ABSTRACT. The effect of water-borne contaminants on the durability of concrete is well-known and cracked concrete is more susceptible to permeation of these contaminants. Consequently, research is attempting to develop concrete that can self-heal cracks, potentially reducing costs of repair and maintenance work on infrastructure projects dramatically. The research described in this paper was carried out to demonstrate the use of microbiologically-induced calcite-precipitation as a means of autonomic self-healing of concrete in a full-scale site trial. The paper describes microbiology and concrete technology investigations carried out to select an appropriate combination of spores and nutrients, and to devise a method for encapsulating these safely within the concrete. It is demonstrated that for the encapsulation method used and the agents chosen it is possible to produce self-healing concrete with similar early-age and mechanical properties to that of normal concrete. This self-healing concrete was then used in a reinforced concrete wall, and the initial findings are described.

Keywords: Self-healing, Bacteria, Lightweight aggregates, Encapsulation

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INTRODUCTION

The effect of water-borne contaminants on the durability of concrete is well-known and cracked concrete is more susceptible to permeation of these contaminants. Consequently, research is attempting to develop concrete that can self-heal cracks; potentially reducing costs of repair and maintenance work on infrastructure projects dramatically. The Materials for Life (M4L) project, a partnership between Cardiff University, University of Bath and the University of Cambridge is aiming to develop self-healing concretes to reduce the repair and maintenance requirements of concrete structures. The project combines research on a number of multi-scale techniques to develop autonomic self-healing within concrete [1, 2].

One approach to autonomic self-healing is the utilization of microbiologically-induced calcite-precipitation. This approach utilises the metabolic activity of bacteria and biomineral precursors embedded within the material to form an inorganic material, usually calcium carbonate (CaCO₃), in the form of calcite, as the healing compound.

There are two key pathways for delivering the healing process: (i) enzymatic hydrolysis of urea [3] and aerobic metabolic conversion of calcium salts [4]. The aerobic metabolic conversion pathway was used in this research and the healing occurs because the bacteria act as a catalyst for the conversion of an organic calcium salt (precursor), for example calcium acetate, to calcite under favourable conditions: the presence of water, oxygen and nutrients. The by-products of the conversion of calcium acetate to calcite are carbon dioxide and water which are compatible with concrete (Equation 1). Furthermore, a weak carbonic acid may form that will lead to carbonation of calcium hydroxide within the concrete leading to a form of enhanced autogenous healing. Figure 1 shows the healing of a 0.4 mm crack by impregnation of *Bacillus cohnii*, calcium acetate and yeast extract [5].

\[
CaC_{4}H_{6}O_{4} + 4O_{2} \rightarrow CaCO_{3} + 3CO_{2} + 3H_{2}O
\]

Eq.1

Figure 1 Precipitation of calcite in a crack as a result of bacterial conversion of calcium acetate [5]

Consequently it is necessary that any ingredient used does not affect the early-age and mechanical properties of the concrete. Once a crack appears the ingredients must become active and form calcite rapidly within the crack without adversely affecting undamaged concrete and steel reinforcement. This restricts the choice of ingredients that may be used.
Furthermore, it is necessary, in order to ensure survival, that the bacteria are added in the form of spores. Consequently upon cracking it is necessary for the spores to germinate.

Laboratory-based experiments have demonstrated the viability of this method for self-healing of concrete in ideal conditions and in controlled environments. However, the implementation of bacteria-based self-healing concrete on a larger scale has not been attempted in Europe and it present a number of challenges: (i) the selection of sufficiently cheap and available microbial self-healing agents, (ii) a means of encapsulating the self-healing agents within the concrete to ensure they survive and do not adversely affect the concrete production process, and (iii) ensuring that the early-age and mechanical properties of the concrete are not significantly affected by the inclusion of the self-healing agents. Research towards meeting these challenges is discussed in this short paper.

**COMPOSITION OF MEDIUM**

To ensure complete self-healing from germination to healing to sporulation, Sharma et al [6] have suggested that it is necessary to use a complex healing agent consisting of carbon and nitrogen sources, sporulation and germination aids, proteins and buffer solutions. Indeed, in vitro tests by Sharma et al [5] at the University of Bath have shown that spores can be germinated to cells within approximately 3 hours in a complex medium that includes germination aids (isonine and alanine) and the presence of sodium ions to carry them across the spore wall.

However, further initial microbiology tests, by the authors, have demonstrated that Bacillus pseudofirmus spores may germinate and grow adequately in the presence of only yeast extract and that complete germination occurs within 24 hours. Although slower than that with the more complex agent it is appropriate for the application of self-healing concrete.

Tests at the University of Bath have further demonstrated that neither calcium acetate nor yeast extract would affect the setting of the cement or hardening of the concrete provided fewer than 10% of the nutrients were released into the concrete during mixing [7].

For the purposes of further research the composition of the nutrient solution was 300 g/l of calcium acetate and 30 g/l of yeast extract. Both values are close to the maximum solubility of these ingredients in water.

**ENCAPSULATION**

Wiktor and Jonkers [4] have previously demonstrated the capability of encapsulating calcium lactate (80g/l), spores of B. alkalinitrilicus, yeast extract (1 g/l) in expanded lightweight clay aggregates (Liapor). For the purposes of the trial the nutrients were encapsulated in perlite, a lightweight aggregate commonly used in microbiological applications as a plant growth media. The properties of perlite are given in Table 1. Using a different approach to Wiktor and Jonkers [4], the nutrients (calcium acetate and yeast extract) was encapsulated separately from the bacteria spores (B. pseudofirmus) to minimise the potential for germination before a crack is formed.
The perlite were impregnated with the nutrients and bacteria by soaking the perlite in the appropriate volume of solution until all solution was absorbed. The composition of the perlite is shown in Table 2.

<table>
<thead>
<tr>
<th>Table 1 Physical properties of uncoated and coated perlite</th>
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<tbody>
<tr>
<td><strong>UNCOATED PERLITE</strong></td>
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<tr>
<td>Apparent density, kg/m$^3$</td>
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<tr>
<td>Loose bulk density, kg/m$^3$</td>
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<td>water absorption</td>
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<tr>
<th>Table 2 Composition of uncoated perlite, per g of perlite, after “impregnation” with bacterial agents</th>
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<tbody>
<tr>
<td><strong>CALCIUM ACETATE, g</strong></td>
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<tr>
<td>Perlite with nutrients</td>
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<tr>
<td>Perlite with spores</td>
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To verify the suitability of perlite in terms of its ability to prevent the nutrients from being released into the concrete, initial tests were carried out in which safranin, a dye commonly used to stain microbial cells, was added to the perlite. These stained perlite were then added to mortar. On inspection of cut faces from the hardened faces it was clear that there was substantial leakage of the dye from the perlite. Further trials considered a number of coatings that could be used to prevent leakage of the dye. It was found that a double layer of protection: consisting of a layer of sodium silicate and a layer of Portland fly ash cement prevented leakage of the dye. The sodium silicate coating was applied by soaking the impregnated perlite in sodium silicate solution until the perlite was completely wet. The perlite was then dried at 20°C for 24 hours. A second layer of sodium silicate was then applied to the perlite, as above, followed by the application of dry cement to the wet sodium silicate surface. The perlite was then cured in water for 48 hours. The properties of the coated perlite (in the absence of bacteria and nutrients) are given in Table 1. Based on comparison of the density of the coated and uncoated perlite it was estimated that the mass of the coating was approximately 70% of the overall mass of the coated perlite.

Tests were also carried out to ensure that the viability of spores (ability to germinate) was retained after impregnation in the perlite. Experiments were conducted under sterile/aseptic conditions by crushing perlite impregnated with *B. pseudofirmus* spores at 0, 3, 15 and 30 days. 1g of each sample was obtained at each time period and serially diluted (10-1-10-9) in test tubes. These were then vortexed for two minutes to provide homogeneity. The viability of spores in terms of colony forming units (CFU) was then determined. The results indicated that whilst the number of viable spores may have decreased steadily over a period of 30 days, from approximately $10 \times 10^9$ to $2 \times 10^9$ (Figure 2), this reduction in viable spores was only around 0.01% of the initial number.
Preliminary Laboratory Concretes

Two preliminary concrete mixes were prepared in the laboratories to assess the effect of the coated perlite (containing nutrients) on the properties of the concrete. It was important to ensure that the nutrients were well encapsulated so that they did not affect the early-age or mechanical properties of the concrete, and that the addition of a lightweight aggregate did not adversely influence the compressive strength of the concrete. The mix proportions for both preliminary concretes are provided in Table 3.

The mixes were proportioned to achieve a cube strength of 40 MPa at 28 days. A w/c ratio of 0.40 was used to account for the use of the weak perlite aggregates. The sand (0/4) content of normally designed concrete mixes (without self-healing agents) was reduced to permit the use of perlite as a replacement, despite the fact that the perlite was coated with some unhydrated cement. Superplasticizer was used to obtain a target slump of 100 to 150mm. In terms of the addition of self-healing agents, Mix M1 contained 1.9% calcium acetate by mass of cement and 0.05% yeast extract by mass of cement. M2 contained 3.8% calcium acetate by mass of cement and 0.1% yeast extract by mass of cement. No spores were added to the perlite for these preliminary trials as the intention was to assess the effects on early-age properties and not to assess self-healing. Coated perlite containing spores were used in the full-scale trial described later.

These mixes both gave the necessary degree of consistence and from visual inspection of the fresh concrete there was no damage of the perlite coating. Perlite is a distinct white colour and is clearly visible in concrete when used without a coating. Both concretes set and hardened normally and could be demoulded at one day. The mean seven day strengths were 33.4 MPa and 27.4 MPa for concretes M1 and M2, respectively. The 28-day water-saturated density of M1 and M2 was 2253 kg/m³ and 2240 kg/m³, respectively.

Figure 2 Viability of spores after impregnation in perlite
Table 3 Mix designs for preliminary laboratory concretes

<table>
<thead>
<tr>
<th>MIX</th>
<th>NUTRIENT CONTENT (% by mass of cement)</th>
<th>CONSTITUENTS, kg/m³</th>
<th>0/4</th>
<th>4/10</th>
<th>Coated perlite (with nutrients)</th>
<th>Coated perlite (for spores)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td>2.6</td>
<td>185</td>
<td>455</td>
<td>655</td>
<td>930</td>
<td>70</td>
</tr>
<tr>
<td>M2</td>
<td>5.3</td>
<td>175</td>
<td>440</td>
<td>505</td>
<td>895</td>
<td>140</td>
</tr>
</tbody>
</table>

Superplasticizer (Glenium® ACE 456) used at a dosage of 1.2 litres per 100 kg of cement in both mixes

After cracking, visual inspection showed uniform distribution of the perlite and it was clear that cracking of the concrete resulted in splitting of the perlite (Figure 3) – thus in practice cracking would lead to release of the self-healing agents. This was anticipated and desired as the perlite aggregates were expected to have a lower strength than the paste or conventional aggregates, ensuring cracking would allow access to the self-healing agents.

Figure 3 Broken cube showing good distribution of perlite (the white spots) in concrete M2

FULL-SCALE TRIAL

Overview

For the full-scale trial, five concrete panels were cast on the same day as part of a series of work with Cardiff University and the University of Cambridge under M4L (Figure 4). There was one control panel with no self-healing system, and the other four had different forms of self-healing systems installed, one of which contained the bacterial self-healing mix M2 described above. The trial was carried out at the A465 Heads of the Valleys section 2 project; a £200M contract to upgrade an 8.1km section of the A465 road between Gilwern to Brynmawr in South Wales, UK, from single to dual carriageway. Costain Group Plc was the lead contractor for the project. The project office compound was used for construction of the trial elements.
The panels were designed to crack at 500 mm above the base slab upon loading by including 16 mm diameter starter bars on the front face up to this point, before changing to an A393 mesh (10 mm diameter bars) to create a weak section in the panel. In addition a capillary network created by a 2D network of 4 mm diameter channels through which healing agents could be pumped under pressure was embedded in the concrete to permit later addition of further nutrients, bacteria or oxygen as necessary (Figure 5). To enable the healing agents to migrate to areas of damage the network was designed for and placed in the zone most susceptible to cracking. The network was created using polypropylene tubes which were removed from the concrete once it had hardened. The network channels were joined using 3D printed joints made from polylactic acid [8].

Figure 4 The five panels cast. The bacteria-based self-healing concrete panel is the third from the left.

Figure 5 Embedded capillary network installed prior to casting of the concrete
Because of the size of the panel, bacteria-based self-healing concrete was only added to the panel at the weak section where cracking would occur (Figure 6). As previously stated the concrete mix used was M2 as given in Table 3. The only difference was that spores were also used. Coated perlite impregnated with spores were used at a content of 45 kg/m$^3$. This equated to approximately $4 \times 10^{13}$ spores per m$^3$ of concrete. The other parts of the panel were made of concrete supplied by a ready-mixed concrete producer of design concrete strength C40/50.

![Figure 6 Panel containing bacteria-based self-healing concrete (all dimensions in mm)](image)

The self-healing concrete was made in a 90 litre tilting drum mixer. Due to the type of mixer used it was only possible to add ingredients whilst the drum was revolving. This meant that the constituents were “dry” mixed for longer than anticipated, and much longer than in the preliminary trial.

To monitor crack healing, the panel was cracked at 36 days after casting by applying load through a threaded bar running through the centre of the width of each panel to a reaction wall designed to act against the force produced by the jack. The bars were placed 1.5m above the base slab into each panel and the reaction wall. The procedure is described in full in [8]. Throughout the site trial the crack width, deflections, strains, permeability and applied loading on the panels were all monitored. This was achieved using a combination of DEMEC pips, optical microscopes, linear variable displacement transducers, load cells, on-site permeability apparatus and a digital image correlation system [8].
Results

The panel was demoulded at two days (Figure 5), which was consistent with the other four panels cast. Although the extended period of mixing had led to damage of the coated perlite through attrition with the coarse aggregates—the perlite being clearly visible due to its white colour in the fresh concrete, there was no noticeable delay in setting or early-age hardening. This suggests that even though the perlite lost its coating during mixing there was insignificant release of nutrients to have an effect on cement hydration. Cubes tested at 7 and 28 days gave mean strengths of 29.1 and 35.1 MPa, respectively. Whilst this was weaker than anticipated it can be perhaps explained by some difficulties with compacting the concrete cubes on site.

The panel was cracked at 36 days and the crack ran through the centre of the self-healing concrete section as designed. Figure 7 shows the initial cracks that occurred upon loading and the residual cracks that remained after unloading. These residual cracks were approximately 0.1 mm in width.

At the time of writing, optical microscope images show a degree of crack healing. However further investigation is required to establish whether this observed crack-healing can be attributed to the incorporation of bacteria, or whether autogenous healing has occurred.

CONCLUSIONS

The following conclusions can be made from the work presented.

1. A combination of bacteria-based self-healing agents have been developed consisting of ingredients that have limited effect on setting and hardening of concrete, but which permit rapid germination and growth of bacterial spores.

2. A coating has been developed that prevents the release of self-healing agents into the concrete prior to cracking.

3. Self-healing concretes cast at full-scale have successfully demonstrated that mechanical properties can be maintained and that setting and hardening is unaffected by the addition of encapsulated self-healing agents.
ACKNOWLEDGEMENTS

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