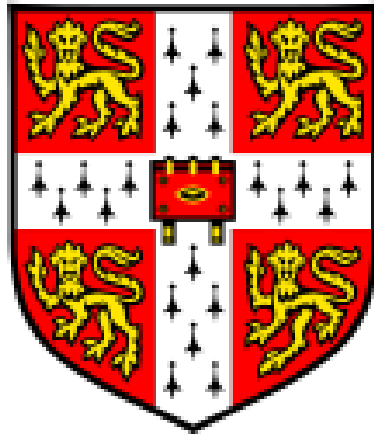


Green Low Strength Engineered Cementitious Composites



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I hereby declare that, except where specifically indicated, the work submitted here is my own original work.

Signed: 

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Technical Abstract

Engineered cementitious composites (ECCs), also known as strain hardening cement-based composites (SHCCs), more popularly named ‘Bendable Concrete’, are mortar-based high strength composites with specific short randomly distributed polymer fibres. They have risen to the forefront of attention due to their performance benefits over concrete. Distinct for having tensile strain-hardening behaviour, vastly increased tensile ductility (3 - 12% strain at failure) and an intrinsic ability to self-heal. By way of reducing the brittle nature of concrete and the introduction of self-healing, as a result of the formation of the many small cracks in the material, ECCs have opened a new world of possibilities to enhance the safety, durability, and sustainability of civil infrastructure.

However, a major concern with ECCs remains the high cement content, due to the removal of large aggregates within their matrix. This high content of cement only further intensifies the strain on sustainability to an already challenged industry. It is reported that more than 11.4 billion tonnes of concrete is being consumed annually worldwide, with 4.08 billion tonnes of cement produced in the year 2018. This production of cement is responsible for 8.6% of global greenhouse gas emissions and stipulates a large influence in the rise of global warming. Additionally, the use of polymeric fibres, implemented to achieve higher strength characteristics, provides an added destructive impact on the materials sustainability. Therefore, to reduce the damaging effect on the environment caused by the use of cement and fibres, the construction industry must direct towards the incorporation of an increased sustainability concern into the design of cementitious materials.

Reducing the percentage of cement used in ECCs or substituting with appropriate alternatives, namely exploring Greener Low Strength Engineering Cementitious Composites could help stem the effect on greenhouse emissions whilst still making strides towards more resilient and robust infrastructure.

With that in mind, this project explores the possibility of designing lower strength ECCs whilst assessing the effect of constitutional parameters. Furthermore, the project set out to investigate the self-healing behaviour and healing rate of greener low strength ECCs under 4-point bending with respect to crack closure and their respective healing rate. The experimental work conducted included unconfined compressive strength tests on hardened specimens through the first 14 days of the material life. To assess the strain-hardening behaviour of each of the mixes, through the

analysis their corresponding stress-strain plots, 4-point bending tests were performed using an electro-servo universal testing machine (Instron 5567) set at a constant displacement rate. Lastly, digital microscopy was used to observe crack formation and to measure crack widths on the samples directly after bending testing had been performed and after the material was allowed to self-heal.

Results showed that the formation of ECC mixes with strength values of 30 MPa and below were achievable with individual samples showing strain hardening behaviour, up to a failure at strain of 0.3%. However, the production of only one singular crack, in each sample, limited the ability to gain higher failure strains. Additionally, the large crack widths recorded (320 μm , 280 μm & 370 μm), due to the formation of only one crack and a decrease of typically high volumes of unhydrated cement particles and pozzolans in high strength ECCs restricted the capacity to self-heal as effectively as expected compared to that of other previously examined ECC mixes in literature with crack widths typically held below 100 μm . It was additionally discovered that the reduction of fibre content below 1% proved detrimental to the ductility performance of the ECC mix. The number of fibres present provided insufficient strength to bridge the crack formed, hence no strain hardening was observed and the smallest failure at strain was recorded across all mixes and when compared against literature.

This work showed the promise of reducing cement content to produce greener lower strength ECC mixes. The benefits become clear with performance gains over that of the fundamentally brittle nature of other cementitious materials. Namely a value of strain at failure was attained that was 20 times greater than that of normal concrete whilst having comparative values for compressive strength. Furthermore, with their intrinsic ability to self-heal, resulting in the eradication of the need for repair and maintenance, these greener lower strength ECCs have opened a new world of possibilities to enhance the safety, durability, and sustainability of civil infrastructure marking a strong stride forward towards more resilient infrastructure.

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1 Introduction

1.1 Motivation - Cement's global challenge

Global demand for cement in the construction industry has increased dramatically over the last few decades (*Figure 1*), driving up the energy consumption and CO₂ emissions related to that of cementitious materials. Cementitious material use is set to further rise as global urbanisation and economic development increases demand for new buildings and infrastructure. Along with other parts of the global economy, the cement industry will need to dramatically cut its emissions to meet the Paris Agreement's temperature goals (United Nations, 2015).

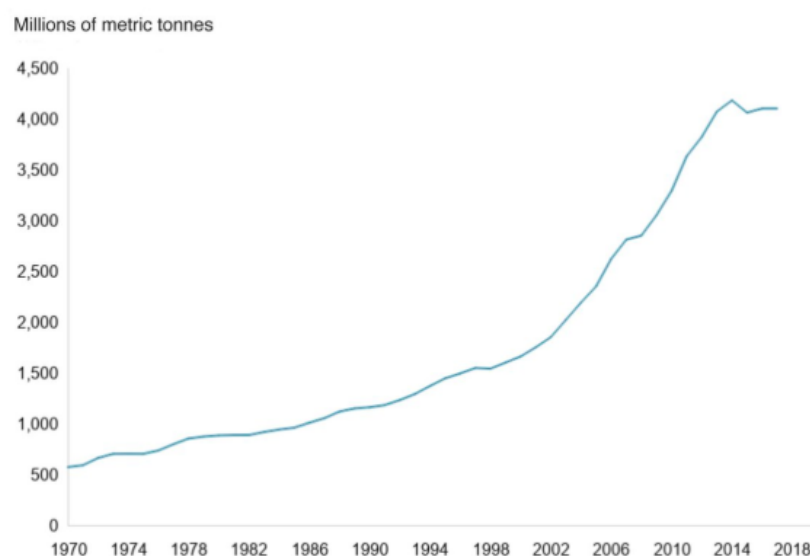


Figure 1- Global Cement Production (Andrew, 2018)

Due to its prevalence in modern infrastructure, concrete is experiencing the most rapid increase in consumption among globally common structural materials; however, the production of concrete results in approximately 8.6% of all anthropogenic CO₂ emissions (International Energy Agency and Organisation for Economic Co-operation and Development, 2015). Initial estimates suggest that cement production returned to 4.08 gigatons (Gt) globally in 2018, a 1% increase, with China being the largest producer of cement accounting for close to 60% of global production (United Nations, 2018). This already outsized footprint is only projected to grow in the coming decades as economic development and rapid urbanization continue globally but more quickly in particular areas across Southeast Asia and sub-Saharan Africa. According to the International Energy Agency, by 2050 cement production could increase by as much as 23% (International Energy Agency, 2019).

This poses a significant challenge for combating climate change. One 2018 study by Chatham House (also known as the Royal Institute of International Affairs) estimated that cement-related emissions will have to fall by at least 16% by 2030 (Chatham House, 2018) if nations are to meet the 2015 Paris Climate Accord target of staying below 2 °C of warming this century.

This challenge becomes more complicated if the longevity of the cementitious systems are considered. Cementitious materials, incorporating concrete, mortar, grout and cement-modified soil, all inherently have low tensile strengths, making them brittle. Cracks caused in cementitious materials are inevitable and have numerous triggers, for example thermal deformation, structural loading, fatigue and stress concentration. This cracking does not automatically denote failure since reinforcement has been widely applied to carry the tensile and shear loads which would have led to the cracking or strain initially. Nevertheless, due to the presence of the cracks, the matrix is more accessible to air, water and aggressive ions which increase the rate of the material degradation. In steel-reinforced cementitious materials, cracks can accelerate corrosion and then the heightened corrosion will result in more cracks (Al-Amoudi & Maslehuddin, 1993). It therefore becomes evident that cracks destructively affect the functionality and diminish the service life of cementitious structures. Huge amounts of money and resources are spent on regular inspection, maintenance, repair and partial replacement of infrastructure every year to protect against material degradation. With the UK investing around £40 billion per year for maintaining and repairing infrastructure, which is mostly made of cement-based materials (H M Treasury, 2010). These conservations are often short term solutions and a second repair is usually necessary within 10-15 years. Moreover, in many situations the structures are inaccessible and maintenance or repair could be rather challenging and costly. These settings include: deep water applications such as offshore wind farms; geotechnical structures such as underground tunnels, shafts, piles, repositories for wastes, foundations, grouting, soil-cement systems, retaining/cut-off walls; and embankment structures subject to aggressive environment such as offshore and earthquake (Al-Tabbaa & Harbottle, 2015). In such circumstances, the demand for development and presentation of high quality, more durable and resilient construction materials, which do not require frequent maintenance and repair, is necessary and beneficial both environmentally and financially.

1.2 Towards resilient infrastructure

One of the main objectives in material science is to develop and manufacture novel materials which mimic natural materials but more efficiently and effectively tackle damage, *i.e.* biometric

man-made materials with built in self-healing capability to repair structural damage. In general, the use of self-healing materials over a traditional crack-control design include:

Reduce cost of repair and maintenance: Reducing disruptions and increasing reliability:

An approach to improve durability sees the development and manufacture of materials to mimic biological systems that more efficiently and effectively tackle damage, *i.e. self-healing materials*. These biomimetic man-made materials have a built in capability to repair structural damage. In general the use of self-healing materials over traditional damage restriction design include:

Increasing sustainability by using less raw material: Self-healing materials will ideally repair damaged areas and regain strength and durability. In doing so, components of the infrastructure can be designed with less redundancy to reduce the use of raw materials. Moreover, due to the self-healing properties of the product, no extra materials will be needed to repair the damaged areas. The combined reduction in raw materials, transportation, processing and extraction will reduce the overall CO₂ emissions and, in turn, help the overall sustainability of the lifetime of the material and the application it is used in.

Reduce cost of repair and maintenance: Reducing the need for maintenance and extending the lifespan of the material used will automatically lead to better, more economical and environmentally friendly constructions and infrastructure.

Reducing disruptions and increasing reliability: Reducing the frequency of repairs or ideally eliminating them completely, will lead to less disruptions due to human intervention.

Since the beginning of the introduction of concrete (Mehta, 2014) in the 1800s, it has continuously evolved in response to new requirements in field applications. Engineered cementitious composites (ECC), also called strain hardening cement-based composites (SHCC) or ‘Bendable Concrete’ (see *Figure 2*), are high cement content mortar-based composites with specific short randomly distributed polymer fibres. They are distinct for the tensile strain-hardening behaviour and tensile ductility, contrasting to the quasi-brittle nature of ordinary concrete and fibre reinforced concrete (FRC).

By reducing the brittle nature of concrete, ECCs have opened a new world of possibilities to enhance the safety, durability, and sustainability of civil infrastructure. With a strategically designed microstructure, ECC features incredible ductility under tension, previously only seen in ductile

metals. ECCs also have the intrinsic capability for self-healing, unlike any current cementitious alternatives (Yildirim, et al., 2015). Cost savings can be realized through more efficient material design, and less frequent maintenance and repairs. However, the long-term environmental impact of ECC over concrete should also consider the carbon footprint of the raw materials.

A major concern with ECC remains the high cement content, due to the removal of large aggregates, which leads to high carbon footprint (Shamarn, Jan 2016), as well as the use of polymeric fibres implemented to achieve high strength characteristics. Hence, reducing the percentage of cement used in ECCs or substituting with appropriate alternatives, namely exploring green low strength ECCs, could turn the tide on greenhouse emissions caused by the production of cement for the same volume of ECCs produced.



Figure 2 - Ductile Behaviour of ECC or 'Bendable Concrete' (Li & Herbert, 2012)

The project will investigate ECC systems with greener cementitious components and fibres with the intent to reduce the quantity of cementitious components and to assess their impact on the capability that ECC mixes relative to that of the resulting composites produced, respectively. I will therefore question if reducing the overall strength capability of the mix, through using these greener approaches will affect properties that are heavily sought after and desirable in standard high cement content ECC mixes.

1.3 Aims and Objectives

With ECCs being a comparatively new and innovative material primarily, designed to produce high performance outputs and more recently attain high self-healing capabilities, many environmental and sustainability issues may have been overlooked as they have not been of primary concern. This project will investigate ECC systems with greener cement components and fibres and assess their impact on the self-healing capability of the resulting composites. With both lower strength properties and the fact that minimal research has been conducted into Greener ECCs, questions remain before progressing further and looking deeper into material microstructural properties.

ECC has a high cement content primarily due to the elimination of coarse aggregates. To reduce these adverse sustainable effects, the key objectives of this project to be examined are:

- Explore the possibility of designing lower strength ECCs and assess whether the strain hardening characteristics (in particular ultimate tensile strain and ductility) are affected by the transition to lower strength mixes.
- Assess the effect of compositional parameters (namely: cement, water, sand and binder substitution) on the mechanical behaviour of the mixes.
- Assess the self-healing behaviour and healing rate of developed GECCs under 4-point bending with respect to crack closure and the rate at which they heal.

This report details the decision of these objectives and outlines the results found.

2 Literature Review

This chapter presents a technical overview of ECCs and its theory. It further looks into comparisons to other cementitious materials whilst considering factors that could affect properties at lower strengths with greener components and exploring compositional variation. It then introduces the general concept, mechanisms and applications of self-healing in cementitious materials and how they can be used as part of a solution for lower strength ECC mixes.

2.1 Engineering Cementitious Composites (ECCs)

2.1.1 Definition and classification

ECCs are broadly classified as FRC (A Bentur, 2007) as it contains fibre in a cementitious matrix. However, under tensile loading, ECCs are strain hardening whereas FRC exhibits a tension-softening behaviour after the appearance of a crack that continues to widen as the load bearing capacity decreases. ECCs then represent a family of materials with ductility being the common feature, with tensile strain capacity typically beyond 2% (Li, 1993) (Li, 2003) . For comparison, the elastic limit for both normal concrete and FRC is reached at about 0.01%. The design basis of ECC is also significantly different from those of high strength concrete (HSC) or Ultra High Performance Concrete (UHPC). HSC and UHPC are designed based on dense particle packing. Instead, the material microstructure of ECCs are designed with prescribed interactions between the microstructural components, based on a body of knowledge known as ECC micromechanics. In other words, the fibre, matrix, and fibre/matrix interface features are deliberately engineered to interact with one another in a certain prescribed manner, when the composite is loaded. Emphasis of this design basis is the reason behind its name **Engineered Cementitious Composites**.

2.1.2 Strain-Hardening and Self-Healing Behaviour

Fibre-reinforced strain-hardening cementitious composites (SHCC), such as ECCs, are newly developed materials which have become more popular due to the extremely high tensile ductility, fracture energy and energy absorption capacity. ECCs exhibit multiple fine cracks (widths below 100 μm) under tension due to the effective fibre bridging and load transfer mechanism (*Figure 3*).

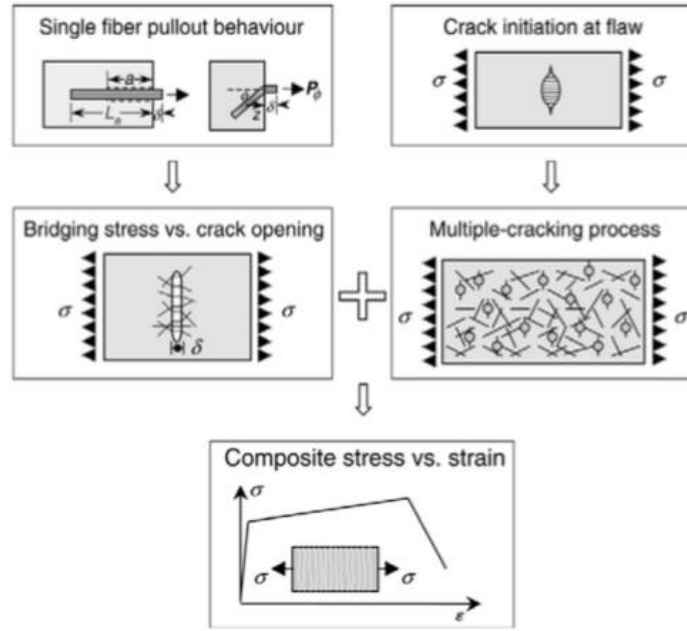


Figure 3 - ECC Fibre Bridging Behaviour (Li & Wang, 2006)

When cracking occurs, fibres take effect to bridge cracks, transferring the load and controlling crack widths. This allows the material to achieve superior tensile ductility using only moderate quantity of fibre reinforcement (1% by volume) (Yang & Li, 2010). The cracking control capacity of ECC could effectively enhance the durability and facilitate self-healing of the cementitious material (Li & Herbert, 2012) .

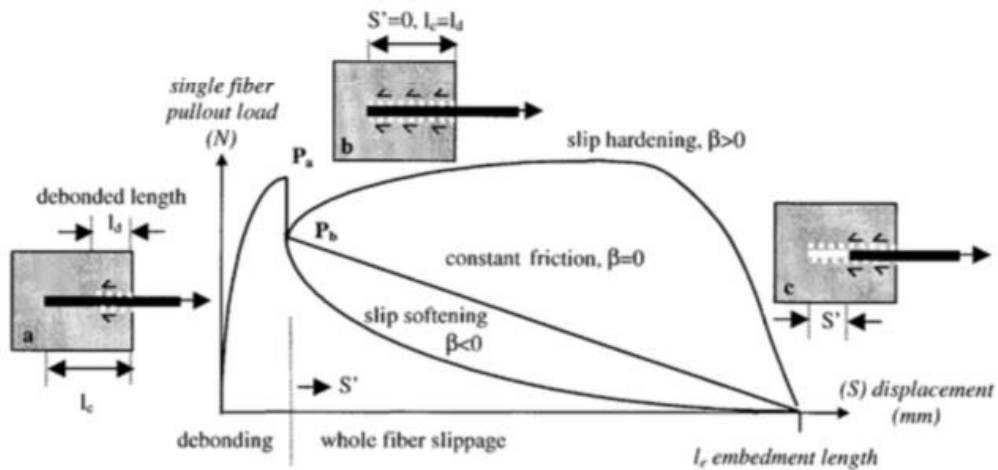


Figure 4 - Single Fibre Pull Out Curve (Redon, 2001)

Strain continues increasing through the numerous cracking behaviours resulting from fibre debonding, fibre pull-out (Redon, 2001) and new crack initiation, and the ultimate strength could

be no less than the first cracking strength (*Figure 4*). Therefore, a high tensile strain capacity (usually $> 3\%$ and more than 100 times that of normal concrete) can be realised in ECC (Weimann & Li, 2003). As shown in *Figure 5*, as the strain increases, the crack width increases initially but levels out afterwards, which means the ductility is realised by generating multiple new cracks to increase the crack number, rather than expanding existing crack widths. The steady-state crack width is an inherent property of ECC and would not be affected by sample scale.

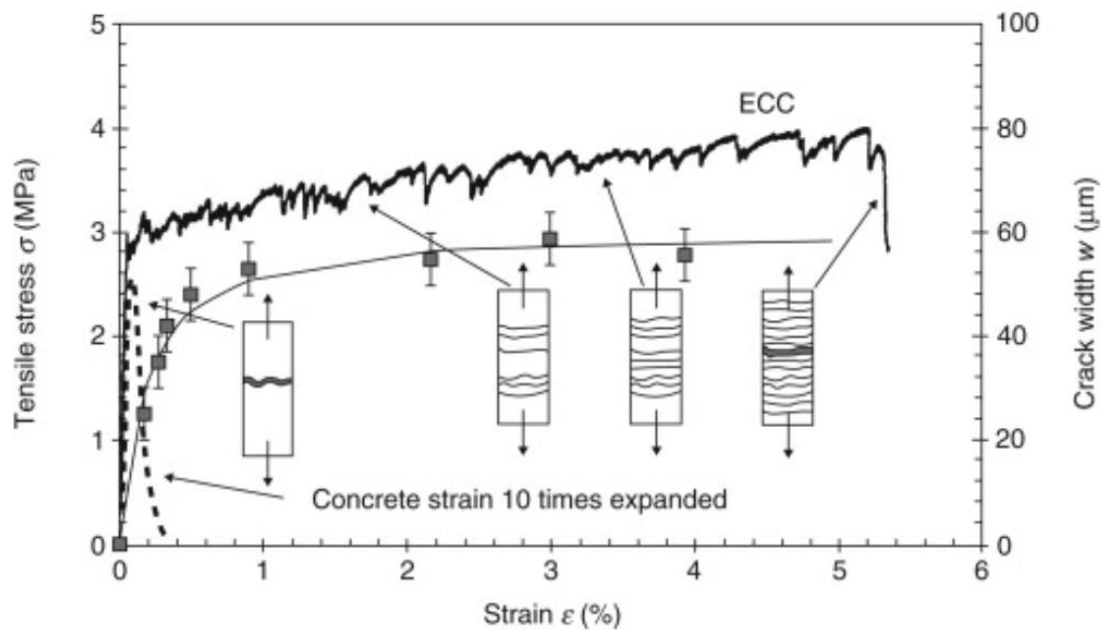


Figure 5 - ECCs vs Regular Concrete Strain Comparison (Weimann & Li, 2003)

The compressive strength of ECC is comparable to normal or high strength concrete, ranging from 20 MPa to 150 MPa. This large range of compressive strength values has been achieved through the variation of compositional elements of ECC mixes. The fibre materials mostly used and studied in ECC are polyvinyl alcohol (PVA) and polyethylene (PE). The main properties of ECC developed to date are provided in *Table 1*.

Compressive strength (MPa)	First cracking strength (MPa)	Ultimate tensile strength (MPa)	Ultimate Tensile Strain (%)	Young's Modulus (GPa)
20-150	3-10	4-20	3-12	18-40

Table 1- Major physical properties of ECC (Van Zijl, et al., 2011)

Even higher properties could possibly be achieved in the future with advanced techniques (Yu, 2018) and according to the demand. The various research aspects so far have included the design theory of ECC (Yang & Li, 2010), considering the effects of composite tailoring with different admixtures, mix proportion, fibre selection and treatment on the direct tensile, compressive and flexural behaviours with their corresponding models (Li, 1997) (Wang & Li, 2007). Its special feature of multiple fine cracking has been proved to greatly enhance the durability under different environmental impacts. The first steps towards full scale applications in different sites in the world have been taken in the U.S., Japan, China (Li, 2003; Zhang, et al., 2013).

2.2 Applications

2.2.1 Current ECC applications

Use and understanding of ECC is growing rapidly around the world. Nevertheless, the material remains a novelty to most practicing engineers and designers. The experiences of current real-world applications of the material provides importance to the development of ECC. Field applications serve many purposes: first, it provides the opportunity to translate basic material properties, high tensile strain ductility of ECC in particular, to enhanced structural performance; second, it provides validation of the usefulness of the unique properties of ECC that cannot be achieved with conventional concrete; third, it provides a feedback path to further development of the material. Thus, the applications of ECC bring life to the development of ECC itself.

Most of the current cases using ECC over normal concrete are driven by the significant improvement in structural resilience and structural durability, with high compressive strength requirements (>50 MPa) that can be offered by ECCs, frequently in association with a need to overcome existing construction or maintenance challenges. Performance capabilities have ruled the decision making in utilising ECCs in damage mitigation of building structures (Fukuyama, 2011), high performance structural applications (Kanakubo, 2007), the application of high performance bridge deck slabs to reduce any tendency of tensile stress build-up between the link-slab and the adjacent pavement to diminish joint damage in pavements (Zhang, et al., 2009) (Lepech & Li, 2009), and more generally the use of ECCs and exploiting their material ductility on the performance of concrete repair (Li & Li, 2010) (Rokugo, et al., 2005) (Alexander, et al., 2012). These applications each typically require high levels maintenance so the introduction of ECC with its high strain capacity and intrinsic self-healing were shown to help prolong the lifespan.

2.2.2 Lower strength ECC applications

Although lower compressive strengths of these ECC mixtures will put limits on applications in structural construction such as beams and columns, lower compressive strengths are nevertheless considered valuable for non-structural repairs related to enhancing durability. High-strength concrete repairs are typically more brittle and, therefore, more prone to cracking than normal strength concrete. Furthermore, high tensile stresses are developed from restrained shrinkage and low stress relaxation due to typically high modulus and low creep of high-strength repair concretes (ACI (American Concrete Institute), 2006). These lower strength ECC are therefore expected to be particularly suitable for repairing relevant structural and non-structural concrete repairs, such as catch basins, maintenance holes, valve chambers, pipe support, road base, sidewalk, curb, gutter, and similar concrete applications (City Of Toronto Transportational Services, 2001). The development and use of recycled tyre rubber to develop lower strength (≈ 20 MPa) low modulus ECC has been demonstrated to provide a basis for more durable concrete repairs with increased performance against that of a comparable cement grout (Huang, et al., 2013).

Another application where the use of lower strength greener ECCs could be beneficial is in in-ground barriers, where it would be used as a physical barrier to protect against the movement of contaminants. Typically, compressive strength requirements of below 15 MPa (Bruce & Bruce, 2003) are required so would suit this project. The introduction of this self-healing materials will only further promote the use of ECCs in such applications due to the maintenance challenges usually faced.

It can be expected that ECC will find applications, in other fields in future, potentially displacing traditional construction materials. The drivers to the implementation of ECC have common themes include enhanced public safety and rapid recovery post event, reduction of time, cost and labour during installation, and reduction of maintenance cost over the service life of the infrastructure. Often times, the use of ECC provides an effective solution to a problem. What makes these value propositions feasible is the combination of unique properties of ECC, including most fundamentally the tensile ductility, durability, and intrinsically tight crack width.

As is well known, the construction industry is highly cost sensitive. The application examples discussed here offer some insights into the economics of adopting ECC in infrastructure applications. Although the unit cost of ECC is similar to some fibre reinforced concrete and repair mortars, comparison is often made with normal concrete which has a lower unit cost. In some

applications, such as ECC bridge deck link slabs, life cycle cost analysis (Keoleian, et al., 2005) shows that the reduction in long term maintenance cost more than make up for the higher initial material cost. In other applications, such as buildings with ECC coupling beams, the initial cost can be lower than previous designs due to the elimination of anti-seismic devices.

2.3 Green Engineered Cementitious Composites

2.3.1 High Volume Fly Ash Mixes (HVFA)

Although much of the work done as of yet on ECCs has been directed towards high strength, high performing mixes. Studies have been conducted that have deeply investigated into the effect of introducing high ratios of Fly Ash into ECC mixes (Yang & Li, 2007) (Sahmaran, et al., 2013). They found these key outcomes.

2.3.1.1 Compressive Strength and Strain Hardening of HVFA Mixes

Increase of fly ash content generally reduces the compressive strength of ECC. As can be seen from the curve above in *Figure 6*, the replacement of cement by class F fly ash generally reduces the compressive strength of ECC at all ages. However, even at 75% replacement of cement (FA/C = 2.8), the compressive strength of ECC at 28 days can still reach 35 MPa which exceeds the nominal compressive strength for normal concrete (30 MPa). No significant strength gain is found in HVFA ECCs between 28 to 90 days. Fly ash is usually considered a beneficial ingredient for long term strength development in concrete due to its pozzolanic properties. In HVFA ECC, however, secondary hydration of fly ash may only reach a very limited reaction degree because the fly ash content is relatively high in proportion to cement and fly ash may remain in the system as fillers even after a long time of curing.

With the introduction of fly ash at a high volume, a successful ECC mix was produced at the very low end of the strength spectrum (90 day strength of 22.2 ± 1.4 MPa) that maintained important ECC properties. This information confirmed that ECC mixes at low strength values are possible to create without losing out on other performance parameters, made possible through changing specific parameters individually to produce successful ECC mixes.

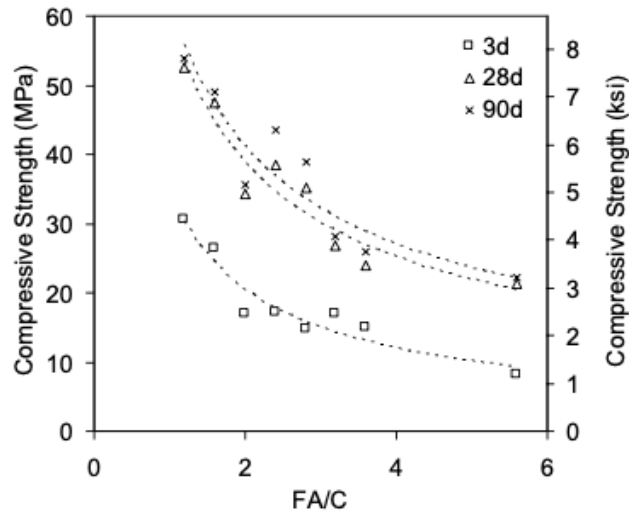


Figure 6 - Compressive strength of HVFA ECC as a function of fly ash content (Yang & Li, 2007)

Secondly all HVFA ECCs exhibit tensile strain hardening behaviour (Figure 7a) at different ages and the tensile strain capacity reaches 2-3.5% at the age of 90 days (Figure 7b). This indicates that the most unique property of ECC, tensile ductility, is retained and is not sacrificed by replacing cement with large amount of fly ash.

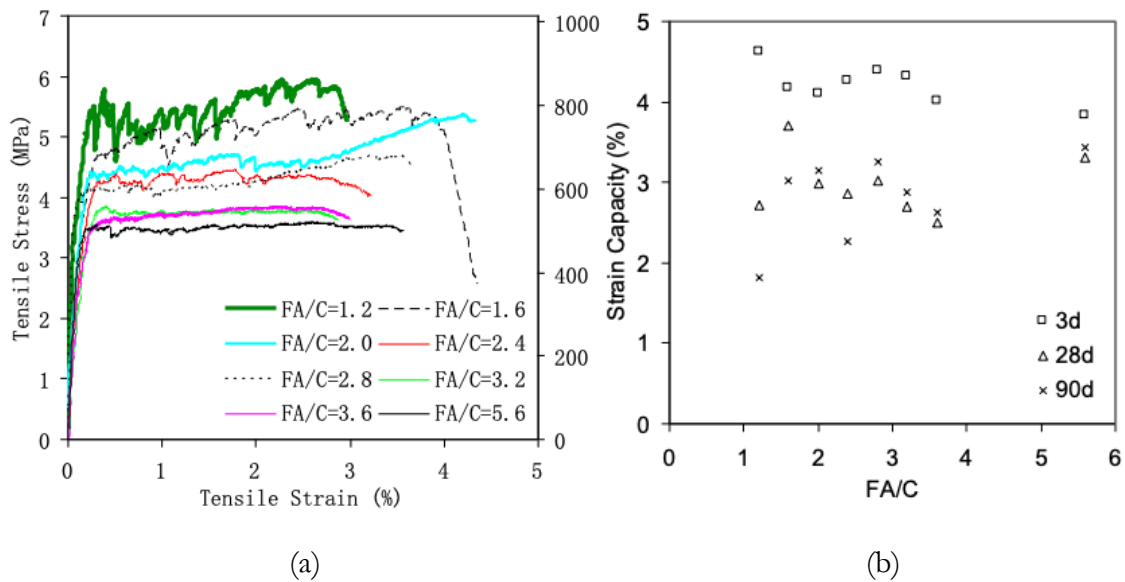


Figure 7 - Tensile behaviour of HVFA ECCs (Yang & Li, 2007) (a) stress-strain curve at 28 days and (b) tensile ductility as a function of fly ash content

2.3.1.2 Crack Behaviour

Crack width of ECC at material hardening stage does not depend on structural geometry and has been recognised as an important material property. The imposed deformation is accommodated by ECC multiple cracks with constant crack width during the strain-hardening stage. The term residual crack width indicates that crack width is measured from the unloaded specimen after the uniaxial tensile test. It was observed in the study that the width of a loaded crack is approximate two times the width of the unloaded one. Each data point in *Figure 8* is an average of 4 or more common specimens and ≥ 20 crack width are measured from each specimen.

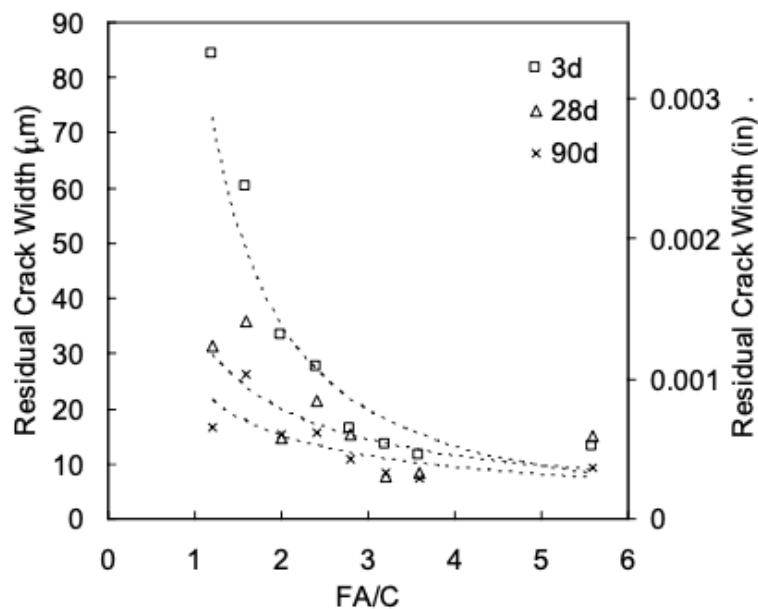


Figure 8 - Residual crack width of HVFA ECCs as a function of fly ash content at different ages

It was found that the crack width reduces as fly ash content increases at all ages. The drop is significant especially in early ages (3 days, *Figure 8*). At the age of 28 days, the residual crack width of some HVFA ECCs can be lower than 10 μm . This observation strongly suggests that HVFA ECC will most likely have lower permeability and better durability even in the presence of microcracks when compared with cracked concrete in which the crack width is not self-controlled and is usually in the range of several hundred micro-meter to several millimetre.

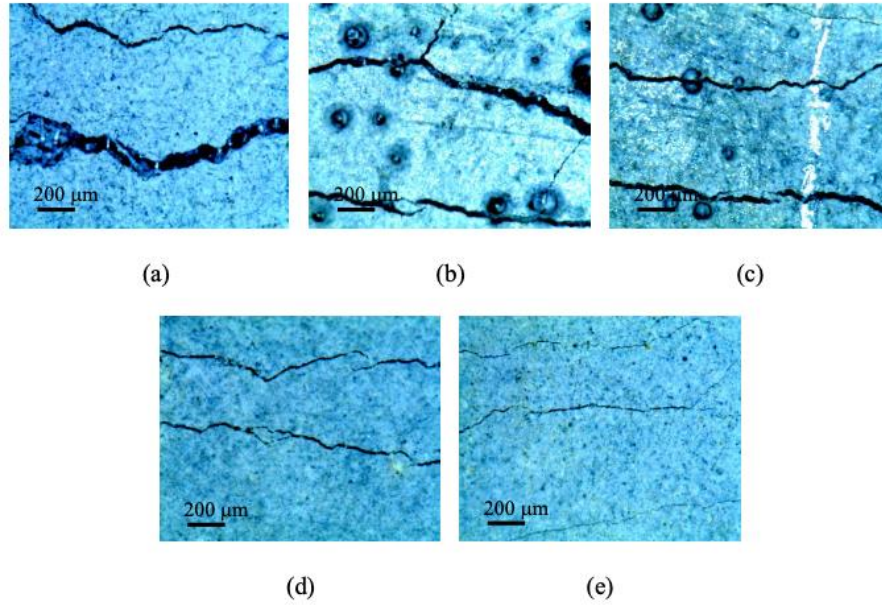


Figure 9 - Microscopic photos of residual crack width of HVFA ECCs at 3 days (REF) where FA/C are: (a)1.2, (b)1.6, (c)2.0, (d)2.8 & (e)3.6 respectively

High fly ash content tends to reduce the crack width in ECC. It was found that high interface frictional bonds (τ_0) restrain the slippage of fibre and are responsible for the tight crack width (Figure 10). Microstructure analysis reveals that high τ_0 is a result of densely packed interface transition zone by unhydrated fly ash particles. Tight crack width has been shown to promote self-healing in ECC and greatly benefit the durability of ECC infrastructure.

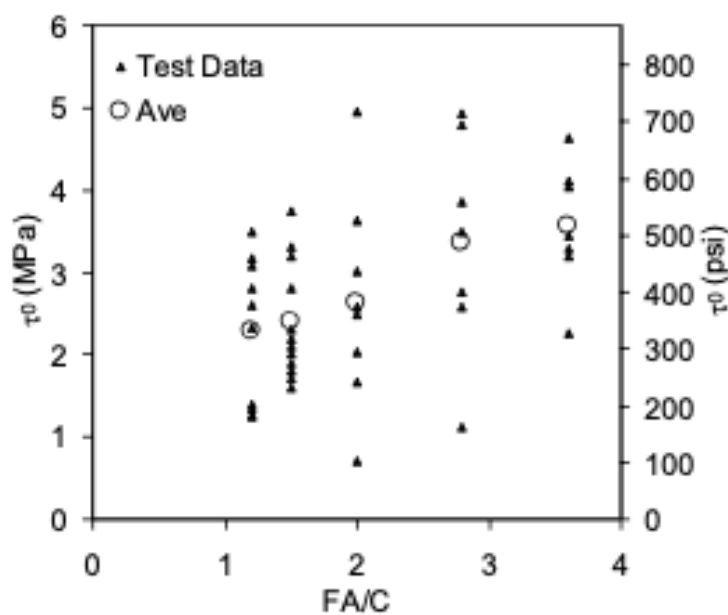


Figure 10 - Frictional Bond Strength of HVFA ECCs as a function of fly ash content at 28 days (REF)

2.3.1.3 Summary

To summarise the literature (Yang & Li, 2007) and (Sahmaran, et al., 2013) demonstrated that:

1. Increase of fly ash content generally reduces the compressive strength of ECC. At FA/C equals 2.8; however, the compressive strength at 28 days can still reach 35 MPa, which is the regular strength grade for concrete in many applications. HVFA ECCs with different compressive strength may be selected for use in different applications.
2. All HVFA ECCs show tensile strain-hardening behaviour, with PSH indexes larger than one. At the age of 90 days, the tensile ductility of HVFA ECCs can still reach the long term stabilized value of 2-3%.
3. High fly ash content tends to reduce the crack width in ECC. It was found that high interface frictional bond τ_0 restrains the slippage of fibre and is responsible for the tight crack width. Microstructure analysis revealed that high τ_0 is a result of densely packed interface zone by unhydrated fly ash particles.
4. Increasing the amount of fly ash in HVFA ECCs tends to improve robustness (reduced variability) of tensile ductility, based on the current analysis.

These findings by Yang and Li indicate that lower strength ECC mixes are not only possible, but the unique ductility characteristics are not impaired. Namely the ductility and tensile strain capacity are not directly linked with compressive strength as had been previously thought. The literature clearly demonstrates that although changing one element of the ECC mix can control and change specific performance parameters, it does so without losing out on other advantageous and distinctive strain hardening attributes linked with ECCs.

2.4 Self-healing

In most construction materials today, self-repair of crack damage is not possible but is highly desirable to extend service life and reduce repair needs and to maintain structural integrity. In concrete, cracks can weaken structures both in strength and in stiffness and can also provide pathways to accelerated deterioration such as carbonation and/or corrosion of steel reinforcement.

Self-healing has been suggested as an effective means for enhancing the service performance and lowering the economic impact of maintenance of concrete infrastructure (Van Breugel, 2007). The contrasts in performance and cost over the life cycle of a concrete infrastructure with and without self-healing capability can be significant (*Figure 11*). While initial cost may be higher, the life cycle cost can be lower for infrastructure whose performance can be self-restored after each damage event, thus preventing accumulated degradation that requires expensive repair. For this reason, recent research in self-healing concrete has drawn much attention in both the technical community and the wider population.

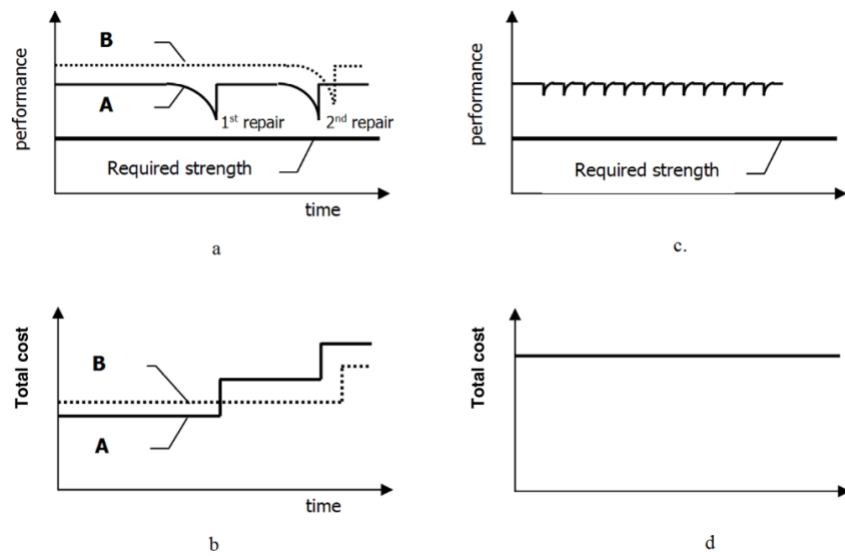


Figure 11 - Conceptual performance and cost of infrastructure over life cycle, for (a)-(b) concretes A (normal) and B (higher performance) without self-healing, and (c)-(d) self-healing concrete. (De Rooij, et al., 2013)

2.4.1 Self-healing in Cementitious Materials

With the introduction and in recent times substantial research into smart self-healing cementitious materials the cracks could be detected and healed by themselves, without outside involvement, it is hoped the the repair and maintenance work on such structures could be reduced or even withdrawn. Self-healing would cause the deterioration to be slowed down, and the durability of constructions to be heightened. The self-healing techniques can be categorised into autogenous healing and autonomous healing with a diverse range of methods that could potentially be applied to different healing environments based on their strengths and limitations.

2.4.1.1 Autogenous/autogenic healing

Inherently, cementitious materials have the capability to heal small cracks to recoup transport and mechanical properties (De Belie, et al., 2018). Autogenous self-healing is categorised by the components of the cement matrix which can take effect to realise self-healing. Physical, chemical and mechanical causes can all initiate this (De Rooij, et al., 2013) which are illustrated in *Figure 12*.

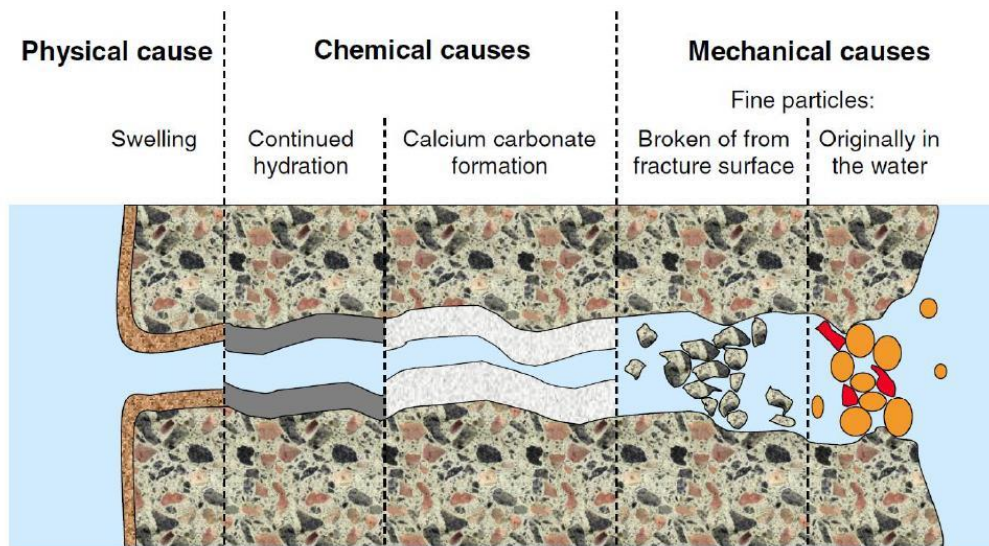


Figure 12 - Different causes of autogenic self-healing in cement-based materials (De Rooij, et al., 2013)

Important mechanisms (shown in the *Figure 13*) include:

1. Recrystallisation of calcium hydroxide ($\text{Ca}(\text{OH})_2$) in the cement matrix.
2. The pozzolanic reactions by supplementary cementitious materials (SCMs) such as fly ash (FA) (Huang, et al., 2016).
3. Continuing hydration of unhydrated cement particles in the presence of water, forming calcium silicate hydrates.
4. Carbonation resulting from the crystallization of calcium carbonate (CaCO_3) on the crack faces in the presence of carbon dioxide and water.

Many factors affect the efficiency of self-healing based on different materials and environmental conditions. Main factors are intrinsic features such as: age and mix design of the cement-based material, crack widths and depths; external healing conditions such as environmental exposure/curing conditions (e.g. availability of water and CO_2 surrounding cracks, thermal

conditions); stress state and steadiness of the cracked state (mechanical loadings); and healing duration (Huang, et al., 2016) (Ferrara & et al., 2018)

Crack dimensions (including crack width, length and depth) regulate the degree of self-healing in a geometrical manner. One of the largest restrictions of autogenous healing is that its efficiency is limited to small cracks only ($<300\text{ }\mu\text{m}$), meaning that the healing efficiency can be significantly boosted by reducing the size of the crack. It has been shown that fibre reinforcement is effective at limiting crack widths (De Belie, et al., 2018).

2.4.1.2 Autonomic healing

Autonomic healing is defined as the recovery due to the presence of engineered additions which were initially not in the material and which can be altered by the designer (De Rooij, et al., 2013). Based on current studies, autonomic healing can be divided into four categories.

1. **Encapsulated autonomous healing:** Tailored additions can be enclosed in capsules which are embedded in the material. Compared with non-encapsulated methods, capsules can store healing agents and control the appropriate time for releasing them for the reaction. Capsules are designed based on either chemical or mechanical (i.e. ruptures) triggers (De Belie, et al., 2018). In a highly alkaline environment, mineral cargos have shown better stability, compatibility and stronger bonding than organic agents. Shell materials were investigated thoroughly which are chosen from parameters such as overall capsule size and dosage to be administered. They include: melamine, acrylate, polyurethane, ethyl cellulose, alginate, polyurea, polystyrene, melamine formaldehyde, polyvinyl alcohol, phenol formaldehyde (Al-Tabbaa, et al., 2017) (Al-Tabbaa & Harbottle, 2015).
2. **Vascular healing:** Similar to the cardiovascular system in the human body, a vascular network can be embedded in the cement material for transporting and releasing liquid healing cargos. The advantage is the healing agents can be supplied from the outside limitlessly, so multiple healing is possible (Van Tittelboom & De Belie, 2013).
3. **Shape memory polymers (SMPs):** pre-tensioned polyethylene-terephthalate (PET) tendons are cast into a concrete member which can be activated externally when cracking

happens and further close/tighten the cracks due to the prestress (Al-Tabbaa, et al., 2017) (Paine, et al., 2019).

4. **Stimulated autogenous healing (or non-encapsulated method):** Based on the intrinsic healing ability of cementitious materials, non-encapsulated additions can be applied to enhance healing efficiency. Specifically, fibres can be incorporated to control and limit crack widths, thus facilitating intrinsic healing (Homma, et al., 2009) (Cuenca & Ferrara, 2017). An alternate approach is using superabsorbent polymers (SAPs) that can be embedded in the matrix to block cracks and store water when wet, and release water slowly when dry to accelerate hydration.

4.2.2 Self-healing in ECCs

The intrinsic self-healing ability of cementitious materials depends heavily on crack widths. The ability to self-control the crack widths makes ECC a very promising self-healing material. The high volume of unhydrated cement particles and pozzolans in ECC also help stimulated self-healing. The crack widths of ECC are typically less than 100 μm , and the value could be further reduced by matrix tailoring.

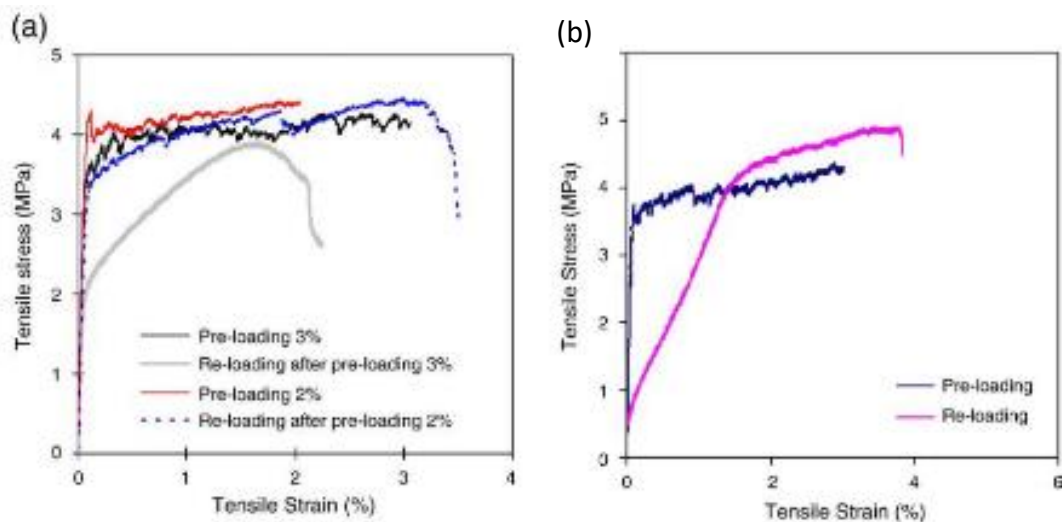


Figure 13 - Stress-strain relationships of ECC (a) pre-loading and re-loading after 10 wet/dry cycles; (b) preloading and re-loading without self-healing (Yang, et al., 2009)

The study of self-healing in ECC was pioneered in 2005 (Yang, et al., 2005) with the ECC mixtures being mostly studied are the standard M45-ECC ($FA/C = 1.2$, $S/B = 0.36$, $W/B = 0.26 \pm 0.01$, $V_f = 2\%$) and ECC with high volume fly ash (HFA-ECC with $FA/C = 2.2-2.8$, $S/B = 0.36$, $W/B = 0.26 \pm 0.01$, $V_f = 2\%$). The self-healing effect was generally studied in terms of the recovery of mechanical and transport properties. Common techniques for investigating mechanical properties are uniaxial tensile tests, four-point flexural tests, splitting tension tests, single-crack tensile test, etc. In general, different strain or deformation is pre-applied based on the ultimate composite strain/deformation capacity of the material, and the recovery of first-cracking strength, tensile strength, strain/deformation capacity, stiffness and the amount of multiple cracking is evaluated after re-loading (Yang, et al., 2009). The example of the recovery of mechanical properties under uniaxial tensile loading is shown in *Figure 13*.

After re-loading, either old cracks could reopen or new cracks could generate (*Figure 14*). This is dependent on the relative strengths of healing products and the matrix. The transport properties having been investigated include water permeability, rapid chloride permeability, gas permeability, diffusion of chloride ions and absorption. Other common parameters characterizing the damage recovery include the change of crack widths and crack number, and the recovery of resonant frequency over the curing cycles, ultrasonic pulse velocity measurements (Yildirim, et al., 2015) (Suryanto, et al., 2016).

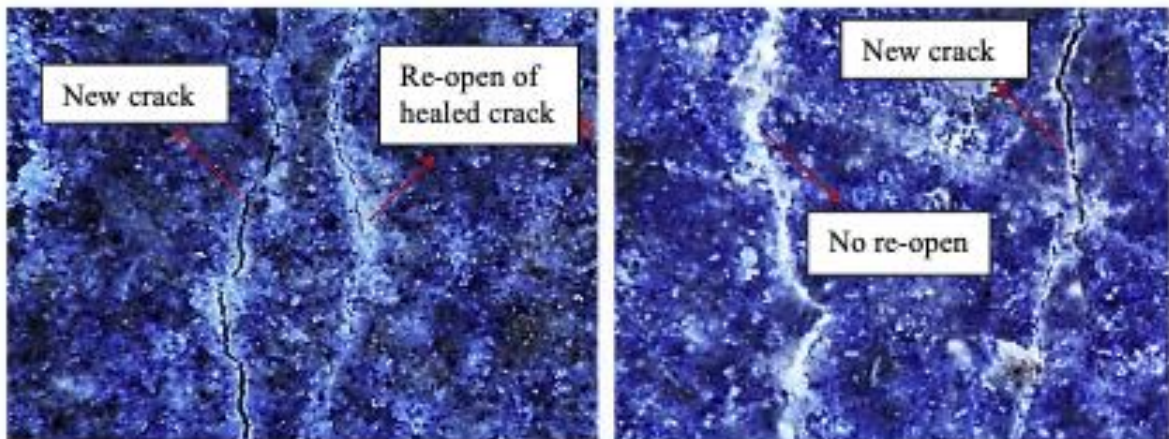


Figure 14 - Cracking pattern of self-healed ECC after reloading (Zhang, et al., 2019)

Various research has been pushed into ECC self-healing such as: looking into a the range of effectiveness of certain curing conditions and environments (Liu, et al., 2017) (Suryanto, et al., 2016), the effect of different types and amount of SCMs on self-healing (Kan & Shi, 2012) the

influence of hydrated lime on the self-healing of M45-ECC (Yildirim, et al., 2015), incorporating two types of SAPs (average size 100 μm and 477 μm) to assess the repeated self-healing of ECC in three different curing conditions (Snoeck & De Belie, 2016) and studies into the autogenous healing behaviour of fibre-reinforced RMC-based strain-hardening composites (RMC refers to reactive magnesia-based cement) without incorporating Portland cement (Qiu, et al., 2019).

These have shown that fibre reinforcement has displayed itself to be a promising method for enhancing autogenous healing of cementitious materials, although a lot of research aspects still remain to be investigated. The healing is only efficient when sufficient healing components, moderate pre-strain and proper conditioning are present. Therefore, further material tailoring, by incorporating other additives, could potentially improve the robustness of self-healing in ECC (Li & Herbert, 2012).

3 Materials and Experimental Procedure

3.1 Materials

The starting point of my mix design was to use a simple cementitious grout with the addition of fibres. This served as the basis for compositional modification and tailoring. The mix was comprised of Type I Ordinary Portland cement (ASTM C150), silica sand with a maximum grain size of 2 mm and PVA fibres with oil coating with the intention to add in Class F fly ash (ASTM C618) to a later mix. Considering the availability and cost of local materials in the UK, the materials below were available for me to use within the lab and used in this study:

1. Cement (C): CEM-I 52.5N High strength Portland cement (BS EN 197-1)(BSI, 2011) was used as the clinker source (supplied by Hanson, UK).
2. Sand (S): Washed and ungraded silica sand (Trade name: RH110) was used as the aggregate, supplied by Minerals Marketing, Redhill, Surrey.
3. Fly ash (FA): Fly ash (Trade name 450-N) with fineness category N (BS EN 450-1)(BSI, 2012), supplied by CEMEX, UK, was applied as a supplementary cementitious material. It is a pozzolan which can extend the hydration period.
4. Polyvinyl alcohol (PVA) fibres: PVA fibres (Trade name REC-15 ×12), shown in *Table 2* were used. These were produced by Kuraray, Japan, and supplied via NTU in Singapore. The properties of these PVA fibres were specially tailored and produced for optimal strain-hardening behaviour (Table 2). They were coated with oil (1.2% by weight) to reduce the excessively strong interfacial bonding.

Density (g/cm ³)	Tensile Strength (GPa)	Elastic Modulus (GPa)	Elongation (%)	Diameter (μ m)	Length (mm)	Surface Oil Coating (%)
1.3	1.56	41	6.5	40	12	1.2

Table 2 - Properties of PVA Fibres

3.2 Mix Generation/ Production of Specimens

The mixing and casting processing method was applied in this study. A Kenwood Chef food blender (4.6L, 1000W) consisting of a stainless-steel bowl and a planetary mixing blade was used for mixing. The procedure of specimen production is described below:

1. Moulds were prepared and lined with a releasing agent to guarantee ease of release from the mould to get the highest quality samples possible.
2. Cement, Sand, Fly ash are mixed together in the mixer dry and mixed on the slowest setting (86 rpm) for 2 minutes to ensure all parts are mixed homogeneously.
3. A solution containing the pre-determined fixed amount of water and super plasticiser was added with slow mixing speed (mode “minimum”) within 1 min.
4. The process was paused for 2 min to allow the SP to take effect (if applicable).
5. The process was then mixed continuously for 5 mins to ensure homogeneity of all constituent parts.
6. Fibres were weighed, broken apart (so that fibres are individually separated rather than clumped together) and added slowly to the mix to ensure complete distribution of fibres.
7. Further mixing for 5 min ensures fibres are mixed in homogeneously.
8. Mix then placed into the 2 variants of moulds (40mm x 40mm x 40mm steel moulds and 300mm x 50mm x 25mm wooden moulds for compression and bending testing respectively) in small steps and compacted fully into all the corners, alongside vibration, so that air bubble and pores that could cause imperfections are eliminated.
9. Final top surfaces of the samples were finished smooth to ensure continuity across all the mixes and in turn the samples themselves
10. Samples were then covered with a plastic film to safeguard against escape of moisture during the first 24hrs before the samples were demoulded to allow testing to occur.



Dry mixing



Adding Water



Adding PVA Fibres



Moulding

Figure 15 - Mix Generation

3.3 Distribution of Fibres

The PVA fibres arrive initially in small bundles in dry conditions as can be seen in Figure 16. This provided some resistance to the mixing process as it was found that these fibres would stay clumped together if added directly into the mixing process without being broken apart by hand. This was a tedious process that had to be done as I couldn't discover how it had been best done in literature previously. Without this fibre distribution within the ECC mix was affected along with the homogeneity of the material.

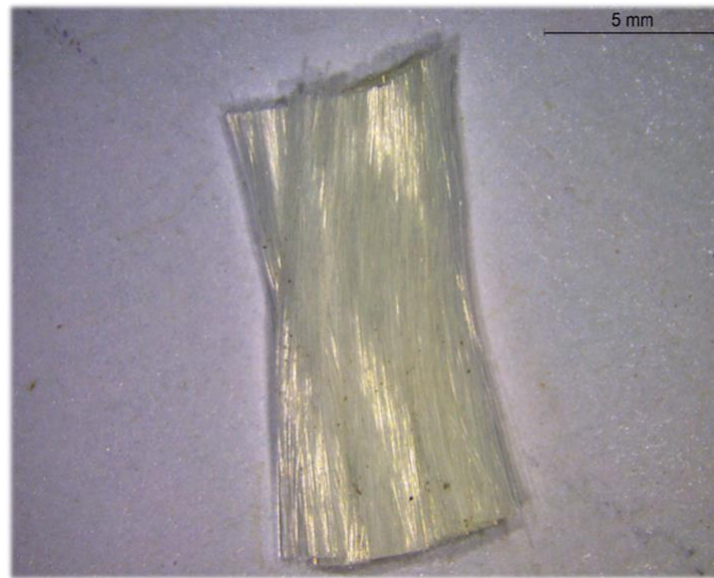


Figure 16 - Cluster of PVA fibres prior to mix production

3.4 Curing Conditions

The specimens are then put aside in a water rich environment within the laboratory to be able to be tested upon day 1, 7 & 14 whilst the cement is still hydrating. Samples were placed and submerged in a water within a sealed container. This method was chosen as application of lower strength/greener ECC mixes would likely be used in ground and would be surrounded by saturated soils therefore would be subject to such conditions in real life applications. As samples would experience the same conditions after damage had occurred, the conditions for healing of the samples post cracking, during the healing stage of my tests, were held the same as previous curing conditions mentioned.

3.5 Testing Methods

3.5.1 Compression testing

Unconfined compressive strength test was conducted on the hardened specimens of a certain age in the compression frame. The unconfined compressive strength is a basic property of cementitious materials, and its variability between samples could give an indication of the quality of sample quality and uniformity, i.e. whether the mixing has been sufficient and the fibres have distributed well in the mixture. Cubic specimens with dimensions of 40×40×40 mm were tested in the 50 kN and 250 kN testing cells with a vertical loading speed of 2400 N/s according to BS EN 196-1 (British Standards Institution (BSI), 2016). Before testing, the specimens were taken out of the curing box, and water on the sample surface was gently dried by using lab paper towel. The unconfined compressive strength is calculated as the failure load P_{max} divided by the loading surface area A , as given by:

$$\sigma_c = \frac{P_{max}}{A}$$

Equation 1 - Unconfined Compressive strength



Figure 17 - Uniaxial Compression Testing on Cubic 40×40×40 Specimens

3.5.2 Bending testing

All flexural tests were performed using an electro-servo universal testing machine (Instron 5567) with a maximum load capacity of 50 kN, controlled by a constant midspan displacement of 0.5 mm/min for all specimens. During the tests the time, applied load P , and midspan deflection δ , flexural strain and flexural stress were simultaneously recorded. Each data point was recorded at a frequency of 50 Hz or every 0.02 seconds. All the collected signals were outputted to a computer controlled dynamic data acquisition system where the results were plotted and stored.



Figure 18 - Experimental set up of flexural 4-point bending testing

Figure 19 illustrates a typically stress-strain plot of ECC materials with associated salient points labelled. Measurements were recorded for stress and strain/midspan deflection at first cracking, maximum stress and failure ($\sigma_c, \sigma_m, \sigma_f, \epsilon_c, \epsilon_m, \epsilon_f$) from the full logged data. The value of stress at failure (σ_f) is also referred to as the Modulus of Rupture (MOR). Stiffness can be measured at various stages throughout the plot with E_0 measuring the initial stiffness, E_e measuring the stiffness in the elastic region and the E_h during strain-hardening. All data was recorded and exported via a csv file to the main computer to be analysed using Microsoft Excel.

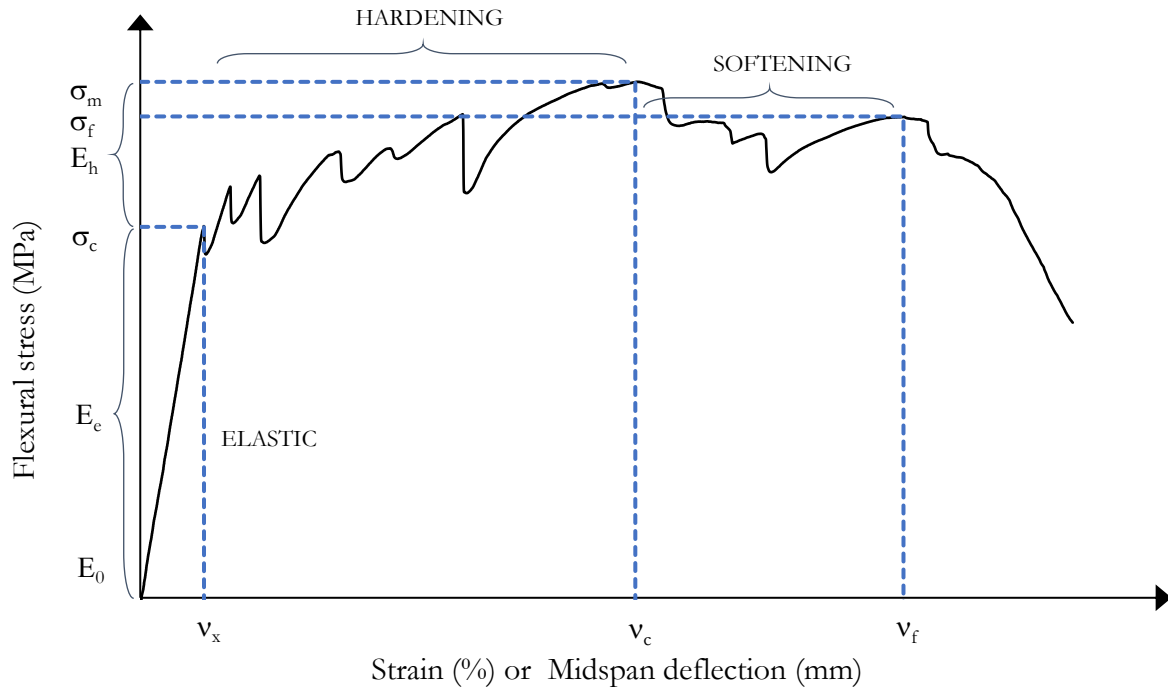


Figure 19 - Typical stress strain plot for strain hardening ECC and corresponding analysis

3.5.2.1 - 3-point vs 4-point bending

For non-homogenous materials, such as engineered cementitious composites it is found that 4-point bending is a much better test of mechanical properties due to the highest or maximum bend stress only occurring under the loading anvil in three point flexural bend tests. In four-point bend tests, the maximum flexural stress is spread over the section of the beam between loading points. Also, a three-point test best applies where the material is homogeneous, such as plastic materials. A four-point test tends to be the best choice if the material is not homogeneous (Szaroletta, 2002), such as composites so that the weakest flaw is actually assess rather than the flaws only found at mid-span directly underneath the load at maximum stress. The stress concentration of a three-point test is small and concentrated under the centre of the loading point, whereas the stress concentration of a four-point test is over a larger region, avoiding premature failure. Although 3-point bending test tend to be more simple set ups and tests that require simpler sample geometry's they tend to lead to much more complex stress distributions through the sample (Ricardo, et al., 2018). Another factor that sets 4-point bending above 3-point bending is also that within the two central loading pins where bending moment is at a constant maximum the shear stress within this region is zero leading to pure bending only within this area illustrated in Figure 20 (Altstädt, 2015).

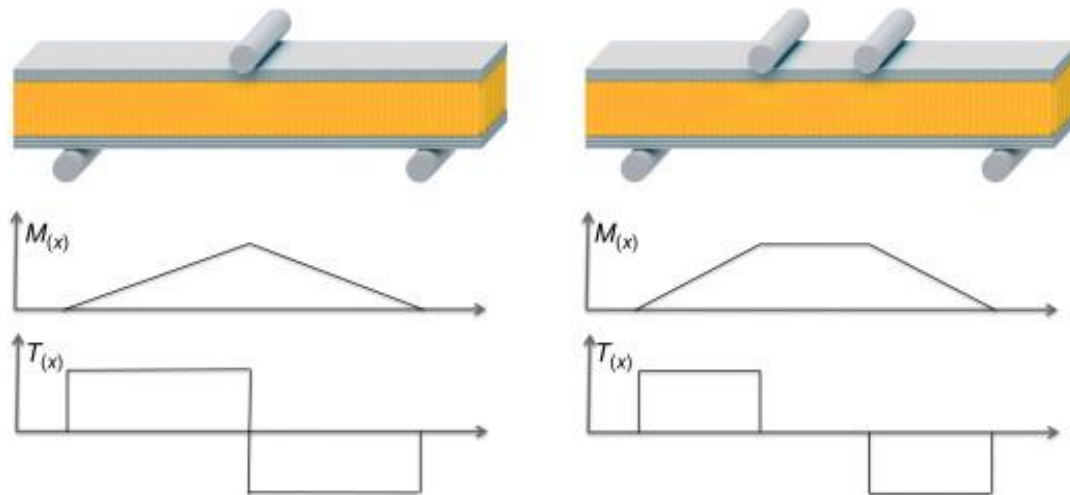


Figure 20 - Illustration of 3 and 4-point bending respectively with corresponding moment and shear diagrams

3.5.3 Microscopy crack testing

Light microscopy was used to observe crack formation and measure crack width on the samples after bending testing had been completed (day 7). The crack width was measured using a computer software, where I marked 3-4 locations along each crack of all samples and collected at 3 or 4 data points at each of these marked location. This allowed me to gain a better average of crack width at each marked location across the length of the crack that had been formed. After the samples had been left to heal for 7 days microscopical observations were conducted again to see any variations in the crack dimensions and pattern.

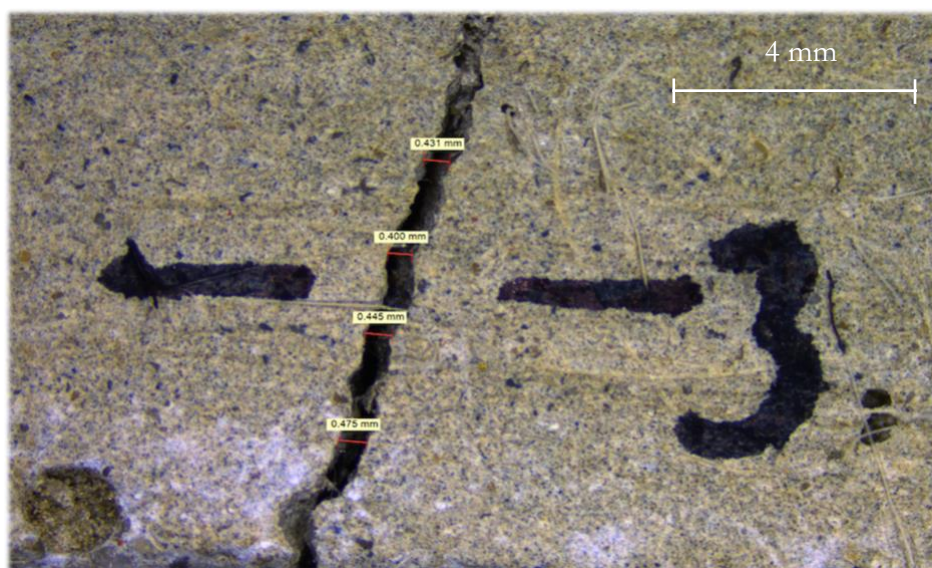


Figure 21 - Example of crack width data collected from one marked location (3rd in this case) on each sample.

4 Results and Discussion

Samples were prepared varying cement, fly ash and fibre contents to investigate the effect of their addition to the performance in lower cement content ECC mixes. Testing methods were outlined in the previous chapter. Compression testing was performed on day 1, 7 and 14 of the material life following demoulding. All bending samples were then cured in water up to day 7 and were then subsequently removed, subjected to a 4-point bending test and the resultant cracks analysed. To assess self-healing of cracks, samples were allowed to heal for 7 days where then crack results were to be reanalysed to identify intrinsic healing abilities in the ECC mixes. *Table 3* shows the composition of all 5 mixes tested. All mixes were tested in triplicates, and the mean values were reported. Any variations are shown in all graphs as error bars.

<i>Mix No.</i>	<i>Cement (wt%)</i>	<i>Sand (wt%)</i>	<i>Fly ash (wt%)</i>	<i>Water (wt%)</i>	<i>Fibre (% by total volume)</i>	<i>Water/binder ratio</i>	<i>FA/C</i>
1	25	62.5	0	12.5	1	0.5	0
2	25		0		0.5		0
3	25		0		1		0
4	12.5		12.5		1		1:1
5	8.33		16.66		1		2:1

Table 3 - Mix compositions tested

4.1 Workability

Notably, all of the mixes produced were initially very stiff, and showed low workability when handling. This provided difficulty in the moulding of each specimen, and ensuring sufficient compaction was achieved. Changes made from Mix 1 into Mix 2 and Mix 3 made a large difference in terms of manufacture of specimens and in reducing stiffness of the fresh ECC mix. A reduction on the volume fraction of fibres was pivotal in reducing the stiffness of the unmoulded mix as the fibres were the main reason of the increased stiffness of cementitious grout. Literature agrees with this finding where reduced slump was measured in samples with a higher volume fraction of fibres (Dasgupta, et al., 2020). Furthermore, in Mix 3, the introduction of superplasticiser allowed the mix to become more compliant or less stiff allowing for facile fabrication of samples. Another additive or viscosity modifier could have affected the mix in a similar manner (Bentz & Snyder,

2013) and could have been proven necessary if the volume fraction of fibres was increased beyond 1%. Mix 1 showed a visible sign separation between the sand and cement within the mix (see *Figure 22*) that was likely caused during the mixing process and the stiffness during mix generation. The fibres however were observed as separated well, as can be seen in a *Figure 21*, which shows the surface distribution of the fibres to be in a random nature with minimal clumping of the fibres.

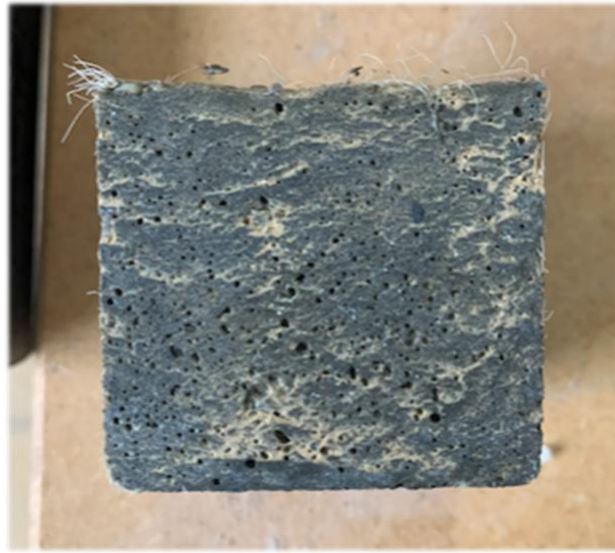


Figure 22 - Photograph of Mix showing some separation of constituent materials

The introduction of fly ash in Mix 4 & 5 made minimal observable impact on the stiffness/viscosity of the initial fresh mix and moulding ease.

4.2 Compressive Strength

One aspect of the project was to assess the unconfined compressive strength of the hardened specimens at 1, 7, 14 and 28 days age. However due to the Covid-19 pandemic, testing was interrupted and none of the 28 day compressive tests or any testing beyond 24 hrs for both Mix 4 and 5 was possible. Data for Mix 4 & 5 has been extrapolated, using average % strength gained in Mix 1-3 and against experimental strength development curves from literature of FA mixes (Yang & Li, 2007). These give a projected history of strength values up to 14 days as a visual comparison. *Figure 23* illustrates the compressive strength of each mix up to day 1.

The small error bars indicate that the uniformity across each individual mix was consistently high and that the production of specimens was effective in making all homogeneous mixes. On average

the standard deviation across all the mixes at every date measured was 1.232 MPa except for in Mix 2. The latter defies this trend, at day 14, where the standard deviation surged to 3.744 MPa with the values of the three specific compression strength data points individually being 30.206 MPa, 30.400 MPa & 23.819 MPa. The lowest reported can be considered an outlier and could be attributed to: 1) a flaw due to the specimen production such as air bubbles not being removed, 2) due to a pre-existing crack/flaw, 3) human error meant transfer of data led to an error in the value recorded or 4) instrument malfunction lead to an incorrect value being recorded.

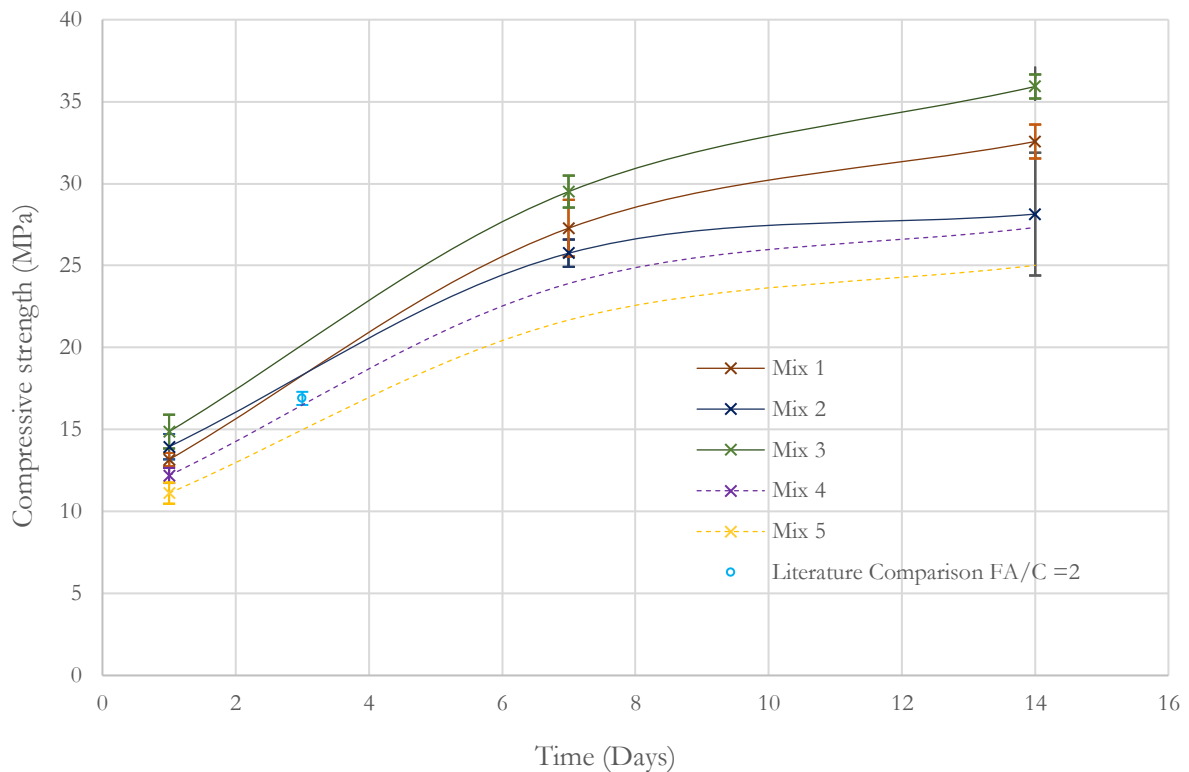


Figure 23 - Compressive strength data of each mix composition at Day 1, 7 & 14 days

Mixes 1-3 were all expected to reach very similar overall strength values as each of them have the same cement content with the same cement/water and cement/sand ratios. The introduction of superplasticiser explains why Mix 3 has a slightly higher strength than Mix 1 and 2 despite the same cement and water/cement ratios being used. A more uniform and homogenous mix may have been created using superplasticiser hence a more predictable chance of the full strength of the samples being found with a minimal variance in error. This is supported through comparative findings (Alsadey, 2012). Furthermore, results are in agreement with previous findings (Yang & Li, 2007), in terms of the negative effect on compressive strength of the addition of fly ash into

the ECC mix. A 10% and 16 % reduction in strength for Mix 4 and Mix 5, respectively, relative to Mix 1 were reported, with the only difference in mixes being the ratio of cement to fly ash used in the binder.

The results for Mix 4 & 5 beyond day 1 were extrapolated based on an average percentage gain in strength across Mixes 1-3. These extrapolated results were compared to a mixture used in (Yang & Li, 2007) which contains a FA/C ratio of 2, which is the same as used in Mix 5. Although ratio of cementitious material/sand used by the authors was higher relative to that of the mix investigated herein, to achieve higher compressive strength, results seem to be on the same order of magnitude, almost exactly matching the suggested projection line of Mix 4. At day 3 this projection lay within the range of results found in literature of 16.9 ± 0.4 MPa. This suggests that this extrapolation of results is acceptable and could be used to formulate conclusions.

4.3 Bending

Strain hardening is perhaps the most important ability/performance characteristic of ECCs. Therefore a 4-point bend test was carried out, on 3 samples per each mix, on day 7 in the process using an electro-servo universal testing machine (Instron 5567) with a maximum load capacity of 50 kN. The test was controlled by a constant midspan displacement of 0.5 mm/min for all specimens to maintain a continuity in testing methodology. Only Mix 1-3 were subjected to 4-point bending experiment (Day 7 of mix testing schedule). Due to Covid-19, bending tests for Mix 4, 5 and a concrete control couldn't be undertaken. Results will show that strain hardening was only achieved in Mix 3 with strain at failure reaching values of up to 0.32% , whilst producing only one singular crack in each sample individually.

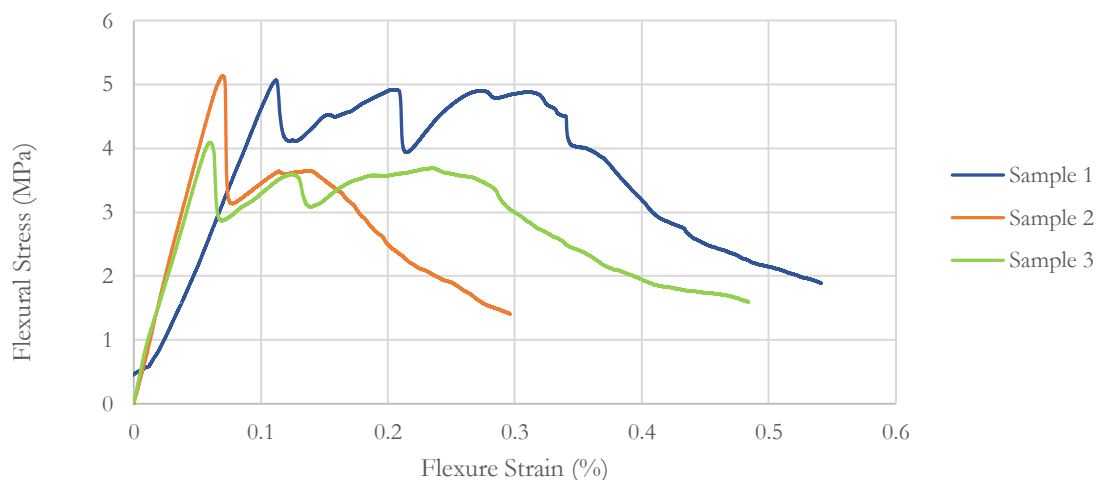


Figure 24 - Stress-Strain results from the 4-point bending test for Mix 1 (Day 7)

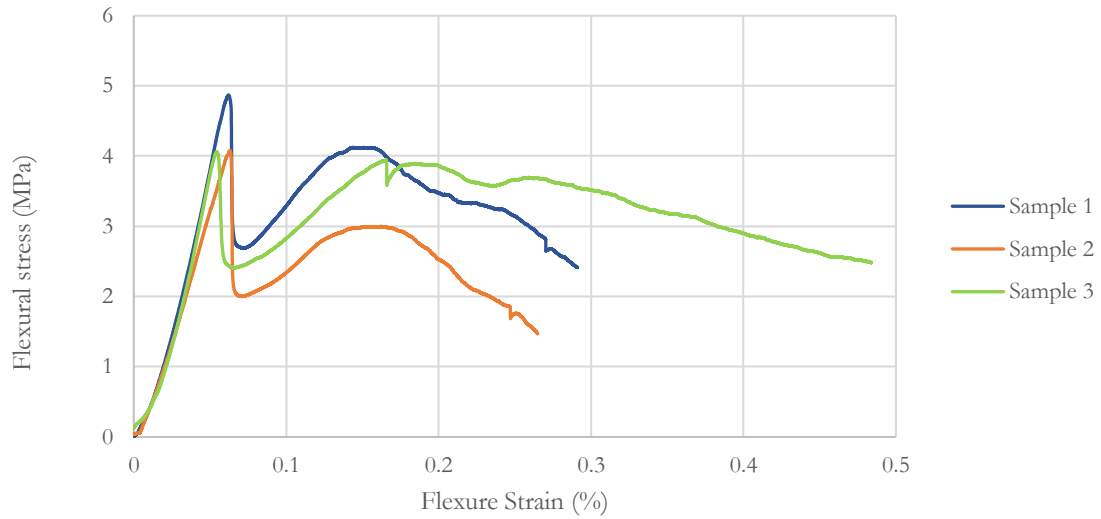


Figure 25 - Stress-Strain results from the 4-point bending test for Mix 2 (Day 7)

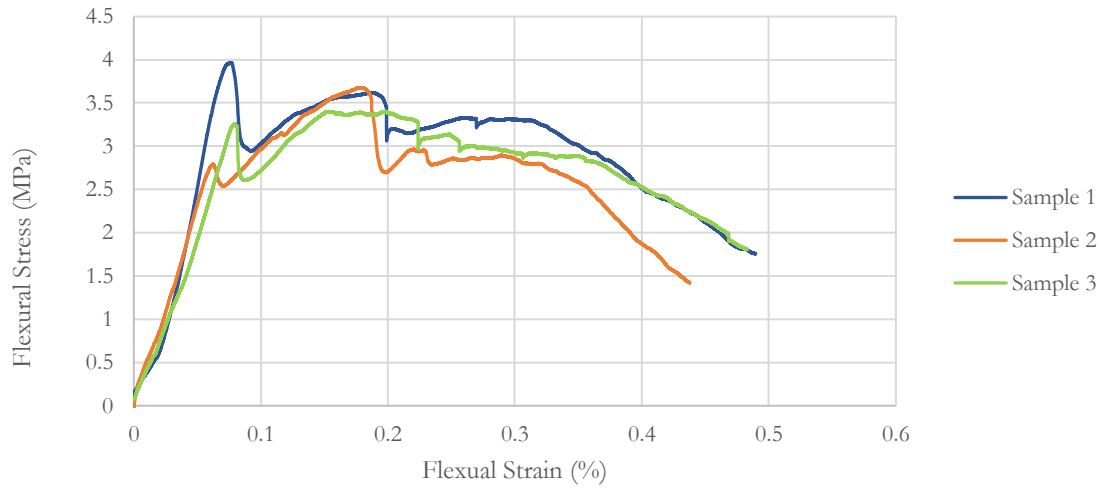


Figure 26 - Stress-Strain results from the 4-point bending test for Mix 3 (Day 7)

Mix no.	Strain at failure - ν_f (%)	Initial stiffness - E_0 (MPa/mm)	MOR 1 - σ_m (MPa)	MOR 2 - σ_f (MPa)
1	0.215	13.176	4.766	4.071
2	0.153	15.564	4.333	3.662
3	0.212	11.201	3.674	3.222

Table 4 - Summary of key findings, across all mixes, through 4-point bending tests

4.3.1 Singular crack production

First and most important to note across all mixes, all samples produced only one visible singular crack. As widely reported the defining ductile ability of ECCs to produce multiple small cracks across the tensile face of the material allows high deformability whilst suppressing brittle fracture (Li, 2019). Multiple cracking provides the tensile ductility or “give” to the material when overloaded beyond the elastic state. However, as can be seen in many of the samples (e.g. Mix 1, Sample 1 see *Figure 24*) more than one blip/drop of stress as strain increased, suggests more than 1 crack had been formed due to this release of energy. This drop that follows after the first cracking could also be the full release of the one singular crack as observed after the samples were removed from the testing rig. An example where this is likely to have happened, where the first cracking did not result in the full crack opening, is in Mix 3 Sample 2. Here the first cracking yielded a drop in stress of only 0.2 MPa. It then rose, beyond first cracking, to a maximum value of stress capacity of 3.67 MPa, akin to that of the other samples first cracking stress capacity suggesting initially only partial release of the crack.

4.3.2 Strain-hardening behaviour

Another dominant performance capability of ECCs is the intrinsic ability to have an increased stress capacity under strain also known as strain hardening (Li, 1993). This was not present in any sample in Mix 1 & 2 but was present in 2 out of 3 samples in Mix 3. Mix 2 shows us that reducing the fibre content below 1% by volume total leads to a clear reduction in performance relative to that of Mix 1. One clear peak is observed (first cracking) in all samples tested, suggesting no possibility of strain hardening beyond initial cracking as intended. A lower strain at failure (0.171%) and a reduced ability to maintain stress leads to a reduction in performance. A reduction of fibre content meant that less fibres were able to bridge the initial crack so the formation of another crack wasn't possible as that stress couldn't be maintained within the crack itself.

Developments were made in Mix 3 in which 2 out of 3 samples (samples 2 and 3) strain-hardened under 4-point bending (stress strain plots shown in *Figure 26*). Here the modulus of rupture (MOR) did not occur at first cracking unlike in the prior two mixes. Sample 3 showed the highest hardening stiffness of 1.64 MPa/mm. However this value is greatly affected by the first cracking stress of this sample, as mentioned prior, with these samples only experiencing partial opening of the initial crack. Stress values at first cracking were 3.32 ± 0.62 MPa and then rose up to a maximum value of stress (MOR) of 3.66 ± 0.28 MPa.

4.3.3 Comparison to Standard Concrete

For comparison a Typical stress-strain curve of a concrete taken from literature is included (Wittmann, 1989), see Appendix A. Although formation of only 1 crack in all samples occurred, if we were to compare against ordinary concrete, it is clear that a gain in maximum strain at failure has been achieved. In the case of Mix 1 and Mix 3 a 2031% and 1998% increase was achieved respectively, where a 1% volume fraction of PVA fibres were used. *Figure 27* confirms reducing the volume fraction of fibres down to only 0.5% hinders the ductility of the material with only a 1388% increase in strain capacity however this is nevertheless a significant increase in performance relative to that of the ordinary concrete mix. However, if one were to compare it against ECCs mixes found in literature the opposite could be said with the strain at failure lying well below the suggested range of failure strains of ECCs, being in the region of 3-12% (Van Zijl, et al., 2011) a factor of 50 out. General modification to aim for greener lower strength applications such reduced fibre content, elimination of the use of additives or the increased water content have led to a unclear reason as to the reduction in performance compared to a standardised ECC mix used in literature - M45-ECC (Wang & Li, 2007).

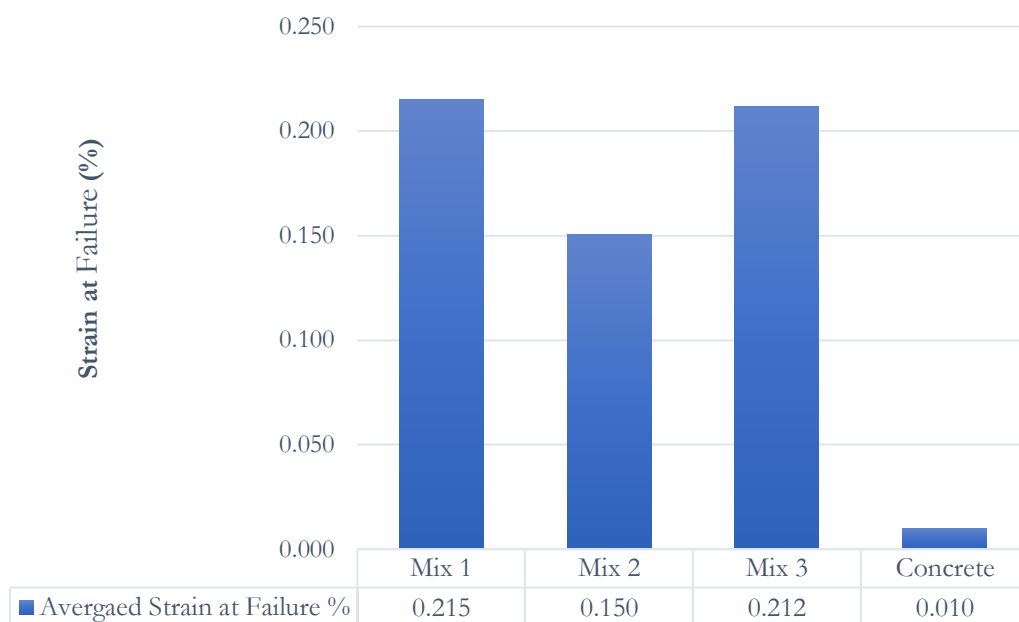


Figure 27 - Strain at failure of Mix 1-3 compared against a standard concrete mix

4.4 Crack Healing

Optical microscopy was used to observe crack formation and measure crack width on the samples after 4-point flexural testing had been completed (Day 7). After healing microstructural characterisation was performed again to assess the damage levels. The crack widths were measured to observe any noticeable changes. *Figure 30* shows characteristic crack analysis for one sample and its images produced using light microscopy before and after 7 days of healing. Here three discrete locations were chosen along the crack length and crack width was measured with four data points at each discrete location. An example is given in *Figure 28* & *29* for Mix 3, Sample 1. Full data was collected across all mixes and their specimens at each individual analysed location.

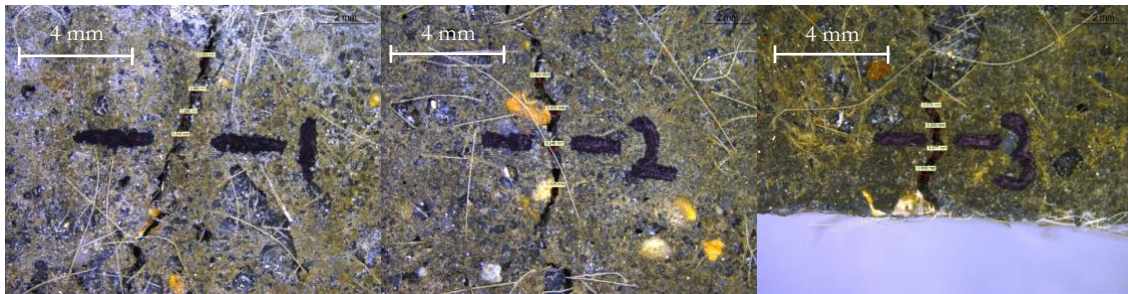


Figure 28 - Crack analysis of each marked location of Mix 3 Sample 1 (Day 7)

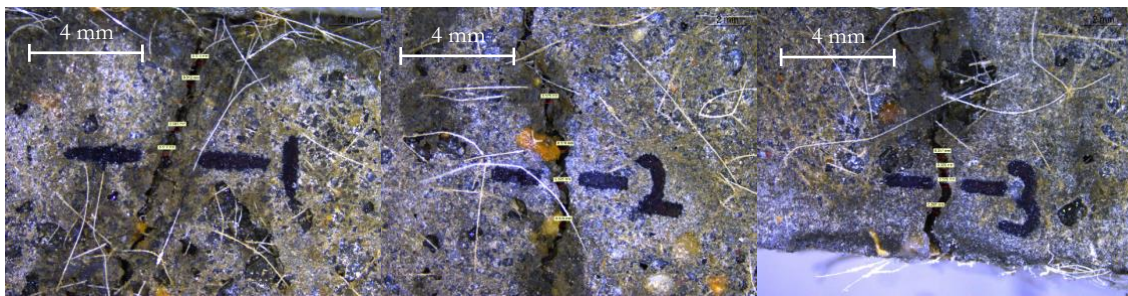


Figure 29 - Crack analysis of each marked location of Mix 3 Sample 1 (Day 14)

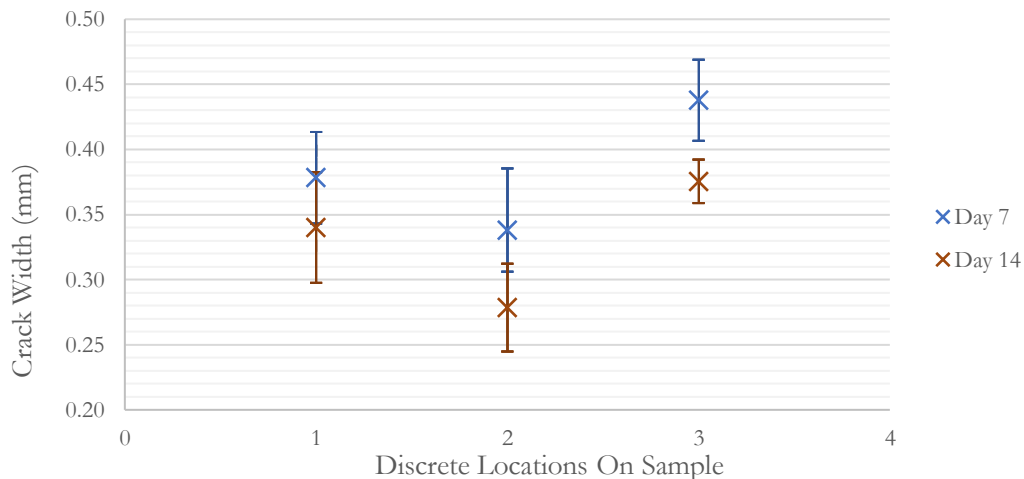


Figure 30 – Subsequent crack width data collected from digital light microscopy of Mix 3, Sample 1

4.3.1 Cracking Prior to Healing

With only 1 crack produced in all of the specimens, larger crack widths were observed compared to what was anticipated, as normally bending in ECC would have resulted in finer and more distributed cracks for the same strain value reached. This is because the bending/strain of the material largely comes from the opening of cracks rather than the flexure of the material matrix itself. With that in mind the crack size of ECC mixes is typically lower than 100 μm (Li, 2003). However as can be seen in *Figure 31* the crack widths analysed averaged at 320, 280 & 370 μm for the three mixes independently, lying well outside the range of typical ECC cracks. The smallest size of initial crack width was produced in Mix 2. This was achieved due to the failure strain of the mix being the lowest as there was not sufficient amount of fibres to bridge the crack after first cracking so the largest strain-softening was felt. This is supported by Mix 1 & 3 exhibiting a failure strain of 43% and 41% greater than Mix 2 resulting in the formation of initial crack size increase of 15% and 28% respectively.

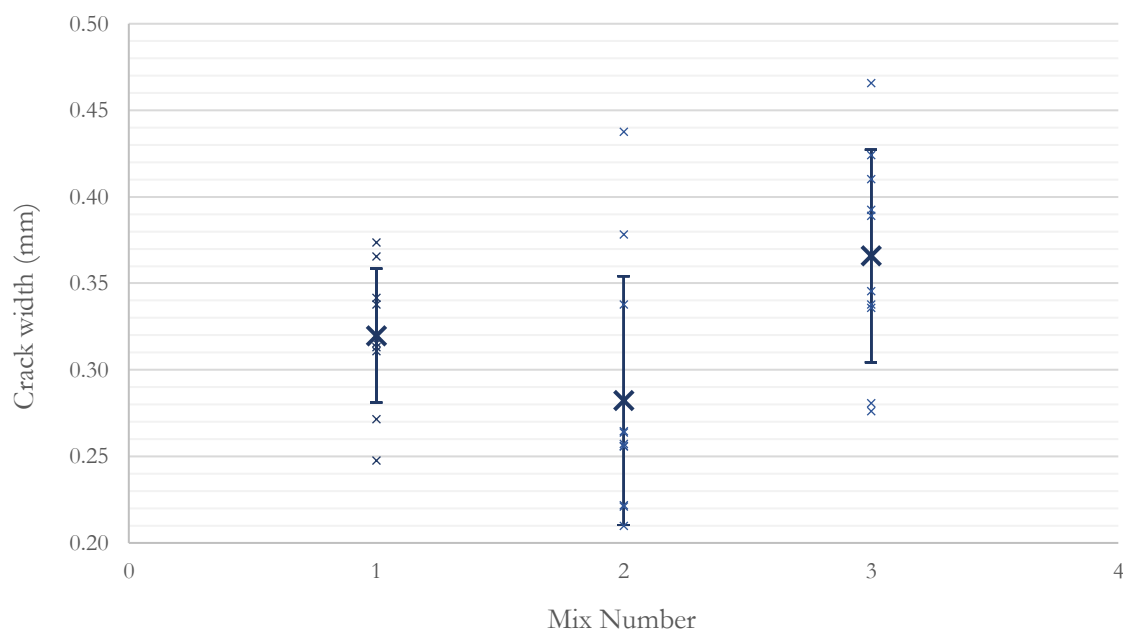


Figure 31 - Initial crack width for mixes 1-3 measured after 4-point bending prior to any healing

4.3.2 Cracking after healing

After the loading/cracking of the sample they were re-submerged in water for 7 days, to assess self-healing capacity. After that period lapsed microstructural characterisation was performed to assess the damage levels. The crack widths were measured to observe any noticeable changes.

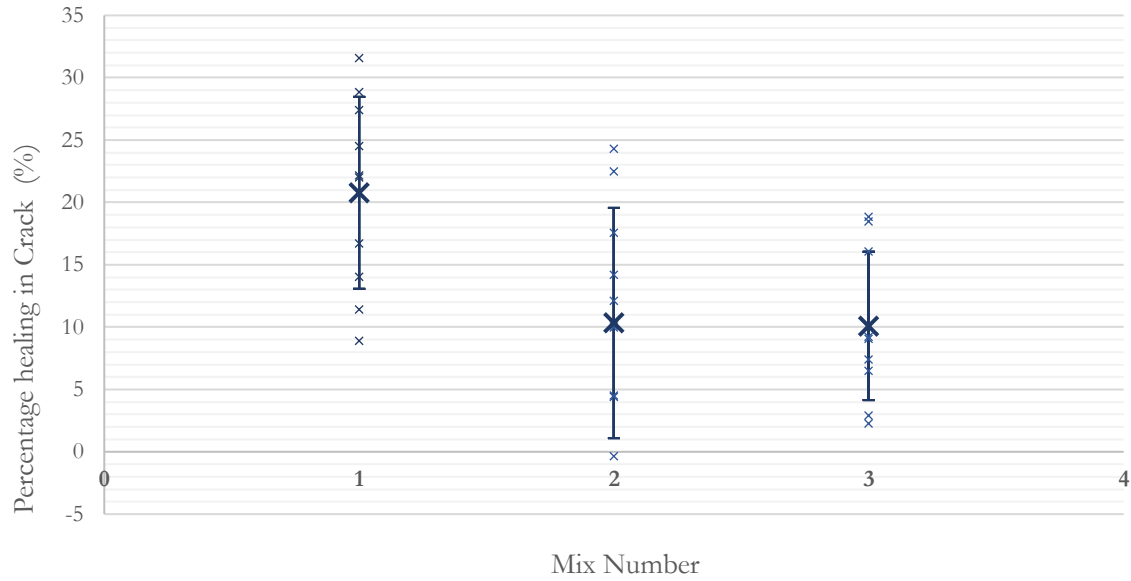


Figure 32 – Percentage healing of crack for each mix tested

All samples showed visible signs of self-healing through a reduction in crack width over the 7 day healing period. *Figure 33* shows the % reduction in crack width (% healed), from day 7 to day 14. Mix 1 resulted in the greatest average reduction in crack width of 20.7% with individual crack locations ranging from an 8.8% reduction all the way up to 31.6% reduction in crack width. Mix 2 and 3 had a lower average reduction of crack width of 10.3% and 10.0% respectively. The lower value of healing found in Mix 2 was again down to the reduction of fibres present bridging the gap of the crack. Studies have shown that, as far as fibres are concerned, fibres within the material can be incorporated to control and limit crack widths (Cuenca & Ferrara, 2017) & (Homma, et al., 2009). The highest initial crack width of Mix 3 lead to the smallest % reduction of crack width confirming the effect of initial degree of damage on the healing efficiency (Yang, et al., 2005).

As can be seen in *Figure 29* on day 14 relative to that of day 7 there is a build-up of a white precipitant on the surface and within the crack of the sample primarily attributed to the formation of calcium carbonate (Van der Zarwag, 2007). Furthermore, some unhydrated cement within the ECC matrix may have also contributed to further hydration and production and precipitation of further hydration products in the crack opening. However, due to the reduction of the cement/pozzolans in my mixes tested relative to that of high strength ECC as reported in literature, the latter contribution of further hydration will be of smaller effect. These chemical causes will all further reduce the crack width through aiding with the healing of the material. Mechanical causes such as fine particles/minerals originating in the water or small particles broken off during fracture

could also aid in the healing process. A more detailed explanation of autogenous healing has been published (De Rooij, et al., 2013).

5 Conclusions

It is discernible that many factors have pointed towards the importance of getting lower embodied energy and carbon intensities (representing resource use and pollutant emissions) in ECCs. As for most fibre reinforced cement-based materials, ECC tends to use higher cement content due to the elimination of coarse aggregates. Furthermore, the use of fine manufactured silica sand and synthetic oil-based fibres also increases the material energy, carbon intensities and cost over that of normal concrete. There is, therefore, a strong incentive to develop greener versions of ECC. Greening of ECC, however, must be performed with care so that the ductility and durability (performance) of the material are not compromised.

The overall homogeneity of the mixes tested was proven to be held to a tight tolerance with the analysis of compression testing revealing that samples produced a narrow spread of results, with low individual standard deviation. However, Mix 1 showed observable signs of separation within the mix itself. The hardened mix had apparent separation between the sand and the cement within the samples. This alongside of the high stiffness/low workability during mix production suggests that improvements would be beneficial to the mix generation to ensure higher quality of sample production. The introduction of further additives or the presence of a viscosity modifier into the mix could make this an achievable concept.

Low compressive strength values of 30 MPa (tested at day 14) were easily achieved through the reduction of cement content relative to that of high performing ECCs, which can achieve strength value ranging up to 150 MPa (Van Zijl, et al., 2011). It was further found and confirmed that the introduction of higher ratios of FA/C decreased the unconfined compressive capacity further, as expected from literature (Yang & Li, 2007).

Results found that the lowering of cement content had major effects in terms of the number of cracks produced within the ECC mixes during flexure testing. The formation of one crack only, led to a decrease of strain at failure to a range outside of that usually denoted with ECCs. Typical ECC mixes are suggested to have a range of failure strains anywhere between 3-12% (Van Zijl, et

al., 2011), however the investigated samples produced values of only 0.215%, 0.150% and 0.212% across the three mixes tested.

Furthermore, the reduction of fibre content below 1% was detrimental to the ductility/elongation performance of the ECC mix. The number of fibres present provide insufficient strength to bridge the crack formed hence no strain hardening occurred and the lowest failure at strain was recorded at 0.150%. One clear peak in stress was seen in each of the samples tested, as seen in *Figure 25*, which confirmed this lack of bridging effect making the matrix behave in a more brittle manner more like regular concrete.

The large crack width, due to the formation of only one crack in all the samples tested, and the decrease of typically high volumes of unhydrated cement particles and pozzolans in high strength ECC have limited the capacity to self-heal as effectively as other tested ECC mixes in literature. Authors have reported recovery close to 100% even for specimens deliberately pre-damaged up to 3% tensile strain (Yang, et al., 2009). However in those examples numerous cracks were observed with widths bellow 100 μm (Li, 2003). As can be seen in *Figure 31*, the crack widths analysed in my experiments averaged at 320, 280 & 370 μm . Lying well outside the range of ECC crack.

In summary, the results stated in this report demonstrate that a reduction in performance characteristics has occurred when lowering the cement content of the ECC mixes. Primarily due to the formation of only singular cracks in each specimen resulting in lower strain at failure being documented. However the benefits of greener lower strength ECCs become evidently clear in contrast to the inherently brittle of concrete, in which ECC could act as a direct replacement for future applications. As such, ECCs may provide a value of strain at failure of 20 times greater that of normal concrete with a comparative compressive strength. Further with their intrinsic ability to self-heal, resulting in the eradication of need for repair and maintenance, these ECCs have opened a new world of possibilities to enhance the safety, durability, and sustainability of civil infrastructure marking a definitive progression forward towards more resilient infrastructure.

6 Further Research

6.1 Short term

With the project being so time limited and the unprecedented situation created due to Covid-19, there are a few elements of my project that could not be achieved in the time frame available. Further, looking back in hindsight other operations could have been looked into to answer any further unanswered questions that have arisen during the progression of the project..

With the project timetable reduced and its short time frame generally, if I were to have greater time, I would have liked to explore the effect of healing time on the crack width of specimens as the intrinsic self-healing of ECCs plays such an important role in providing beneficial properties over that of more well used cementitious materials. In this project the samples only had 7 days after initial cracking to show healing capabilities. Ideally day 14, day 28 and beyond would have been analysed to give an enhanced understanding of the reduction of crack width with age and to see if at all this allowed the material to fully heal. An increase of time would have further given the opportunity to see how a healed sample would have performed if 4-point bending tests would have been re-run. These loading-healing cycles would have given valuable insight into the extent to which mechanical performance could have been regained under self-healing.

Fly ash comprised a large part of my literature prior to starting experimental work, therefore, I would explore further and try to confirm the benefit of using high volumes of fly ash in my green lower strength ECCs. As we saw an increase of fly ash content reduced the compressive strength of ECC therefore it would be interesting to see whether it would also provide what was necessary in increasing the number of cracks produced and help these mixes exhibit strain hardening behaviour. Questions remain regarding whether this would reduce the observed crack width? Or if there would be an increase in the strain at failure of the lower strength mixes?

The exploration of other Supplementary Cementitious Materials (SCMs) or a combination of various components could lead to further developments towards greener or higher performing materials. Other questions that appeared during the manufacture of the samples, that would have provided further insight, would be to quantify more accurately the effect on the workability. This could have been achieved through performing flow table tests/slump tests much like in methods performed in literature (Dasgupta, et al., 2020). This would further give an indication into the effective distribution of fibres within each mix matrix.

Additionally, the stress strain plots in *Figures 24, 25 & 26* initially suggest the release of energy/reduction of stress at more than one location along the specimen or could have been the release of 1 singular crack in stages. With only the chance to look at the samples after failure it was an almost impossible task to quantify which part of the crack opening had led to which individual drops/blip in stress (as per the stress-strain plots). With this being said, a method of viewing the sample, such as a timestamped video, during loading would have provided extra information in terms of identifying the generation of specific crack openings. With the introduction of numerous cracks this could also provide extra data to pinpoint which cracks opened at which drop in stress.

6.2 Long term

Longer term, further expansion of this research could include:

Larger Scale Laboratory Testing: Larger scale testing of all behaviour of the mixes whilst also assessing them in soil applications within the lab would be necessary in terms of gaining a fuller understanding of the materials nature.

Full Scale Field Testing: This will allow us to see the behaviour of the greener lower strength ECCs real field applications and conditions, outside of the lab environment, in order to be able to replace that of other low strength cementitious materials.

Whole Life Cost (WLC): A WLC calculation should be done in order to establish the financial benefits due to the introduction of green low strength ECC. The WLC should include:

1. Initial cost of procurement including constituent material costs.
2. Operational costs including running costs and one-off costs associated with the materials.
3. Maintenance, repair and disposal costs.
4. Non-financial environmental cost associated with the constituent materials and construction methods.

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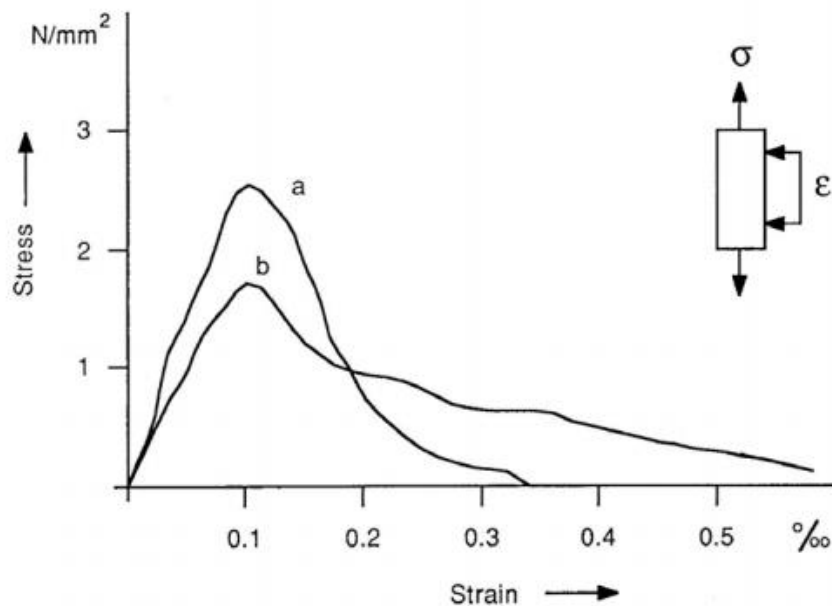
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8 Appendix A



Completed stress-strain diagram for normal concrete (a) used as a comparison against ECC mixes produced. Taken from (Wittmann, 1989) “Structure and Fracture Mechanics of Composite Materials”. Note the unit of strain used is permille ‰ (part per thousand parts) not percent % (part per 100 parts).

8.1 Risk assessment Retrospective

The main risks in this project primarily arose from using machines during testing and manufacture, the use of hazardous materials and respiratory risks. Both hazardous substance risks and mechanical risks were assessed fully. A low risk of inhaling dust was present due to the presents of dust from fine particles, due to pouring of component materials, but was a avoided with a use of a dusk mask and increasing ventilation in the work area by opening windows. Furthermore, there was a low residual risk due to handling materials like cement that can cause burns. This was avoided by the use of gloves and lab coat as well as eye protective goggles at all times whilst in the lab. A low residual risk of HAVS (Hand Arm Vibration Syndrome) was present due to the use of a vibration table but this was avoided by not using the vibrating table when not need and not touching or resting on the table when in use.

If I was to risk assess the project again, I would have fully planned the manufacturing and testing procedure in advance so that the risk assessment could give specific risks for specific equipment as opposed to equipment in the generalised sense.

9 Appendix B

Disrupted lab work all due to closure of the lab during red phase of university corona virus plan and due to nationwide lockdown. Due to closure of labs the amount of data collected was roughly half that of what I was expecting to produce. Therefore a distinct shift in my report was made towards literature review as the results section had been thinned.

- Bending testing – New Civil Engineering Building - West Cambridge
 - Mix 4-6 - 3 further mixes had been prepared (Mix 4 &5) introduction of fly ash and (Mix 6) standard concrete comparison. These were all going to be subjected to 4-point bending testing using the Instron machine in the new civil engineering building on 20/03/2020
- Unconfined Compression Testing – Geotechnical Engineering Lab- Main site
 - Mix 1
 - Day 28 – 27/03/20
 - Mix 2-3
 - Day 28 - 03/04/20
 - Mix 4-5
 - Day 7– 20/03/2020
 - Day 14 – 27/03/2020
 - Day 28- 10/03/2020
- Optical Microscopy (Leika Microscope) – Geotechnical Engineering Lab -Main Site
 - Mix 1
 - Day 21 (14 Day healing time)
 - Mix 2-3
 - Day 21 (14 Day healing time)
 - Mix 4-6
 - Day 14 (7 Day healing time)
 - Day 21 (14 Day healing time)

I had to move back home limiting the amount of photos that I could have utilised in my final report.