Biomimetic Vascular Networks in Concrete for Self-Healing Applications
IIB Project 2015/2016

Technical Abstract

Concrete is the most widely used construction material in the world. However, as concerns for the environment and a drive to provide a more sustainable infrastructure intensifies, questions surrounding the environmental cost of concrete are becoming a focus of policy makers, environmentalists and businesses in this sector. The manufacture of cement, the key constituent of concrete, accounts for up to 7% of the world's CO_2 emissions. Therefore, any method which can increase the lifespan and reduce the carbon footprint of our buildings will have a strong, positive, global effect.

Concrete is a quasi-brittle material and as such, although strong in compression, it is susceptible to cracking when loaded in tension. These cracks are an intrinsic feature of reinforced concrete, with their size constrained by steel reinforcement bars. These cracks can become extremely problematic if they are allowed to propagate. The reason for this is because water can pass up these cracks and cause corrosion of the steel, thus compromising the durability of the overall structure. The annual cost of repairing concrete structures affected by corrosion amounts to 775 million GBP.

A solution to this ever prevalent issue is the integration of self-healing concrete into the design of such structures which is a solution explored in this report. Inspiration has been taken from nature and a biomimetic vascular network has been incorporated into concrete samples as a vehicle for this self-healing. The core idea is that this network, embedded in the concrete, will deliver a healing agent through the material's internal structure, directly to the point of a crack. This is in order that the crack can be sealed against the ingress of aggressive substances.

A thorough literature review is laid out, evaluating recent developments in the field of self-healing concrete, with emphasis on tubular encapsulation. A review of how concrete is waterproofed on an industrial level has also been presented. It was found that the use of interconnected vascular networks for self-healing purposes in cementitious materials have not been widely explored. The work of previous 4th year students which has addressed this topic previously was outlined. It was found that it was possible to manufacture a network with a 3D printer. Initially curved networks were made from PLA but eventually these networks were simplified in shape for ease of construction and printed in the more brittle ABS as it cracked more reliably. The first choice of healing agent was cyanoacrylate-based adhesive. However this blocked the tubes when injected and did not allow for repeated cycles. A colloidal silica healing agent was also used but this set up was only able to achieve a maximum load recovery

of 31% and stiffness recovery of 47%. It was found that the healed sample absorbed 250% less water than the cracked, unhealed, control sample. Therefore it was decided to investigate in this project the ability for a vascular network to prevent water ingress at a crack. The property that would be compared between samples was the sorptivity coefficient.

A network without the need for plastic tubes was conceived. This was to allow for reliable cracking of the samples and network at the same time, which had been hard to control in previous tests. In some of the first designs polyolefin heat shrink was used as formwork for the network. However it was not rigid enough to provide a viable option for more complex tube shapes. Instead electrical cable was used to create multi-tube networks. These networks were cast in small reinforced prismatic concrete samples. When tested in three-point bending the networks were found to crack reliably in the same location as the surrounding concrete. A waterproofing agent was then injected into the samples down the network which was released into the cracks, providing the concept of vascular healing in concrete using hollow networks.

Four different waterproofing agents were used within the system. They all decreased the amount of water absorbed into the concrete to 9-40% of the control samples. Two of these were designed to deposit a film on the concrete surface as a barrier against water. Of these, the sample treated with tung oil provided the most effective barrier. The reason for this is thought to be because it was the more viscous of the two so left a thicker coating on the crack face. The other type of waterproofing was a penetrating sealant. These were siloxane-based however the agent with nano-sized particles provided the best reduction in sorptivity out of all four agents. As a penetrating agent the small size of the particles is a significant advantage as it means that it is able to pervade deeper into the concrete, creating a thicker, and therefore more effective, boundary to water ingress. It was shown that repeated coatings had little effect on the reduction in sorptivity coefficient measured, but it was not established if this was because one coat was sufficient or because not enough coats were applied to try and develop a trend. Finally the effectiveness of single and double network tubes were compared. For the high viscosity, film-forming agent the smaller-diameter double tube system meant that there was not a good flow down the tubes so the single tube network presented the best reduction in sorptivity coefficient. For the lower viscosity, penetrating agent the opposite was true as the double tube allowed fuller access to the crack face area.

Vascular networks have shown themselves to be an efficient and quick way, in a situation when time can be of the essence, of blocking the ingress of water into a structure after cracking. The use of hollow networks instead of polymer tubes has proved to be a more reliable way of ensuring that the network cracks with the concrete. For the small samples the methods employed here are adequate however for manufacture to be scaled up other approaches should be used, e.g. 3D printed biodegradable-polymer tubes.

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1 Introduction

1.1 Motivation

Concrete has been used as a building material for millenia. The development of concrete as a building material was pioneered by the Romans, who built impressive concrete structures such as the Pantheon made from a mortar made up of a volcanic, pozzolan ash and water [1]. Throughout the centuries concrete technology has further developed and the invention of Portland cement in the 19th century meant that concrete became a mainstream construction material for a wide array of buildings and structures [2].

Whilst being arguably the most versatile construction material used today, the use of concrete does not come without its disadvantages. Concrete is particularly susceptible to deterioration mechanisms. One of the main reasons for this is its propensity for cracking. Concrete cracks when loaded, and due to shrinkage, expansion and creep [3].

A structure that has cracks needs to be repaired because, as well as adding to a loss in strength due to a reduction in section, they also provide sites for water ingress which leads to corrosion. This is the chemical phenomenon where steel rebar within reinforced concrete structures degrades if moisture and oxygen are present. This leads to further cracking and spalling of the concrete covering, therefore reducing overall strength and compromising the integrity of the structure. A primary reason that moisture and oxygen reaches the rebar, which should have sufficient concrete cover as specified by building standard codes, is due to the cracking of the concrete, leading to an ingress of water. It is therefore very clear that there is a strong motivation to find a solution for this deterioration problem.

1.1.1 Economic and Environmental Considerations

Cracking, and ultimately corrosion, is the main cause of damage to reinforced and pre-stressed concrete structures. The National Association of Corrosion Engineers (NACE) conducted a study for the U.S. in 1998 and concluded that the ageing infrastructure is one of the most pertinent issues that society faces today. It predicted that 15% of the 583,000 highway bridges in the U.S. are structurally deficient due to corroded steel and steel reinforcement. The direct costs due to this problem are estimated to be 8.3 billion USD, with indirect costs due to traffic delays caused from repairs and replacements at least 10 times these costs [4]. In the UK the Department of Transport predicted that the annual repair costs for concrete structures affected by corrosion are 775 million GBP [5]. Furthermore, moisture-related failures often occur within the façades of high-rise buildings within 5 yeas o constuction [6]. In fact, the cost of repair due to these moisture-related problems in the façades was found to be 55.6% of the total construction cost [7].

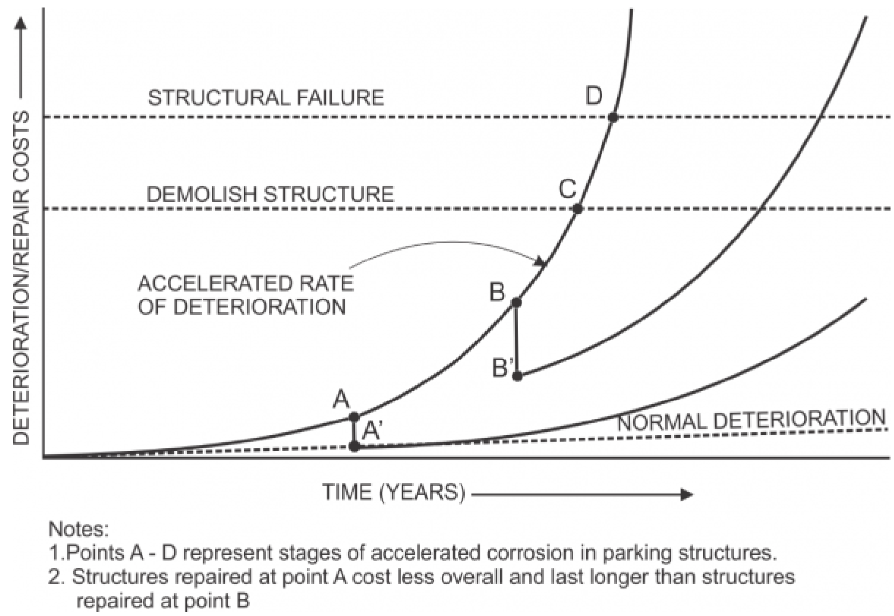


Figure 1: Parking Structure Deterioration Curve (Taken from [8])

Figure 1 shows the impact of deterioration on repair costs and the service life of a concrete structure. An accelerated rate of deterioration would be mostly due to the corrosion mechanisms previously mentioned. Therefore it can be seen that the life span of a concrete structure which experiences corrosion, whether or not repairs are carried out, is exponentially less than a structure which experiences no extraordinary deterioration. As such, if the lifespan of a structure is sufficiently long then there would be significant advantages in trying to incorporate a way of reducing any deterioration into the design in order to reduce this rate.

Alongside the financial costs it is also relevant, from a sustainability angle, to consider the environmental costs of rapid deterioration of a concrete structure.

Structures with long life spans present an opportunity for the infrastructure to become more environmentally friendly. A study by DEFRA in 2005 found that the construction industry accounted for up to half of the U.K.'s total CO_2 emissions [9]. Globally, the production of Portland Cement accounts for 5-7% of all total CO_2 emissions [10]. This figure is set to rise in the future as construction and development in rapidly developing countries continues to boom.

The cost of demolishing a building and rebuilding when measured in embodied energy is ten times greater than the value if the same building was maintained, repaired and reused. That equates to the use of around 1 million gallons of petrol versus the use of 100,000 gallons for a 50,000 square foot building [11]. These are clearly very large savings that can be made if the effort is made to preserve buildings throughout their life span and ensure that the need for the construction of new buildings is eliminated.

1.2 Aims and Objectives

This project aims to build on current research in investigating the use of a biomimetic vascular network (i.e. a three-dimensional hollow tubular array which provides internal access for a healing agent to a damage site) for self-healing, more specifically waterproofing, in concrete. The objectives of the project are:

- To review the current research and literature related to waterproofing concrete and the use of vascular networks for delivering a healing agent. This will identify the areas for improvement and the fields for further study.
- To conceive of and design a vascular network suitable for applying waterproofing agent to a damage site.
- To investigate the quality of waterproofing achieved, draw conclusions and identify the areas for improvement and further research.

1.3 Report Outline

This report is divided into five sections. Section 1 introduces the motivation and outlines the aims for this project. Section 2 reviews the literature and the studies which are pertinent to this project. It identifies the areas for improvement and how the current research will be taken forward. Section 3 describes the experimental method that is used within the project, detailing the materials, equipment and methodology used, thus linking the processes with the overall project aims. Section 4 lays out the results from the experiments and examines the consequences of the findings. Finally, Section 5 summarises conclusions derived from the project and suggests possible future research that could be undertaken. At the end of the report references are listed and supplementary information is appended.

2 Literature Review

This section presents a summary of the academic literature which is relevant to this project. There is a focus on results previously recorded and the opportunities for further exploration

2.1 Definition for Self-Healing Materials

Throughout recent work into self-healing many attempts have been made to consistently define terminology and create definitions to be used in the field of self-healing materials. RILEM (The International Union of Laboratories and Experts in Construction Materials, Systems and Structures) concisely defines self-healing as *any process by the material itself involving the recovery and hence improvement of a performance after an earlier action that had reduced the performance of a material* [12].

Clearly from this definition it can be seen that the principles invoked in designing self-healing materials are very different from in traditional material design. The former aims to manage the damage as it occurs but the latter aims to prevent any damage happening in the first instance [13]. The process of waterproofing a structure after cracking occurs falls into this RILEM defined definition. This is because the process recovers the natural waterproofing ability that a concrete structure possesses which was compromised by the existence of a surface crack.

2.1.1 Desired Attributes for Self-healing Materials

Li and Yang (2007) outlined a framework of the key attributes for ideal self-healing materials against which robust self-healing can be compared [14]:

1. **Pervasiveness**: The healing mechanisms should be ready for activation at any time;
2. **Stability**: The healing mechanism should remain active throughout the whole life span of the structure;
3. **Economics**: The solution should be financially viable;
4. **Reliability**: The healing mechanism must be effective in a wide range of environments and should have a very low failure rate;
5. **Quality**: The mechanical properties of structure should return to pre-damage levels or better;
6. **Repeatability**: It should be possible to heal multiple damage events.

2.1.2 Autogenous and Autonomic Self-healing

RILEM goes on to further categorise self-healing into two distinct types [12]. *Autogenic* healing is the process of self-healing in which the components used in the process are already present in the material (without specific design for self-healing). An example of this is when the rehydration of concrete in water can reinstate the hydration process with small pockets of unhydrated mix [15]. Conversely *autonomic* self-healing uses components that are engineering additions, and would not be normally found in the material. Concrete has the ability to autogenically self-heal, however autonomic self-healing has been the focus of a lot of work in recent years with many methods developed. The proposed methods can either be active or passive: active release modes require human intervention in the healing process; passive release modes require no human involvement in their execution. The advantage of the passive mode is that the need for labour-intensive human inspection, repair and maintenance is eliminated, however the active method inspires greater confidence in the end user as they have a higher level of control.

2.2 Self-healing in Cementitious Materials

Van-Tittelboom studied self-healing in polymers and separated the methods for cementitious self-healing into three categories [16]:

- **Intrinsic, or autogenic healing**: The chemical composition of the concrete is optimised or additional materials are added to exploit the natural self-healing mechanisms;
- **Capsule-based healing**: Microcapsules containing a healing agent are added to the mix which are designed to rupture as a crack propagates through the structure;
- **Vascular healing**: A network of hollow tubes inside the structure are linked to a reservoir of healing agent which is designed to be released when the network is broken by a crack.

The disadvantages of intrinsic healing include the fact that the crack size must be heavily restricted in order that reliable self-healing can take place, and the fact that it has a low reliability and a high sensitivity to external environmental factors. However, it is a low cost option for self-healing. Capsule-based healing and vascular systems aim to address these issues. Capsules, once ruptured and used, cannot be replenished. Often it is this original site of failure that will be the most stressed part of the structure, so it is likely to experience multiple failures - this highlights a serious disadvantage. However, vascular networks can be replenished with a healing agent and the network is much more likely to rupture in case of a crack when compared to a microcapsule. Serious care must be made when deciding how a network such as this will be fabricated, its scale and and other practical issues such as how to install it into a concrete structure.

2.3 Biomimetic Vascular Networks

Figure 2: SEM image of vascular network in adipose tissue (Taken from [17])

Biomimetics is a field of study in which biological processes found in nature are applied to the development of materials, synthetic systems and machines in the field of science and engineering [18]. The vascular network, or vascular system, is a series of vessels that deliver nutrients and oxygen to, and remove metabolic by-products from, the organs in the body [19], i.e. they are comprised of hollow tubes which carry fluids through a material matrix. The complex shape of the network (see Figure 2) means that every part of the body is serviced by it.

It is interesting at this stage to highlight the differences between the different vascular networks found in nature. For example, while for both plants and animals the network is made from tubes going in two different directions which are designed to move nutrients and water to all parts of the organism, there are striking contrasts. In animals the circulatory systems are pump operated by the heart. However in plants they are operated by gravity or water tension. Furthermore, plants only have water solutions flowing through the tubes whereas animals have cells moving through the networks also [20].

One of the objectives for the network design in this project is to be able to provide maximum coverage of the concrete. This is something that is seen in the architecture of the respiratory system, a vascular network, in the human body. This is a network that starts at the trachea and after 23 bifurcations reaches the alveolar sacs. It is a very viable method for optimum oxygen access to the tissue and also the subsequent carbon dioxide removal. This architecture could be replicated in a concrete structure for the delivery of a waterproofing agent.

It should be noted that care should be taken when implicating nature within new scientific functions. The reason for this is due to the fact that often biological systems are optimised for more than one function. The vascular system is a source of nutrients, hormones, and waste products, to name but a few materials transported. Othe functions include maintaining homeostasis and fighting disease and infection in the body. This means that the functionality of a vascular system is not solely driven by the need for effective and efficient distribution, but instead is designed to be a vessel for the many thousands of processes that the body needs to survive, and to have sufficient redundancy to allow survival from injury or illness. Therefore, when considering a biomimetic solution to an engineering problem, it would not be sensible to directly copy what is seen in nature as this may include unwanted complexities and functionalities [21].

2.4 Use of Biomimetic Networks in Concrete

2.4.1 Tubular Encapsulation of a Healing Agent

The use of biomimetic networks has largely been used up to this point as a vehicle for self-healing agents that are used to restore the strength to a failed sample. Proof that this is a viable method of delivery within this field means that this kind of network could be also used to deliver other agents directly to the site of failure.

The first work with embedded networks in concrete was carried out by Carolyn Dry at the beginning of the 1990s [22]. Dry encapsulated an air-curing cyanoacrylate adhesive in an array of 16 brittle, hollow glass fibres which were cast into square prismatic beams. The samples were loaded in three-point bending to the point where the glass fibres were heard to have broken and then the samples were left to heal for 14 days. Upon further retesting the loaded samples were found to be able to carry more load on the second bend test, with an average increase of 15%, whereas the control samples did not [23].

In further research, Dry experimented with hollow networks of tunnels coated with a brittle sealer [24]. The tunnels each contained one part of a three-part methyl-methacrylate adhesive. Upon the tubes' cracking the three parts met and formed a solid plexiglass-like matrix within 24 hours curing time. This bond restored the full strength of the sample and increased the flexibility of the concrete beam. Difficulties with this method of encapsulation include the need for a removable former around which the concrete is cast to make the hollow internal network.

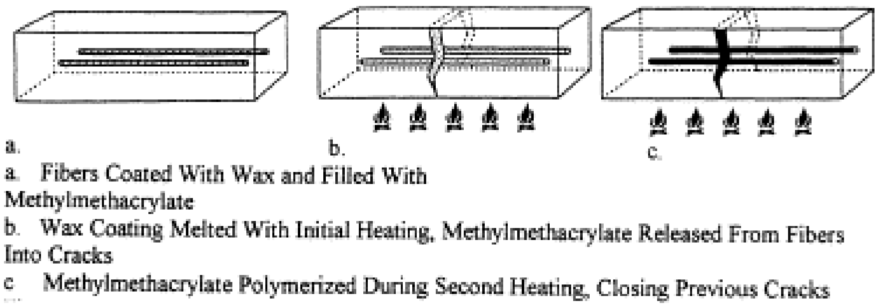


Figure 3: Design for timed release of polymerizable chemicals to repair cracks and fill pores by melting of the coating on porous fibers. (Taken from [25])

In 2000, Dry investigated designs for the internal release of sealants, adhesives and waterproofing chemicals to reduce the permeability of concrete. The active mode design involves the fast release of a liquid methylmethacrylate from inside wax-coated, hollow, porous-walled polypropylene fibres into concrete to fill the voids. This is shown diagrammatically in Figure 3. The wax-coating is melted when a low heat is applied to the surface of the concrete which triggers the chemicals' release. The heat is then raised, polymerizing the methylmethacrylate in the cement pores. The results showed that the samples which had the chemicals released internally had a lower permeability than those which had the methylmethacylate applied on the surface and this is shown in Figure 4.

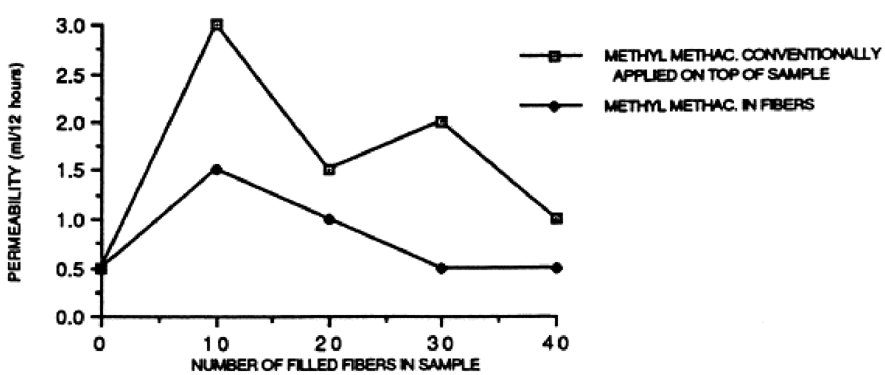


Figure 4: Design for timed release of polymerizable chemicals to repair cracks and fill pores by melting of the coating on porous fibers. (Taken from [25])

2.4.2 Networks Connected to the External Environment

Work by Joseph et al. (2010) revealed that the encapsulation of a healing agent in such a way as Dry had done meant that the release of the healing agent was hindered. This was due to the negative pressure forces that build up at the end of the sealed tubes as the healing agent was released [26]. To this end, he revised the experimental set up to incorporate four hollow capillary tubes, open to the atmosphere at either end, filled with wicking grade, 5 sPs cyanoacrylate adhesive healing agent as shown in Figure 5.

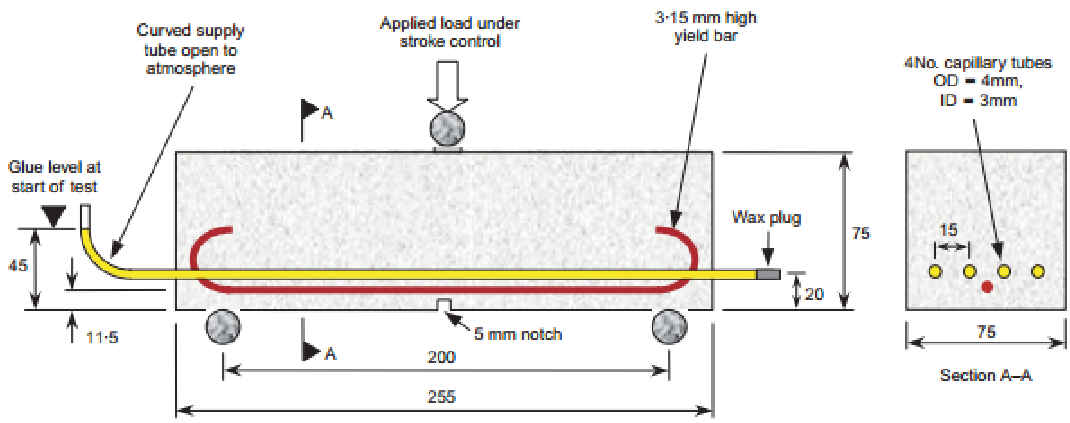


Figure 5: Revised experimental set up by Joseph et al. featuring tubes exposed to external environment (Taken from [26])

Results from this experiment indicated that the initial crack had been credibly healed because upon reloading cracks appeared in other locations instead.

The main advantage of having the tube connected to the external environment is that the healing agent can be delivered multiple times to the structure, which is not the case when the tubes are entirely encapsulated. Further work was done on providing an external, elevated 'repairing agent tank' where the head difference meant that the agent flowed around the embedded network [27]. Dry herself did further experimentation and also concluded that it was better to provide an external reservoir, where the healing agent is released after the crack forms [28].

2.5 Current Implemented Approaches to Waterproofing Concrete

2.5.1 Development of Waterproof Concrete

The technology in use today for waterproofing concrete structures includes waterproof membranes, surface coatings, and waterproofing agents (i.e. polymers, pore blockers, silicones, and pore liner penetrants, amongst others). Each of these methods comes with its own advantages and disadvantages. Methods of application include integral mixing and as an external membrane however the method that will be focused on in this project is the application as an external coating.

External Coating

An external coating is applied on the surface of a substrate either by dipping, spraying or brushing. It prevents water penetration by blocking the external pores. A coating is normally applied as a repair at some stage during the working life of a structure as a method of improving its life span. These coatings are made up from polymer, silane or siloxane-based additives.

It has been shown that the use of silicate containing compounds can improve the water resistance of a structure on application [29]. Furthermore, silanes and siloxanes produce a similar water resisting effect when exposed to cyclic wetting and drying conditions [30]. Heritage work with historic masonry has indicated that mortars treated with siloxanes have an improved resistance to water ingress via the capillaries. However, the water vapour permeability was not hugely influenced by this treatment [31]. Polyurethane and epoxy resins were shown to perform well in resisting water absorption and chloride permeability [32].

There have been further studies into the differences between the performances of organic and water-based solvent coatings. These found the organic versions to be the more effective as a waterproofing agent [33]. However, Nolan et al. (1995) carried out research that revealed that silane water repellent was the more effective in reducing water absorption in the concrete found near the surface of a sample [34]. The ultimate thickness of the coating is another factor that affects its efficacy [35].

Recent developments in the external coating market incorporate the use of nanotechnology, however this technology is still at the formative stage. However, there has been research to show that a silane-clay-nano composite reduced the moisture of absorption when compared to a silane-only coating [36].

2.6 Previous Work by 4th Year Project Students

Studies into self-healing concrete have been a focus of CUED for a number of years and since 2013 4th year project students have been making progress with the use of biomimetic vascular networks for self-healing purposes in concrete [37] - [38]. Most of the students' work centred around the use of 3D printed networks, casting these in concrete, cracking the samples and delivering adhesives to the crack sites in order to recover the strength of the small concrete sections.

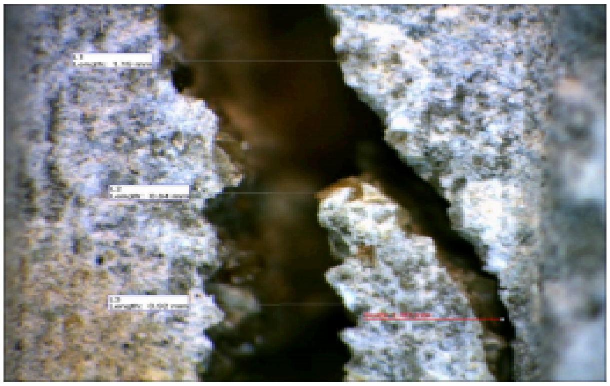
Initially interconnected networks were printed in PLA (Polylactic acid) and the healing agents used were cyanoacrylate-based adhesives. The progression of the network design is show in Figure 6. Two different viscosity agents were used. After a curing time of 48 hours, strength recovery was found to be 72% for the low viscosity samples and 65% for the high viscosity. The stiffness recovery was 74-100% for the low viscosity samples and 69-77% for the high viscosity samples [37]. It was decided that cyanoacrylate was not an ideal healing agent as it solidifies when air reaches the cracked network meaning that only one healing cycle was possible in the experimental configuration used in this project.

(a) (b) (c)

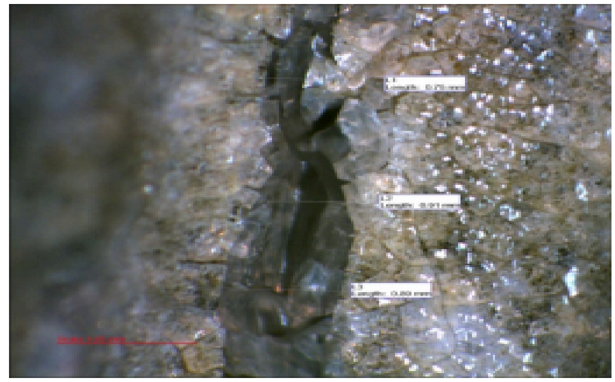
Figure 6: Different concepts for vascular networks (a) The model design in Cubify® Invent™(b) Output from 3D printer in PLA (c) Individual network with supports cut away demonstrating flow with no leaks or blockages (taken from [37])

In further experiments the PLA was replaced with the more brittle Acrylonitrile Butadine styrene (ABS). A colloidal silica healing agent was circulated through the networks due to the safety concerns surrounding the use of a glue in a pressurised system. It was found that a greater coverage of the crack face by the healing agent was possible in the specimens with more delivery tubes. An increased crack face coverage lead to a greater recovery of the specimen properties. A maximum load recovery of 31% and stiffness recovery of 47% was found for this system with this healing agent. However the cracks were observed to be 96% obscured after 28 days of healing and fully obscured after 28 days of healing (see Figure

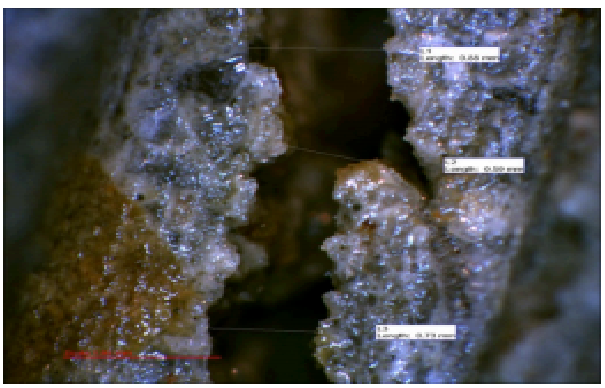
7). Furthermore, the healed specimens had a sorptivity coefficient of 159% of an uncracked control whilst the sorptivity of the cracked control was 401% of the uncracked equivalent [38].



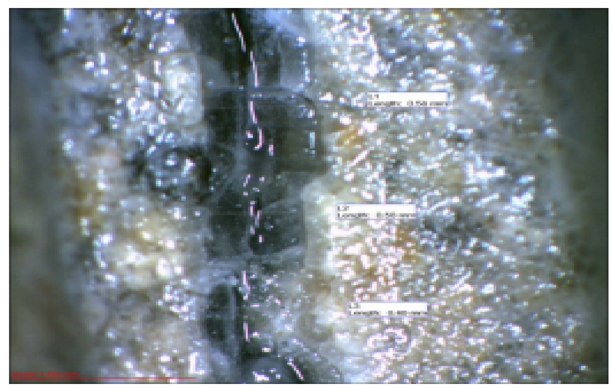
(a) Control sample (10 days)



(b) Healed sample (10 days)



(c) Control sample (28 days)



(d) Healed sample (28 days)

Figure 7: Progression of crack healing (taken from [38])

2.7 Proposed Research

This project aims to use the research which has been already carried out in this field and the information presented in Section 2 as the foundations for the project, ensuring that the work carried out is both relevant and original.

The method of waterproofing that will be used will be an external coating, delivered through the vascular network. When the concrete cracks the waterproofing agent will be delivered down the tube, spill out at the crack and the crack surface will be coated with the waterproofing, thus eliminating the ingress of water up into the concrete. Whilst it should be noted that water and chlorides can diffuse through the concrete and ultimately cause corrosion, this project will focus on the ingress of water and chlorides through external cracks.

The research shows that the use of an interconnected network presents the best chance for administering a waterproof coating over multiple cycles.

Some of the issues that will need to be addressed include:

- Designing an embedded tube system that will be brittle enough to crack under loading but be tough enough to be able to withstand the casting process.
- Considerations about the scalability of any experimental system as the end goal is for this process to be included in future concrete structures.

The potential use of biomimetic vascular networks in the field of self-healing concrete is clear, and the extension of replacing the healing agent with a waterproofing agent is also clear. This is especially true when considering work by Schlangen (2013), where he concluded that certain types of self-healing do not increase strength at all but do decrease the permeability of a structure [39]. It should be noted that some of the self-healing agents used in previous studies can take weeks to cure or to provide a barrier against the water ingress. This gives ample time for water to enter up into the crack so this project will aim to provide a quick method of providing a waterproof barrier in the event of a structural failure. It is therefore with this in mind that this project will address the waterproofing, and hence sorptivity, of a cracked structure via the use of biomimetic vascular networks.

3 Materials and Methods

The following section outlines the materials used in this project and the experimental preparation and methods.

3.1 Waterproofing Agent

Four different waterproofing agents were used:

Tung Oil

Tung oil is a drying oil obtained from the pressing of the seed from the nut of the tung tree (*Vernicia fordii*). It is normally used as a natural finish for wood, including on musical instruments. It does not penetrate the surface but instead it stays on the surface and dries upon exposure to air. The resulting coating is transparent, waterproof and flexible. It is composed primarily of eleostearic acid, with small amounts of oleic, linoleic and palmitic glycerides [40]. Tung oil has been shown to reduce water sorption in lime binders in walls [41] and on concrete surfaces [42].

Tung oil is naturally very viscous, therefore to allow it to be injected down the narrow tubes it was mixed with white spirits at a ratio of 1:1 as specified by the manufacturers.

Alkyl Polysiloxane Sealant

As previously discussed in Section 2.1.1, silanes and siloxanes are effective in reducing the water permeability of a structure. Silanes and siloxanes are known as breathable sealants. This is because they permeate into the pores in the concrete, but only bond to the sides, leaving the pores open. This means that liquid water stays out but water vapour can evaporate through, which helps preventing spalling and flaking of the concrete [43].

The sealant employed in this project was an alkyl polysiloxane sealant, called Thompson One Coat Water Seal Ultra, which is used in many industrial applications. It is milky-white in appearance and has a similar viscosity to water so there was no need to dilute it in order for it to be injected.

Acrylic Sealer

Acrylic sealers are a low cost waterproofing option that, when applied, form a thin protective film on the surface of a concrete surface. They are very quick to dry, however the resulting film is thinner than that produced by a lot of the other industrial sealants so it wears faster and requires reapplication sooner [44]. The acrylic sealer used in this project was the Plastikote© Premium Gloss Clear Sealer. There was no need to dilute this sealer as it had a similar viscosity to water.

Nano Waterproofing Sealant

Nano waterproofing agents are an example of a new development in the waterproofing market with not a lot of research into their suitability. Nano-sealants are silane-based, creating a breathable barrier, but the silane particles are nano-sized. This means that the sealant can penetrate much further into the concrete than the normal silane mixtures. The nano-sealant used in this experiment was nanoShell Stone which has the ability to impregnate a concrete surface to a depth of 15 mm [45]. There was no need to dilute this sealer as it had a similar viscosity to water.

3.2 Concrete Materials

The cement was a mixture of Ordinary Portland Cement (PC) (CEM-I, 52.5), sand (maximum particle size 2 mm) and tap water, combined in a ratio of 1: 1.5: 0.4 (cement: sand: water) by weight. Due to the small size of the samples there were no large aggregates included. This composition allowed a good workability of the mix and meant that it could flow around the small spaces in the moulds and around the formwork.

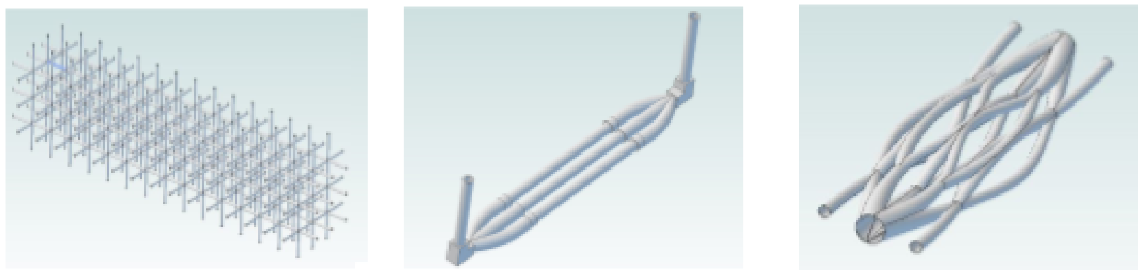
3.3 Network Materials

ABS

Acrylonitrile butadine styrene (ABS) is a thermoplastic polymer which is lightweight and is easily able to be injection moulded and extruded. Therefore it is used with the 3D printer in this project.

Polyolefin

Polyolefin was another material which was used in the network design. It is a material which shrinks radially when heated to between one-half and one-sixth of its diameter.



(a) (b) (c)

Figure 8: Different concepts for vascular networks (a) An orthogonal network array based on [46] (b) A simple interconnected network made from constituent parts (c) A vascular network with interconnected tubes of varying diameter, mimicking blood vessels (adapted from [37])

3.4 Design and Manufacture of Vascular Network

3.4.1 3D Printed Network Design

In previous experiments the network has been 3D printed. The most effective geometry was found to be tubes with an inner radius 4mm and an outer radius 5mm. This size of tube meant that manufacturing errors were avoided, whilst the mechanical properties of the section were retained. Figure 8 shows the different network designs considered when comparing the literature, which uses a simple interconnected network [46], and those that directly mimic the more complex vascular networks observable in nature. The network shown in Figure 8b was chosen to limit the complexity of parts and allow a modular approach to be taken towards the assembly of the network, as the individual network components were to be printed separately and assembled before casting. Initial experiments were carried in this project out using these 3D printed networks.

The tubes are printed using an additive manufacturing technique in which successive layers of print material are deposited on top of one another, thus creating a 3D object.

The tubes were printed on a Cubex^{TM} 3D printer, shown in Figure 9. The parts were taken from previous designs and were printed in Acrylonitrile Butadiene Stryrene (ABS). Previous tests indicated that this was a preferable choice of polymer when compared to Polylactic Acid (PLA), which is the other polymer that the Cubex^{TM} printed with, as it was more brittle, therefore more likely to crack with the concrete [38].

However during the initial experiments issues arose that indicated that a better method could be developed for this project. In other previous tests where crack size was controlled, a crack size of 0.8 mm was decided upon to ensure that the polymer tube had also cracked [38]. However after the initial experiments it became clear that for the smaller-sized samples being used in this project a much smaller crack size would be preferable. This is because due to the

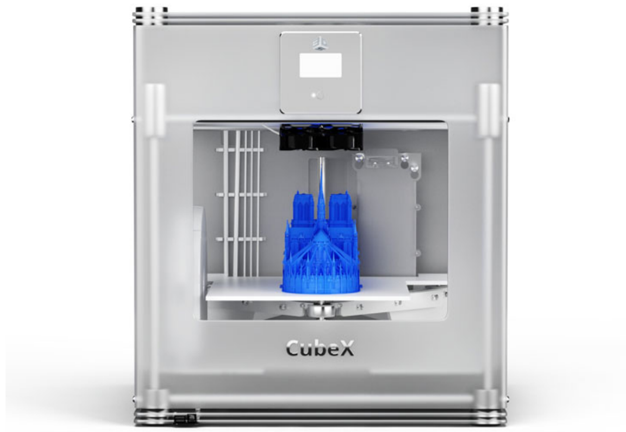


Figure 9: Cubex^{TM} 3D printer used to print network parts [47]

overall relative dimensions of the concrete a crack this large would not mimic what would be found in a real structure. The issue with the previously used 3D printing methods, therefore, was that there was no certainty that the polymer tube would have cracked at this significantly smaller crack width. It was decided to develop a new method of casting tubes that would involve a removable former. This removed the polymer, leaving a purely concrete section, such as some of the samples prepared by Dry [24].

3.4.2 Polyolefin Heat Shrink

The first approach to solving this problem was to use polyolefin heat shrink. The set up for this is shown in Figure 10. Thanks to holes drilled into the moulds at each of the ends, it was possible to feed the polyolefin through the whole sample, again along the main central axis, with the idea being to shrink the heat shrink after demoulding and remove it, thus leaving a hollow tube going through the concrete sample. A 2:1 polyolefin tube which had an initial diameter 2.5 mm was used, which shrunk to 1.25 mm on heating. Due to the flexible nature of the heat shrink it had to be mounted on a 2.5 mm diameter steel bar in order for it to be able to hold its shape and remain in plane during casting.

Concrete was cast around the tube and the sample was left to harden for 24 hours, removed from the mould and then a heat gun was used to shrink the polyolefin tube. However, the hot air did not reach far enough into the concrete sample to be able to shrink the polyolefin in the centre, but it was found that the polyolefin tube itself could be successfully pulled out of the sample using pliers. This left a hollow tube through the centre of the concrete.

This therefore provided a very viable way of providing a straight, single-tube network. Having said that, as the aim in the long run was to mimic the network shown in Figure 8c, with multiple tubes which had the ability to curve and be flexible in casting, there was room

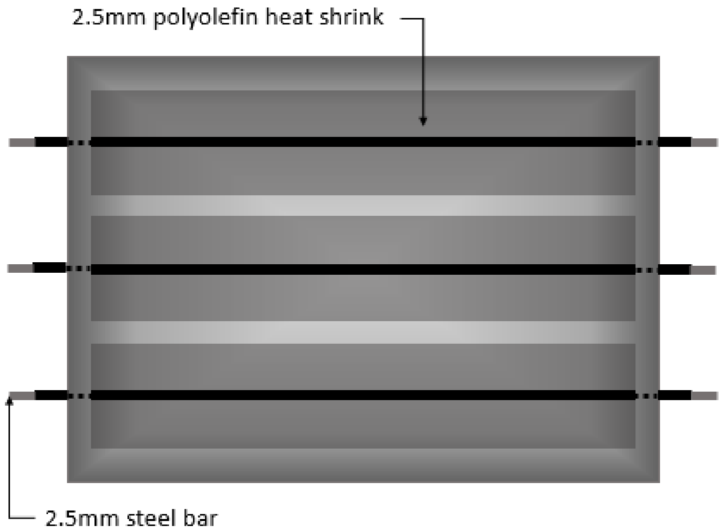


Figure 10: Set up of polyolefin network formwork for three square prismatic moulds

for improvement in this method as the need for tube being mounted on a rigid steel bar was not entirely optimal. Other more viable designs are set out below.

3.4.3 Electrical Cable Formwork

An electrical cable was used as an alternative formwork for the network. The cable was flexible so could be bent into the required length and was easily removed after casting due to the fact that the concrete did not make a good bond with the smooth, insulation surface. Furthermore, the cable was not hollow so retained its shape even when submerged in concrete.

Single Tube Networks

The polyolefin tubing was replaced with a 2.5 mm diameter insulated electrical cable. The casting method was the same as the one used previously, however this time the cable was secured through the holes in the mould using tape at the ends to keep it taut. The cable was coated with a release agent, which is also normally used to coat the mould, to enable it to be extracted easily after casting.

Multiple Tube Networks

The method was further developed to create networks with multiple tubes. In the single-tube casting process it was easy to keep the single wire taut and in plane, however when the network was altered to a shape similar to that shown in Figure 8b there was a tendency for the electrical cable to sag out of plane, which would have inhibited the flow of any agent in the future.

Figure 11: Half cast mould with black speaker cable as formwork for multi-tube network (grey reinforcement bar, discussed in Section 3.2, is also visible)

To resolve this, the concrete sample was cast in two parts. The bottom half was cast and allowed to harden for 24 hours. Following this, speaker wire (two separate insulated wires lightly bonded together) was coated in release agent and was fed through the hole in one end of the mould and out of the other. The wire was then split for the length that would be covered in concrete, leaving the two ends as one single wire, protruding from the end of the mould. The network was arranged in the cast as shown in Figure 11. The individual wires each had a diameter of 1.90 mm. The remaining concrete was cast so the mould was full and the samples were allowed to harden for a further 24 hours. Finally, the samples were demoulded and the cable was peeled apart at the ends and pulled out from the concrete.

3.5 Mould Preparation

The samples were 40 mm x 40 mm x 160 mm in size, making them portable and minimising material waste. The specimens were reinforced with a 2.7 mm diameter wire which was cut into 210mm lengths and bent into a 'U' shape with 10 mm of anchorage bent through 90°at either end. This was glued onto the bottom of the mould using a silicone adhesive.

3.6 Example Procedure for Specimen Preparation

3.6.1 Mould and Mortar Preparation

The reinforcement was glued into the mould and the cable was threaded through the holes in the ends of the mould and secured with electrical tape. The internal faces of the mould and the cable were then lightly oiled with a release agent to ensure that a clean removal of the specimen would be possible after casting.

The constituent elements of the mix were measured out separately. The sand and the cement were added to the mixer together and were mixed for 2 minutes. Then the water was slowly added over the next minute. The final mixture was mixed for a further 2 minutes to ensure a homogeneous mix was fed into the mould.

3.6.2 Casting

The mortar mix was poured into the mould in three parts. After each third had been poured in, the mould was slammed several times onto the preparation table to ensure that it was laid evenly and that any air bubbles in the mixture were able to escape. For the specimens with a two-tube network, which were cast in two halves, each half was poured in two parts and the same slamming action was carried out to ensure an even casting without bubbles.

3.6.3 Curing

The specimens were allowed to set for 24 hours in the moulds (unless specified otherwise) then they were carefully demoulded and labelled. The cables, which were longer than the length of the mould, were delicately removed using pliers. Most of these cables came out easily, due to the use of the release agent, however for those that did not, the copper wire at the centre of the cable was removed first and then the remaining insulation could be easily removed. The samples were then transferred to a water bath to cure for one week. After drying out for 24 hours all the specimens were notched to a depth of 3 mm across the centre of the base of the specimen using a diamond blade cutter. The specimens were cracked using a three-point bend test and injected with a waterproofing agent.

3.6.4 Injection of Waterproofing Agent

Except for the controls, which had no waterproofing agent applied to them, each of the samples had one end covered with tape and pressure applied using a finger, then 5 ml of agent was injected into the uncovered end. For all the samples the agent was seen to come out of the crack, see Figure 12. The samples were then allowed to dry for a further 24 hours.

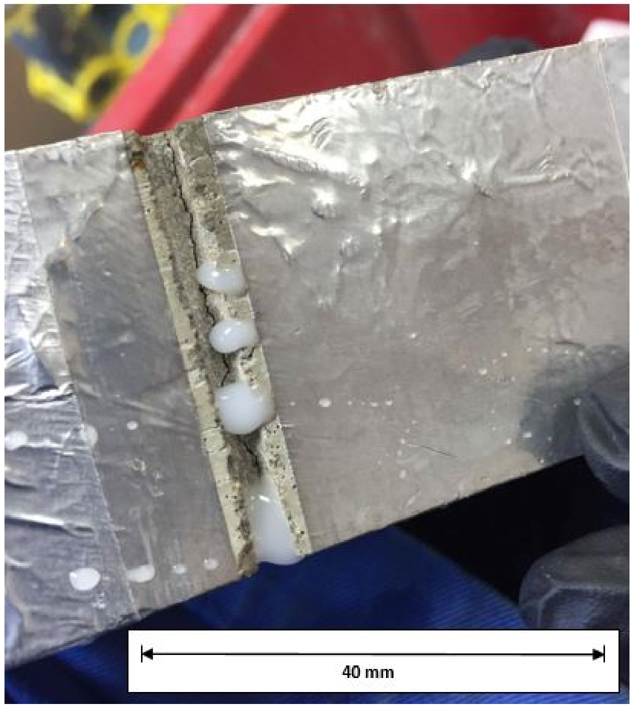


Figure 12: Injected waterproofing agent seen to have come out of the crack

3.7 Experimental Methods for Testing Sorptivity of Waterproofed Sample

3.7.1 Three-Point Bend Testing

The specimens were cracked in a three-point bending test using an Instron® 5967 Dual Column Tabletop Universal Testing System with a 30kN load cell attached. Two small metal brackets were glued, with a two-part epoxy resin, to the bottom of the sample, separated by a distance of 30mm. These provided mounts for the clip gauge, which measured the crack opening displacement, to be attached, see Figure 13.

All specimens were cracked using the same load regime of a loading rate of 0.125 mm/min. The machine was programmed to stop loading when the clip gauge measured a reading of 0.3 mm (this is the maximum crack width in concrete buildings as recommended by Eurocodes [48]).

3.7.2 Sorptivity Testing

The term sorptivity is defined as a measure of the capacity of the medium to absorb or desorb liquid by capillarity [49]. Sorptivity is being used increasingly as a measure of concrete resistance to exposure in aggressive environments [50].

The cracked specimens were prepared for the sorptivity test by almost completely covering

Figure 13: Set up of three-point bending test on Instron® with strain gauge attached on bottom face

the lower face with aluminium foil tape. This stopped the unwanted entry of water in the undesired areas, only allowing water up through the crack and a small gap of a few millimetres either side. The initial mass of each sample was measured using a mass balance which had an accuracy of ±0.5 g. Then the specimens were placed in a water bath filled with distilled water, with the samples submerged so the full base of the crack was covered, meaning that water could be drawn up into the crack by capillary action. This experimental set up can be seen in Figure 14.

At the times $t = i\ minutes$ (where $i = n^2$ for $n = 1, 2, 3, 4, \ldots, 16$) the samples were removed and their mass recorded and then replaced. This allowed the mass of water absorbed by the uncovered area of the specimen to be calculated for each time period, that is:

$$w(t) = m(t) - m_0$$

Where $w(t)$ was the mass of water absorbed at a time t, $m(t)$ was the mass of the specimen at the time, t, and m_0 was the initial dry mass of the sample. The absorption per unit area (g/m^2) for that suction period was then calculated by dividing *w(t)* by the area of the uncovered surface. This value, i, was then plotted against $\sqrt{t}$. The gradient of this was the sorptivity coefficient of the specimen ($g/(m^2 s^{0.5})$) as per RILEM TC 116-PCD [51].

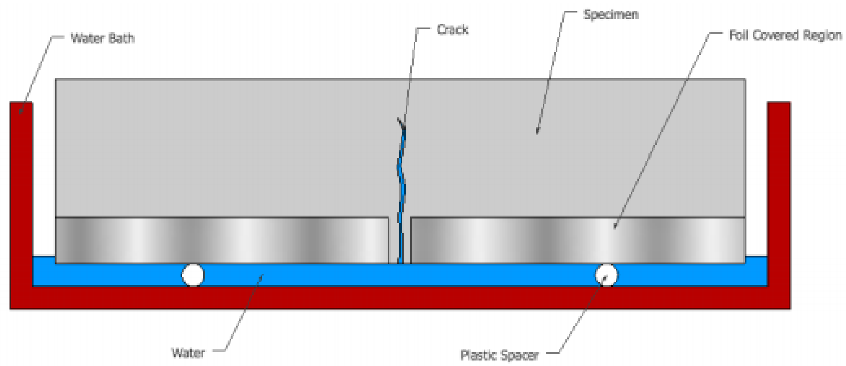


Figure 14: Diagram showing the experimental set up for the sorptivity testing (Taken from [38])

(a) (b)

Figure 15: Sorptivity laboratory set up (a) aluminium foil covering bottom face of sample, leaving a few millimetres either side of crack (Taken from [38]) (b) Laboratory set up of sorptivity test

3.8 Experimental Method for Testing Viscosity

A Brookfield DV3T Rheometer was used to measure the viscosity of the tung oil. A 10 ml sample was placed in the rheometer sampling chamber and a SC4-27 spindle was inserted, see Figure 16. The sample was left to settle for five minutes. In order to erase the shear history, pre-shearing with a rate ranging from 0 to 30 s^{-1} was carried out for one minute. The shear rate was then varied from 8.5 s^{-1} to 60 s^{-1} and then was ramped back down to 8.5 s^{-1}. A shear stress vs. shear rate relationship was obtained. The viscosity was calculated to be the gradient of the linear regression of the ramp down section of this relationship .

Figure 16: Sampling chamber for Brookfield DV3T Rheometer [52]

4 Results and Discussion

4.1 Preliminary Testing

Initial tests were carried out in order to assess if the network system was a viable way of reducing the sorptivity of a concrete sample. The network was designed to be made from a PLA tube. This choice was based on material selection carried out by Kimber (2014) [37]. The two waterproofing agents that were used in these experiments were the tung oil and the alkyl polysiloxane sealant (referred to as siloxane or siloxane sealant).

Before the waterproofing agent was injected the samples were cracked using the Instron® machine. The control samples did not have a tube in them. For these initial investigatory tests the samples which were going to be waterproofed were loaded until a cracking sound was heard. However, the cracks were not all evenly sized. The reasons for this include the anisotropic nature of the cast concrete and differences in bonding between the tube and concrete. The control samples were loaded until the crack was seen to reach half way up the sample, giving relatively constant crack widths.

Table 1: Summary of results from preliminary investigations

Specimen	Mean Sorptivity Coefficient ($g/(m^2 s^{0.5})$)	Standard Deviation
Control	0.137	0.053
Tung oil	0.143	0.117
Siloxane Sealant	0.060	0.058

After cracking and injection of the waterproofing the sorptivity tests were carried out, the results of which can be seen in Table 1. A reduction by over 50% in the sample sorptivity can clearly be seen between the control specimen and the specimen that had been injected with siloxane sealant. This indicates that this is a viable self-healing mechanism and choice of waterproofing. The tung oil sample did not show any reduction in average sorptivity however. The standard deviation of the sorptivity coefficient for each type of sample was calculated for the set of data accumulated from the repeats. It can be seen that the standard deviations, when compared to the means, are relatively much larger for the siloxane sealant and tung oil samples than for the control samples. This variability within specimen results can be explained by the difference in crack size that has been mentioned earlier. It could also be due to the viscosity of the injected materials which is something that is touched on later in the report.

Overall these results were encouraging as they showed that this was a method that could be taken forward to improve sorptivity of cracked cementitious samples. For future experiments however it was decided that the variability could be minimised by eliminating the polymer tubes in the section, and it was therefore decided that purely concrete sections should be used. These tubes will be cast in all the samples, including the controls. It was also decided that further sealants should be used which had lower viscosity as the clear difference between the two agents' viscosity was thought to have had an effect on the results. These changes in experimental method were designed to remove the variability and reduce the standard deviations observed.

4.2 Viscosity Testing

From the viscosity testing it was found that the tung oil and white spirits mixture had a dynamic viscosity, μ, of 17.5 mPa.s (methods for this calculation can be found in Appendix B). The other three waterproofing agents were water-based therefore the viscosity for water was assumed, i.e. 1.002 mPa.s [53].

These results indicate that there will be a larger variability, therefore larger standard deviations, in the results for the tung oil samples as they are less easily pumped down the tubes, especially for the smaller diameter tubes.

4.3 Testing Different Waterproofing Agents

Figure 17 shows the resulting sorptivity coefficients for single-tube specimens treated with the four different waterproofing treatments. These results were obtained over two different tests and have therefore been normalised with respect to the control sorptivity in each test.

That is to say that to normalise each of the results, the mean sorptivity coefficient for each type of waterproofing was divided by the mean sorptivity coefficient for the control from the test, giving a non-dimensional value. The agents could not all be tested with the same concrete batch due to constraints with the number of moulds available.

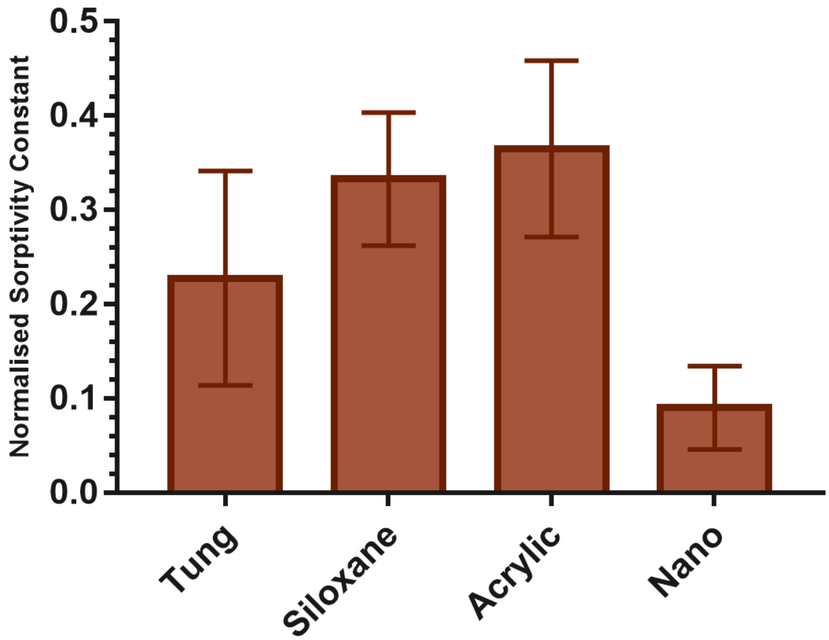


Figure 17: Graph showing the normalised sorptivity coefficients for samples treated with different waterproofing agents)

From this graph it can be seen that the addition of the waterproofing agent through the vascular system has significantly decreased the value for the sorptivity coefficient for all the different waterproofing agents.

The smallest reduction comes from the acrylic sealer, which is not a surprising conclusion as this is the cheapest type of waterproofing agent. The acrylic sealant creates a thin film on the surface of the crack. This means that unless the acrylic fully covered both sides of the internal crack faces there would still be the chance for water to be absorbed up the crack and into the concrete by capillary action. Due to the brittle nature of the acrylic only thin layers can be applied at a time in order to to reduce the risk of cracking.

Tung oil offered the second best waterproofing capabilities for this system. This is another example of an agent that deposits a coating on the face of the concrete. However as seen in the viscosity experiments the tung oil is much more viscous than the acrylic. Therefore it would flow much slower over the crack face and would leave a thicker residual coating which would be more effective than the thinner acrylic alternative in repelling water away from the crack. However, in reality, a thick coating on the outside of a concrete structure could lead to further issues as it would inhibit the travel of water vapour. This means that the water vapour leaving the concrete would build up when it reached the surface which would lead to spalling, flaking and other surface deterioration. Another point to note is that the tung oil leaves a dark stain on the concrete (see Figure 18) and strong smell which are both undesirable attributes for repairing concrete structures.

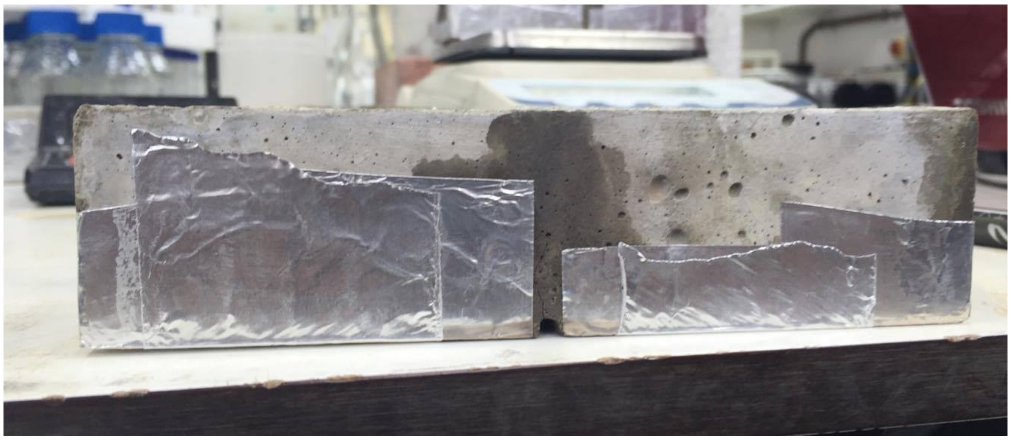


Figure 18: Sample treated with tung oil clearly showing dark stain around crack from the oil

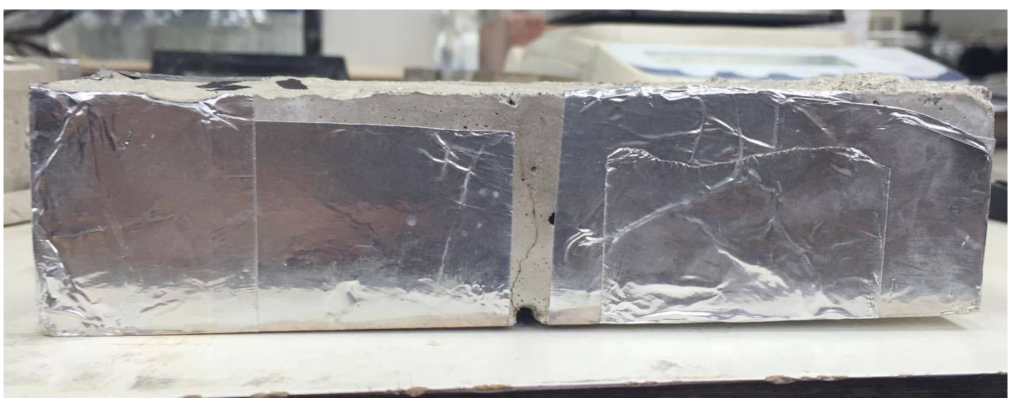


Figure 19: Sample treated with siloxane sealant which has been completely absorbed, leaving no residual staining

It is further possible to compare the siloxane sealant and the nano-sealant. These two agents have very similar chemical compositions, with the main difference being the size of the particles, thus the penetrating distance of the sealant. From Figure 17 it can be seen that the nano-sealant was over three times as effective as the industrial siloxane sealant at reducing the sorptivity of the sample. The greater depth of penetration means that a thicker boundary against external water is created within the concrete, thus reducing the chances of water ingress. Furthermore, the nano-sealant was by far the most effective sealant used in this project - compared to the control sample the sorptivity coefficient has reduced by an order of magnitude. The penetrating sealants do not stain the outside of the concrete but are completely absorbed, see Figure 19.

These results indicate that the biomimetic vascular network is an effective way of waterproofing a crack in a cementitious material and different waterproofing agents provide a range of efficacies.

Figure 20 shows the physical difference between the surface coating and the penetrative method of waterproofing. These images were taken on the Scanning Electron Microscope (SEM).

Here Figures 20a and 20b show the composition of the control concrete sample. The two main constituents, Calcium-Silicate-Hydrate gel and ettringite (the needle-like constructions) can be observed.

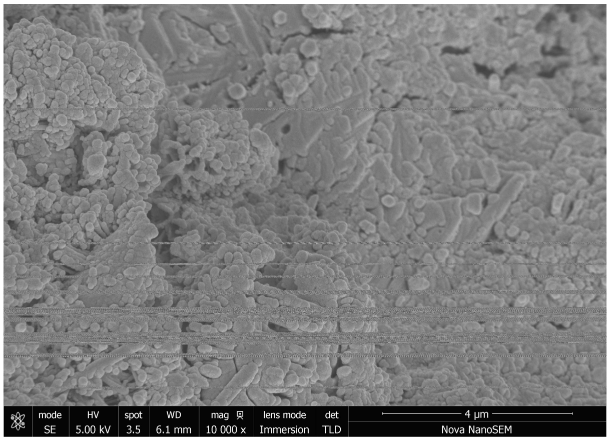
Figures 20c and 20d clearly show the effect of the tung oil on the concrete surface. There is a thick coating completely covering the fine details that were observable in the control samples. Vortices can be seen in the surface patterns which gives the impression that the tung oil was 'splashed' over the crack surface. This thick coating suggests that water would have difficulty penetrating through to the concrete, although small cracks can be seen, especially in Figure 20d.

The SEM yielded slightly lower quality images for the samples which had been treated with the siloxane sealant, however they still give a good impression of the mechanics involved in the waterproofing. Figures 20e and 20f bear much more of a resemblance to the control samples than to the samples treated with the tung oil. This is because this type of sealant is a penetrant and is designed to soak into the concrete and coat the inside of the pores and not sit on the surface like the tung oil.

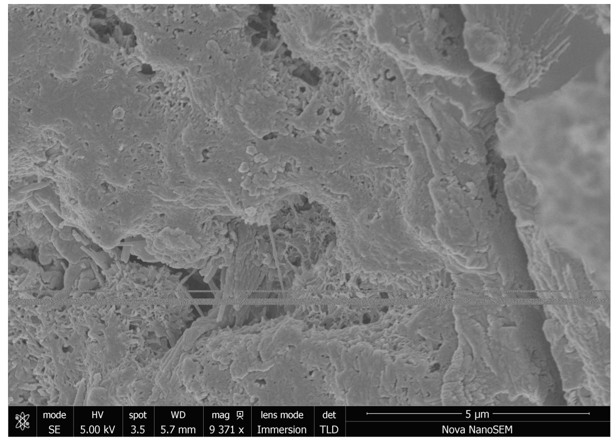
4.4 Testing Repeatability of Self-Healing Mechanism

As defined by Li and Yang (2007) one of the attributes of self-healing materials is *repeatability*, i.e. the idea that the system in place should be capable of healing multiple damage events [14]. It was outside the scope of this project to investigate the degradation of the self-healing agents after application. However the effect on the sorptivity coefficient with multiple coatings of the waterproofing agent was investigated, the results for which are shown in Figure 21. The sorptivity coefficients have been normalised in the same way as described in the previous section for ease of comparison.

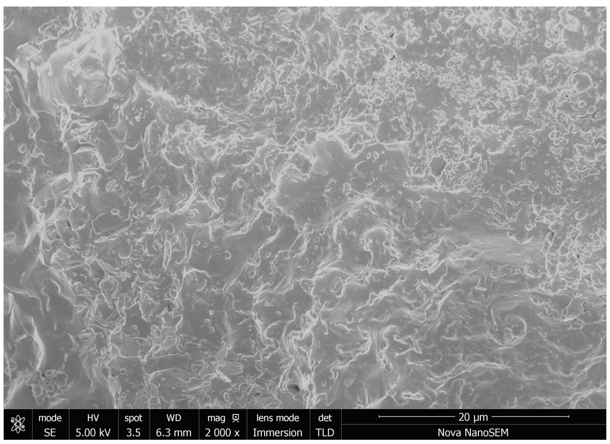
It can be seen that for all the three coatings, for both the tung oil and siloxane samples, there is a significant reduction in the sorptivity coefficients for the specimens with the self-healing networks when compared to the control samples.



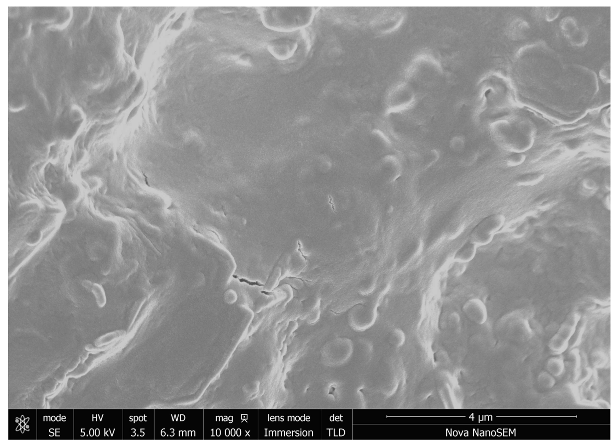
(a) Control sample 20 μm scale



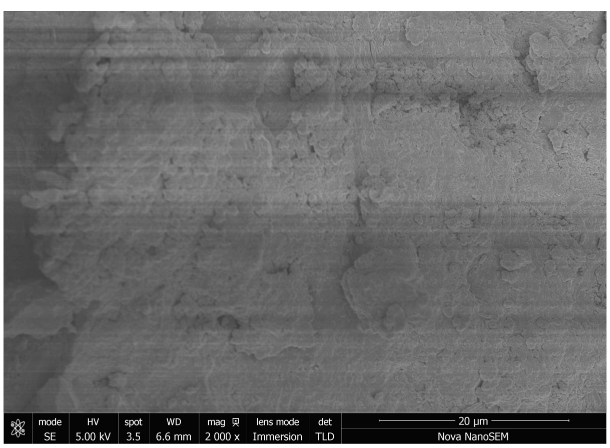
(b) Control sample 5 μm scale



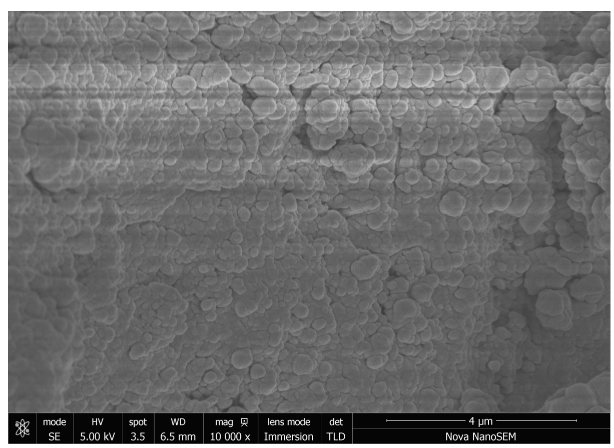
(c) Tung oil sample 20 μm scale



(d) Tung oil sample 4 μm scale



(e) Siloxane sealant sample 20 μm scale



(f) Siloxane sealant sample 5 μm scale

Figure 20: SEM images of concrete samples treated with different waterproofing agents

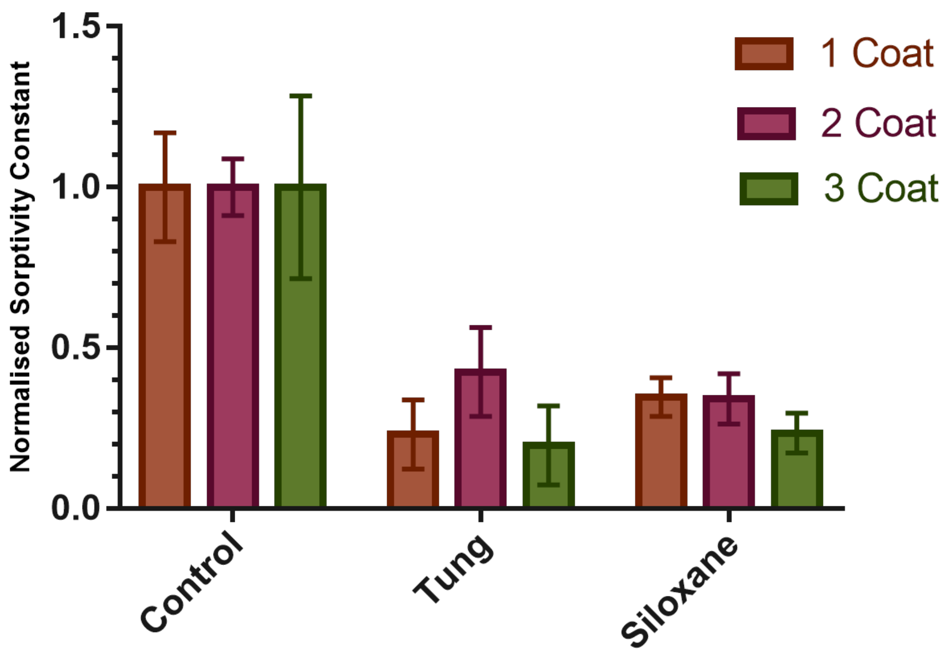


Figure 21: Graph showing how the sorptivity coefficient changes for samples with multiple coats of waterproofing agent

It is hard to draw any other conclusions from the data for the tung oil presented in Figure 21 as there is no obvious trend. As discussed before the standard deviations present for these samples are large and the reason for this could be due to the the higher viscosity, making the tung oil harder to pump. What can be seen though is that the addition of extra tung oil layers does not diminish the ability of the concrete to resist the water ingress. A suggestion for further work, therefore, could be to investigate if the multiple layers of tung oil do in fact make a difference at all, or if the initial coating provides the maximum resistance to water ingress.

There is also not a clear trend for the siloxane sealant samples. However, it could be expected for there to be a reduction in the sorptivity coefficient for samples which had been treated with more coats of a penetrating waterproofing agent. The reason for this is because the sealant is fully absorbed into the concrete, creating a barrier below the surface. The more coats that are added to the concrete, the thicker this barrier becomes and the depth of concrete that is able to repel water increases thus reducing the amount of water that would be able to be absorbed through the crack. Further tests would have to be completed to try and develop a trend and to see if there is a maximum number of coats after which the sealant cannot penetrate any further, therefore levelling off the sorptivity coefficient.

These tests aimed to see the effect of multiple coats but also the repeatability of the mechanics of the vascular network. Referring back to Figures 20c and 20d it can be seen that the tung oil leaves a thick residual coat. Therefore it could be that multiple coats of tung oil could block up the network tubes, impeding the agent's access to the crack site. With the penetrating siloxane sealant this would not be the case unless the concrete became saturated with the waterproofing agent and the sealant had to sit on the surface of the tubes and crack site. From the three coats applied in this experiment the tubes do not seem to have been obstructed.

The coatings that are applied to cracks do not have the same lifespan as a concrete building does. Therefore they would need to be reapplied via the networks at least once. As such, it should be ensured that the tubes are large enough that if there is a risk of blockage the flow of healing agent is not significantly hindered.

4.5 Testing Different Network Geometries

4.5.1 Sorptivity Results

One of the concerns surrounding the inclusion of tube networks within a concrete structure is about the effect that they would have on the section stiffness and therefore the overall bearing capacity of the structure. However, for the section size tested in this project, the reduction in the value for the second moment of area was negligible (this is shown in Appendix C). For the network to have a significant effect on the value for I_{yy}, i.e. to reduce it by at least 5%, a tube diameter of around 20 mm would be necessary for this section. However, the encouraging results from this project suggest that the size of tube chosen at this stage are adequate for the lower viscosity waterproofing agents to reduce the sorptivity coefficient. As such, there is no need to compromise the stiffness by choosing larger diameter network tubes.

Figure 22 shows the change in the normalised sorptivity coefficient for the networks with one and two tubes for both the tung oil and the siloxane sealant. The set up with two tubes was hoped to mimic the bifurcations found in nature and allow the healing agent to reach a larger proportion of the crack face.

For both of the network geometries the sorptivity coefficient was significantly reduced when compared to the mean. For the tung oil the sorptivity coefficient increased when more tubes were added. This means that there was an increase in the water that was absorbed by the concrete. The reason for this is most likely because the tung oil was not able to easily move down the tubes and into the crack. Figure 23 shows how the tung oil has spread out over the face of the crack from the specimen with one tube in the network (a void can be seen just above the tube). Figure 24 shows the same thing except this time the specimen

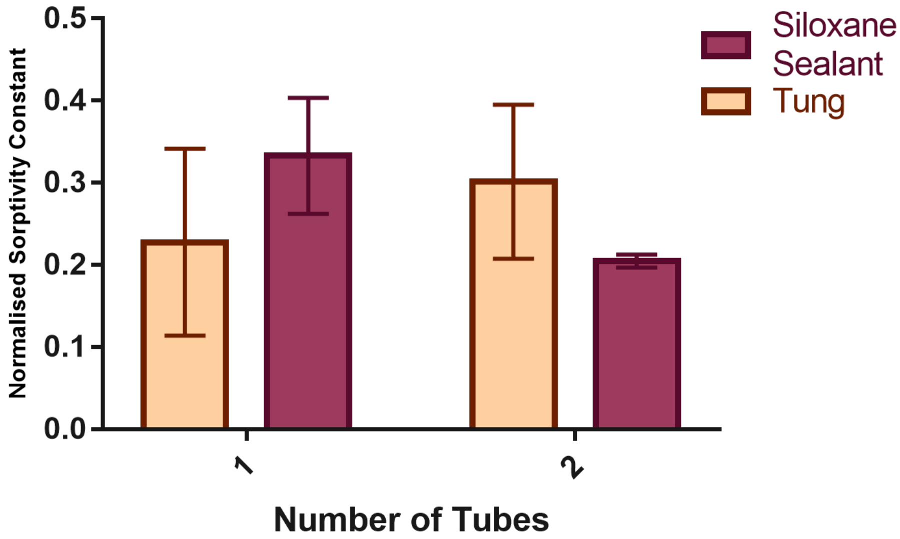


Figure 22: Graph showing how the sorptivity coefficient changes for samples different tube network geometries

has a two-tube network. The tung oil has had a dye added to it and shows up as ochre on the grey concrete surface. It is easy to see that the tung oil covers much more of the face of the concrete in the one-tube network than in the two-tube network. As seen in Section 3.2 the viscosity decreases exponentially as the proportion of tung oil decreases. Therefore reducing the amount of tung oil would allow better flow through the network, however this could compromise the resulting sorptivity coefficient.

The results for the tubes injected with sealant show a different trend however. There is a marked reduction in the amount of water absorbed by the sample which had two tubes when compared with the sample that only had one. As discussed by Heywood (2015), the greater number of tubes embedded in the concrete, the higher the crack face coverage [38]. In this instance, the viscosity of the siloxane sealant was low enough that the flow was relatively unaffected by the small diameter of the pipes and a greater area of the crack face was reached by the sealant, where it was then absorbed. Therefore the addition of further tubes within the concrete could lead to an even lower sorptivity coefficient. Further investigations into the optimal number of tubes for full face coverage could be carried out.

4.5.2 Viability of Network Construction Method

As can be seen in Figure 23 a fairly sizeable void has formed next to where the cable was originally running though the sample. The reason that this is present in the sample is be-

Figure 23: Single tube network injected with tung oil

Figure 24: Two tube network injected with tung oil

cause the air bubble may well have become bonded to the electrical cable during casting, therefore did not manage to leave the concrete before hardening, resulting in a void forming. This image highlights another issue that could be present when casting extra tubes in concrete: during the plastic phase of freshly lain concrete bleeding or water gain (sedimentation) results in the formation of a water or water and entrapped air gap on the undersides of reinforcing bars [54]. This leads to a poor surface finish and cracks developing above the cast tubes which would be highly undesirable. To minimise these effects the concrete should be vibrated and well compacted to maximise the air expelled during the casting stage.

Another question surrounding this method of network manufacture is if it is a viable option when scaled up to be used in real concrete structures. For this project the samples were small and there was only one bifurcation of the embedded tubes, therefore it was easy to remove the cable formwork from the concrete, leaving the hollow network. However, when scaled up this is not likely to be as easy, as the network will expand in three dimensions and will have multiple branches. Therefore there is still further refinement necessary in this method before it will be able to be implemented on an industrial scale.

5 Conclusions

5.1 Key Findings

Self-healing concrete is a very relevant way of achieving the sustainability levels needed in order to reduce the environmental and economic impact that infrastructure has on society. Previous research into this area highlighted that one way of tackling this ever-growing issue is through the implementation of a biomimetic vascular network as a means of creating self-healing concrete. These studies were used to provide a basis for the design and testing of such a system for this project. The main aim of the project was to create a network that minimised the water ingress at a crack face soon after the damage had occurred. It was proposed to use a network connected to the environment.

In contrast with previous 4th year projects in this area, it was decided that the tubes would not be made from polymer. The reason for this was to ensure that the network cracked at exactly the same time as the surrounding concrete to ensure an effective mechanism. From previous studies it was found that due to a mismatch in stiffnesses between the tubes and the concrete they did not both reliably crack at the same time. Considerable effort was spent investigating the best approach to building a polymer-free network. It was decided that the tubes would be made up of temporary formwork which would be removed after casting the concrete, leaving purely concrete sections, much like some of the initial self-healing experiments carried out by Carolyn Dry [24]. This was decided to ensure that the tubes would crack at exactly the same time as the surrounding concrete. Different materials were used as a removable former, including polyolefin, but eventually electrical cable, coated in oil, was selected. This cable was easily removed after casting, leaving a hollow network. This set up allowed both single-tube and double-tube hollow networks to be cast in the prismatic concrete samples.

Four types of industrial waterproofing agent were selected for injection into these networks. These four types could further be divided into two types of waterproofing agent: surface coatings and penetrating sealants. These agents were allowed to dry for 24 hours after application. It was found that they all significantly reduced the sorptivity coefficient of the samples to 9-40% of the control samples. It was the more viscous surface coating which was the more effective as it deposited a thicker coating on the concrete, and it was the penetrating sealant with the smaller particle size which was able to provide a better barrier to water ingress as it had the ability to penetrate to a deeper depth.

It was found that a larger number of coatings of tung oil did not seem to make a difference to the sorptivity, but the variability in these results may also be due to the high viscosity of this healing agent. The addition of more coats of the penetrating sealant was beginning to show signs of a decreasing sorptivity coefficient, further tests in this area would confirm this.

The viscosity of the healing agents was shown to have an effect on the efficacy of the different geometries of vascular system. For the high viscosity tung oil, decreasing the network tube diameter had a negative effect on the ease of the flow of the oil and a larger crack coverage was not seen. However, the siloxane sealant was able to flow equally well through both the larger and smaller tubes, and the sorptivity coefficient was the smallest for the two-tube system, suggesting that this network design allowed a fuller coverage for the sealant.

To summarise these key findings they should be compared against Li and Yang's frame work for 'robust' self-healing materials outlined in section 2.1.1:

1. **Pervasiveness**: Depending on the size of the tubes and the viscosity, the healing agent was able to be delivered effectively to the site of the crack immediately.

2. **Stability**: The healing agents used will be chemically stable within the concrete however further research should be done to establish if the network tubes are small enough to close up, e.g. by autogenous self-healing. ;

3. **Economics**: The sealants used in the project are the standard sealants used on concrete these days. The labour involved in making the networks could be a large economic factor in the implementation of this project;

4. **Reliability**: Reliability was improved from previous experiments by the removal of the polymer networks, which were replaced with hollow tubes which cracked reliably. Further experiments into the optimal tube size and viscosity of the agent would allow the agent to reach the crack site reliably.

5. **Quality**: All healing agents significantly reduced the sorptivity of the concrete samples to between 9 - 40% of the control sample value.

6. **Repeatability**: It was shown that it was possible to repeat the experiment with consistent results.

5.2 Improvements

The main improvement would be to develop the method of network casting. It is clear that having the hollow tubes within the concrete is an effective way of delivering a self-healing agent to the site of a crack. However there are questions surrounding the scalability of this method. One way of modifying the current method would be to cast a biodegradable network in the tube, which after degrading would leave the same hollow network as seen in this project.

Issues surrounding this type of network manufacture would include how to hold the network in place, how to extract the materials from the network once they have started to degrade and how to construct the network. One method which is being used in bioengineering for applications such as hip implants, is the use of a biodegradable polymers. Examples of these polymers include PLLA and PLGA which have different degradation rates, depending on their chemical composition. These plastics are easily 3D printed which has been shown to be a quick and effective way of manufacturing multiple, complex tubes. Further improvements to this system would include a damage sensing component which would create an automatic network that could inject the self-healing agent directly to the damage site upon cracking without the need for human involvement, thus creating a smart structure. Ultimately, the system developed in this project should be coupled with other self-healing mechanisms to allow a cracked sample to not only be waterproofed but also for the strength to be regained, such as in the experiments described in the Literature Review. The combination of such systems would mean that the network design would need to be refined in order to minimise blockages and other issues associated with the injection of the healing agents designed to improve strength that have been discussed in previous reports [38].

5.3 Recommendations for Future Work

The results from this project have highlighted areas for which there is opportunity for further investigation. Detailed below are a number of ways in which this work could be taken forward and they are split into short term projects which may be investigated within a year and long term projects that will require a longer time investment.

5.3.1 Short Term Projects

- **Investigation into optimal network tube size for different healing agents:** Different waterproofing agents have different viscosities, therefore it may be that a 'one-size-fits-all' approach would not be best if different waterproofing agents are required, e.g. for different environments. Consideration of the injection or pumping technique would be necessary.

- **Combine with self-healing networks used for restoring strength:** Once the network has been installed it would be pertinent to see if it was a viable way of transporting multiple healing agents to a crack, all targeting different performance criteria.

- **Investigation into biodegradable printed networks in concrete:** A biodegradable printed network will provide greater complexity in the shape of a network, especially when considering larger structures. It will also mean that the sections remain entirely concrete so the networks are likely to crack at the same time as the surface concrete.

- **Design of vascular network to heal other failure modes:** Only tensile cracking was considered in this project, however this is not the only way that cementitious materials crack. Further refinements could be made to ensure that a network would crack due to shear and shrinkage cracking. Ultimately the network should be able to crack reliably for all failure modes.
- **Design and embed members into larger members:** It would be recommended to test the networks on larger members that are more likely to be used in industry in order to establish the groundwork for when these networks are embedded in concrete structures.

5.3.2 Longer Term Projects

- **Computational modelling of damage and healing in vascular systems:** A computational model for self-healing using vascular networks may be able to be developed if studies for the finite element analysis of progressive damage in self-healing composites (Nguyen, 2012), lattice modelling methods for fracture patterns and recovery (Joseph, 2008) along with other studies are combined [55]- [56].
- **Investigate methods of visualising the healing process:** Due to the fact that cracks are often contained within the structure, novel methods such as CT scanning are currently being used to view the internal healing within specimens [57]. Along with other imaging methods, this could provide a very viable way of monitoring the flow network within a structure and is an avenue which should be further explored.

The application of self-healing cementitious materials in concrete structures in the future will reduce an economic and environmental burden. The concept of vascular networks, which are an intrinsic biological part of almost all living things, can be implemented in concrete structures and the work here has shown the impact that their inclusion could have on a structure's durability. Progress must still be made but this is an obvious step forward in a world of smart and sustainable infrastructure.

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Appendix A: Risk Assessment Retrospective

At the beginning of the project a risk assessment was carried out. It identified chemical, mechanical and office-based hazards. This process allowed much more careful consideration of the risks involved with the project and helped in creating a safe system of work. Many safety measures were undertaken throughout the project, mostly these were involved with the production of the concrete. For example, goggles and a dust mask were worn when mixing the cement. Furthermore, gloves were always worn when handling the chemicals in the lab. No issues were reported throughout the project which implies that the initial risk assessment was successful in identifying the hazards present.

Appendix B: Viscosity Calculations

Table 2: Dynamic viscocities of substances used in project

Substance	Dynamic Viscosity (mPa.s)
Water	1.002 [53]
Tung Oil	256
White Spirit	0.74-1.64 [58]

Equation (1) is a simple model of how the viscosity for a mixture is calculated, where X_i is the molar proportion of each component and μ_i is the dynamic viscosity of each component [59]. In this case the mixture was a tung oil and white spirit, mixed at a ratio of 1:1.

$$ln(\mu) = X_1 ln(\mu_1) + X_2 ln(\mu_2) \tag{1}$$

Appendix C: Second Moment of Area Comparisons

Table 3: Reduction in second moment of area of concrete section with tube networks when compared to solid section

Number of Tubes	Diameter of Tubes (mm)	Reduction in I_{yy}
One	2.5	0.0009%
One	22	5.4%
Two	1.9	0.0005%
Two	19	6.0%