

# “SARCOS” PhD Meeting

Self-healing As preventive Repair of COncrete Structures

Novi Sad, Serbia

7<sup>th</sup> – 9<sup>th</sup> March, 2019



## BOOK OF ABSTRACTS

## FOREWORD

The 1<sup>st</sup> SARCOS PhD and ECI Meeting was organized at Laboratory for Materials in Cultural Heritage, Faculty of Technology, University of Novi Sad, from 7<sup>th</sup> to 9<sup>th</sup> of March, 2019 in Novi Sad, Serbia, with the aim of presenting PhD/ECI's actual research on self-healing of concrete structures. An intention of the meeting was that each participant highlights "How should the self-healing/repair topic improve during the next years to ensure their success?" with the issues in her/his research which still need to be developed. The presentations of participants opened the discussions during the round table sessions focused on finding solutions how researchers could overcome their scientific problems and challenges. Hopefully, this could serve as a basis for initiation of possible future collaborations among PhD students and ECI researchers within the SARCOS action network and beyond.

The meeting also provided excellent opportunity for participants to get introduced with the Laboratory for Materials in Cultural Heritage (HeritageLab), its activities and equipment. The hands-on practical training included methods for characterization of cement-based materials such as XRF, FTIR, Hg intrusion porosimetry, thermo-vision measurements, UV-Vis spectroscopy, microscopy (portable and polarization), as well as practical training in the Laboratory for Polymer Materials (PolyLab) and in the Microbiology Lab.

Within this meeting, a scientific field trip to Belgrade, the capital of Serbia, was organized with an expert guidance to the concrete landmark objects such as Avala Tower, Ada Bridge, Blok 23 residence quarter and Genex Tower.

This e-book consists of abstracts of the presentations given by the participants at the 1<sup>st</sup> SARCOS PhD and ECI Meeting. More information can be found at the Action Website: <http://www.sarcos.eng.cam.ac.uk>.

We hope that the 1<sup>st</sup> SARCOS PhD/ECI Meeting was a fruitful experience for participating researchers which opened new opportunities for PhD students from SARCOS action network to meet each other and discuss on among peers their research progress, as a way to ensure success of their work, giving a positive impact to the COST Action 15202 "SARCOS".

## **COST CA 15202 Self-healing as preventive repair of concrete structures “SARCOS” PhD Meeting – Novi Sad, Serbia, 7<sup>th</sup> –9<sup>th</sup> March 2019**

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**Venue:** University of Novi Sad, Faculty of Technology  
Bul. cara Lazara 1, 21000 Novi Sad, Serbia

**Meeting Dates:** 7.3.2019 – 9.3.2019

**Chaired by:** Dr Bojan Miljevic, ITC Manager  
Dr Marta Roig Flores, ECI Manager  
Prof. Dr Jonjaua Ranogajec, Host Institution

### **OBJECTIVE /SCOPE**

The SARCOS PhD Meeting was organized with the aim of presenting PhD/ECI's ongoing research on self-healing of concrete structures. The intention was that each participant highlighted at the end of talk “How should the self-healing/repair topic improve during the next years to ensure their success?” the issues in their research which were still to be developed. This would open the discussion as a round table where they would possibly find a solution on how to overcome a certain scientific problem. This could initiate possible future collaborations between PhD students and ECI researchers within the members of SARCOS action. The issues on how to apply for the STSM and/or Conference Grant were discussed as well. The second part was focused on the introduction to the Heritage Lab activities and its equipment with the hands-on practical training of participants.

**Organized by:** Prof. Dr Jonjaua Ranogajec,  
Prof. Dr Siniša Markov,  
Dr Bojan Miljević,  
Dr Marta Roig Flores,  
Dr Snežana Vučetić,  
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## Agenda

Thursday, 7.3.2019		Friday, 8.3.2019	
08:30 - 09:00	Registration	09:00 - 10:30	Presentations of PhD/ECI research @Blue_room
09:00 - 09:15	Welcome words @Blue_room		
09:15 - 10:45	Presentations of PhD/ECI research @Blue_room		
10:45 - 11:15	Coffee break @room 102	10:30 - 11:00	Coffee break @room 102
11:15 - 13:15	Presentations of PhD/ECI research @Blue_room	11:00 - 13:00	Presentations of PhD/ECI research @Blue_room
13:15 - 14:15	Lunch break @room 102	13:00 - 14:00	Lunch break @room 102
14:15 - 16:00	<b>Hands-on practical</b> - separate agenda provided for groups A-F - Laboratory for materials in cultural heritage - Microbiology laboratory - Laboratory for polymer materials		
16:00 - 17:30	<b>Discussion round table - networking with coffee and cookies</b> <i>Groups 1-3 @room 101, Group 4 @Blue_room, Group 5 @Laboratory</i>		
17:30 - 19:00	Guided visit to Gallery of Matica srpska	17:30 - 18:00	Closing remarks @Blue_room
19:00 - 21:00	Dinner at Pivnica Gusan, Zmaj Jovina 4, passage	18:00 -	Free time

Saturday, 9.3.2019	
08:15 - 08:30	Gathering in front of the Faculty of Technology
08:30 - 10:00	Bus trip to Belgrade
10:00 - 12:00	Scientific excursion: introduction – Part 1
12:00 - 13:00	Lunch break
13:00 - 15:30	Scientific excursion: old and modern concrete buildings – Part 2
15:30 - 17:00	Return to Novi Sad

## **PRESENTATIONS OF THE PHD/ECI RESEARCHERS**

### **-PLENARY LECTURES-**

## Presentations

Polymer Flexible Joints as an Alternative External Repair Method in RC Structures.....	8
Innovative and sustainable masonry strengthening system for the rehabilitation and improvement of urban resilience.....	10
Development and evaluation of self-healing cement composites incorporating inorganic admixtures and hydraulic phases.....	12
Repair of civil engineering structures using a low environmental impact mortar.....	14
Sustainable bio-based earth mortar with self-healing capacity .....	16
X-ray microcomputed tomography of self-healing in-ground barrier materials incorporating microcapsules.....	18
Effect of Addition of Pre-Soaked Expanded Perlite on Self-Healing Performance of Cementitious Composites .....	20
Modelling autogenous healing of High-Performance Fibre-Reinforced Cementitious Composites (HPFRCCs) via a discrete model.....	22
Advanced surface methods for the preventive repair of concrete structures .....	24
Self-healing in soft soils improved with geopolymers .....	26
Alkali Activated Self-healing Concrete Structures.....	28
Self healing in high strength concrete with super absorbent polymer (SAP) .....	30
Description of the method for observation the microstructure of mineral binders in the state of stress .....	32
Innovative SuperAbsorbent Polymers for crack mitigation and increased service life of concrete structures .....	33
On water tightness and crack closing in self-healing concrete.....	35
Crack healing of glass fibre reinforced concrete using crystalline admixtures.....	37
Influence of TiO <sub>2</sub> nano-sized particles solutions on self-healing properties of concrete structures .....	38
Engineered Cementitious Composites designed with particle size optimization .....	39
Preventive repair of concrete substrates using epoxy resin coatings .....	41
Self-Healing Performance of Cementitious Composites Under the Continuously Increased Permanent Mechanical Loads.....	42
A biomimetic design of vascular network in self-healing cementitious materials .....	44
Self-healing capacity evaluation of UHPFRC with Crystalline Admixtures and Nanoparticles by means of Permeability Tests .....	46
Synergistic strategies for the simultaneous extension of service life and maximisation of waste incorporation for ultra sustainable marine concrete structures.....	48
Nano-scale Modified Cementitious Composites for Bio-inspired Self-healing Ability.....	50
Synthesis and characterization of core/shell particles for developing self-healing mortars..	52
Self-healing Geomaterials .....	53
Overview of the research of laser assisted automate tape laying process for production of advanced composite materials for self-healing .....	55

Screening of new alkalophilic sporogenic bacterial strains and evaluation of their biocalcification potential.....	57
Self-healing materials for the repair of infrastructure.....	59
Utilization of lightweight aggregate in the bacteria-based self-healing concrete.....	61
Ad-hoc sensors for the non-remote monitoring of concrete for the non-destructive characterization of concrete durability.....	63
Discussing Effective Ways to Promote Self-Healing Concrete .....	65
Self-Healing in Cementitious Materials.....	66
Crystalline admixture impact on crack healing of concrete reinforced with glass fibres .....	68
Late-age Autogenous Self-healing Capability of Cementitious Composites .....	69
Shaking table tests on scaled model of traditional brick masonry building .....	71



## Polymer Flexible Joints as an Alternative External Repair Method in RC Structures

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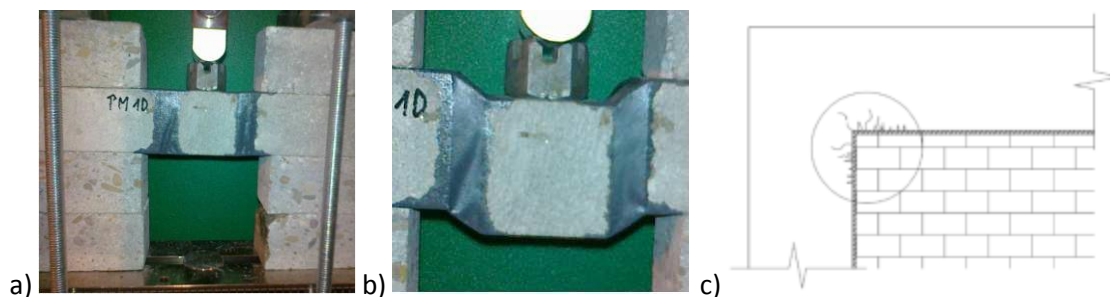
Advancements in technology and material sciences lead new materials to be used in civil engineering, too. Polymer based new material called Polyurethane PM is one of these newly developed solutions, which primarily designed to be used as flexible joints in various types of constructions. In my studies, the usage of polymer flexible joints (PFJ) between masonry and concrete elements is currently being investigated.

The idea of using Polyurethane PM as flexible joints is not very new, the story is started almost a decade ago at Cracow University of Technology (CUT). Many researches are already done in the past regarding the material properties and its possible usage areas [1-4]. My research focuses on the usage of PFJ between different structural elements, namely concrete and masonry, in order to mitigate hazardous seismic effects on these elements as well as entire buildings. It is observed that during seismic excitations an ordinary solution of using classical type mortars lost their bonding features easily, thus in-plane and out-of-plane failures occur as shown in Fig. 1. This causes loss of lives and properties, and therefore affecting governments and societies adversely. The mechanism is emerged due to the stress concentration increment around the boundary zones between concrete elements and masonries. When lateral loads demand high deformations, classical mortar approaches fail and either crushing or debonding damages occur, see Fig. 1.



**Figure 1: In-Plane (a) and Out-of-Plane (b) failures and their schematic visualisation (c).**

In order to overcome this obstacle, an innovative solution of using PFJs is thought. The polymer material has strong bonding and hyper-viscoelastic features that enable large elongation capability, besides, it is also durable under high compression affects thus it protects different structural elements against crushing failures. In terms of self-healing perspective, the material is preventing the expansion of micro cracks that might occur at concrete boundary zones. These cracks are due to the cyclic loads and they tend to expand during seismic excitations. Strong bonding features of the PFJs lead crack widths to be minimized. Details are shown in Fig. 2 [5].



**Figure 2: Shear test of PFJ: before (a), after (b) [5] and symbolic micro-cracks on concrete (c).**

The research in the scope of my PhD studies is started approximately a year ago. Literature scanning and reviewing past researches were the fundamental steps. Following that, some numerical analyses are done in order to comprehend the behaviour of PFJ both in small and large size structures. The research so far aims to understand the effectiveness of material in terms of damping, frequency and stress mitigation areas. The primary results give promising outcomes that PFJs can be used in real structures. Possible self-healing features are not investigated yet, however, laboratory tests on real size specimens are being prepared and are going to be tested in the next months of this year. The material will also be tested as an external repair solution for filling the cracks. Some images of the on-going project are given in Fig. 3. According to the results of numerical analyses, it is highly expected that PFJs might provide sufficient and long-term protection for the structural elements including concrete, hence permanent and durable solution can be found.



**Figure 3: Laboratory test samples at Slovenian National Building and Civil Engineering Institute (ZAG).**

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## **Innovative and sustainable masonry strengthening system for the rehabilitation and improvement of urban resilience**

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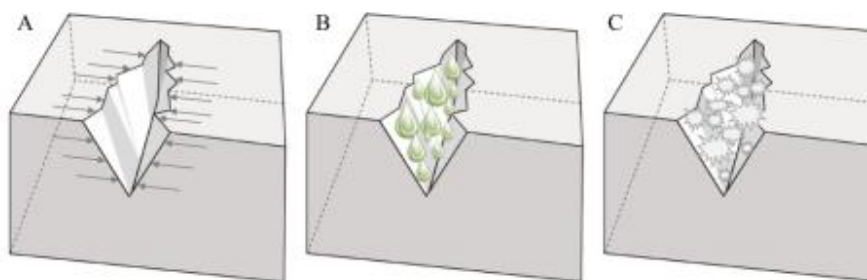
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The main objective of the PhD research is to develop a strengthening system designed to feature strain hardening behaviour, high ductility and proper adhesion to masonry substrates, as part of a simple process of application that allows versatility to architectural solutions. This system is meant to be applied to masonry structures showing deficient behaviour against seismic actions, or walls with low load carrying capacity. Cement matrix composites are a relatively recent class of materials that show mechanical properties adequate for this application. Additionally, they can be tuned to present self-healing features and good behaviour to elevated temperatures.

Several levels of damage can be imposed to an overlay strengthening system after the application in a structural element. For higher static or dynamic actions the level of damage is associated to large cracks with no autogenous self-healing capacity. Nonetheless, when considering a range of low to medium monotonic or cyclic actions, the levels of damage are associated to smaller cracks. These small cracks can show autonomic self-healing capacity, provided that the system engineered properly.

A self-healing process can be defined as a spontaneous in-situ repair of micro-damage occurring in the materials as consequence of weathering or overloading processes [1]. With basis on experimental studies and practical experience, it is believed that self-healing of cracks in cementitious materials is a combination of several chemical and physical processes. In the literature several possible causes for the self-healing phenomena are presented: formation of calcium carbonate or calcium hydroxide; hydration of the unreacted cement or cementitious materials; expansion of the hydrated cementitious matrix in the crack flanks (swelling of C-S-H).

Mortars exhibit autogenous self-healing properties due to the composition of the cementitious matrix. That healing process can be improved by adding specific materials to the original composition promoting the autonomic healing. The materials used are mainly pozzolans, fly ash, special expansive agents and bacteria, see Figure 1 [2]. The restriction of the crack width is generally achieved by the incorporation of fibres, such as in engineered cementitious composite (ECC), which promote the autonomic healing. The most used fibres in this case are polyvinyl alcohol (PVA). ECCs present a quite unique crack pattern because, instead of one single large crack, multiple cracks form in the ECC matrix and the maximum crack width remains below 60  $\mu\text{m}$ . Several researchers investigated the water supply mechanisms and the possibility to mix super absorbent polymers (SAP), also called hydrogels, into cementitious materials to provide additional water, see Figure 1b. These polymers can absorb large amounts of liquids and swell significantly, forming a soft and insoluble gel. The effects of self-healing on the recovery of mechanical properties of lime-based mortars with commercial admixtures and purpose-designed binder coated granules has been studied by De Nardi et al. [3], see Figure 1c. The experimental evaluation was done by means of a methodology based on the comparative analysis of pre-damaged/healed and undamaged specimens. The results obtained by the authors show that the presence of crystalline admixtures speeds up the sealing of the cracks and the recovery of the mechanical properties. This effect is more obvious for specimens exposed to open air, in which condition the autogenous healing capacity in the reference mortar was almost zero.



**Figure 1 - Improved autogenous healing by: a) restriction of the crack width; b) water supply; c) improved hydration and crystallization [2].**

The development of a fibre reinforced mortar with improved self-healing behaviour and mechanical performance is being carried now. In order to evaluate the influence of the type of fibres in the self-healing it was decided to use two type of fibres, synthetic polyacrylonitrile fibres and natural sisal fibres. Additionally, a commercial crystalizing agent was used to study the combined effect to the behaviour of a natural hydraulic lime matrix. The self-healing obtained under wet/dry cycles will be evaluated at two different ages, 28 days and 90 days, and for two different levels of damage.

The results obtained in the experimental study will be used to validate and improve the viability of the overlay strengthening system as a sustainable and resilient solution for the rehabilitation of masonry structures.

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# Development and evaluation of self-healing cement composites incorporating inorganic admixtures and hydraulic phases

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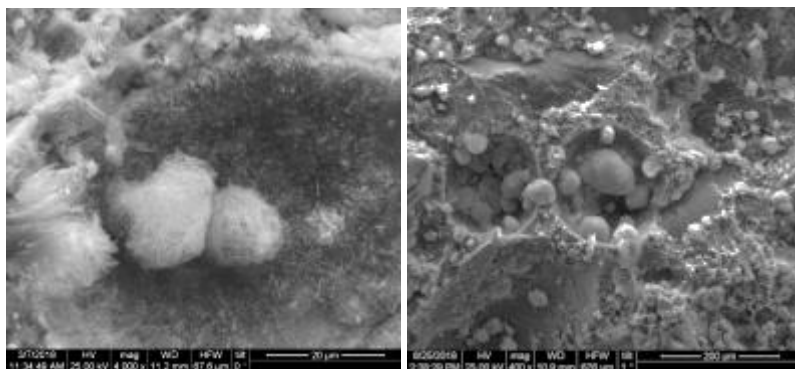
## OBJECTIVES

The present post-doctoral research project is taking place within the framework of the collaboration of NCSR "Demokritos" research centre and TITAN S.A. Cement Company, under the auspices of ISN foundation.

The main objectives of my project include the development and evaluation -at laboratory scale- of different groups of self-healing cement composites that incorporate different types of healing agents, such as mineral admixtures, crystalline admixtures and hydraulic cement phases.

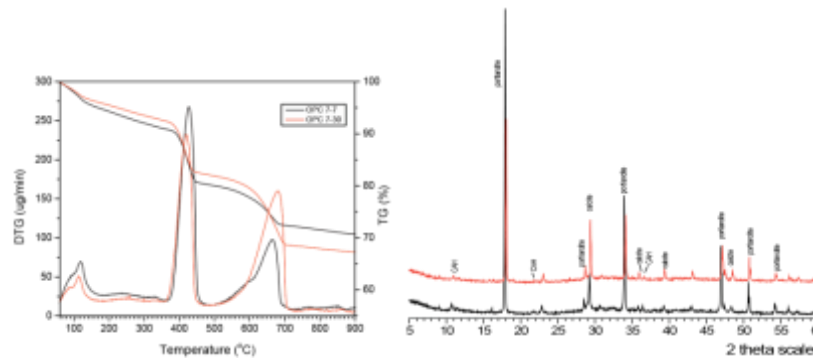
More specifically, the aim of the project is two-fold. Firstly, the self-healing action of the different admixtures is studied, including the characterization of the self-healing products and the boundary conditions affecting the phenomenon such as the effects of curing time before damage, extend of healing period, conditions of healing and damage degree. Secondly, the present project is focused on the investigation and development of methodological approaches for the assessment of self- healing efficiency of the cement composites.

The first part of the project is related to the study of the healing mechanism of cement composites incorporating inorganic admixtures. The healing products formed inside cracks are characterized as to their mineralogy by means of WDXRF, XRD, SEM/EDS and DTA/TG.



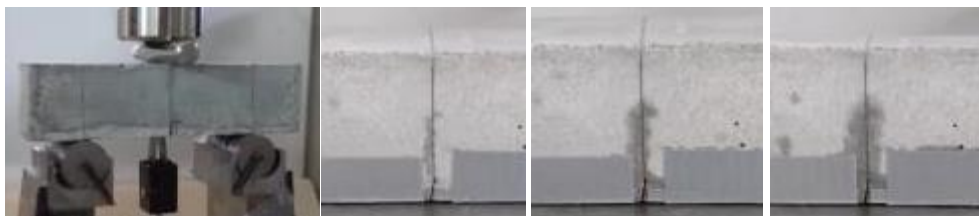
**Figure 2: Secondary products forming inside cracks of cement composites incorporating a crystalline admixture (left) and an expansive agent (right)**





**Figure 2: Thermogravimetric (DTG) and crystallographic (XRD) analysis of healing products after 7 and 28 days of healing**

Secondly, self-healing efficiency is assessed by examining the performance of specimens after their healing regime and contrasting it to that of untreated specimens. CMOD controlled flexural tests are applied to prismatic specimens, in order to ensure a reproducible and controlled level of damage. More specifically, self-sealing efficiency is assessed by measuring the capillary water absorption and water permeability of cracked mortar specimens, whereas, the recovery of mechanical properties is examined by applying controlled levels of compressive stress.



**Figure 3: Measurement of the capillary water absorption of cracked mortar specimens before healing**

Overall, the self-healing/ repair topic has gained a lot of attention in the last decade, during which, very important developments have taken place [1]. Today there are many promising self-healing systems researched [2], nevertheless, there is a knowledge gap related to the boundary conditions of their healing efficiency (e.g. damage degree). Moreover, there is a lack of universal methodologies for the assessment of their healing efficiency [3]. This shortage acts as a barrier in the developing self-healing systems research as there isn't yet a common expression for the quantification of the efficiency for specific performance requests.

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## **Repair of civil engineering structures using a low environmental impact mortar**

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Cracking is almost inevitable for concrete structures, especially for reinforced concrete structures. Without causing a risk of collapse, it accelerates degradation processes, reduces their service life and requires repair. The maintenance and repair of civil engineering structures is difficult due to the multiple origin of degradations (chemical, physical and mechanical). However, the repair and rehabilitation techniques and strategies for concrete structures have been evolving for several years. The research of repair materials optimizing the repair and structure durability is a part of a constructive and preventive approach to extend the life of concrete structures, as well as the safety of users. Different solutions/strategies are available: classical (removal of contaminated concrete and addition of a repair material, e.g.) or innovative (self-healing e.g.) solutions. The main objective of the present PhD is to design innovative repair materials with low environmental impact and self-healing capacities.

Self-healing of cementitious materials is already studied since many years in several laboratories in Europe (Delft University [1], Icitech-Politécnica De Valencia Universitat [2], Politecnico Di Milano [3], Ghent Universito [4], etc...). These studies look at different types of self-healing techniques such as autogenous and encapsulated autonomous self-healing [5]. The present research work focuses on the repair materials characterized by an autogenous healing capacity stimulated by the use of mineral additions due to the continuous hydration of unhydrated binder grains [6] and crystalline admixtures reducing material permeability [1-3, 7].

Firstly, self-healing hydraulic materials are designed and optimized by varying the following parameters: mineral addition (fly ash, blast-furnace slag) and crystalline admixtures types, their contents, chemical activators and curing type. To determine their healing capacity, mortar samples are cracked at 28-day-old using the three points bending test and the healing kinetics is monitored by means of optical microscope. In parallel, the hydration process (hydration products, hydration kinetics, porosity) and macroscopic properties (mechanical strength, delayed deformations, permeability, chloride diffusivity) are also characterized by means of several experimental techniques such as calorimetry, thermogravimetry, etc. Secondly, a new experimental test will be developed in our laboratory to analyze the repair capacity of the mortars previously designed and their durability. A special attention is given to the bond strength between the repair material and substrate, as well as to their deformational and physico-chemical compatibilities. The development of this new experimentation will be complex. Indeed it requires to combine different measurement technics such as the image correlation and acoustic emission, and different test methods for measuring bond strength and transport properties.

As a PhD student since October 2018, attending this meeting will be a very constructive experience to evolve and enrich my background information in the field of rehabilitation of reinforced concrete. I would be delighted to have the opportunity to discuss this topic more deeply with expert researchers and other PhD students which will definitely have a huge impact on my work. Moreover, having profitable discussions with researchers from different

laboratories can be a great chance to find new innovative ideas. All of the above reasons, aligned with my full motivation and curiosity to intervene and resolve concrete issues have encouraged me to apply for this meeting offered by you. My participation will be a great start for my work.

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## Sustainable bio-based earth mortar with self-healing capacity

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### OBJECTIVES

Bio-based construction solutions can boost durability and indoor environmental quality - for free - and can continue to store carbon sequestered by trees and other organic materials. Studies are missing on performance-based design of bio-based composites for retrofitting and refurbishment of the built environment. My research team is bonded by a unique goal: to develop novel bio-based multi-functional systems to provide a solution for significant energy savings, structural durability as well as indoor wellbeing and comfort. Therefore, we design bio-based materials by tackling moisture buffering while ensuring the right chemical and mechanical properties to produce smart behaviour that in the future will feed the Building Management System.

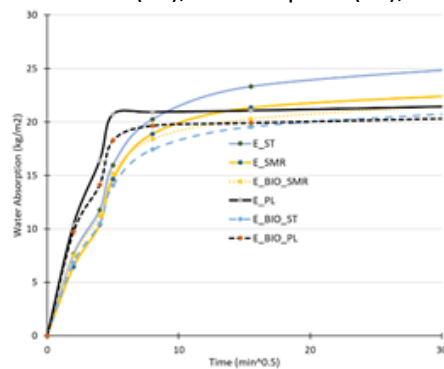
The research is focused in novel bio-composites formulations with agro-wastes exposed to aging phenomena and using bio-consolidation mechanisms to increase durability, minimising simultaneously the embodied energy and Global Warming Potential. Therefore, properties related to hygrothermal, mechanical and self-healing capacity of a sustainable bio-based mortar repair system for earth construction are presented here.

Currently, there is very scarce characterisation of materials that directly compare earth, bio-fibres and self-healing behaviour. The performance analysis of hygrothermal and structural behaviour gives an indication of the performance and durability of the samples. Beside this, using hygroscopic materials with well-controlled ventilation systems can reduce heating and cooling energy consumption by up to 10% and 30%, respectively, when applying hygroscopic materials with well-controlled ventilation systems. Therefore, moisture buffering needs to be analysed for each composite as it can also reduce energy use through latent heat exchanges caused by the adsorption and desorption of water vapour also achieving a healthy level of humidity. Bio-based materials generally have good humidity buffering properties and are passive solutions made with a combination of earth, wood, oil palm stalks, cork and specific organic fibres, contribute to the stabilisation of fluctuations in relative humidity and maintain a healthy indoor environment quality (IEQ). Beside this, these materials present several other advantages: large availability, low mass densities, cell structures, low thermal conductivity, high sound absorption properties and no harmful effects on health.

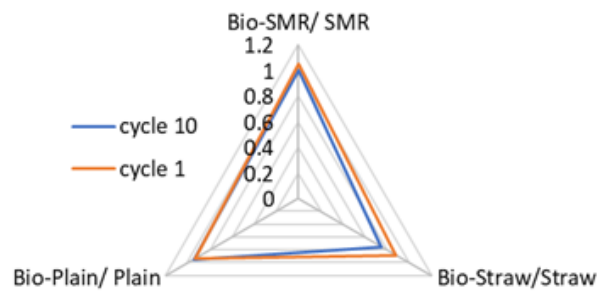
Prismatic and 0.1mx0.1m earth-based mortars with bio-fibres, with and without self-healing behaviour, were cast with plain mixes, saw mill residues (SMR), straw fibres, wool fibres and wood pellets. The bio-based agent added to the bio-based composite consists of bacteria. Processes of self-healing can be natural, biological or chemical. In these processes, different approaches have been designed and assessed by researchers [1][2]. The focus here is on biological self-healing processes, where these are based on biological precipitation of microorganisms (bacteria or fungi) resulting in the filling of cracks and voids. Factors such as pH, temperature and moisture content are crucial for the survival of the microorganisms [3].

Mix-designs were casted with and without self-healing behaviour, resulting in samples to test flexural and compressive strength, capillary, porosity, pH and the Moisture Buffering behaviour (MBV). Figure 1 presents the water absorption via capillary of earth-based mortars with straw (ST), SMR, plain (PL) with and without the bio-agent. Figure 2 presents the MBV

ratio between earth-based mortars with and without the bio-agent and the following bio-fibres: straw (ST), SMR or plain (PL), after 1 and 10 cycles (1 cycle=24h).



**Figure 1: Water absorption via capillary of earth-based mortars with straw (ST), SMR, plain (PL) with and without the bio-agent.**



**Figure 2: MBV ratio between mortars with and without the bio-agent for earth-based mortars with: straw (ST), SMR, plain (PL), after 1 and 10 cycles.**

Adding bio-based agent to an earth matrix with SMR, straw and even plain compositions has promising rules for hygric properties, as initial water absorption rate tends to reduce. MBV values range between 0.8 and 2.3 g/(m<sup>2</sup> %RH), where straw composites presents the best performance. Adsorption and absorption rates seem to be correlated, where water adsorption tends to reduce from 1 to 10 days for all samples with the bio-agent.

Pores connected with cracks allow the flow of aggressive agents that can trigger further pathological phenomena compromising the durability of the materials. The environmental and economic impacts could be reduced by using durable composite materials with effective self-healing capacity. Concurrently, the built environment looks to use bio-based construction solutions for repair/retrofitting to boost sustainability [4].

Compressive strength is enhanced at least 15% when the bio-product is used in earth-based mortars. Therefore, mortars where agro-wastes are combined with bio-product show a better performance. For the incoming years, the self-healing technics should work in the feasibility and the cost effectiveness of their use, to make them attractive for the industry, reducing also the CO<sub>2</sub> emissions.

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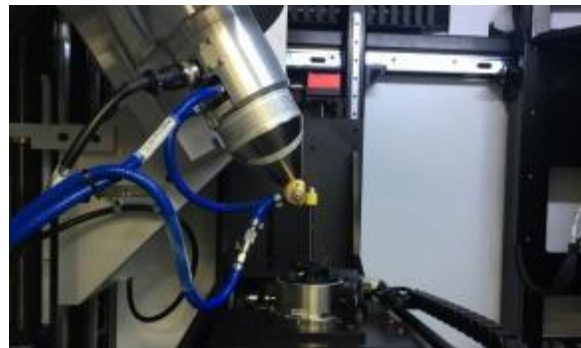
# **X-ray microcomputed tomography of self-healing in-ground barrier materials incorporating microcapsules**

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## **INTRODUCTION**

The main advantage of micro-CT ( $\mu$ CT) is the ability to perform 3D imaging in a non-destructive way. It returns a 3D distribution of the local linear attenuation coefficient of the material, and specialized rendering software allows for visual inspection of this 3D volume. The most common lab-based setup is the standard cone-beam  $\mu$ CT. In this setup, the conical X-ray beam makes geometrical magnification possible by positioning the object under investigation at any position between the X-ray source and the detector (Fig. 1).

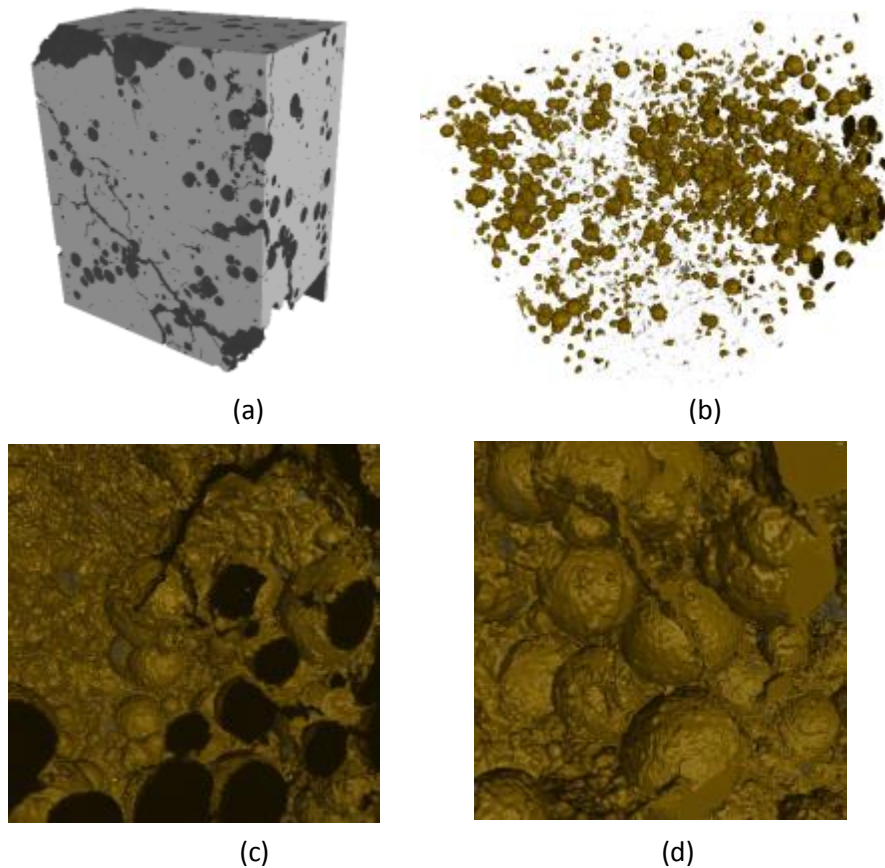


**Figure 1: Setup of the  $\mu$ CT test**

## **MICRO-CT ANALYSIS**

In this study, the  $\mu$ CT is applied to in-situ monitor the crack-triggered rupture of microcapsules in the in-ground barrier materials. Thies microcapsules, manufactured by Thies Technology Inc with polyurea as the shell material and sodium silicate as the cargo material were adopted. Thies microcapsules were added in cement grout, which was then mixed with kaolin clay to produce clay-cement in-ground barrier material samples. A sample with the size of 10×10×10 mm was cracked using the servohydraulic compression frame after 28-day curing. The sample was then tested by a micro-CT device (Nikon XT H 225 ST).

The rupture of microcapsules triggered by cracking is revealed by the reconstruction of CT scan results. After compression, several cracks can be observed on the cross section in Figure 2(a), and most cracks passed through several microcapsules and triggered the shell rupture. The microcapsules can be extracted from the clay-cement matrix based on the grey value, and Fig. 2(b) shows the fairly uniform dispersion of microcapsules in the clay-cement sample, which increases the chance of propagating cracks passing through microcapsules. The roughness of the shell material can be seen by zooming in on the microcapsule and such surface texture can enhance the adhesion to the clay-cement matrix (Fig 2c-d).



**Figure 2: CT scan 3D image reconstruction results of (a) clay-cement matrix incorporating microcapsules; (b) extraction of embedded microcapsules; (c,d) crack-triggered rupture of microcapsules**

## CHALLENGE AND FUTURE WORK

In practice,  $\mu$ CT is often used as a quantitative tool in addition to qualitative analysis and the complex 3D quantitative analysis can be prone to some errors caused by effects including image noise, voxel size, discretization and imaging artefacts. One of the main limitations of  $\mu$ CT in this study is the lowest achievable voxel size compared to the size of the clay-cement sample. Another problem is the operator dependency of the image analysis. Due to the variation in sample size, shape and composition, as well as the tube voltage and current, no fixed and generally accepted protocols exist for  $\mu$ CT scanning of cementitious materials. This means that many parameters can be chosen arbitrarily for image analysis such as the grey value threshold and void diameter threshold, leading to less accurate results of image segmentation and recognition of cracks and microcapsules.

In conclusion,  $\mu$ CT is a promising non-destructive tool for the investigation of the rupture of microcapsules triggered by cracking and subsequent self-healing processes, and such analysis can improve the understanding of self-healing mechanism and the assessment of the performance of different self-healing approaches. Future work on the development of a standard and reliable protocol of  $\mu$ CT analysis is needed to obtain convincing visual results to examine the healing efficacy of the microcapsule-based self-healing system.

## ACKNOWLEDGEMENT

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# Effect of Addition of Pre-Soaked Expanded Perlite on Self-Healing Performance of Cementitious Composites

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## OBJECTIVES

The current study includes the effects of addition of pre-soaked expanded perlite aggregates on the microcracking formation and self-healing performance of engineered cementitious composites (ECCs). For this purpose, ECC mixtures were produced with different ratios of pre-soaked expanded perlite aggregates, portland cement, fly ash, silica sand), poly-vinyl-alcohol (PVA) fibers and high water reducing admixture. Together with the basic mechanical property characterization of sound ECCs, self-healing tests performed on specimens included the investigations of the flexural strength, deflection capacity, ductility ratio, initial stiffness and rapid chloride permeability measurements before and after self-healing occurrence. Moreover, microscopic observations were made on microcracks to monitor the changes in the cracks. The results indicated that average microcrack widths decreased with the increase of the pre-soaked expanded perlite aggregate content and the number of microcracks decreased with the curing time. For the future, it is planned to reduce the production cost of ECC mixtures by improving the self-healing performance of expanded perlite aggregates added to ECCs.

## INSTRUCTIONS

The construction sector has a significant place in sustainable development of countries. Although the cost of building design and materials is cheaper at the first sight, the wrong design, mechanical and environmental loading effects cause the need of repair and retrofitting over time. Associated with these needs, the ultimate cost of structures increases dramatically, even to levels predetermined during the initial design stage. This case is an important obstacle in the construction of sustainable buildings. To counteract these negative effects and lower/eliminate repeated repair applications, ECCs which are characterized with the strain-hardening response have been developed to take advantage of high ductility and confront durability issues [1]. Strain-hardening response of ECCs comes along with the steady-state multiple microcracking formation. Furthermore, the average widths of these cracks in ECCs remain in the range of 100  $\mu\text{m}$  or less [2] enabling the occurrence of pronounced autogenous self-healing. Moreover, the superior ductility of ECCs allows it to be sustainable, with long service life and high durability [3]. The main aim of the current study is to investigate the self-healing performance of ECCs incorporated with pre-soaked expanded perlite aggregates (PS-EPA). The motivation was not only to use PS-EPA as internal water reservoirs for triggering the autogenous self-healing but also using them as flaws in dense microstructures of ECCs to better restrict the widths of microcracks by lowering the matrix fracture toughness values. Preliminary results clearly indicated that addition of PS-EPA at different ratios instead of silica sand successfully lowered the matrix fracture toughness values which further lowered the widths of microcracks and helped more pronounced autogenous self-healing (Figure 1). The author of current research believes that utilization of perlite in ECCs for different purposes (i.e. tailoring microcracking behavior, shrinkage properties and autogenous self-healing) rather than only lowering the overall weight of the materials will pave the way for new lines on innovative ECC-related research. This is also important for proposing new ways of using perlite



aggregate in cementitious systems considering the fact Turkey possesses the 74% of world's perlite reserves. [4].

Mixture	(a) Before air curing (28 days)	(b) After air curing (28+30 days)
ECC_0		
ECC_10		
ECC_20		
ECC_30		

Figure 1: Self-healing of ECC microcracks containing water-saturated expanded perlite aggregate

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## Modelling autogenous healing of High-Performance Fibre-Reinforced Cementitious Composites (HPFRCCs) via a discrete model

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The ongoing Ph.D. research is part of the project ReSHEALience (Horizon 2020), which has the aim to develop an Ultra High Durability Concrete (UHDC) and a Durability Assessment-based Design (DAD) methodology for structures, to improve durability and predict their long-term performance under Extremely Aggressive Exposures (EAE), i.e. chloride induced corrosion and chemical attack.

The improvement will be supported upgrading Ultra High Performance Fibre Reinforced Concrete with new functionalities for marine structures and infrastructures and for geothermal/biomass energy plants. This goal will be achieved, among the other activities, through the development of a theoretical model to evaluate ageing and degradation of UHDC structures in EAE extending the modelling to predict the lifespan. The model development will be part of a new durability-based design approach for the use of UHDC, aimed at assessing the structures durability and Life Cycle Analysis, to achieve an increase service life of 30%, and a long-term reduction of maintenance costs. The modelling activity will be validated against an extensive experimental campaign performed in the framework of the project and will be tested through long-term monitoring in six full-scale proofs of concept that UHDC in real conditions has the expected enhancement in durability.

Looking closer and specifically at the PhD research program, in last months the activity has been totally focused on the modelling part of the project ReSHEALience, which has the purpose to assess aging and degradation of UHDC and predict its service-life under EAE conditions. A multi-physics model that links the effects of chemical reactions, transport of ions, diffusion of moisture and heat, in cracked and un-cracked concrete conditions, corrosion and damage initiation and propagation, has to be formulated and implemented in a finite element code. Furthermore, not only macroscale models for chemo-diffusion and corrosion arise in the ReSHEALience project model-chain, but also a discrete mechanical model at mesoscale, rooted in Lattice Discrete Particle Model for Fibre Reinforced Concrete [1].

The developed numerical tool, which will take into account both the aggressiveness of the environment and self-healing of UHDC, is required to be employed not only to support the experimental results of the project but also to simulate the behaviour of real structures in service conditions.

The autogenous healing, thus, represents one of the aspects which must be evaluated and implemented into the model to make it reliable and suitable to reach the fixed goals.

Talking of the self-healing, this capability of cement-based materials, in recent times, has gathered the increasing interest of concrete professionals and scientific community. As demonstrated by many authors over the decades since its discovery in 1836, the autogenous healing of concrete would lead to a considerable recovery of physical and mechanical properties, once damage occurred. Referring to HPFRCCs, self-healing is expected to perform even better, as fibre reinforcement is usually one of the strategies to make standard concrete's autogenous healing reliable and predictable, which would be randomly scattered otherwise. Thus, HPFRCCs are inherently prone to experience such phenomena because of their mixture constituents.

The recent SMM (Solidification-Microprestress-Microplane model M4) model for concrete, which makes use of a modified microplane model M4 and the solidification-microprestress

theory, is able to reproduce the concrete behaviour, e.g. creep, shrinkage, thermal deformation, aging, and cracking from early age up to several years. The moisture and heat fields, as well as the hydration degree, are obtained from the solution of a hygro-thermo-chemical problem which is coupled with the SMM model. This numerical framework needs to be extended to incorporate the self-healing effects and, in particular, the effect of delayed cement hydration, which is the main cause of the self-healing for young concrete. The coming update model will be also able to simulate the effects of cracking on the permeability and the opposite restoring effect of the self-healing on the mechanical constitutive law, i.e. the microplane model [2]. Once defined the numerical model, a validation against experimental data will be needed: the reference experimental results are expected to be provided by means of four-point-bending tests on HPFRCC specimens, performed up to controlled crack opening and up to failure, respectively before and after exposure to different conditioning environments. Then, the recovery of stiffness, ductility and load bearing capacity will be evaluated [2, 3].

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## Advanced surface methods for the preventive repair of concrete structures

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### OBJECTIVES

The ongoing research proposed for the author's doctoral thesis deals with the development of more durable surface treatments, based on the penetration of nanomaterial dispersion through the concrete pores. This type of treatments aim to consolidate the cementitious matrix of the treated substrate with compatible products such as nanosilica dispersions [1,2]. But considering other types of nanoparticles, such as nanotitania or nanoalumina, different properties can be incorporated in the existing substrate, such as photocatalytic ability or resistance against fire [3,4].

The doctoral thesis proposal involves the production of ad-hoc nanoparticles dispersions, in order to assess the influence of significant parameters, such as the nanoparticle size distribution, the nanoparticle concentration, the nanoparticle activity,... on the effectiveness of the treatments on consolidating the treated surface and/or improving other properties such as the photocatalytic ability or the resistance against fire. [5]

But also the development of sensors for monitoring the effectiveness on the treatment penetration through the concrete pores and on improving the resistance against the penetration of water and/or aggressive agents is considered.

The doctoral thesis is starting and then only some preliminary results can be shown. The first results have been obtained in the framework of the STSM of Ruben Beltrán in the lab of Prof. Emilija Fidanchevski at the Faculty of Technology and Metallurgy (Skopje). During this STSM, the nanoparticles were obtained through different sol-gel synthesis methods and different mechanical activations (Agate mortar, Attritor mill and planetary mill) to later characterizing them, dispersing them and obtaining the final products for applying as surface treatments.

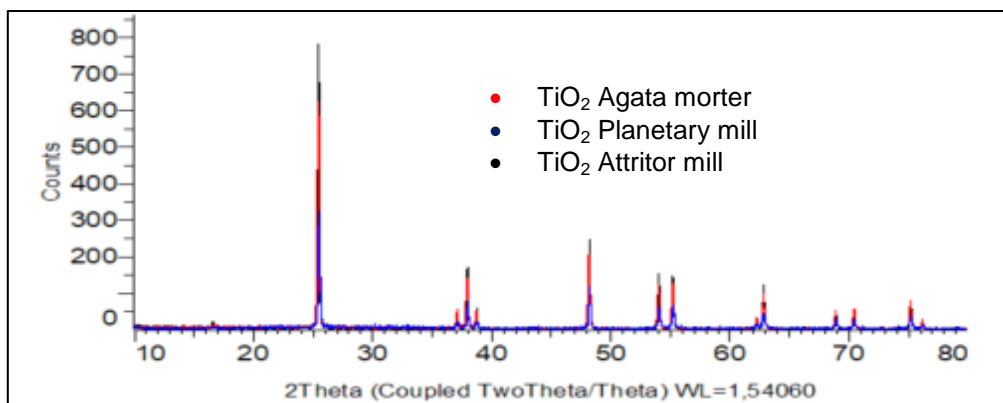
Currently, the design and implementation of resistivity sensing arrays for monitoring the penetration of the treatment are being carried out. An integrated system for the remote monitoring of the electrical resistivity has been produced and is being calibrated on laboratory samples.

The next step will be a further STSM of Ruben Beltrán (expected at February 2019) at the lab of Prof. Jonjaua Ranogajec (Faculty of Technology, University of Novi Sad, Serbia), the colloidal dispersions for applying as surface treatments will be obtained.

### Some preliminary results

In this first part developed in Macedonia, the synthesis of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{TiO}_2$  were carried out by the sol-gel method. Subsequently, part of these samples was calcined to check in the future how this parameter can affect the behaviour of surface treatments in the base-cement material. In addition, samples were also prepared to which different mechanical activations were made. For the commercial  $\text{TiO}_2$  in the Rutile and Anatase phase, the Agate mortar, Attritor mill and planetary mill; for the commercial  $\text{SiO}_2$  the Attritor mill and for the commercial  $\text{Al}_2\text{O}_3$  the Attritor mill. In total, 14 different types of powder were obtained among the 3 compounds.

In the first results obtained in DRX of the  $\text{TiO}_2$  in the Anatase phase, it can be observed that it is the same compound with small variations in intensity peaks depending on the mechanical activation chosen. Figure 1.



**Figure 3: Different mechanical activations of  $\text{TiO}_2$  Anatase. Comparison.**

Later, the characterization of each of the compounds will be completed and it will be seen how after dispersing them and applying them to the surfaces of base-cement materials they affect them.

### How should the repair topic improve during the next years to ensure its success?

The development of repair treatments with improved durability and improved properties is essential for incorporating actual concepts such as sustainability and raw materials optimization to the construction industry in the case of existing structures. Nowadays, the ageing of the civil infrastructures is occurring in the worldwide, and huge inversions for repairing processes are made by the different countries. In this sense, it is highly important to develop treatments highly compatible with the treated substrate, guaranteeing longer service life for the reparations, but also able of adapting the existing constructions to the actual requirements of the modern societies, mainly focussed on the citizens' wellness. In this sense, incorporating added functionalities such as photocatalytic activity or resistance against fire appear as promising alternatives.

But also registering "the history" of the repair is highly interesting for improving the repair topic during the next years. The development of sensors able to inform about the effectiveness of the repair treatment and about any fail occurring along the service life of the repair structure is a powerful tool to ensure the success of the repair methods in the next years.

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## Self-healing in soft soils improved with geopolymers

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### ABSTRACT

Sustainability and the negative impact of waste on our environment requires elimination of waste through reducing consumption, reuse, recycling of all waste and industrial by-products. Natural resources can be conserved when waste/by-product materials are used as full or partial replacement materials to virgin raw material. In the Geotechnical field, the use of green binders, such as geopolymers binders, on the improvement of the strength and stiffness of soft soils instead of the Portland cement have been recently studied [1,2,3]. Concerning the self-healing in improved soils, it is a subject that only very recently started to be studied through the use of bacteria or biopolymers [4,5].

Geopolymer binders are materials with cementitious properties, obtained from the dissolution of materials rich in silica ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) in a chemical activator solutions with high pH. A wide range of products can be derived from industrial by-products depending on materials composition, production processes and technologies. Fly ash (FA) with low calcium (Ca) content are an example of a by-product material frequently used in this type of binders. Different reasons led to the use of FA, one of them it is the pozzolanic nature that greatly improve the strength and durability. In addition, many researchers have shown that FA is an ideal component for the production of geopolymers due to the high content of aluminosilicate, which is an essential raw material that can be activated with an alkaline activator to achieve the geopolymer process. The use of this type of binders presents several advantages, mainly in the environmental point of view, since they allow a reduction on the carbon dioxide ( $\text{CO}_2$ ) emissions associated to the production of Portland cement, the reduction of wastes on landfills, the conservation of natural resources and the reintroduction of wastes in the economy.

Geopolymers can exhibit a wide variety of properties. All these properties, such as shrinkage, fast or slow setting, compressive strength, acid resistance, fire resistance, thermal conductivity and other characteristics depend on the type of raw material used. Also, the self-healing capacities of this kind of materials have been investigated. In fact, in its microstructure, a geopolymer has unreacted particles that are likely to react by applying some process (e.g. curing temperature or other) that can result in the production of additional gel that may confer self-healing properties to the material. The motivation for developing these self-healing cementitious materials is therefore, to achieve materials with improved durability and service life and reduced lifetime costs, both in terms of money and environmental impact.

Only few studies have been published concerning the improvement of soils with geopolymers binders but the results are very promising. This is one of the main goals of my PhD thesis. In this context, the evaluation of the self-healing characteristics of geopolymers used in the context of soil improvement would be very innovative and important to address. In particular, how the geopolymers characteristics can provide self-healing properties to improved soils in terms of shear strength and deformability? The challenge to address in the SARCOS workshop is how to design an experimental campaign in order to evaluate this self-healing characteristics in soft soils provided by geopolymers added to improved its mechanical characteristics. By

using conventional or special triaxial tests? Which stress paths should be adopted? The use of dynamic methods, like bender elements piezoelectric transducers, would be a good measure of self-healing in the material? Chemical tests like SEM, XRD-EDS and others should be of any use? At what curing age should the tests be carried out?

The answer to these and other questions would be of great support in the design of an experimental campaign to bring light to this very innovative and subject. Even though geopolymers are not cement based materials, as an alternative eco-friendly binder, the interest of their self-healing properties is indubitable even in the sense that they could serve as a mean of comparison with the self-healing characteristics of the cement based materials.

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## Alkali Activated Self-healing Concrete Structures

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### OBJECTIVES

With this research abstract I hereby would like to state my motivation to attend “SARCOS PhD Meeting on Self-healing Concrete Structures” organized by University of Novi Sad – Faculty of Technology in Novi Sad, Serbia 7 – 8 March 2019.

The PhD meeting theme “Self-healing Concrete Structures” is of a special relevance to my professional goals and career objectives, and therefore I would highly appreciate to be given an opportunity to attend this meeting gathering keen PhD students and Early Career Investigators from all over the Europe.

My research topic is “Alkali-activated self-healing concrete structures”. It is known fact, that appearance of small cracks in building materials is unavoidable, not necessarily causing a risk of collapse for the structure, but certainly accelerating its degradation and diminishing the service life and sustainability of constructions. The self-healing phenomenon is an important aspect for construction technology which prolongs the service life of infrastructures. The key notion of this concept is that minor damage in concrete structures is not an issue as long as it is counteracted by a subsequent autonomous process or removing or healing the structural damage. The potential of mineral precipitating bacteria for crack remediation and durability improvement have been thoroughly investigated in different studies.

In my research self-healing of alkali activated concretes (AAC) are tested. Proposed AACs can be used as alternative to concrete, and have emerged as solution to overcome the problem of massive usage of natural raw materials and possible way to reduce CO<sub>2</sub> emissions. Useful utilization of the industrial waste (such as fly ash used for AAC production) is much more attractive alternative than to deposit it on the waste sites. Recycling of industrial waste into new materials reduces possible pollution of the environment and decreases the costs of waste deposits, as well as saves resources of manufacturing of new materials and building units.

My research tries to respond to two issues: industrial waste and by-product usage in the production of alkali-activated concretes (AAC), therefore minimizing usage of non-renewable natural resource and application of genetically-modified bacteria for eco-friendly engineering and true self-healing process of constructions to enhance prolonged service life of infrastructures.

I think that self-healing/repair topic should progress in the field of alternative materials used for load-bearing constructions (for example, alkali-activated concrete made from different by-products and waste materials) to ensure their success in the future. To demonstrate a new approach of self-healing process in alkali activated concrete with ameliorated structural properties and true self-healing activity by genetically-enriched spore-forming bacteria towards sustainable and green construction technology.

Personally, as a special added value of this PhD meeting I find the opportunity to network with other students and professionals from the Europe, which is an exceptional opportunity for

scientific experience exchange in the field of SARCOS. This meeting will highly improve my personal development as a young scientist interested in the SARCOS field.

As requested in the Call for Application I am forwarding to you my CV and recommendation letter from my supervisor in Riga Technical University Prof. Dr. Sc. Ing. Diana Bajare.

I am hopeful you will find my application as a worthwhile investment. My attendance at this training is a wise investment and I would highly appreciate to be one of the selected participants.

Sincerely,  
Laura Dembovska



## Self healing in high strength concrete with super absorbent polymer (SAP)

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After the second half of the twentieth century, high strength concrete (HSC) was developed and began to be used in large scale. In order to produce the HSC it is necessary to reduce the water/binder ratio (w/b) and achieve an adequate workability to cast the concrete. To do so, it is needed an adequate granulometry of the aggregate, the use of mineral additions and super plasticizers. This way, an internal dense and refined structure of the concrete is produced. However, due to rapid development of porous and fine networks within the cement paste, which generates a higher capillary tension results in a significantly larger autogenous shrinkage, causing the concrete to crack and can compromise its structural integrity. To mitigate this effect, a technique has already been successfully employed, the use of superabsorbent polymers (SAP) in the concrete, it promoting the inner cure of the HSC [1].

SAP has an intrinsic property of absorbing moisture and fluids from the environment, it is a hydrogel that can increase up to 500 times its size, retaining the liquid in its structure without dissolving. There are some promising pioneering studies suggesting SAP as a concrete healing agent, which there are two mechanisms: the blocking of cracks by the swelling of the SAP (sealing) and filling of the cracks with hydrates from the hydration of the anhydrous cement (self-healing) through the SAP acting as curing agent. The most extended work that investigated the concept was developed by [2] that demonstrated the self-healing of the material. It is believed that the internal healing promoted by the SAP, is able to self-heal the cracks in the concrete forming new gels of CSH and calcium carbonate.

However, the sealing and healing phenomena, depends of the size of the crack, the implementation of reinforcement is necessary, so the cracks remain in an acceptable size for self-healing to occur. Several techniques and methods have been developed to perform crack inspection, they are often inaccessible or invisible, such as in underground structures and infrastructure such as bridges and highways [1]. This kind of application is the target of this research, in this study is proposed to investigate the thin cracks formed just after the concrete crack, since self healing would be favourable in this situation, because there would be cracks that can not be repaired or be easily visualized, though this cracks compromise the structure of the concrete. It is not known if SAP can enhance the self healing of the concrete, however it is acknowledged that these cracks can self heal without SAP, it is needed time and the sealing effect is not achieved.

Although there are studies showing that the use of SAP as a self-healing agent is efficient, there is a gap if the use of SAP used in high-strength concrete to mitigate autogenous shrinkage also has the potential for self-healing. The studies so far have been carried out on concrete of normal (50MPa) or low (20 MPa) strength classes and the commercial SAPs used are not intended to mitigate autogenous shrinkage.

### 1.1 Research question

"What degree of healing does a superabsorbent polymer with the function of mitigating autogenous shrinkage achieve in a high-strength concrete?"

### 1.2 Hypothesis

If SAP mitigates autogenous shrinkage by minimizing the phenomenon of self-desiccation by promoting internal cure of concrete, it would also be able to improve the hydration of the anhydrous cements present in the high-strength concrete and to heal cracks when they occur, outside and slowly releasing into the concrete.

### 1.3 Objectives

The general objective proposed for this work is to analyse the self-healing phenomenon of a high strength concrete promoted by the use of superabsorbent polymers used to mitigate autogenous shrinkage. The focus is to verify the effectiveness of self-healing and sealing of cracks, as well as the optimal amount of SAP needed to promote healing. It is expected that the dosage of the cementitious composite can be used in practical applications and numerical modelling. For this purpose, the following specific objectives were proposed:

- Develop a trace of high strength concrete reinforced with steel bars, and additions of various quantities of superabsorbent polymers. For this, it will be necessary to verify the workability and to evaluate the mechanical properties, so that, even with the addition of the SAP, the cementitious composite remains high strength.
- To quantify the self-healing capacity of the HSC represented by three recovery indicators: mechanical (measuring the load recovery index); durability (determining the index of recovery of durability) and geometric (determining the index of closure of the crack).

The study is divided in four stages: pilot study (determine the highest amount of SAP in the mixture still being HSC; verification of healing by resistance gain; verification of healing by the recovery of the durability properties and characterization of the healing products observing the closure of the crack, using DIC techniques.

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## Description of the method for observation the microstructure of mineral binders in the state of stress

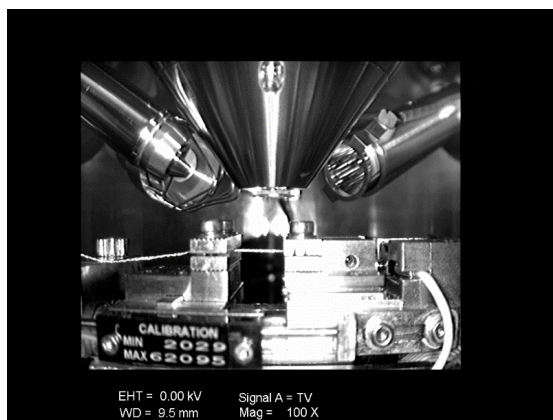
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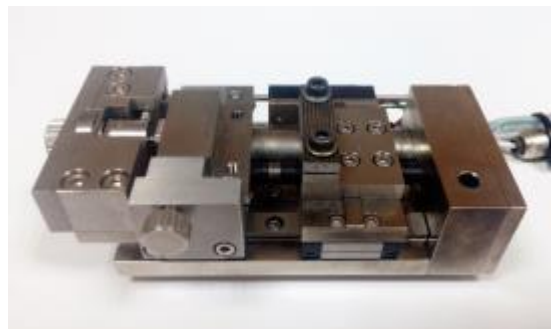
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The article describes the results of observation of changes in the microstructure of mineral binders modified with PET and PP polymer fibers subjected to tensile and compressive stresses. The tests were performed using a Zeiss EVO MA 10 scanning microscope equipped with SE, VPSE and BSD detectors and a Bruker EDFL XFLASH 6/30 detector and also a Deben MICROTTEST tensile stage with a maximum load of 200 N. Samples of the tested materials were prepared in the form of 10 cm length tiles , 2 cm wide and 0.5 cm thick. The research involved the same fibers as well as the matrix itself. The aim of the conducted research was to determine the value of permissible stresses preceding the destruction of the tested materials and to determine the destructive force.

The research results presented in the article are preliminary tests that enable the assessment of the possibility of using a stress table (mounted in the scanning microscope chamber) in tests of selected mechanical features of self-healing concrete. On the basis of preliminary tests, the possibility and usefulness of simultaneous observation of microstructure and changes occurring in materials after application of an increasing load was confirmed. The figures below show the image of the EVO MA 10 scanning microscope chamber and the mounted tensile stage used in the above-mentioned tests.



**Figure 4: Tensile stage in scanning microscope chamber**



**Figure 2: Tensile stage**

## **Innovative SuperAbsorbent Polymers for crack mitigation and increased service life of concrete structures**

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Most of the deteriorating mechanisms acting on concrete structures are related to the ingress of aggressive agents inside the structures. Even before reaching its hardened state, a cement-based composite is subjected to the formation of cracks especially due to the effects of shrinkage during the early ages. The formed porosity of the material can become the perfect path for the ingress of those aggressive agents. After the crack formation the water intrusion cause a drop in the pH of the concrete that can lead to steel corrosion; the ingress of chlorides causes the de-passivation of the protective film, the intrusion of CO<sub>2</sub> can cause carbonation and both processes can accelerate the corrosion.

Recent research has shown that superabsorbent polymers (SAPs) can be used to reduce crack formation due to autogenous shrinkage by means of internal curing. Besides that, this material has been considered of great interest in the study of smart self-healing materials where the promotion of self-sealing is also investigated [1-5].

Superabsorbent polymers (or hydrogels) are a natural or synthetic water-insoluble 3D network of polymeric chains cross-linked by chemical or physical bonding. They possess the ability to take up a significant amount of fluids from the environment (in amounts up to 500 times their own weight).

In the industry side contractors are searching for a way to decrease shrinkage cracks and to obtain a watertight structure. This is especially important for tunnel elements, underground parking garages, basements, liquid containing structures, pavements, etc. Nowadays, contractors are often forced to apply crack repair right after construction, due to the formation of shrinkage and thermal cracks at early age. The shrinkage could be overcome by using SAPs as they may provide internal curing to the construction element: they absorb water in the fresh concrete mix, and provide it to the cement particles at the right moment in the hydration process when they need it, in this way reducing the autogenous shrinkage. In hardened concrete, they may seal occurring cracks, as they swell in contact with intruding water. This will lead to more watertight structures. The SAPs will subsequently promote autogenous healing of the crack since they provide water for further hydration of unhydrated cement particles and calcium carbonate precipitation, leading to even more tight structures and possible regain of the mechanical properties.

In this context, the project named as "iSAP - Innovative SuperAbsorbent Polymers for crack mitigation and increased service life of concrete structures" aims to reduce shrinkage cracks by means of internal curing resulting from the addition of superabsorbent polymers. In addition, the construction elements will possess self-sealing and self-healing characteristics. In that way, the service life of the concrete structure will be prolonged.

Different polymers (commercial available and in-house produced) are being tested to verify the potential for mitigation of autogenous shrinkage in cement pastes and concretes and also to evaluate their influence in the fresh (setting time, workability, air content) and hardened states (mechanical strength). The self-sealing effect is currently being studied by means of water permeability tests with cracked and uncracked concrete specimens subjects to low and

high pressure water flow. Two commercial polymers have been selected for further testing with concrete while the in-house polymers still need to be optimized.

The next stage of the research will be to test those polymers with regards to the self-healing effect in order to achieve an optimal combination of different polymers that can lead to internal curing, self-sealing and self-healing.

So far the main challenges being faced for the self-sealing testing and that should be tackled again during the self-healing studies are based on the techniques from preparing cracked specimens in a controlled way. Most of the times, achieving an specific range in the crack widths can be an exhaustive part of the work, resulting many times in the loss of a considerable amount of test specimens.

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# On water tightness and crack closing in self-healing concrete

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## INTRODUCTION

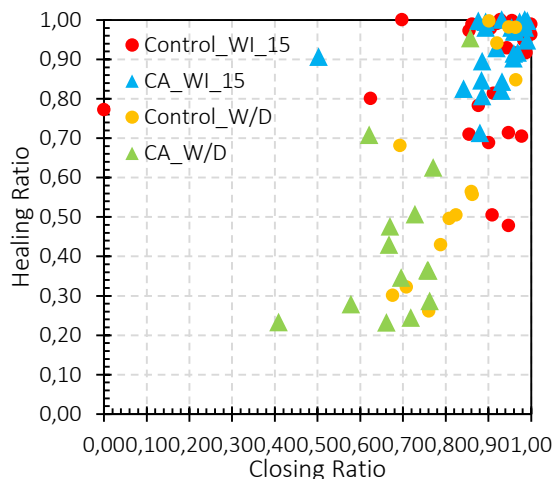
Concrete has a natural self-healing capability to seal small cracks, commonly referred to as autogenous healing. This capability is mainly produced by continuing hydration and carbonation, which are very limited in efficiency. It has been reported that it is able to close visually cracks, and for early age cracks (2-3 days), this crack closing is mainly produced during the first 28 days healing underwater [1, 2]. Several concrete designs and self-healing agents have been proposed to improve self-healing [3], such as crystalline admixtures (CA), which are permeability reducing admixtures thought to provide self-healing properties. The lack of knowledge on CA and the non-standardized methods to evaluate self-healing in concrete [4] complicate the analysis of the different healing products and the evaluation of self-healing.

## VISUAL CRACK CLOSING vs. WATER TIGHTNESS

This work shows the experimental results obtained in terms of early age (3 days) crack closing and recovery of water tightness (method described in [2, 5]) in concrete samples with and without 4% of crystalline admixtures (concrete composition in Table 1). After 42 days healing underwater (WI\_15) and under wet/dry cycles conditions (W/D), the results show that crack closing was generally higher than the recovery of water tightness (Figure 1), especially under wet/dry cycles conditions (W/D). The presence of CA led to a better relation between the two properties. Thus, it can be inferred that crack closing not always corresponds directly with an improvement of water tightness.

**Table 1. Concrete mix used in the experimental work.**

Material (kg/m <sup>3</sup> )	w/c=0.45	
	Control	CA Concrete
Cement II/A-L 42.5 R	350	350
Water	157.5	157.5
Gravel (4-12 mm)	950	959
Natural sand	899	875
Steel fibers	40	40
Limestone powder	50	36
Crystalline Admixture	-	14
<b>Avg Slump (cm)</b>	<b>13</b>	<b>16</b>
<b>Avg Compressive str. (MPa)</b>	<b>55</b>	<b>61</b>



**Figure 1. Healing Ratio vs Closing Ratio for control and CA specimens [4].**

## **SURFACE vs. INTERNAL HEALING**

To evaluate if autogenous healing and self-healing promoted by CA was being produced internally or only on the external surface of concrete samples, two strategies were followed: 1) to verify the presence of precipitates in the internal crack after cutting specimens transversally to the crack with a saw, and 2) removing 1 cm of the upper part of concrete samples, which showed the deposits closing the cracks, and repeating water permeability tests.

The first test showed that the crystal precipitation occurred only up to 0.5-1 cm depth, however, the cut process could also have detached the crystals inside the crack, if any. For the second strategy, internal water permeability was tested in 26 specimens precracked up to 0.35 mm at the age of 2 days, put to heal at the age of 3 days and were let to heal for 6 months under water immersion at 30°C, to ensure complete (or almost) healing in terms of water permeability. After cutting the external disks with the healed crack and performing water permeability test again, water was able to flow as through open cracks, reducing Healing Ratios from 1.00 to 0.50-0.90.

## **CONCLUSIONS AND FURTHER LINES OF RESEARCH**

The results shown in this work indicate that high efficiencies obtained in terms of crack closing do not translate directly to similar improvements in terms of water tightness recovery. This could also happen when analysing the correlation between other durability properties, and thus, further investigations are needed to evaluate the self-healing consequences in concrete. In addition, despite the improvement measured in terms of water tightness, the phenomenon reported was to a great extent a surface process, with internal cracks remaining open and permeable. These results are of great importance in order to predict the durability properties of concrete elements where self-healing is involved or expected and therefore, future lines of research should cover these points to ensure the safety of self-healing concrete elements.

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# Crack healing of glass fibre reinforced concrete using crystalline admixtures

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## OBJECTIVES

Self-healing methods of concrete is many and can be used on based on goals and technologic possibilities. Glass fibre reinforced concrete (GRC), which is used in experiments is sprayed and during spraying fibre and concrete is mixed together. One of GRC uses usually is facade material which is, 12-18 mm thin. Different types of racks usually appear on the face of panels or on fixing points due to different lodes like wind lode, long term lode and impact lode. Some of these cracks are only aesthetic like on face of the panel and some can be structural, like those that appeared on fixing points. Self-healing techniques that can be used for sprayed GRC is limited by nozzle size of spray gun and mixing technique. Most suitable method is to use crystalline admixture. Crystalline admixture also has dual positive influence – crack healing and permeability reduction ability. Crystalline admixture reacts with cement and forms calcium silicate hydrates. These additives also help to reduce concrete porosity and water permeability [1]. Difference between crystalline additives which are permeability reducing admixtures and hydrophobic additives are - crystalline additives are hydrophilic nature [2]. Due to Belie et. al. [3] crystalline admixtures performance is largely influenced by mix design. Better effect can be noticed with HPFRC. But still healing process for 0,3 mm width cracks is relatively slow from few weeks to several months.

In this research Aalborg white cement and 1% of crystalline admixture from SIKKA is used. Samples were made and pre-cracked after 7 and 28 days using four-point bending test. Samples were healed in water and outside environment where temperature fluctuated between +5°C and -10°C, and humidity were above 85%. Sample crack size were measured by microscope after pre-cracking and after healing process. During research different challenges must be overcome. Some of them is pre-crack sample crack size deviation reducing, crack size that can be healed and healing capacity out side water environment. In conclusion samples which were held in water could heal wider cracks comparing samples in outside environment after the same time.

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## Influence of TiO<sub>2</sub> nano-sized particles solutions on self-healing properties of concrete structures

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TiO<sub>2</sub> nanoparticles usually have been used as cement coatings according to their ability to break down dirt or pollution and then allow it to be washed off by rain water. Up to now we have worked on design of coatings with photocatalytic and self-cleaning properties which were applied on three types of substrates: non-porous, porous and highly porous. The research was focused on investigation of four systems where nano TiO<sub>2</sub> (3 and 10 wt.%) was impregnated onto different mineral carriers (synthesized Zn-Al layered double hydroxides (Zn-Al LDH) and three different types of natural clay). The impregnation of the nano TiO<sub>2</sub> onto Zn-Al LDH carrier was accomplished by vacuum evaporation prior to mechanical activation (planetary mill) and only by mechanical activation (realized in an attritor). The impregnation of nano TiO<sub>2</sub> onto natural clays was realized only by mechanical activation, but in two different mills: planetary mill and attritor.

The obtained composite powders (ZnAl LDH-TiO<sub>2</sub> and clays-TiO<sub>2</sub>) after impregnation were characterized from the aspect of granulometric composition, mineralogical composition, photocatalytic activity and particle morphology. The composite powders that showed the greatest photocatalytic activity, which was in the direct correlation with the way of impregnation, the content of nano TiO<sub>2</sub>, particle size distribution and their crystallinity, were further used for the coatings preparation. The prepared coatings were applied onto mineral substrates with different porosity and then examined from the aspect of photocatalytic activity, self-cleaning and durability.

Surface properties (roughness, hydrophilicity and micro-hardness) and functional properties (photocatalytic activity and self-cleaning efficiency) were studied in order to define the optimal formulation of the applied coatings. The effect of the photocatalytic behavior of the coated substrates in terms of self-cleaning ability was assessed by photodegradation of Rhodamine B, performed before and after durability tests. The obtained results showed that photocatalytic activity of the applied coatings generally depends on the procedure of the TiO<sub>2</sub> impregnation onto/into the carrier and the loaded TiO<sub>2</sub> content.

The durability of the photocatalytically active coatings was tested through modified rinsing procedure and adhesion tests.

The purpose of future work is based on our earlier experience; properties of the designed TiO<sub>2</sub> coatings and their compatibility with a cement hardened substrate. The further work is going to estimate the self-healing functionality in order to seal the already formed cracks in the hardened concrete. The obtained TiO<sub>2</sub> forms will be impregnated into the inorganic cementitious matrix according to one of the aforementioned procedures and characterized by the aspect of granulometric composition, mineral composition, and particle morphology. TiO<sub>2</sub> nanoparticles, by possessing many unique features, will significantly improve the functionality and durability of the repaired concrete materials by accelerating the formation of calcium-silicate-hydrate (C-S-H) gel into the cementitious matrix. It is expected that the newly formed system will improve mechanical and physical properties of the cement/s TiO<sub>2</sub> system, including shrinkage, creep, porosity, permeability and elasticity.

**Key words:** self-healing, TiO<sub>2</sub> nanoparticles, concrete structure

## Engineered Cementitious Composites designed with particle size optimization

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Fiber reinforced high-performance cementitious composites possess high damage tolerance and durability compared to other types of cementitious composites for their significantly higher deformability under direct and indirect tensile stresses [1-3]. Engineered Cementitious Composites (ECC) which is a special type of fiber reinforced cementitious composites, can exhibit enhanced autogenous self-healing properties arising from the mutual effect of multiple cracking behavior with micron-sized cracks and abundant unhydrated binders available in their composition [4-5]. In this study, ECC mixtures are designed and produced with local materials and the proportions of the ECC ingredients are determined by using Andreasen and Andersen particle size optimization model to achieve a densely packed and compacted structure. Mechanical and self-healing performance of the produced composites are investigated and compared with the conventional ECC mixtures. As mechanical properties compressive and flexural strength values are determined at 7, 28 and 90 days. In addition, self-healing performance are evaluated in terms of flexural strength and deflection capacities under four-point bending. Nondestructive methods such as rapid chloride ion penetration test, electrical bulk resistivity, and sorptivity are also used to reveal the self-healing. In addition, crack closure rates are determined by using micrographs of the cracks obtained throughout the experimental program.

ECC is normally produced with quartz sand with size range of 0-200  $\mu\text{m}$ . An ECC mixture is developed with the combination of 4 different aggregates with different size ranges by using modified Andreasen and Andersen particle size optimization method to obtain a closely packed matrix.

According to the model [6] target particle size distribution (PSD) curve is obtained with the equation (1):

$$P(D) = \frac{D^q - D_{\min}^q}{D_{\max}^q - D_{\min}^q} \quad (1)$$

Where,  $P(D)$  is the ratio of the particles smaller than  $D$  in total solid materials,  $D_{\min}$  is the minimum particle size,  $D_{\max}$  is the maximum particle size and  $q$  is the distribution module.

After the development of the target PSD curve, the optimum combination of the available aggregates is determined to obtain the closest fit to target curve. In our case the optimum aggregate distribution is obtained for the volumetric ratios obtained 27.2%, 11.7% and 61.1% for 0,45  $\mu\text{m}$ , 0-200  $\mu\text{m}$  and 100-300  $\mu\text{m}$  sizes, respectively for a distribution modulus of 0.53.

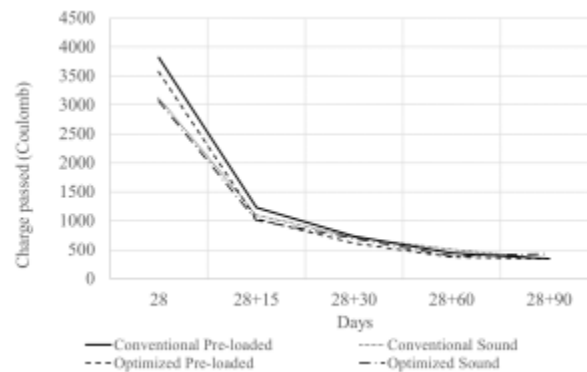
Self-healing behaviours of the conventional ECC with quartz sand of 0-200  $\mu\text{m}$  and the optimized ECC with the combination of sands are determined. For this purpose, puck specimens of  $\varnothing 100 \times 200$  mm are produced to be used in Rapid Chloride Ion Penetration (RCP) test and beam specimens of  $360 \times 50 \times 75$  mm are prepared for Ultrasonic Pulse Velocity (UPV) tests. 28 days after casting, specimens are preloaded. Up to 90 days after the preloading UPV (Table 1) and the electrical charge (Figure 1) values are determined for both the virgin and preloaded specimens.

**Table 1. UPV test results**

UPV (m/s)	28	28+15	28+30	28+60	28+90
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	days	days	days	days	days
<b>Conventional Sound</b>	4525	4597	4792	4923	5064
<b>Conventional Pre-loaded</b>	3871	4386	4563	4706	4839
<b>Optimized Sound</b>	4447	4750	4827	4901	5128
<b>Optimized Pre-loaded</b>	4147	4392	4492	4561	5389



**Figure 1. RCP test results**

The packing density and self-healing properties of the mixture produced by using modified Andreasen and Andersen particle size optimization are found to enhance the self-healing behaviour.

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# Preventive repair of concrete substrates using epoxy resin coatings

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## ABSTRACT

In the growing construction industry, especially in the field of large area floors, there is a need to ensure a suitable floor for more significant loads. Among many methods which can prepare floor on chemical compound aggression and mechanical loads, epoxy resin is commonly used as a coating layer. Recently, this method has developed as an alternative preventive repair method [1] in contrast to limited pure concrete which can be only grinded or brushed. The concrete substrate without protection layer can be easily destroyed during normal exploitation. Epoxy resin coating layer has the properties which extend the floor life. This usually allows a satisfactory adhesion of the concrete substrate to be obtained [2, 3]. During designing floor anyone is analysing thermal shock influence on the coating. Overheated of the coating occurs when the protecting layer has low strength adhesion and the floor is exposure to forklift trucks mechanical loads.

To provide preventive repair for concrete substrate, in this study the concrete substrate surface was textured by many methods before applying epoxy resin. Pull-off test results show that adhesion properties of the coating layer can be increased which has additional impact on improving thermal shock resistance. Epoxy resin needs specially prepared concrete substrate surface before applying. But manufacturers recommended methods are labor-intensive and expensive. Moreover, there is a higher probability of failure during preparing concrete substrate surface. In the authors' opinion there is a need to search for a way to avoid these steps during the construction process of epoxy resin floors or try to find an alternative preventive repair method for concrete substrate care.

This research is dedicated a study conducted to evaluate the effect of texturing of the surface of concrete substrate on its adhesion to epoxy resin. The study investigates a total of seventeen types of textures: after grooving, imprinting, patch grabbing and brushing. The texture of the surface of the concrete substrate was prepared during the first 15 minutes after pouring fresh concrete into molds. The epoxy resin was laid after 28 days on hardened concrete substrates. To investigate the adhesion of the epoxy resin to the concrete substrate, the pull-off method was used. The results were compared with the results obtained for a sample prepared by grinding, normative minimal pull-off adhesion values and the values declared by the manufacturer. During this study twelve out of fifteen tested samples achieved a pull-off adhesion higher than 1.50 MPa. It was found that one of the imprinting texturing methods was especially beneficial. This preventive repair method shows that there are a lot of ways to minimize concrete substrate surface prepare work process with additional bigger pull-off strength. But there is a need to overcome some technological problems in texturing process for the best texturing method types or to find alternative texturing method which can be easily and commonly used.

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# Self-Healing Performance of Cementitious Composites Under the Continuously Increased Permanent Mechanical Loads

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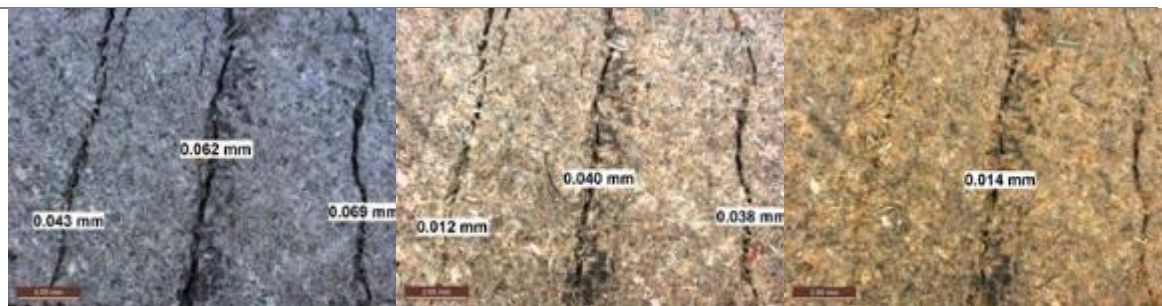
## OBJECTIVES

This study focuses on the determination of self-healing performance of Engineered Cementitious Composites (ECCs) under continuously increasing permanent mechanical loads. For the purposes of the study ECC mixtures were produced with different pozzolanic materials (e.g. Class-F fly ash [ECC\_FA] and ground granulated blast furnace slag [ECC\_GGBFS]). Autogenous self-healing evaluation was made on 180-day-old specimens subjected to continuously increased sustained mechanical loading and further cured for 150 days. In addition to mechanical property characterization of ECCs, self-healing assessment was made in terms of changes in flexural properties, ultrasonic pulse velocity (UPV) measurements and microcrack measurements. Results revealed that ECC\_FA samples showed better performance under continuously increasing mechanical loads compared to ECC\_GGBFS samples. Results of the current study are believed to present more conservative self-healing performance of late-age ECC specimens subjected to constant and continuously increasing mechanical loading conditions.

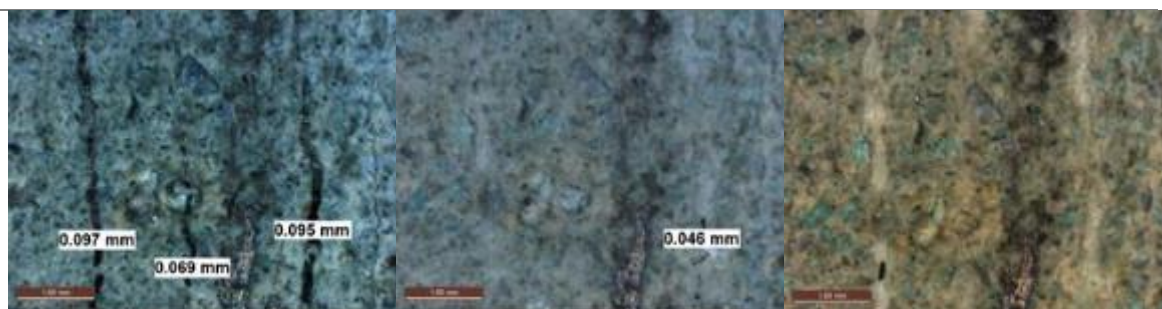
## INSTRUCTIONS

Although the service life for infrastructures is at least 75 years according to European standards, it has been observed that many buildings have been damaged and deteriorated after 20 or 30 years due to mechanical impacts and environmental conditions that they have been exposed to throughout their lives. One of the reasons for this short-lived performance of infrastructures is high brittleness of conventional concrete. Engineered Cementitious Composites (ECCs) are one of special materials that counteract the high brittleness of conventional concrete materials through their increased ductility coupled with multiple micron-size microcracking occurrence [1,2]. However, although widths of cracks are relatively limited in ECCs, concrete materials are always under constant stress in real-life that means crack widths will be larger than most of self-healing studies which tests the specimens in the unloaded state that causes certain amount of crack closure upon load removal. To simulate real-life conditions in a more rigorous way, self-healing performance of ECCs was therefore measured under increasing sustained loading conditions. Moreover, 180-day-old specimens were used for the analyses so that an understanding of late-age self-healing capability of cementitious composites could be developed. The results of the study showed that the fly ash containing composites had a higher rate of healing compared to composites containing slag under continuously increasing loads (Figure 1). Moreover, results revealed that self-healing performance of medium-age composites can be quite high. As another testing method of autogenous self-healing capability of ECCs, UPV testing was found to be inadequate to precisely capture the changes in microcracks as a result of self-healing occurrence upon further water curing.

### ECC\_FA



### ECC\_GGBFS



(a) 180 days

(b) 180+30 days

(c) 180+90 days

**Figure 1.** The appearance of microcracks surfaces after continuous water curing

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## A biomimetic design of vascular network in self-healing cementitious materials

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### ABSTRACT

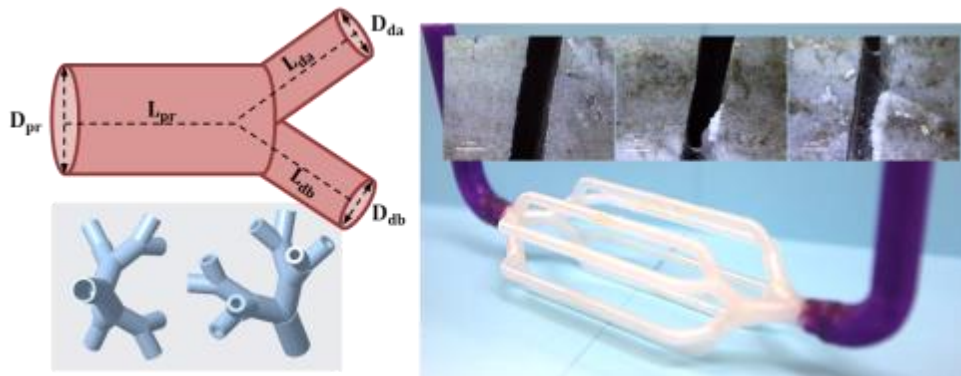
Most of our infrastructure are made out of cement-based materials. However, they all crack no matter how carefully being stored or reinforced. The repairment of those damaged infrastructure leads to significant cost. Self-healing in cementitious materials is an intrinsic process to some extent (called autogenous healing) as it relates to the nature of the cement components. However, autogenous healing is limited by the degree of damage – the crack needs to be roughly below 200  $\mu\text{m}$ . To address the limitations functional engineered additions are being developed and embedded in the cementitious matrix (autonomic healing). Encapsulation-aided healing has been one of the most popular and successful approaches based on the release of limited quantities of stored healing agent from embedded reservoirs such as polymer capsules<sup>[1-3]</sup>, glass capsules<sup>[4,5]</sup>, ceramic capsules<sup>[6]</sup> and mineral capsules<sup>[7]</sup>. However, this technique has difficulties in ensuring long-term service life and repeated damage repair due to the uncertainty of capsules breaking and limitation in providing enough healing agents for large cracks.

Vascular networks are becoming an immersing technique of achieving resilient infrastructure. A vascular system houses the healing agents within its hollow channels or interconnected networks which are incorporated within the cement matrix. And is the only type of self-healing methods that has the capability to address different scales of damage in cementitious materials.

To date, previous research on vascular mainly focused on developing individual hollow tubes<sup>[6,8]</sup> or gridded networks<sup>[9]</sup> for self-healing cementitious material. These designs have the limitation on the healing area and sensitivity of diagnosing cracks.

The main aim of this work is to develop a novel vascular network inspired by nature for self-healing in cementitious systems. To achieve this, a biomimetic vascular network was designed and generated following Murray's Law for circulatory blood volume transfer. The designed structures were constructed through 3D printing and assessed in cement-based matrix. Mechanical testing assessed the compatibility of the system with the surrounding matrix as well as the functionality of the network in delivering and releasing healing agents at the location of damage. This initial proof of concept work confirmed the ability of this system to deliver healing after multiple damage events (Figure 1).





*Figure 5: Self-healing vascular network design and crack healing process in 7 days*

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# Self-healing capacity evaluation of UHPFRC with Crystalline Admixtures and Nanoparticles by means of Permeability Tests

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## INTRODUCTION

Self-healing is the capacity of a material to repair itself autogenously from a damage. Materials with this feature could give a chance in the civil engineering field, improving durability in structures and reducing ordinary maintenance operations over the years. A generic concrete, under load, always suffers cracks due to its low tensile strength. These cracks are not so relevant from the mechanical point of view, but they can allow the entry of aggressive chemical agents that start the degradation process inside the element. Under the same load, UHPFRC has evinced a better behaviour than traditional concretes in terms of deformations and cracks opening, showing lower deformations due to its high resistance and smaller cracks, in the range of  $0,02 \div 0,04$  mm, for this reason it turns out to be the most suitable to evaluate self-healing.

## EXPERIMENTAL PHASE

This MSc's thesis is being performed in a collaboration between *Politecnico di Milano* and *Universitat Politècnica de València*. The purpose of this work is to evaluate the self-healing capacity of Ultra High-Performance Fibre-Reinforced Concrete (UHPFRC) using Crystalline Admixtures and Nanoparticles, and compared to traditional and high performance concrete types. In order to promote self-healing capacity in UHPFRC different additives have been used in the mix: Penetron Admix, Cellulose Nano-Crystal (CNC) and Cellulose Nano-Fibres (CNF). Additionally, Alumina Nano-Fibres (Nafen), have also been added to UHPFRC to study their effect in a better crack control, and thus, self-healing of the cracks produced. by means of permeability tests.

Different type of concrete with different resistance values were manufactured: H0 - C25/30, H1 - C80/90, HB3 - C150, HB4 - C135. In total, 14 different dosages were fabricated adding to the basic dosages of UHPFRC (HB3 and HB4) different concentrations of crystalline admixtures and / or nanoparticles. Each concrete batch had 4 concrete beams of 100x150x750 mm with  $\varnothing 8$  lower reinforcing bars and  $\varnothing 6$  stirrups that will be pre-cracked at different cracking levels (in total 56 beams). Together with the production of the beams, to characterize them, every time were fabricated 4 cubes of 100x100x100 mm to test the compressive strength at 7 and 28 days and 2 smaller prisms of 100x100x500 mm to perform the four-point bending test at 28 days and the invers analysis.

The second step of the experimental phase will be to perform a pre-crack in the prisms through four-point bending test, targeting 2 different crack levels:

- small crack or crack closed, reached during loading in central high level of the DEMEC, with a residual crack  $< 0,05$  mm for no fibers concretes and  $< 0,05$  for UHPFRC, and the last,
- large cracks, with a residual crack after unloading at the reinforced level of  $0,1 \div 0,2$  mm for no fibers concretes and  $1 \div 2$  for UHPFRC.

Once the specimen is cracked, using a Microscope and a Crack Width Meter is possible to perform a cracks analysis and a map of them, to know the exact number of cracks in the MEDEC's area, their actual position and above all their size.

For the preparation of the samples for the permeability tests, smaller samples will be sawed from the concrete beams. In this way, 4 specimens with the same cracking level (small or big crack) and 2 specimens with no cracks will be extracted from one beam end, where no cracks were produced.

At the age of 28 days, all the specimens will be sawed in a size of 150x150x50 mm, and afterwards will be subjected to the water permeability test, carried out putting a specimen under a water pressure of 2 bar for 5 minutes and weighting the amount of water that is able to pass through the specimen (gH<sub>2</sub>O/min). The preliminary results, as expected, experimentally confirmed that the bigger is the crack, the higher is the water flow which can pass through it. Consequently, if a crack closes after the healing process, the water flow should diminish.

After the water permeability test, half of the cracked specimens will be subjected to the chloride permeability test to calculate the chloride depth penetration (mm). Preliminary tests have been performed to verify the procedure using a PVC tube, with a height of 60 cm and outer diameter of 75 mm, glued to the specimen with a high-performance sealant (Sikaflex). The tube is filled with salt water with a concentration of 35 g NaCl/l and every tube is filled for a total height of 50 cm. The other half of the cracked specimens will be directly submerged in salt water for 1/2 months to self-heal. Spent their time in the tank, they will be tested at chloride permeability to evaluate whether cracks have been closed in terms of chloride penetration, and if this is confirmed by the results of the tests compared to the previous ones.

The purpose of this work is to provide insights in the self-healing reactions produced in UHPFRC, with several additions, and how this self-healing is translated to a water tightness recovery and chloride penetration protection.

## ACKNOWLEDGEMENT

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# **Synergistic strategies for the simultaneous extension of service life and maximisation of waste incorporation for ultra sustainable marine concrete structures**

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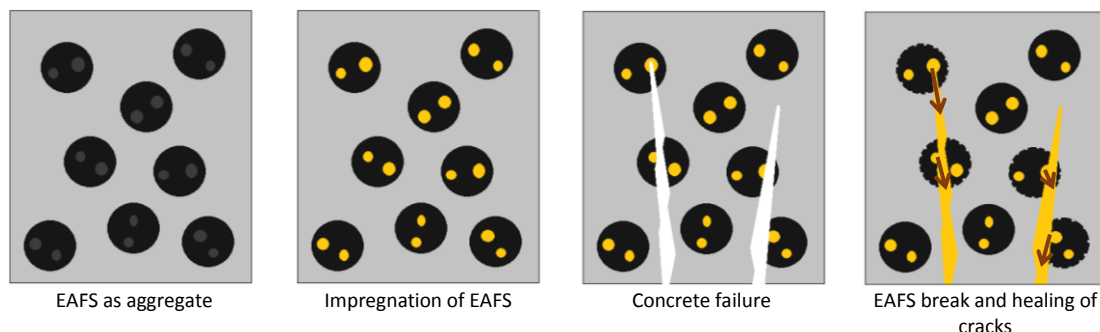
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## **ABSTRACT**

In order to drastically increase the sustainability of marine and other concrete structures, this study proposes the development of research in order to simultaneously reduce the resource intensiveness associated to their fabrication and maintenance, as well as by drastically extending their service life. In the specific case of marine environments, the concrete structures are exposed to several physical and chemical interactions that affect its strength and stiffness leading to the beginning of a cracking process that contribute to their premature deterioration. This deterioration is due mainly to the alkali-silica reaction, attack by sulphates and the penetration of the chlorides that induce corrosion of the reinforcement steel bars, which added to the strong loading and abrasive effects, resulting in further cracking and disintegration [1-2]. The resolution of these problems, which are recognized as urgent, will be pursued by maximising the incorporation of wastes in concrete design, whether as aggregates, fibres or even as part of innovative hybrid binder systems. The extension of service life will be further enhanced by promoting the intrinsic self-healing processes, as well as through the engineering of these processes in order to maximise stiffness and strength recovery after induced damage events. These developments will be put forward by searching for synergistic strategies that multiply benefits and will have as main supporting technology the extreme cracking control and outstanding tensile toughness typical of ECCs (Engineered Cementitious Composites) and UHPFRCC (Ultra High Performance Fibre Reinforced Cementitious Composites).

To accomplish these objectives the research method is divided into three phases. The first phase intends to development alternative binder systems incorporating significant amounts of wastes or recycled materials, such as fly ash (FA) and electric arc furnace slag (EAFS) powder. Considering that these alternative binder systems may present several challenges considering the durability performance and the ability to promote self-healing mechanisms, hybrid binder systems also containing Portland cement and other cements will be considered in the design process. The second phase concerns the incorporation of waste materials in the UHPFRCC compositions, whether as aggregates (electric arc furnace slag) or fibres (for example, recycled tyre steel fibres). To improve the UHPFRCC's sustainability, the design mixture will be performed with seawater instead of tap water, since 96.5% of the existing water in the earth is in the seas and oceans [3]. Particle packing methods such as the Andreasen&Andersen model, or the procedures used to optimize fluid concrete mixtures such as the self-compacting concrete, will be employed to design the innovative and sustainable UHPFRCCs. The third phase comprises the characterization and further development of the self-healing mechanisms in the UHPFRCC performed. A combination of strategies to achieve the final goal is object of intense research. These strategies encompass the incorporation of fibres to restrict the crack width, as well as the addition of agents to improve hydration and crystallization (FA and EAFS powder). By taking advantage of the high porosity of electric arc furnace slag (EAFS), one of the strategies to develop will be based on the impregnation of this industrial by-product with magnesium oxide (MgO), sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) or water to promote crack healing, either

by the ongoing hydration promoted or by providing additional water (Figure 1). The impregnation can be applied in EAFS powder or EAFS as an aggregate, through the use of the vacuum technique, which will allow a more efficient absorption [4-5]. Although these techniques seem promising, several risks are identified, and additional strategies need to be considered at this stage.



**Figure 6: EAFS impregnation to promote the self-healing of marine concrete structures**

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## ACKNOWLEDGEMENT

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# Nano-scale Modified Cementitious Composites for Bio-inspired Self-healing Ability

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## OBJECTIVES

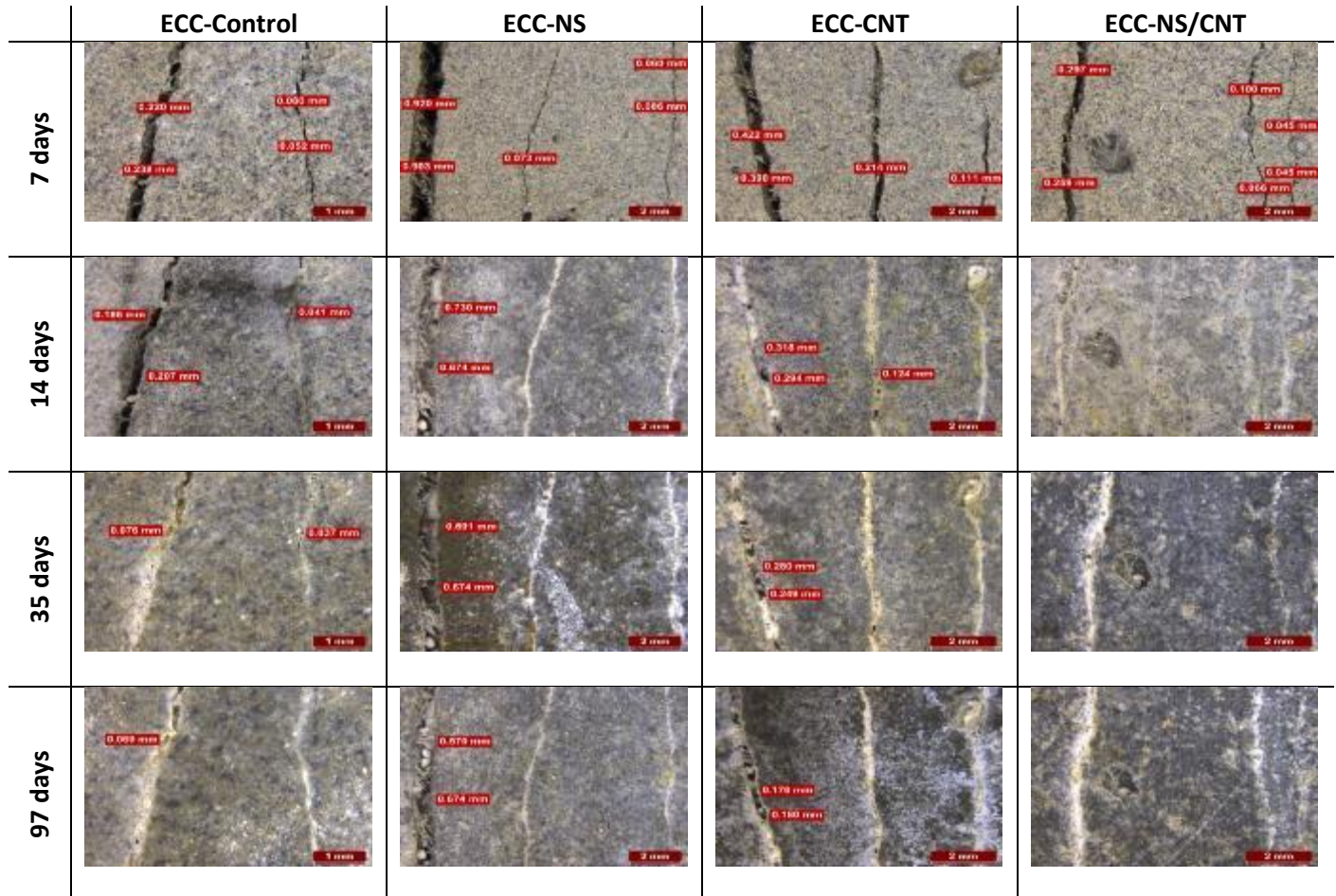
The study of author comprises the enhanced autogeneous self-healing properties of engineered cementitious composites (ECCs) to precisely monitor the damage and healing of cracks at the same time. To do this, nano silica (NS), carbon nanotubes (CNTs) and carbon fibers (CFs) were utilized in ECC mixtures. The self-healing of sound and pre-loaded specimens were assessed based on electrical resistivity (ER), rapid chloride permeability tests, resonance frequency and freeze and thaw cycles. Compressive and flexural tests of sound and preloaded specimens were determined with the investigation of their flexural strength, deflection capacity, ductility ratio, initial stiffness and energy dissipation capacity. Microstructural analysis (SEM-EDX, XRD, TGA-DTA) was also conducted to understand the self-healing compounds of healed cracks. The study indicated that enhanced mechanical recoveries and durability properties can be attained through robust self-healing with the tailoring of NS, CNTs and CFs in ECC mixtures. The improving of self-healing/repair during next years to ensure longevity of infrastructures is closely related to specifying inclusive round robin test system. As the accumulation of knowledge on the self-healing phenomenon has increased, reliable and accurate common test systems may provide beneficial results in real structures. Also, a precise evaluating life cycle of self-healing structures may provide new insights to stakeholders of construction industry such as companies, governmental bodies, trade associations. This may bring more self-healing oriented building design and applications so that construction industry can perform sustainable transformation in the forthcoming years.

## INSTRUCTIONS

Although structures are designed to serve approximately between 50-100 years in accordance with their types, they become out of service even before approaching their life span. Consequently, structures can need repair/retrofitting in some cases while reconstruction can be also required too. In this case, reconstruction makes the cost two-fold within service life while repair/retrofitting also become substantially costly with regard to its scale. From this point of view, Engineered Cementitious Composites (ECC) [1] have been developed in order to contribute for more sustainable construction industry in terms of economical and environmental issues. Thanks to such composites, it is possible to obtain composite behaviors that tolerate several mechanical loading, exhibit restrained crack development (mostly smaller than 100  $\mu\text{m}$ ) and damage-resistance. As it is known, existing cracks in concrete structures can have potential to close by healing products under the circumstances of limited crack widths. Existing cracks of concrete structures can be healed by various healing products (hydration of unhydrated cementitious materials, calcite formation, swelling in the edges of cracks etc.) [2] with self-healing concept. Recently, self-healing is one of the research area that is also intensively studied by the researchers [3]. The main idea of the author's study is to develop healing products by enhancing the quality and expedition at higher level thus exhibiting performance like a crack-free concrete with the integration of ECC mixtures and self-healing property similar to self-healing talent (bio-inspired) of living creatures. In the study, efficiency of self-healing products are improved by nano technology products [4] that have led new developments recently in many fields. Accumulation of knowledge in the thesis of the author may bring more development of self-healing mechanisms of nano modified cementitious composites and different aspects for discussions existing in the literature related to self-



healing. Also, it will be possible to observe the characteristic effects of nano scale materials on the mechanical and durability properties in cement-based systems along with triggering more stable self-healing mechanisms (Figure 1). The other novel subject investigated in the study is the characterization of self-healing products occurring at different ages. Investigation of self-healing robustness in different ages will provide novel findings for literature since investigation will offset the problem of unpredictable crack occurrence of concrete.



**Figure 1.** Representative photos of microcracks of different ECC specimens.

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## Synthesis and characterization of core/shell particles for developing self-healing mortars

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### OBJECTIVES

Self-healing mortars with encapsulated active particles have attracted the interest of both materials science and construction industry, as a promising approach for addressing issues of materials' degradation and reduction of structures' lifetime due to mechanical damage (cracks). This PhD aims at the development, synthesis and characterization of core/shell particles, as well as the evaluation of their healing ability when they are incorporated in traditional and modern mortars.

The first part of our research is focused on the synthesis of inorganic particles by modifying the Stöber method, in order to produce uniform silica particles in the micro/macro scale. Subsequently, sodium silicate and different types of expansive mineral admixtures will be integrated in the core of the silica particles for acting as healing-agents. The characterization of the particles synthesized is based on electron microscopy (SEM/TEM) and dynamic light scattering.

The second phase of the PhD research addresses the surface modification of the core/shell particles and their incorporation in the cement matrix. The aim of this phase is to optimize mixing parameters such as concentration, particles distribution and adhesion to the matrix, minimizing any potential defect on the binder.

Finally, the evaluation of microcapsules' healing efficiency after their incorporation into the cement matrix will be carried out. Strength recovery will be evaluated through controlled damage of the specimens and measuring of the compressive strength after the healing process. It is a fact that recovery of strength and durability is high when cracks and pores are blocked effectively with healing products. Furthermore, healing ability will be characterized by water permeability and water absorption tests.

The complexity of this system, as well as, the absence of sufficient experimental results is the reason why researchers have not yet successfully combine all the above parameters to achieve efficient sealing and strength recovery of concrete matrix.

So far, the results concern the parameters that affect the size and morphology of the formed silica particles, as well as the study of the methodology for the healing ability assessment.

Overall, the main goal of this research is to develop the appropriate methodology for producing core/shell microcapsules that will satisfy the above requirements and synthesize composite mortars with enhanced self-healing abilities.

### ACKNOWLEDGEMENT

The author acknowledge the support from EU COST Action CA 15202 "SARCOS" (<http://www.sarcos.enq.cam.ac.uk>). This PhD research has been co-financed by the European Union and Greece through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH – CREATE – INNOVATE (AKEISTHAI – Self-healing and self-sensing nano-composite conservation mortars, MIS 5031866).

## Self-healing Geomaterials

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### OBJECTIVES

This project aims to enhance biocement to enable autonomous self-healing following damage/deterioration. To date two studies have been undertaken to investigate the augmentation of microbially induced calcium carbonate precipitation (MICP) to enable self-healing of biocemented soil. Montoya and Dejong [1], and Botusharova [2] demonstrated the ability of MICP treated soil structures to self-heal, in principle, and provided evidence that dormant bacteria can survive within biocement and germinate upon damage to the encapsulating precipitate, to enable healing through calcium carbonate precipitation. The nutrients and precursor chemicals (cementation media) required for the healing process were injected into the biocement, following chemical or physical degradation. To enable a truly autonomic process, the cementation media will need to be delivered automatically, from within the soil matrix.

### METHODOLOGY

The use of carrier materials to immobilise cementation media within the biocement will be explored. Through a study of literature and evaluation of nutrient supply options; expanded perlite (Harborlite 800), diatomaceous earth (Celite S) and natural fibres (jute, coir) were selected as potential carrier materials. The suitability of these carriers for nutrient storage has been investigated by loading the carrier materials with concentrated cementation media consisting per litre of deionised water; Oxoid CM001 Nutrient Broth 20g, urea 133.33g, ammonium chloride 66.67g, sodium bicarbonate 14.13g, calcium chloride dihydrate 49g. 1g samples of carriers were soaked for 24 hours in the media and dried at 90°C between loadings. A drying temperature below 100°C was used to mitigate against depolymerisation of the lignin in the fibres and decomposition of the sodium bicarbonate. Nutrient release was explored by immersing loaded carrier materials in deionised water and standard cementation media. MICP has been tested in aqueous solutions using nutrients released from loaded carriers to determine if all cementation media constituents have been immobilised effectively.

To explore the effect of a nutrient loaded carrier material on the MICP process, twice loaded expanded perlite was mixed with silica sand. The sand had been washed in 3% HCL solution to remove carbonates and autoclave sterilised. This mixture was wet pluviated into 50ml centrifuge tubes containing one pore volume of bacteria and cementation media. Triplicates with and without carriers (controls) were prepared. 1mm holes were pierced in tube bases for vacuum extraction. Tubes were inverted in an incubator at 30°C between treatments. Supernatant was extracted on weekdays over a 14 day period. Use of a vacuum assisted extraction of supernatant, and percolation of media into the sand. Ureolytic *Sporosarcina ureae* bacteria will be used throughout this project. *S. ureae* was grown on LB agar amended with urea. Pure cultures were used to inoculate liquid broth and cultured in at 30°C, 150rpm. Vegetative cells were harvested for experiments by centrifugation at 3200rpm for 20minutes and washing with Phosphate Buffered Saline. Following this preliminary test apparatus is being prepared to produce biocement columns of diameter 38mm, length 70mm, connected to a peristaltic pump to supply and extract the media. Specimens produced will be subjected to unconfined compression testing, repeated following deterioration and healing. CaCO<sub>3</sub> content will be determined by mass loss on ignition. Miniature CPT is planned to be utilised as a means of determining uniformity of MICP.

## PRELIMINARY RESULTS

The capacity for immobilisation of the cementation media by the carrier materials was determined following three loadings, as shown in Figure 1a, followed by nutrient release in Figure 1b.

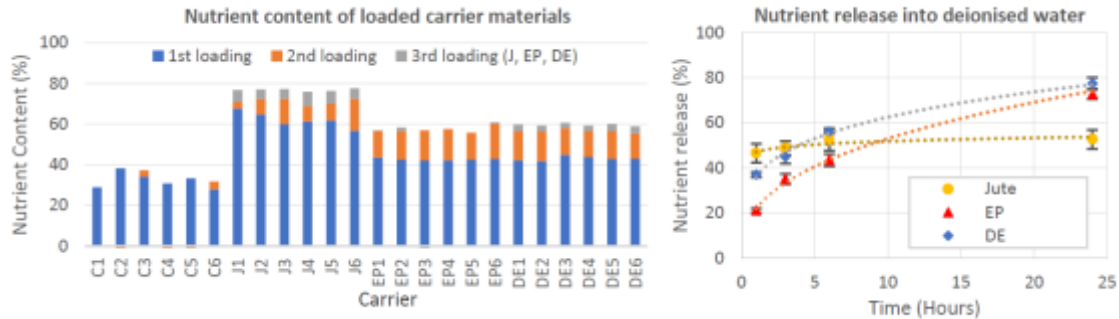


Figure 1: Immobilisation capacity of carriers (a) and nutrient release profiles (b)

Due to the relatively low immobilisation by Coir this wasn't used in further experimentation. The brittleness of Jute following immobilisation has led to subsequent studies with Hemp fibres, which are yielding favourable results.

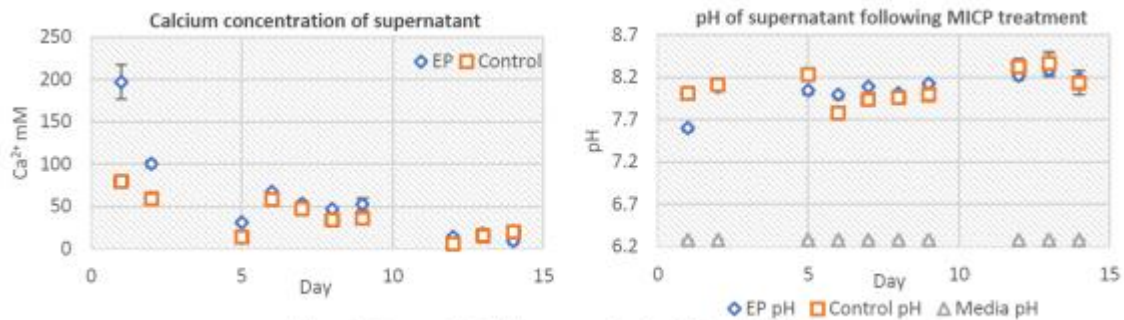


Figure 2: Supernatant calcium concentration (a) and pH (b)

The process of loading fibres and mixing with sand is to be explored further, fibres will potentially further enhance the strength and durability of the biocement. The loaded powders are easily separated after drying and can be evenly distributed within the sand using a pestle and mortar, for small samples. The loaded fibres clump together more firmly. It can be seen in Figure 2a that the elevated calcium ion levels due to leaching of media from carriers is more prominent following the first two treatments, with equilibrium seemingly having been reached soon after. Tests will be undertaken to determine healing resulting from the remaining immobilised media. Carrier nutrient retention needs to be improved for long-term healing. Using geopolymer coatings will be explored as a solution.

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# Overview of the research of laser assisted automate tape laying process for production of advanced composite materials for self-healing

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## OBJECTIVES

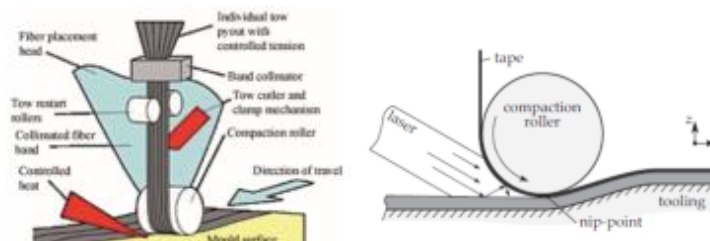
In the frame of my PhD thesis, the automated processes for the production of parts of composite materials will be investigated. Those composite parts can be used for different application as well as for self-healing of concrete structures. Focus in the PhD thesis is put on the automated fiber placement (AFP) technology and automate tape laying technology (ATL) and the equipment used to perform those technologies as well as production parameters.

The main purposes of the research are:

- Define the technological parameters of robotic AFP and ATL manufacturing processes (automatic tape laying) that will enable the production of a composite structure on the spot, without further processing into a traditional autoclave furnace or in a press (usually used).
- Comparison of manufactured composite structures using automated procedures with composite structures produced under the same conditions with traditional compression pressing procedure using a press.
- Demonstrate the advantages of applying robotic procedures for the production of composite structures.

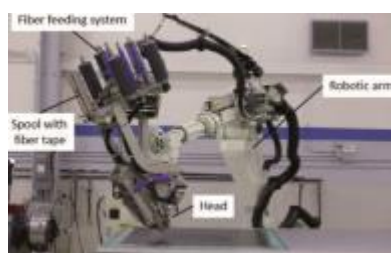
## INSTRUCTIONS

The essence of automated fiber placement (AFP) technology and automate tape laying technology (ATL) is to lay composite layer with 0,25" width unidirectional prepreg tape. Each single tape is laid down by a robotic system. Each layer can be laid with different orientation, which benefits a structure capable to carry load in their required direction. Each tape is pressed to the mould by a roller for proper compaction. The essence of this technology is shown in Figure 1 [1].



**Figure 1. AFP/ATL technology**

The AFP/ATL systems are typically individually suited for a particular application, however, each of those systems has a typical component, such as: head with compaction roller; fiber feeding system; robotic arm; control panel (fig. 2).



**Figure 2. AFP/ATL technology - components**

An important part of the whole system is control software. Typically, AFP/ATL producers provide a dedicated software together with the system. The software controls the robot motion and technology parameters like tape laying speed, compaction force, heat source temperature. The software can also analyse fiber direction and perform simulation. The system presented in Figure 2 is designed for small part manufacturing [2].

Commercially available AFP/ATL systems can work with 3 types of composite materials: thermoset prepreg ; thermoplastic prepreg; dry fiber (unsaturated). Each material is supplied on a standard spool as a unidirectional tape. The most common material system used for structure build are thermoset materials. New generation AFP/ATL systems are equipped with a laser heat source to allow thermoplastic materials processing [3].

A combination of system with thermoplastic materials with an aim to achieve in-situ parts fabrication can be very beneficial from the cost stand point. That technology has been used in industry for several years. Nowadays, it can be observed application of in-situ thermoplastic composite technology. The research is still being conducted to obtain a high material quality by means of using in-situ AFP/ATL technology with thermoplastic materials [3,4]. Thermoplastic composites have several advantages [7]: good damage tolerance properties; superior chemical resistance; non-limited storage time; recyclability. These advantages make thermoplastic composites a very interesting material for structures parts manufacturing, not only from the cost perspective but also from structural strength capability stand point. The main advantages of AFP/ATL system are: producibility; fiber direction accuracy, part to part repeatability; low amount of material waste. AFP/ATL systems have also several disadvantages and limitations. Typical limitations are related to the mould shape, compaction roller diameter, head geometry etc [2-4].

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## Screening of new alkalophilic sporogenic bacterial strains and evaluation of their biocalcification potential

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In recent decades, microbiologically induced carbonate precipitation (MICP) has been extensively studied, due to its application in various engineering practices. The bacteria with biocalcification potential receive more attention as they may be used for the healing of minor cracks in historical monuments, old buildings or for development of self-healing bioconcrete [1]. Microorganisms can influence the precipitation of carbonaceous minerals, specifically calcium carbonate ( $\text{CaCO}_3$ ) through metabolic processes such as organic acid utilisation, urea hydrolysis and denitrification [2]. The result of these metabolic processes is change in the pH and/or the concentration of dissolved inorganic deposit. It is believed that bacterial cell surfaces act as heterogeneous crystal nucleation sites in supersaturated matrix with  $\text{Ca}^{2+}$  and  $\text{CO}_3^{2-}$  ions. Moreover, microbes can alter the saturation state of an undersaturated solutions, by catalysing mineral precipitation [3]. Among all microorganisms from soils and sludges, bacteria with biocalcification potential are the one that can modify soil characteristics through MICP [4]. The aim of present study was isolation and characterization of alkalophilic sporogenic bacterial strains from different sources and investigation of their biocalcification activity.

The samples of soil, sludge and water were collected from various locations: the Danube river, the Danube-Tisa-Danube Canal (DTD), cement factory waste water, deposit of limestone near the Bešenovo lake, and the Beli Majdan cave in mountain Fruška gora. After sampling, the first step was investigation of the occurrence and diversity of alkalophilic sporogenic bacteria in the samples. The specific scheme of isolation is followed, in order to obtain only the growth of selected bacteria. In the selection step, only colonies with the same macromorphologic characteristics as referent strains (Table 1) were chosen for the further investigations.

Table 1. Referent cultures with bioprecipitation potential

Referent bacterial strain	Metabolic pathway	MICP reactions
<i>Sporosarcina pasteurii</i> DSM 33	ureolysis	$\text{CO}(\text{NH}_2)_2 + \text{H}_2\text{O} + \text{Ca}^{2+} + \text{Cell} \rightarrow 2\text{NH}_4^+ + \text{Cell-CaCO}_3$
<i>Pseudomonas stutzeri</i> ATCC 17588	denitrification	$\text{CH}_3\text{COO}^- + 2,6\text{H}^+ + 1,6\text{NO}_3^- \rightarrow 0,8\text{N}_2 + 2\text{CO}_2 + 2,8\text{H}_2\text{O}$ $\text{Ca} + \text{CO}_2 + 2\text{OH}^- \rightarrow \text{CaCO}_3 + \text{H}_2\text{O}$

A total of 74 isolates from the Danube river, 66 isolates from the Danube-Tisa-Danube canal and 43 from other sampling locations passed through complete isolation procedure. The isolated colonies were purified on modified R<sub>2</sub>A agar plates and characterised on the basis of Gram staining, KOH test, size, shape, mobility, ability to form endospores, catalase and oxidase test. Only a few bacterial strains showed ureolytic and/or denitrifying activity, indicating the presence of carbonate precipitating potential. The bioprecipitation was detected by rapid method of the addition of  $\text{CaCl}_2$  solution in Urea broth and by monitoring the bacterial growth on Urea- $\text{CaCl}_2$  agar. The occurrence of inorganic deposit into bacterial colonies was detected by stereomicroscopy and FTIR analysis (Figure 1).



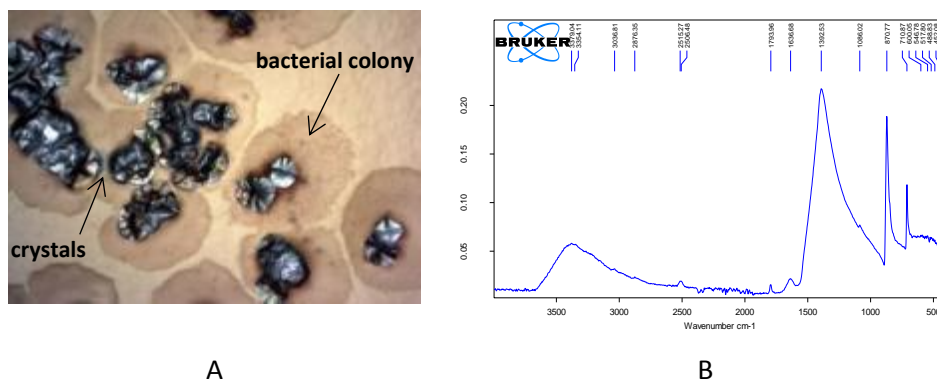


Figure 1. Analysis of inorganic deposits into new bacterial strain on Urea-CaCl<sub>2</sub> agar

A) Microscopic picture (x100); B) FTIR analysis

According to the available literature, environmental factors, such as temperature and pH value, have a great influence on the bacterial growth [5]. Toward this aim, the growth of the chosen bacterial strains was observed in a wide range of pH values (5 – 11) and temperatures (20 – 44 °C). For 95% isolated strains, an optimal pH value of the growth is between 9 and 11 units. The optimal temperature of the growth of 65% of isolated strains is 37 °C, while for the rest of isolates the optimal temperature is between 20 and 30 °C.

Based on the obtained results, it can be concluded that the tested bacterial strains have a great potential to be used self-healing agent for cementitious materials in different environmental conditions.

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## Self-healing materials for the repair of infrastructure

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### OBJECTIVES

Repair and maintenance of infrastructure is a global concern. In Europe, half of the annual construction budget is spent on repair and maintenance but still 50% of repairs fail prematurely [1]. In recent years, there has been an increasing interest in self-healing properties for the repair and maintenance of concrete structures [2][3] e.g. bio-based mortars for repair, and self-healing cementitious materials [4][5]. Self-healing mechanism can be naturally observed in cementitious materials, and further healing mechanisms can also be triggered by externally applied healing agents [6] [7]. Most research has focused on developing self-healing concretes, with later work developing cementitious repair mortars with self-healing properties [8][5]. However, a major problem with concrete repairs is maintaining an effective bond between the conventional concrete substrate and the externally-applied repair mortar, often caused by material incompatibility (e.g. the elastic modulus).

Research suggests that autogenous self-healing is most effective for small crack widths, with the maximum crack healable being 200µm [9]. Engineered cementitious composites (ECCs) are ultra-ductile fibre-reinforced cementitious composites based on the theory of micromechanics [10]. ECCs are characterised as having narrow crack widths [10], one of the fundamental properties for achieving improved efficiency of the autonomous self-healing mechanism [10]. Recently, research has examined the performance of strain-hardening cementitious composites with biological agents [4]. Until now however, there are limited studies regarding ECCs with healing agents for the repair and maintenance of concrete structure.

Self-healing repair materials have not dealt sufficiently with both the (in)compatibility and durability characteristics of the material and associated repair/substrate system. In addition, no previous studies have fully investigated the impact of different repair application methods such as hand applied, spraying, and/or pouring. Until recently, there has also been no reliable evidence on how the corrosion of steel reinforcement within the parent concrete could affect the self-healing performance - this will also be examined in the present study. The aim of this research will be to develop efficient cementitious self-healing materials for the repair of reinforced concrete. Objectives will include investigating:

- The mechanical and durability properties of the material, such as compressive and flexural strength, bond, permeability, and resistance to chloride ingress.
- How the self-healing properties influence the durability and lifespan of the repair material.
- The influence of different application methods (hand applied, sprayed and pouring) on self-healing performance.
- The influence of reinforcement corrosion and chloride ingress on self-healing performance.

The experimental program devised to fulfil the objectives above will involve mechanical characterisation (e.g. compressive strength, flexural strength, bond strength), microstructural

analysis (e.g. Scanning electron microscopy (SEM) and X-ray diffraction (XRD)) and assessment of the durability (e.g. resistance to chloride ingress/migration)

The self-healing cementitious materials developed in this research could significantly reduce the potential maintenance requirement for concrete structures. Therefore, this study makes a significant contribution to the field of concrete repair and the results could help contribute to the definition of guidelines for the repair of concrete structures using self healing repair materials.

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## Utilization of lightweight aggregate in the bacteria-based self-healing concrete

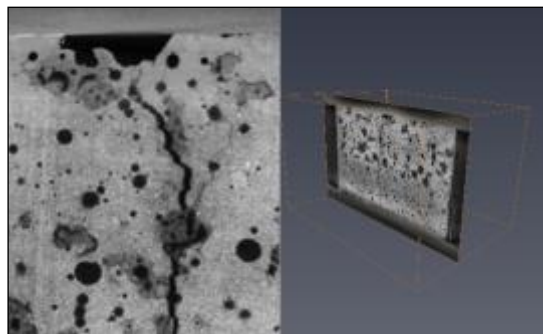
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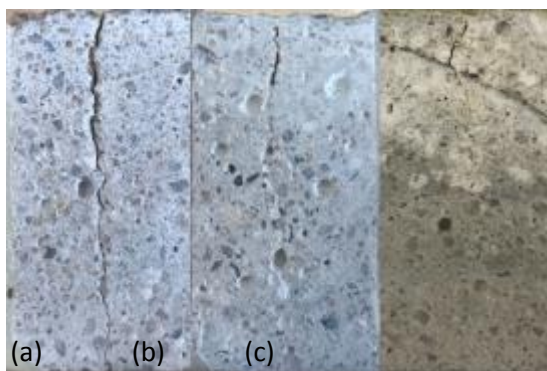
Within the concrete structure, aggressive ions' ingress through cracks may cause seriously corrosion of reinforced steel and reduce the durability of concrete. In order to enhance the autogenous healing capacity of concrete, self-healing concrete which involves addition of specific self-healing agents are introduced. Microbially induced calcite precipitation (MICP) is the biological process through which micro-organisms exert calcite or calcium-bearing minerals. In this study, perlite and aerated concrete granules were used as the carrier for the bio-agents respectively, *Bacillus pseudofirmus* DSM 6950 was selected as the bacteria strain, after 1-week healing, precipitated products were observed within the crack and on the surface

**Figure 7: CT scan of the aerated concrete granule sample**



of mortar samples, and complete crack closure was observed via optical microscope. Permeability coefficient dropped down remarkably as well (Table 1). SEM and EDX confirmed that most of the healing products within the crack were calcium-based crystal which was highly thought to be calcite. However, according to the results of CT scan (*Fig. 1*), self-healing products were only formed on the shallow surface, large gaps were found to exit inside the deep crack. Qian et al [1] observed the similar trend, as they stated that the healing production were firstly formed along the width direction, then along the longitudinal direction, finally along the depth direction. But loose self-healing products were noticed to form within deeper crack when the samples were completely split along depth (*Fig. 2*) [2], which may suggest the possible inaccuracy of CT scan terms of crack determination. There are some possible reasons of the shallow healing phenomenon. Firstly, based on the phenolphthalein test on cracking surface, almost whole cross section presented pink colour in the bio-based samples, which means the pH value within crack was beyond 8.3 which may be too high for the bacteria, Secondly, hypoxia is another important issue in the bacteria-based concrete. According to the phenolphthalein test results, carbonation process can hardly take place inside the crack which indicated that the healed crack strongly prevents the ingress of carbon dioxide. Similarly, it reasonable to suggest that the pathway of oxygen can be blocked as well, and bacteria are more likely to move to the surface where more oxygen are available. Thirdly, in the wet-dry cycle which was used in this study, with the gradually rising of water level, bio-agents may be brought to the surface. And last possible reason is the insufficiency of bio-agents, while based on the chemical reactions, the theoretical amount of precipitated healing products is not enough for completely filling the crack. Further research will focus on determining the reason for the surface precipitation of healing productions and improve the healing efficiency along the depth.

**Figure 2 Cross section along depth, (a) REF, (b) Perlite sample, (c) Aerated concrete granule sample**



**Table 2: Water permeability coefficient**

Sample	Pre-cracked water permeability coefficient (cm/s)	Post-cracked water permeability coefficient (cm/s)	Post-healed water permeability coefficient (cm/s)
REF	5.08E-08	5.61E-04	3.59E-04
Perlite sample	2.03E-08	4.11E-04	4.63E-05
Aerated concrete granules sample	4.01E-08	4.62E-04	4.15E-06

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## Ad-hoc sensors for the non-remote monitoring of concrete for the non-destructive characterization of concrete durability

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### OBJECTIVES

The research ongoing presented in the present study deals with the remote monitoring of the durability parameters related to the reinforced concrete structures performance as a highly promising alternative for the non-destructive characterization of concrete [1-3].

The general objective of the study is the design, development, fabrication and implementation of integrated wireless sensor systems to be embedded for the continuous monitoring of the concrete performance through the measurement in real-time of multiple variables: temperature, humidity content, resistivity, pH, presence of chloride ions,...

Different types of sensors are being developed, using the electrical resistivity as indirect parameter for evaluating both the fresh state properties of concrete and the hardened concrete properties. The resistivity is used as the indirect parameter for evaluating the concrete moisture content and for detecting the penetration of aggressive agents from the concrete surface (chloride ions and/or carbonation depth, even adapting resistivity and RH/T probes in the same sensing module to obtain the simultaneous variation of the different variables). A remote data-acquisition system is being also implemented in the integrated sensor.

### Research carried out

The research ongoing is on the very first stages, and preliminary tests of two different types of sensors have been carried out:

- a) A miniaturized two-point sensor for evaluating the setting process of concrete mixes has been tested on laboratory conditions (Figure 1-Left).
- b) An array of 5 sensors for assessing the water exchange through the concrete surface has been tested for both the uptake and the loss of water (Figure 1-Right).



Figure 1. Sensors developed. Left – For assessing setting process of concrete mixes, Right – For detecting water penetration through the concrete surface.



In the figure, the results concerning the ability of the sensor array (Figure 1-Right) for detecting the water penetration through a dried concrete surface are included. It can be from the figure that the electrical resistance values decrease at different times depending on the depth of each sensor, with longer times of response for the deeper sensors.

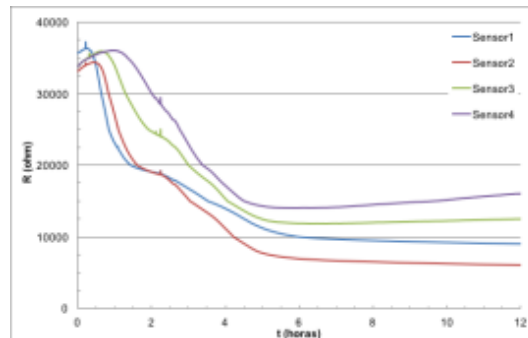


Figure 2. Electrical resistance at the different levels of the sensor array embedded in a concrete sample.

### Research to be developed: how to ensure the success of self-healing/repair topic during the next years?

The application of this study to the self-healing and external repair topic is highly promising from the perspective of a non-destructive and reliable characterization of the materials performance. The continuous monitoring of the electrical resistivity of self-healing materials will allow to obtain the information in real time about the evolution of the cracks, from the formation to the healing process. It is interesting to note that the methodology is applicable to independent of the self-healing approach considered.

By the other hand, the array of sensor will be highly applicable for evaluating the effectiveness of the external repair methods, both concerning the penetrability of the treatment and related to the water-tightness and resistance against the aggressive agent penetration of the treated surface.

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### ACKNOWLEDGEMENT

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## Discussing Effective Ways to Promote Self-Healing Concrete

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The number of timber and steel structures, especially in developed countries, are getting high due to their sustainable properties as a result of environmental pollution and global warming. On the other hand, concrete is still one of the most important building materials in the world because it is common and economical in most of the countries. Therefore, finding new ways to improve sustainability properties of concrete is crucial. Recently, self-healing concrete has been one of the most striking development in concrete technology. Undoubtedly, minimizing cracks is always an important issue to be concerned. Self-healing concrete promises a good solution for this challenge by repairing cracks with the healing agents like bacteria. Although this technology seems prominent, realizing widespread usage needs much more effort because of many challenges to be overcome. Since it is an expensive and new technology, self-healing concrete should be promoted in a convenient way in order to make further progress. In this current study, both qualitative and quantitative research methodologies are being performed in order to monitor the awareness towards self-healing concrete among universities and firms, and find out true ways for promotion of self-healing concrete. Questionnaires and interviews have been held only in Turkey up to now. Initial results show that most of the firms are either not aware of self-healing concrete nor believe that it could be widespread in the future. On the other hand, academicians with material science and representatives of leading firms believe its contributive future. The stakeholders basically argue that universities should put effort on disseminating benefits of self-healing concrete and related programs should be established in order to orient more students to work on self-healing concrete. In addition, multidisciplinary has been highlighted for better understanding and prospective developments in both self-healing concrete and other cementitious technologies.

Key Words: Self-healing concrete, promotion ways, sustainability, bacteria

### ACKNOWLEDGEMENT

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## Self-Healing in Cementitious Materials

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### Abstract

After acquiring my master in October 2017 from the Physics School of the Aristotle University of Thessaloniki, I realized that my enthusiasm for expanding my knowledge of material science remains. Hence, I decided to continue my career through my doctoral thesis at the same university and start my research on "Self-healing in cementitious materials".

Initially, I worked with the effect of nanomaterials on the self-healing capability of cement, the addition of nano-particles in cement-based materials has been proved beneficiary in terms of pozzolanic reaction and early strength gain. For that purpose, nano-SiO<sub>2</sub> and nano-CaO were incorporated in cement pastes in different proportions. The produced pastes were tested in terms of mechanical, physical and microstructure properties. The formation of secondary crystals into the nano-modified structure resulted in: porosity refinement, strength increase and the most important, dense structure by the presence of fully compatible products that reinforce the material's structure [1,2]. In pastes where cement-lime was combined, NS had lower influence due to different mechanisms acting in parallel. CaO nanoparticles not only benefit cement properties, but also the mechanism of their action favors calcite precipitation. Also has a vital role in terms of healing through secondary calcite formation at very early ages, which even in a low percentage (0.5%w/w of binder) is very active. Lime-cement pastes with nanoparticles contain a considerable amount of available portlandite which at later ages can contribute to further calcite precipitation. Even at 28days, healing crystals seem to be formed in wide cracks. Portlandite content increase and healing "bridges" on cracks enforce that claim.

Then I expanded my research by testing compositions that combine nanomaterials with silicate salts. Despite the wide application of sodium silicate solutions, the mechanism by which they act and improve the performance of the concrete is not fully understood [3]. Has been investigated the influence of silicate salts (Na and K) and nanoparticles as well as the influence of the curing environment on the mechanical and physical properties of cement pastes. The samples with sodium silicate salts were more effective in relation to potassium meta-silicate. The role of nanoparticles needs investigation as they seem to form dense structure and managed to fill empty spaces with an excess of C-S-H phases [4]. SEM imaging revealed a number of flake and needle-like crystals in the pastes which were likely the primary reason for the mechanical properties. Overall, the presence of silicate salts in cement systems seems to play a significant role in self-healing procedure.

Recently my interest turned to the effect of crystallites on the ability of self-healing. Different types of crystallites were tested to observe the different effects and have been exposed to different environments; sea water, water, and moisture chambers. Crystallites, aided by the presence of humidity, form idiomorphic crystals that block cracks and pores resulting in reduced porosity [5]. The hydrophilic nature of crystalline admixtures helps the components to react with water and cement particles in the concrete to form calcium silicate hydrates and pore-blocking precipitates in the existing micro-cracks and capillaries. The resulting concrete exhibits significantly increased resistance to water penetration under stress. High-performance fibre reinforced cementitious composite (HPFRCC) were produced in the laboratory. Specimens were pre-cracked at 28 days and the achieved cracks width were in the range of 0.10–0.50 mm. Furthermore, microstructure observations and Ultrasonic Pulse Velocity tests have been conducted. Based on the outcomes, self-healing related indicators have been also

defined. The results show almost perfect healing capability for specimens healed under seawater, better than for specimens healed in water while inadequate for the wet/dry exposure in both of the crystalline types.

The success of self-healing in cement-based materials can only be ensured through thorough research and repeat measurements. I firmly believe that the exchange of knowledge and the ability to work in other laboratories helps researches to better understand their topic. Therefore, I decided to apply for a position to the Ph.D. Meeting at Novi Sad.

One of the biggest questions that need further investigation and is not yet totally clarified is the correlation between the time when the pre-crack occur and the self-healing capability. In particular, in the case of early-age pre-crack concrete, the mechanism is affected mainly by the autogenous healing of cement or from the admixtures. It would also be very interesting to create a pre-crack in late-age in order to investigate the remain self-healing capacity.

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## ACKNOWLEDGEMENT

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## Crystalline admixture impact on crack healing of concrete reinforced with glass fibres

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### OBJECTIVES

Self-healing concrete has been taken a noteworthy place in the concrete field. Depending on factors such as features of concrete production technology and application most suitable self-healing method can be chosen.

Glass fibre reinforced concrete (GRC) is a widely used cement composite. One of the most popular usage is faced panels` production [1], thus this material is exposed to direct outdoor environmental conditions. As not just resistance aspect but also visual aspect is important for such material appeared cracks should be healed.

Taking into account the specificities of production technology of GRC crystalline admixture has been chosen as most suitable self-healing method for GRC.

In this research concrete based on CEM I 42.5 N (Cemex Ltd, Latvia) reinforced with glass fibres is studied. To ensure crack healing 1 % Penetron Admix has been added to studied concrete. Four-point bending test was used to pre-crack samples after the first 7 days of curing at  $20\pm 2^{\circ}\text{C}$  in water media samples. Two different environments were ensured for healing process – half of the samples were kept at  $20\pm 2^{\circ}\text{C}$  in water media and another half – at the variable temperature (from  $+5^{\circ}\text{C}$  to  $-10^{\circ}\text{C}$ ) and humidity 80-90% that reflect outdoor environmental conditions. The microscope was used to measure the size of cracks before and after 14 days of healing process.

As expected at water media healing process is more promising than at outdoor environmental conditions.

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### ACKNOWLEDGEMENT

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# Late-age Autogenous Self-healing Capability of Cementitious Composites

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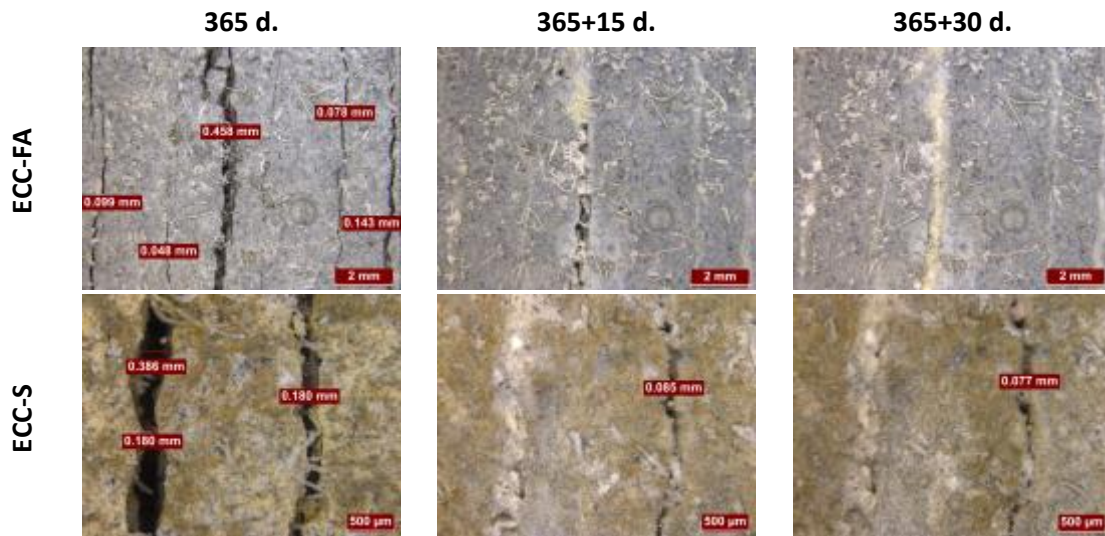
## OBJECTIVES

Engineered Cementitious Composites (ECCs) are materials with distinctive tensile properties and strain-hardening response similar to very ductile metals such as aluminium. Tensile strain-hardening response of ECCs comes along with the formation many multiple microcracks. The formation of these tiny multiple microcracks not only bring about superior mechanical properties but also significantly enhance resistance against many commonly encountered durability issues troubling reinforced concrete structures. On top of all, these tiny microcracks can close by themselves without external interference which provides unique attribute to ECCs known as autogenous self-healing. Main driving mechanisms for autogenous self-healing of ECCs are reported to be ongoing hydration reactions and calcium carbonate precipitation [1]. However, the effect of former on closing of cracks starts to decrease with time as the unhydrated cementitious particles significantly decrease in number with the further progress of hydration in time. This therefore raises the question whether the late-age autogenous self-healing of ECCs would be at similar level to that observed at early-ages or not. The main objective of the current research was therefore to observe and accelerate the late-age autogenous self-healing performance of ECCs by proposing different mixture proportions and curing conditions.

## INSTRUCTIONS

Within the scope of current study, ECC mixtures were produced with low-lime Class-F fly ash (FA – ECC-FA) and ground granulated blast furnace slag (ECC-S), as different mineral admixtures. To observe the late-age autogenous self-healing performance of ECCs, specimens were initially cured for a year. After, cylindrical specimens were almost failed under splitting tensile loading to create microcracks with different characteristics. Accelerated carbonation curing (in a special cabinet with  $50\pm 5$  °C,  $50\pm 5\%$  RH, 3% CO<sub>2</sub>) was applied to specimens kept inside and outside of tap water. The crack width measurements of preloaded specimens were made with 15-day carbonation curing intervals of specimens [2] until completion of 90 days of complete further curing. Changes in the microcracks of ECC specimens were shown in Figure 1 for the selected specimens of mixtures. As can be seen from the views presented in this figure, microcracks as large as 458  $\mu\text{m}$  were healable depending on the ECC composition and further curing conditioning/time. The maximum autogenously healable crack width measurement recorded for this study (458  $\mu\text{m}$ ) was the highest in the current literature, according to author's best knowledge. The level of healable crack width was similar and/or higher than those obtained by autonomous self-healing techniques such as in the case of utilization of expansive mineral admixtures, bacteria and encapsulating agents, as reported by the latest SARCOS review paper of different self-healing perspectives in cementitious systems [3]. Overall, the author concludes that autogenous self-healing capability of ECCs can be enhanced significantly by proper mixture proportioning and arranging of further curing conditioning/time and for future studies, different further curing techniques that may increase the autogenous self-healing performance of ECCs should be sought.





**Figure 8: Representative microscopic views of 1-year-old specimens after limited periods of further carbonation curing [2]**

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## Shaking table tests on scaled model of traditional brick masonry building

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### INTRODUCTION

Providing seismic resistance of buildings is important and actual topic worldwide, especially in older masonry buildings that are characterized by high consumption of energy and a high risk of partial or complete destruction during earthquakes. Traditional brick masonry building is representative for the majority of the existing masonry building stock on the territory of Balkan region and beyond. Most of them are constructed in the second half of XX century and they are still in function as hospitals, schools, residential or individual buildings and historic monuments.

For the structures which are not specially designed with seismic rules and codes as old traditional masonry buildings are, there is necessity to perform tests for assessing their safety and for designing appropriate seismic upgrading. Many of these buildings are built in regions that have been strongly affected by medium and high magnitude earthquakes in the past. For each significant event, specialists have gathered relevant information with respect to their protection, which calls for verifications based on experimental testing.

To obtain the experimental values of the main parameters (physical-mechanical and chemical characteristics of the built-in material, strength and deformability characteristics, ductility capacity and energy dissipation capacity of the structural elements and structures as whole), different testing techniques are applied in practice. The experimental investigation of models on a seismic shaking table is the most corresponding way of investigation from the aspect of dynamic structural behaviour during real earthquakes.

### SHAKING TABLE TESTS – PROCEDURE AND RESULTS

According to the abovementioned, it has been decided to choose traditional brick masonry building as prototype structure, since this type of masonry buildings is representative for the majority of the existing masonry building stock in Republic of Macedonia, but also on the territory of Balkan region.

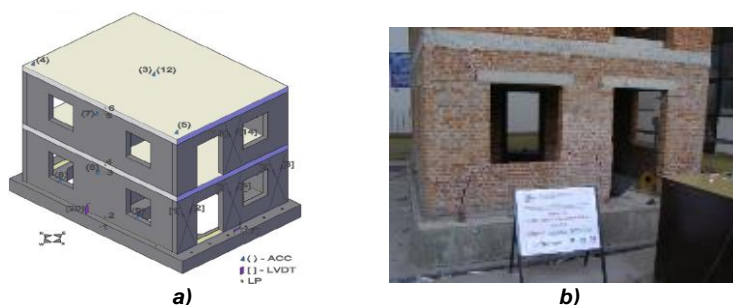
The proposed experimental programme includes:

- Design and construction of a model to a scale of 1:2 of hypothetical traditional brick masonry building with RC floor and roof structures, using the gravity neglected type of modelling, (Fig. 1.a).
- Instrumentation of a scaled model, (accelerometers, displacement transducers and strain gages), for monitoring of dynamic response of the model.
- Shaking table testing of the scaled model by gradual increase of the intensity of input earthquake excitations, aimed at monitoring the progressive development of cracks and the phases of dynamic behaviour of the model, i.e., defining the elasticity limit (occurrence of the first cracks)

In order to achieve the upper stated goals, a programme was adopted for experimental testing under earthquake excitations performed by gradual increase in intensity for the purpose of

monitoring the progressive development of cracks, the modification of the dynamic characteristics, the phases of behaviour and the failure mechanisms. The model has been subjected in its W-E direction to three characteristic earthquake effects (El Centro, 1940; Petrovac N-S, 1979, and Northridge, 1994). The selection of these time histories has been performed from the aspect of both seismic hazard and structural response, i.e., simulated are two main types of earthquake: local (Northridge) earthquake and distant (El Centro and Petrovac) earthquakes.

Analysing the induced damages, it may be concluded the main failure mechanism is transferred in the lower zone and in the final stage it results in occurrence of typical shear cracks of the order of 0.5 - 2.5 cm due to dominant shear stresses. Damages are further on expanded to the bearing walls (Fig. 1.b)). Considering the general behaviour of the model under dynamic effects and under the last applied effect inducing considerably large cracks, it is concluded that the building behaves as a rigid body in the elastic range. At the state of occurrence of the first shear cracks, the loads acting on the walls in the excitation direction are considerably increased which results in larger cracks occurring in these walls, (in plane failure mechanism) and development of damages up to a state close to failure.



**Figure 1: a) 3D view of the scaled model with the instrumentation; b) Damage (cracks) to the facade wall**


## CONCLUSION

In providing the earthquake protection of existing structures, the experts are permanently challenged by the fast development and the improved performance of new materials and techniques. However, the implementation of particular retrofitting or strengthening methodology depends on the extent it has been investigated. The delicate problem of proving the effectiveness of the selected consolidation, retrofitting or strengthening system can be successfully overcome by using the methodology of design assisted by testing. Based on experimental data obtained from site testing of buildings, as well as the results of the seismic hazard analysis, the laboratory testing of models gives very reliable data on the seismic behaviour and stability, pointing out the weak points of the structures. These data are of a great importance for further analysis and development of an appropriate methodology for seismic strengthening or retrofitting.

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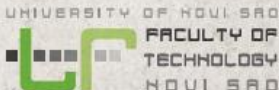
## ABOUT THE HOST



**LABORATORY  
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- Detailed diagnostics
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- Development of tailored cleaning and protective materials
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- Establishment of new testing methods

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## METHODS AND EQUIPMENT

LABORATORY FOR MATERIALS IN CULTURAL HERITAGE examines historical and contemporary building materials, performs scientific research in development of novel (multi)functional materials for cleaning and protection of heritage objects, and establishes modern methodologies for testing of materials functionality and impact. LABORATORY takes active role in a range of supportive activities in conservation-restoration works. It performs:

- diagnostic assessment of historical materials (of inorganic, organic and biological origin) in architectural heritage and artworks,
- identification of painting technologies (mediums, binders and pigment palates) in wall and easel paintings,
- revealing of causes, mechanisms and levels of degradation,
- expert recommendations regarding optimal healing, conservation and restoration,
- treatment monitoring.

In the field of novel materials design, LABORATORY has developed substantial expertise concerning cleaning and protective materials embracing natural phenomena: self-cleaning (UV and VIS), photocatalysis, bacterial activity, denitrification, hydrophilicity, self-healing, etc.

LABORATORY conducts testing, laboratory and *in situ*, using comprehensive chemical, structural and micro-structural analysis (XRF, FTIR, XRD, SEM-EDS, optical microscopy, IR thermographic imaging, optical spectrophotometry), textural (Hg and low temperature gas adsorption porosimetry), surface and mechanical characterisation (roughness, hardness, drilling resistance, contact angle measurements), and thermic and microbiological analysis, including artificial weathering tests (in climate chamber), zeta potential and particle size measurement.

MOBILE EQUIPMENT brings high level of adaptability to diverse working conditions *in situ*, respecting principles of non-invasive and micro-invasive diagnostics.

MOBILE LABORATORY EQUIPMENT includes XRF system, FTIR device, DRM system, optical stereomicroscope, colorimeter/spectrophotometer and IR thermographic camera.



## REPRESENTATIVE PROJECTS AND CASE STUDIES



- **Medieval basilica Arača, Vojvodina, Serbia, 13<sup>th</sup> century** - examination of brick, mortar, render and stone;
- **Orthodox church in Bačka Palanka, Vojvodina, Serbia, 18<sup>th</sup> century** – consultancy regarding the selection of optimal cleaning procedure and materials for fresco paintings;
- **Monastery Krušedol, Fruška gora, Serbia, 16<sup>th</sup> century** – solving conservation obstacles through revealing of painting technology and degradation mechanisms;
- **Antique (Roman) mosaics, Skelani, Bosnia and Herzegovina** – development of compatible mortars for conservation;
- **Medieval Bač Fortress, Vojvodina, Serbia, 14<sup>th</sup> century** – scientific support to holistic conservation approach: determination of production technologies of constitutive building materials (brick, render and mortar), revealing of degradation causes and mechanisms, as well as design and development of tailored materials for denitrification, self-cleaning and long-term protection;
- **Kalemegdan Fortress, Belgrade, Serbia** – diagnostic assessment of 18<sup>th</sup> century stone materials;
- **Petrovaradin Fortress, Novi Sad, Serbia, 18<sup>th</sup> century** – examination of brick, mortar and stone;
- **Count Hadik Portrait, Vojvodina, Serbia, 18<sup>th</sup> century** – determination of painting technology using non-invasive and non-destructive analysis (reveal of original painting below the newer layers), which assisted art-conservators in cleaning, restoration and presentation of the portrait;
- **Monasteries Bođani and Žiča, Serbia** - examination of fresco and secco paintings;
- **Provincial Parliament and Government buildings, Autonomous Province of Vojvodina, Novi Sad, Serbia** - recommendations for cleaning, consolidation and protection of stone façade elements;
- **Innovative multifunctional materials** – self-cleaning and protective materials highly compatible with building mineral substrates (brick, concrete, stone, mortar, render, façade paints), developed under FP7-funded project HEROMAT (*Protection of Cultural Heritage Objects with Multifunctional Advanced Materials*; [www.heromat.com](http://www.heromat.com)); ww
- **Protection of murals on residential buildings in City of Novi Sad, European Capital of Culture 2021** – murals painted by regional and world famous artists on residential buildings have been protected with self-cleaning material developed in the LABORATORY;
- **Training courses for conservation professionals** in application potentials of modern laboratory equipment, investigation of decay phenomena and development of novel tailored solutions for cleaning and protection. Training courses received Annual Award of the Serbian Conservation Society in 2018.





Scientific expertise and state-of-the-art equipment meet in the LABORATORY to assist conservation community region-wide. A number of projects during the last decade have improved LABORATORY skills, expanded competencies and built capacities, developing stable partnerships with heritage institutions and professionals.

LABORATORY acts as a centre of knowledge and modern analytical instruments which enable *historic materials* to tell their story of design, construction, various usages and decay, and help restorers to develop individual approach to conservation, protection and care.



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