# Calcite biomineralisation for the repair of damaged concrete

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## Abstract

UK Civil Nuclear sites contain significant volumes of concrete infrastructure, including both external and internal structures. As a consequence, different concretes are exposed to differing environmental conditions, resulting in variable mechanisms and rates of concrete degradation. For example, external structures may be exposed to salt water and freeze-thaw cycles, while internal structures may be exposed to high temperatures and/or high levels of radiation.

Key to minimising the degradation of concrete structures is the reduction of concrete permeability. High permeability permits the ingress of damaging chemical compounds such as sulfate, and permits carbonation which may damage steel-bearing reinforcements. Consequently, techniques to reduce permeability will improve durability of the concrete. Specifically, very low permeability concrete is highly desirable for radiation shielding structures, which must be impermeable to radioactively contaminated air and liquids.

Microbially-induced calcite precipitation (MICP) may provide a low-cost, low-carbon method for the reduction of permeability in aged or damaged concrete infrastructure. The method used in this study relies upon the ureolytic capacities of the bacterial strain *Sporosarcina pasteurii.* We treat fractured concrete cores in the laboratory and show that our newly developed concrete treatment protocol successfully reduces hydraulic conductivity by at least 2 orders of magnitude in concrete samples collected from UK Civil Nuclear sites. We utilise X-CT imaging to quantify and visualise the calcite deposited within the fracture network present in the concrete samples. Our research indicates this treatment protocol can significantly reduce concrete permeability and thus could be deployed to increase the longevity of degraded concrete nuclear assets.

## Introduction

Concrete makes up a large proportion of infrastructure and assets comprising the built environment. This results in concrete and cement being exposed to a broad range of environmental conditions. Concrete is therefore subject to many forms of deterioration, typically leading to the formation of cracks or fractures; and consequently increased permeability, leading to even further deterioration due to corrosion of reinforcement. Traditional repair methods used depend upon the purpose of the concrete structure and include patching with concrete or bitumen, or the injection of new concrete or other grouting materials into deeper fractures [1], [2]. These methods are expensive in both time, materials, and have associated negative environmental impacts.

The development of low embodied carbon materials and methods for the repair of concrete may contribute to reducing the environmental impact of concrete usage.[3] The use of industrial waste materials to act as nutrient sources for microbial growth may also allow contribution to a sustainable 'circular economy'.[4] Microbially induced calcite precipitation (MICP) relies upon the ureolytic activity of bacteria to trigger biomineralisation. This process occurs via the enzymatic breakdown of urea to produce ammonium and carbonate ions. These carbonate ions will bond to any free calcium ions present in the system, and in a high pH environment the formation of calcium carbonate (calcite) will be promoted.

Most laboratory studies of the treatment of concrete have relied on pouring/dripping treatment solutions onto concrete blocks or immersing blocks fully in treatment solutions.[5]

Neither of those approaches may be practical for in situ application. In comparison, injection enables treatment fluids to applied to non-horizontal surfaces such as walls and roofs, and also allows application into internal fracture networks. Most studies treating natural/created fractures and artificially planar fractures have relied on visual assessment of the repair at the surface.[5] Few studies have investigated strength gains from MICP treatment of damaged concrete. In this study we (1) inject the MICP treatment solutions under controlled conditions, (2) visualise and quantify the deposition of calcite in the core using X-μCT and (3) demonstrate that MICP can effectively regain strength in an initially fragmented concrete core.

## Materials and methods

### Concrete Sample Collection and Preparation

Concrete blocks were provided by Babcock Marine Ltd, taken from concrete caissons/dock blocks used as part of a dry dock structure at Devonport Royal Dockyard facility, within HMNB Devonport. The dimensions of each block were W: 100 cm, L: 181 cm, H: 80 cm, weighing approximately 3.3 tonnes.

From these blocks, a 36 mm diameter by 72 mm length core was cut to produce samples suitable for laboratory scale testing. An unconfined compressive strength (UCS) test was conducted to determine the UCS value of the concrete and also to artificially induce fracturing. Once visible fracturing was observed the core was then split along the predominant fracture into two halves through impact with a chisel.

The core was reassembled, wrapped in heat-shrink tubing and confined at 1000 kPa in a core holder for 1 hour to compress the halves of the core firmly together. After this, the core was vacuum-saturated with tap water and scanned via X-μCT. Finally the core was remounted in the core holder and a confining pressure of 1000 kPa was applied to ensure no by-pass of MICP treatment fluids around the core.

### Bacterial growth and preparation for injection

S. pasteurii was grown from cryopreserved stock cultures in a solid medium consisting of 5.5 $gL^{-1}$ Yeast Extract (Sigma-Aldrich), 5 $gL^{-1}$ sodium chloride (Fisher scientific), 0.4 $gL^{-1}$ D-glucose (Sigma-Aldrich), 0.4 $gL^{-1}$ $K_2HPO_4$ (Sigma-Aldrich), 20 $gL^{-1}$ urea, and 15 $gL^{-1}$ agar (Sigma-aldrich). Urea was added aseptically after autoclaving. A single bacterial colony was then transferred into a liquid growth media consisting of 5.5 $gL^{-1}$ Yeast Extract (Sigma-Aldrich), 5$gL^{-1}$ sodium chloride (Fisher scientific), 0.4 $gL^{-1}$ D-glucose (Sigma-Aldrich), 0.4 $gL^{-1}$ $K_2HPO_4$ (Sigma-Aldrich), and 20 $gL^{-1}$ urea (Sigma-Aldrich). Urea was added aseptically after autoclaving. The culture was incubated overnight at 30 $^oC$. The culture was then centrifuged at 6000 G for 7 minutes. The supernatant was discarded, and the bacterial cell pellet resuspended in mains tap water to an $OD_{600}$ of 1.0. This solution was prepared immediately prior to injection into the core.

### MICP Treatment and Permeability Measurements

A HPLC pump was used to inject water and treatment fluids through the core. Initial absolute permeability (units $m^2$) was determined during injection of tap water by controlling the flow rate at the pump, and measuring the differential pressure across the core. This calculation utilised Darcy's law (formula below) to measure permeability ($k$):

$$q = -\frac{k}{\mu}\nabla p\,.$$

Where $k$ = permeability ($m^2$), $\mu$ = dynamic viscosity of the fluid (Pa.S), $\nabla p$ = pressure drop (Pa), and $q$ = instantaneous flux ($m^3/s$).

Treatment cycles consisted of seven main injection stages through the core, interspersed with water pulses to prevent blockage of the pump and tubing. For each bacterial and cementing solution injection stage, 5 ml of fluid was injected per cycle at a flow rate of 0.1 ml/min. Cementing solution consisted of 111 $gL^{-1}$ calcium chloride (Sigma-Aldrich), and 60 $gL^{-1}$ urea (Sigma-Aldrich).
The order of these injection steps for a single treatment cycle are listed below (Table 1). Permeability measurements were taken with water after each treatment cycle.

Table 1: Treatment Cycle Steps for Cycles 1-6

| Treatment Step | Treatment Solution | Flow Rate (ml/min) | Duration (minutes) | Total Volume (ml) |
|---|---|---|---|---|
| 1 | Bacterial Injection | 0.1 | 50 | 5 |
| 2 | Static Period | N/A | 120 | N/A |
| 3 | Water Injection | 0.1 | 20 | 2 |
| 4 | Cementing Injection | 0.1 | 50 | 5 |
| 5 | Static Period | N/A | 960 (overnight) | N/A |
| 6 | Water Injection | 0.1 | 20 | 2 |

Between treatment cycles, the tubing lines and pump were flushed thoroughly with tap water. This treatment cycle was repeated several times.
After 6 treatment cycles, permeability had decreased significantly. At this point, the flow rate for all injection stages was halved to 0.05 ml/min , with the treatment duration doubled to maintain the same volume of treatment fluid as in Cycles 1-6 (**Table 2**).

Table 2: Treatment Cycle Steps for Cycles 7-9

| Treatment Step | Treatment Solution | Flow Rate (ml/min) | Duration (minutes) | Total Volume (ml) |
|---|---|---|---|---|
| 1 | Bacterial Injection | 0.05 | 100 | 5 |
| 2 | Static Period | N/A | 120 | N/A |
| 3 | Water Injection | 0.05 | 40 | 2 |
| 4 | Cementing Injection | 0.05 | 100 | 5 |
| 5 | Static Period | N/A | 960 (overnight) | N/A |
| 6 | Water Injection | 0.05 | 40 | 2 |

Tomography (X-μCT) Method

X-ray micro computed tomography of the concrete core was carried out using a Nikon XT H 225 LC X-ray computed tomography system. This generated a 2D stack of projections from the scan. The core was scanned once before MICP treatment, and once afterwards. Following reconstruction, pre- and post-treatment stacks were aligned using the registration software Elastix.[6] Thresholding and processing of the stacks was performed using the FIJI distribution of ImageJ [7] which allowed the solid components (concrete: cement matrix or aggregates) to be distinguished from the void or fracture spaces. The data was binarized based on grey values (255/White or 0/Black), with 255 representing concrete/calcite, and 0 representing void/fracture space. By subtracting the pre- and post-treatment binarized stacks, it was possible to visualise where calcite was deposited within the fracture network. It was also

possible to count the number of these voxels, which corresponds to a measurement of deposited calcite volume.

## Results and discussion

Permeability was observed to continually decrease with each treatment cycle completed (Fig 1). After 9 treatment cycles a permeability reduction of 3 orders of magnitude was achieved. After 9 treatment cycles, the core was removed from the core holder and imaged under X-μCT. X-μCT analysis revealed that the initial fracture network within the core had been coated with a new solid phase, (i.e. calcite), see Fig 2. Based on X-μCT data, the measured volume of the precipitated phase was 46.42 $mm^3$.

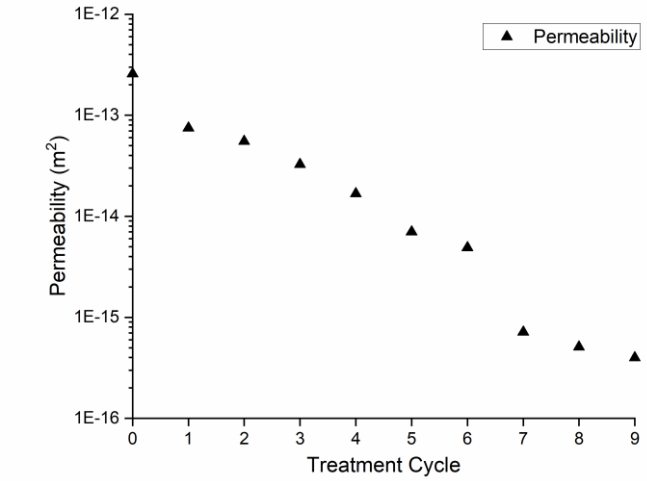


Figure 1: Permeability change vs. Treatment Cycle Number

The initial measured compressive strength of the core was 14.41 MPa. After X-CT imaging another UCS test was conducted, and the new compressive strength measured as 1.58 MPa, indicating that ~10% of the initial strength of the concrete core had been regained via MICP treatment.

## Conclusion

In this study we have demonstrated that MICP treatment via controlled injection can be used to effectively reduce the permeability of damaged concrete, and also results in strength gain in an initially fragmented concrete specimen. Using X-μCT we show that the location of the calcite precipitated maps onto the initial fracture network within the core and that calcite was precipitated along the full length of the core (72 mm length).

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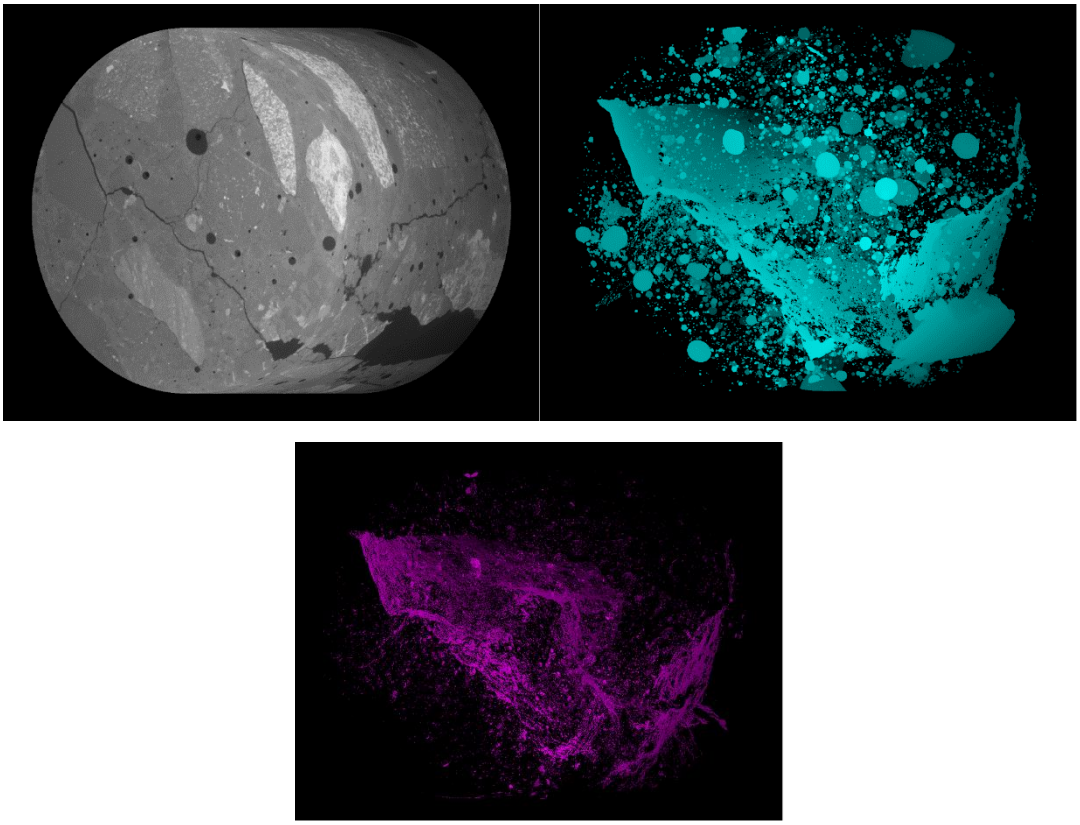

Figure 2: X-CT visualisations of the concrete core, showing front ¾ view relative to the base of core. 3D reconstruction of the scan shows that fractures are present in the concrete, along with voids and aggregate pieces (top left). Image segmentation allowed the initial void and fracture space to be visualised pre-treatment (top right, Cyan). Subtracting the post-treatment void space from the pre-treatment void space allowed direct visualisation of the location of calcite in the fracture network (lower image, Pink).

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