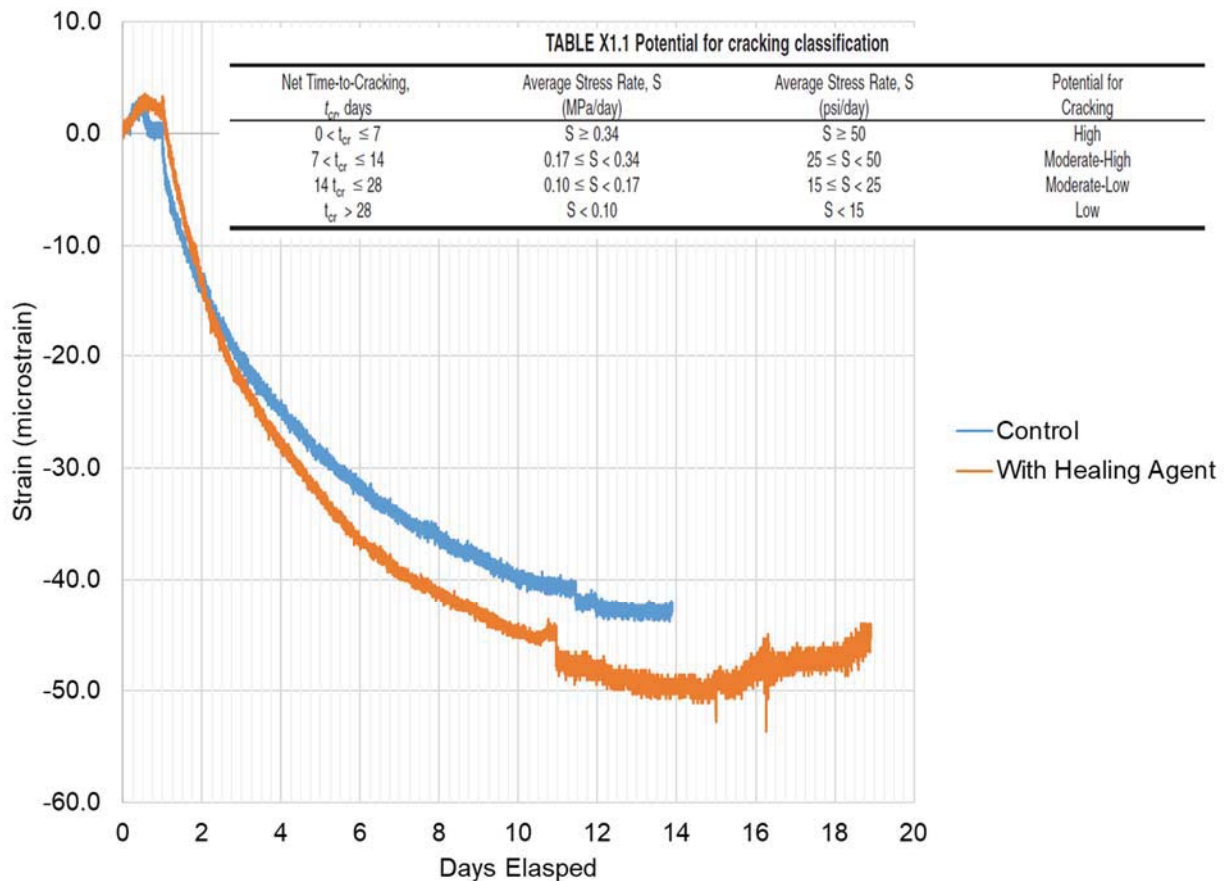


Figure 11. Strain development for concrete with and without Self-Healing Bacteria pellets

Discussion

Reclamation's testing program for Basilisk Concrete's SHC Agent and Liquid Self-Healing Repair System ER7 demonstrated that there is potential for this technology in our infrastructure, however, due to budget and time limitations further testing was not possible using the modifications provided by the supplier to address the deficiencies found during our testing. While Reclamation's intent was to identify an ideal application of the SHC in the field, additional testing will enhance our knowledge of the products and address those concerns discussed in this report.

Testing was able to indicate that there was no difference in compressive strength when comparing the conventional concrete and the SHC Agent specimens. Still, testing reflected notable differences in performance between the conventional specimens and the SHC Agent specimens when addressing freeze/thaw resistance. As the freeze/thaw trials indicated, the SHC Agent specimens experienced significant deterioration due to swelling produced by the activated bacteria. Basilisk Concrete's changed recipe or recommendation to grind down the SHC Agent polymer pills could have resolved this issue and produced more favorable results however further testing was not performed by Reclamation to validate this.

Bond testing also did not produce the theorized results, Reclamation's desire to take advantage of the self-healing nature of the Liquid Self-Healing Repair System ER7 on a mechanically weakened substrate did not produce the strong bond between a substrate and overlay that was hoped for. Still, Reclamation theorized that several factors could have been changed to receive a definitive result. During the trials, two techniques were attempted: application of only Part A to the substrate and application of both Part A and B together. Treatment of the substrate with Part A was executed by allowing the liquid to seep into the concrete, no specific time was chosen before the overlay was cast. For the combination of both Part A and Part B, Part A was applied to the concrete substrate and allowed to seep in, there was no specific time taken by Reclamation before Part B was applied and allowed to seep in before casting the overlay. Future adjustments to the pretreatment of the concrete specimens are recommended to truly assess the healing capacity of both Part A and B. Prior to this trial both Reclamation and Basilisk Concrete were aware that the Liquid Self-Healing Repair System ER7 was not designed for this specific approach and the treatment process would have to be fine-tuned during the testing process. While Reclamation was not able to continue with additional trials it is recommended that future testing completely rule out this type of application. Therefore, further testing is encouraged to determine the potential of the ER7 system to heal micro-fractures on a mechanically weakened substrate and development of a stronger bond to an overlay.

Bacteria pellets can be seen on formed surfaces – this may be remedied with the finely crushed pellets, further investigation may be required if aesthetics are a concern.

Future Outlook

Moving forward, as Reclamation continues to learn more about Self-Healing concrete technologies and work with Basilisk Concrete we will continue to rely on further independent studies to tailor this technologies advantages to our infrastructure. This research study has found that bacterial based Self-Healing concrete has potential for use in different structures, including irrigation canals, parking structures, and retaining wall repairs. Our laboratory tests for the SHC Agent, Liquid Self-Healing Repair System ER7, and Self-Healing Repair Mortar MR3 reflected both positive and areas of improvement for the technology.

When evaluating the SHC Agent, application and mixing of the polymer pills to standard concrete ingredients was straightforward. After calculating the desired volume of polymer pills based on the size of placement (17 lb/yd³) there was no additional handling or precautions required for the product. Reclamation's concern with introducing a bacterial product to our infrastructure was addressed by the supplier through independent research for their MSDS and compliance with European regulations. Discussions were able to verify that the bacteria was food grade and safe to handle as defined by the European Parliament (2000) Directive 2000/54/EC on the protection of workers from risks related to exposure to biological agents at work. No issues were evident during mixing of the polymer pills or adverse effects on the final product. During earlier discussions with the supplier it was pointed out that as the polymer pills degrade within the concrete placement the appearance of white spots may be evident on the exterior of the placement. These spots later faded and were not of concern. As Basilisk Concrete continues to improve on their SHC Agent, including identifying an alternative food

source for the bacteria and improving on the performance of freeze/thaw resistance there is potential for Reclamation to continue to invest in this technology. At the start of this research study the price of the product ranged from \$38/yd³ to \$200/yd³ and this price is expected to decrease and become comparable with the price of conventional concrete. It is expected that the Self-Healing Concrete market will grow to 24% by 2024 [16] and further advances will provide Reclamation with the opportunity to restart trials on the latest versions of Basilisk Concrete's products moving forward to field trials. Successful field trials could then lead to implementing the benefits of this technology to concrete repair projects throughout our infrastructure.

Reclamation's laboratory trials with the Liquid Self-Healing Repair System ER7 produced variable results. During preliminary discussions with the supplier the product was chosen to investigate its self-healing nature on a mechanically weakened substrate after micro-fractures from concrete repair surface preparation were developed. It was theorized that the Liquid Self-Healing Repair System would seep into the micro-fractures and provide self-healing, this in turn would provide a more favorable media for the overlay. Bond testing was performed by casting six bottom slabs of varying configurations as shown in Table 2. Due to the nature of this test the application of the Liquid Self-Healing Repair System (Part A plus Part B) was modified from its intended use and the variable results received do not reflect a true outcome. While the early results indicated that the two solutions of ER7 (Part A plus Part B) together acted as a bond breaker, it is believed that prolonging the period of healing after both solutions were applied prior to casting the overlay may have resulted in a more favorable outcome. Bonding was achieved when using the ER7 (Part A) solution on its own as shown in Table 5. Further testing to validate this theory is needed to fully understand the advantages and limitations of this type of application.

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