Effect of Supplementary Cementitious Materials on Mechanical Properties and Self-Healing Efficiency of Engineered Cementitious Composite

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Submitted to the
Institute of Graduate Studies and Research
in partial fulfillment of the requirements for the degree of

Master of Science
in
Civil Engineering

Eastern Mediterranean University
August 2018
Gazimağusa, North Cyprus

Approval of the Institute of Graduate Studies and Research

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ABSTRACT

Concrete is considered as a fundamental construction material as it has high compressive strength and fairly low cost. However, brittleness behavior and limited tensile strength can be remarkably observed in conventional concrete and accordingly, crack formation easily occurs. Many researchers have dedicated considerable efforts to adjust the brittleness characteristic and other weak points of conventional concrete and thus, they come up with the development of a new type of concrete, which is named engineered cementitious composite (ECC).

In this experimental study, it is aimed to investigate the effects of replacing slag (S) 100% with fly ash (FA), in different proportions with limestone powder (LSP), and glass powder (GP); on workability, mechanical properties, microcracking behavior and self-healing efficiency of ECC. For this purpose eight different ECC mixtures were prepared; at first reference sample is produced with a certain amount of cement and slag as a binder, then slag is replaced 100% with fly ash, 5, 20 and 40% with LSP and 20, 40 and 60% with GP by total mass of slag. In order to determine and compare the effects of slag, fly ash, LSP and GP on workability, mechanical properties and behavior and self-healing efficiency of ECC; flow test, compressive and splitting tensile strength tests at different compressive loading levels, stress-strain diagrams tests under compression were conducted. In addition, self-healing efficiency of eight different ECC produced were determined by means of ultrasound readings and stereomicroscope measurements. Here, cracks were created on the surface of specimens by preloading them up to ultimate splitting tensile strength. After that, microscope and ultrasonic test were utilized to observe and determine the self-healing recovery rate. Overall, replacing slag with LSP up to 5% and with GP up to 20% displayed significant improvement on mechanical properties of ECC and a high relative comparative of self-healing efficiency with ECC-Ref. Calcium carbonate and C-S-H gels were observed to be the prominent healing products.

Keywords: Engineered cementitious composites (ECC), self-healing efficiency, supplementary cementitious material (SCM), glass powder (GP), limestone powder (LSP)

ÖZ

Beton, yüksek basınç dayanım özeliği ve de düşük maliyetli olması sebebiyle en temel inşaat malzemesi olarak kabul edilir. Ancak, betonun gevrek yapıya sahip olması, ve de düşük çekme dayanımı sebebiyle çatlak oluşumu olasılığı yüksektir. Bu yüzden geleneksel beton üretiminde bu konulara dikkat edilmesi gerekmektedir. Pek çok araştırmacı, betonun kırılganlık karakteristiği ve diğer zayıf özeliklerini azaltmak veya gidermek için önemli çabalar harcamış ve böylece, yüksek çimento içerikli kompoze bir beton tipi üretilmiştir (MKB).

Bu deneysel çalışmada, cüruf (C) yerine %100 uçucu kül (UK), farklı oranlarda kireç tozu (KT) ve cam tozu (CT) kullanılmasının; betonun işlenebilirlik, mekanik özellikleri, çatlak davranış ve MKB kendi kendini iyileştirme verimliliği üzerindeki etkileri araştırılmıştır. Bu amaç için sekiz farklı MKB karışımı hazırlanmıştır; ilk referans karışımı, çimento ve cüruf bağlayıcı olarak hazırlanmıştır. Daha sonra cüruf yerine uçucu kul % 5, 20, 40 oranlarında ve kireç tozu % 20, 40 60 oranlarında cüruf toplam kütlesi ile değiştirilmiştir. Bu karışımların etkilerini belirlemek ve cüruf etkileri ile karşılaştırmak için işlenebilirlik, mekanik özellikleri ve davranışı ve kendi kendini iyileştirme verimliliğini tesbiti için; işlenebilirlik testi, basınç ve yarmada çekme dayanımı testleri, farklı basınç düzeylerinde çatlak oluşumu ve giderilebilmesi çalışmaları için basınç yükü altında gerilme - şekil değiştirme diyagramları testleri yapılmıştır. Buna ek olarak, üretilen sekiz farklı MKB betonunda kendi kendine çatlak oluşan çatlakların giderilmesi işlemi öncesi ve sonrasında ses dalgaları ile firkin tesbit edilebilmesi deneyleri yapılmıştır.

Genel olarak, %5 cüruf, kireç taşı ve cam tozu ile önemli MKB betonun mekanik özellikleri ve de çatlak giderilmesi tesbit edilmiştir. karbonat ile kendi kendini onaran bir yüksek göreli karşılaştırmalı olarak görüntülenen %20 C-S-H jelleri olmak gözlendi önde gelen şifa ürünleri.

Anahtar Kelimeler: İşlenebilirlik, puzolanik malzemeler, kendi kendini onaran beton, kompozit beton (MKB), cam tozu (CT), kireç tozu (KT).

ACKNOWLEDGMENT

I would like to express my sincere gratitude to those who helped me and gave me any kind of support they can throughout this study. First and foremost, my deepest appreciation goes to my supervisor Asst.Prof. Dr. Tülin Akçaoğlu for her continuous support during my Master studies and research, her patience, kind advice and willingness to help me anytime. I had a pleasure to know and work with such a smart, professional and kind woman. Her guidance in conducting the experiments step by step and building the content of the thesis means a lot to me. She had an enormous contribution to what I have done, and I cannot imagine a better mentor for my studies.

Besides my supervisor, I would like to thank the laboratory staff, especially Mr. Ogün Kılıç and Mr.Orkan Lord for all their guidance and help. I would like to extend my deepest gratitude to my dear friends Mahdi Alnono and Shahnoza Abdulkhamidova, who did not hesitate to assist me during preparing the study specimens and writing the thesis. Moreover, they gave me confidence and encouraged me to do my best.

My honest gratitude and deepest appreciation goes to the civil engineering department staffs for what they have done for me and for the unlimited support that I have received during my study and my laboratory work.

Last but not least, I thank my family for giving me the opportunity to pursue education abroad, love and empathy they have shown me in whatever I do. I would not be able to come this far without their continuous support and inspiration.

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LIST OF ABBREVIATIONS

BFS	Blast Furnace Slag
CEM	Cement
ECC	Engineered Cementitious Composite
FA	Fly Ash
GP	Glass Powder
HRWR	High Range Water Reducing
LSP	Limestone Powder
MOR	Modulus of Rupture
OPC	Ordinary Portland Cement
PP	Polypropylene Fiber
PVA	Polyvinyl Alcoholic Fiber
S	Slag
SCM	Supplementary Cementitious Material
SP	Superplasticizer
W	Water
Γ	Flowability
W/B	Water-Binder Ratio

Chapter 1

INTRODUCTION

1.1 Background of the Study

Recently, concrete that sufficiently possesses considerable compressive strength has been utilized for several structural engineering projects and objectives. However, the brittleness property is still clearly displayed for conventional concrete. It has been observed that the relation between the compressive strength and brittleness characteristic is often incremental and thus accordingly, many possible constraints and restrictions can be created, which limit the use of high-strength concrete in many structural applications. Producing high ductile concrete in seismic regions is highly recommended as a result of its high seismic response and its ability to absorb the energy. Consequently, it was predicted that the improvement of cementitious ingredients with high ductility would be precious and useful for structural functions. Widespread research has come up with a composite material termed engineered cementitious composites (ECCs) that possess features of high strength concrete with increased tensile strain capability (Hillerborg, A. 1983).

Cracking reduces the durability of concrete constructions by generating pathways for detrimental agents to penetrate to the structure and possibly attack the reinforcing steel or the adjacent concrete that surrounds the steel bars. Cracks can also deteriorate the structure by deleteriously influencing the mechanical properties of the concrete. Thus innovating a concrete, which can heal itself and recover the lack of the performance due to cracking occurrence is greatly desirable and indispensable. Studies have revealed that cracks possess the capability to heal and seal themselves during time when water is used as a curing condition. It has been proved that the permeability of damaged cementitious materials is gradually reduced as long as water is permitted to flow throughout the cracks. This reduction in permeability is in fact because crack widths are diminished as cracks are clogged with healing products. In some intense cases that display small crack widths, cracks can be entirely healed, therefore augmenting the durability of the damaged concrete (Granger et al, 2007)

ECC type concrete that has been developed in the last decades; may lead to create safer, more durable, and maintainable concrete infrastructure that is economical and constructed with conventional construction equipment. ECC reveals ductile behavior with maximum two percent by volume of short discrete fibers. Normal concrete breaks in a brittle manner when subjected to flexural load. Nevertheless, ECC forms a very high curvature before failure at significantly higher loads. Great inelastic deformation in ECC is achieved due to multiple micro–cracks having widths limited between 60 and 100 micron (about half the diameter of human hair) (Ramya et al, 2014).

Engineered Cementitious Composite (ECC), also titled as bendable concrete, is an effortlessly molded mortar-based composite reinforced with particularly designated short random discontinues fibers, usually polymer fibers. Different from normal concrete, ECC exhibits a strain capacity, which is ranging from 3 to 7%, comparison with 0.1 % for ordinary Portland cement (OPC). ECC thus behaves more like a ductile metal rather than a brittle glass (as does OPC), resulting in a wide-ranging variety of functions and applications. ECC similarly looks like ordinary Portland cement-incorporated concrete, except that it does not incorporate coarse aggregate and can bend under strain. ECC has been widely utilized in a diversity of civil engineering applications; ECC displays superior characteristics such as high ductility and improved strain-hardening characteristic, which can be effective for applications that require seismic resistance due to its high damage tolerance, its ability to absorb energy and bend under shear (Ramya et al, 2014).

The amount of cement used in ECC is approximately five times higher that is utilized in conventional concrete, which results in high shrinkage, increased hydration heat and higher expenses needed for construction. The augmented employment of ECC with high utilization of cement brings about significant increase in the amount of CO_2 emissions, considerably contributing to cause health risks due to pollution and leads to global warming. Therefore, in order to suppress the deleterious results of using cement in high proportions and to improve the mechanical and durable properties of ECC, supplementary cementitious materials that characterize pozzolanic behaviors such as fly ash, glass powder, silica fume and granulated blast furnace slag are successfully substituted with cement (Altwair et al, 2012).

1.2 Significance of the Study

It has been found that the crack width is the prominent parameter that significantly affects the durability of concrete. Limited tensile strength of concrete and its brittleness characteristic result in forming wide cracks, which endanger the durability. Moreover, self-healing of wide cracked concrete is difficult to achieve. To address this problem, ECC concrete is produced with its remarkable ability to generate tight controlled cracks and thus, improve the durability and highly display self-healing efficiency and capacity. In addition, the second importance of this study is to investigate the effect of utilizing different proportions of supplementary cementitious materials (SCM) on mechanical properties of ECC and ECC self-healing efficiency. It is worth noting that measuring the optimal percentage of SCM that has a considerably effect on self-healing efficiency and capacity is one of this study objectives.

1.3 Objectives of the Study

The main objectives of this research can be summarized as follows:

1) To evaluate the optimal performance materials required to produce engineered cementitious composite concrete. Moreover, trying to identify the obstacles and restrictions that may reduce the efficiency of work to be remedied in the later research.

2) To investigate the effect of supplementary cementitious materials on workability, compressive and tensile strengths, microcracking behavior under uniaxial compression and with self-healing efficiency of ECC by water intrusion method.

3) To determine the optimal SCM that possesses the significant effect on ECC in terms of mechanical properties.

4) To evaluate the compressive strength recovery, by means of compressive strength measurement and ultrasound readings after one month of water curing healing process.

5) To investigate the self-healing possibility of the produced eight different mixes, with water intrusion method; by measuring the surface crack widths of each ECC both before and after healing process using stereo microscope with 210X magnification.

1.4 Outline of the Study

This research consists of five chapters that are arranged as follows:

Chapter one is about the introduction of this study and in chapter two; literature review about the topic of the study is summarized. Chapter three explains the methodology used to achieve the objectives of this study. Results and discussions are demonstrated and analyzed in chapter four. Lastly, chapter five displays the conclusions of this study.

Chapter 2

LITERATURE REVIEW

2.1 Introduction

Concrete is considered as a fundamental construction material as it has high compressive strength and fairly low cost. However, brittleness behavior and limited tensile strength can be remarkably observed in conventional concrete, thus crack formation easily occurs. Crack formation adversely affects sustainability of structural buildings as a result of the leakage of certain deleterious liquids and gasses that results in serious damages to concrete buildings and due to possibility of wide crack formation. In order to minimize the impact of formation of the cracks and their propagation, steel reinforcement is combined with concrete to suppress the width of cracks. Yet, it has been shown that steel is not sufficient to completely restrain forming the cracks. Accordingly, cracks might propagate widely and the steel reinforcement might be subjected to harmful environmental conditions resulting in steel corrosion. Therefore detection, precaution and maintenance of cracks become indispensable. However, maintenance of crack is considered troublesome and costly at the same time due to its invisibility and inaccessibility.

Cracks have been recognized as a prominent parameter that profoundly impact the durability of concrete, which occur as a result of external factors, harsh conditions and lack of work and concrete qualities. Brittle property of concrete leads to the structure becoming less durable. Therefore, ductility improvement of concrete becomes an urgent need and highly desirable. Moreover, reducing the width of the cracks to the farthest extent has become a significant point of attention through different techniques. Self-healing phenomenon is deemed one of the most effective techniques to eliminate cracks, which is usually associated with continuing hydration reactions and crystallizations in concrete. Using ECC may significantly enhance the fulfillment of self-healing, and accordingly, improve the long-term performance and durability of concrete structure.

2.2 Definition of ECC

Recently, many researchers have dedicated considerable efforts to adjust the brittleness characteristic of conventional concrete and thus they come up with the development of a new type of concrete, which is named engineered cementitious composite (ECC) displaying durable characteristics under wide range of environmental conditions. ECC is a new category of ultra-ductile fiber reinforced concrete depending on the theory of micromechanics design firstly originated in the beginning of 1990s at Michigan University (Li VC, 1993).

ECC comprises cement, fine aggregate and fiber. The amount of cement used for this type of concrete is five times higher than what used for normal concrete resulting in increasing the CO_2 emissions, serious health risks and severe environmental damages. Therefore, distinct supplementary cementitious materials (SCMs) such as fly ash (FA) and slag (SL) are substantially substituted to cement in order to diminish the deleterious effects of cement. Their pozzolanic properties display improvements to fresh and mechanical properties of concrete. Moreover, SCMs can be easily available and require relatively lower cost compared to cement (Yildirim et al, 2015b).

ECC is also considered as a bendable concrete, which is distinguished by high-ductile characteristics from 3 to 7%, tight crack width ranging from around 60 μm to about 100 μm even if it is exposed to high deformation and relatively low proportion of fibers (0 – 2 %). It has been demonstrated that ECC possesses metallic properties after the first occurrence of cracks, which is due to its remarkable tensile strain-hardening behavior resulting from fiber-matrix interaction. ECC displays several chemical and physical features that assist it to be an ideal material for self-healing purposes (Weimann MB, Li VC, 2003).

ECC exhibits outstanding mechanical properties; compressive, flexural and tensile compared to the fiber reinforced concrete. Moreover, water permeability of ECC can be reduced and multiple orders of magnitude lower than what is exhibited for conventional concrete and thus, this brings about better durability and self-healing efficiency. The fracture toughness of ECC is high as a result of strain hardening after cracking and therefore damage tolerance is significantly high. Interaction that happens between fibers and cementitious materials generates many micro cracks with a controlled width rather than creating big width cracks, thus, because of these unique characteristics mentioned earlier, this bendable concrete is well resistant to corrosion as well as it assists the cracks produced to heal on their own without any external interference (Van Tittelboom et al, 2013).

2.3 Structural Applications of ECC

ECC can be deemed as a flourishing material for broad area of functions involving maintenance of buildings, minimizing the effects of earthquakes in seismic zones due to their high ductility, remarkable strain capacity nature and strain hardening property when exposed to tensile load. ECC can represent several types according to the purpose of its use; self – compacting ECC is specially prepared for large-scale structure practices (Kong et al, 2003), high strength ECC at early ages is intended for functional applications, particularly in transportation infrastructure projects (Wang and Li, 2006a) for the applications, which minimum dead load is required light-weight ECC is designed (Wang and Li, 2003), Green ECC is prepared to improve the sustainability of materials and reduce the effects of harmful substances to the environment (Lepech et al, 2007) and Self-healing ECC plays an indispensable role in improving mechanical properties of concrete even after damage occurrence. (Li and Yang, 2007).

It has been proved that this bendable concrete has propagated in many different applications such as full-scale constructional buildings, infrastructure and transportation projects and water and energy buildings fields. ECCs have been utilized to renovate and repair existing buildings in case any damage happens. Moreover, concrete having self-consolidating property can be designed as a precast structural element. Figure 1 illustrates that conventional expansion joint substituted with an ECC slab panel in a bridge deck as it is highly capable of tolerating harsh weather conditions (freeze–thaw cycles). Furthermore, noticeable features of ECC significantly assist the deck to expand and shorten without causing any damage as it behaves similarly as an expansion joint. This improves the sustainability of infrastructure and other structures due to its advanced and unique properties.

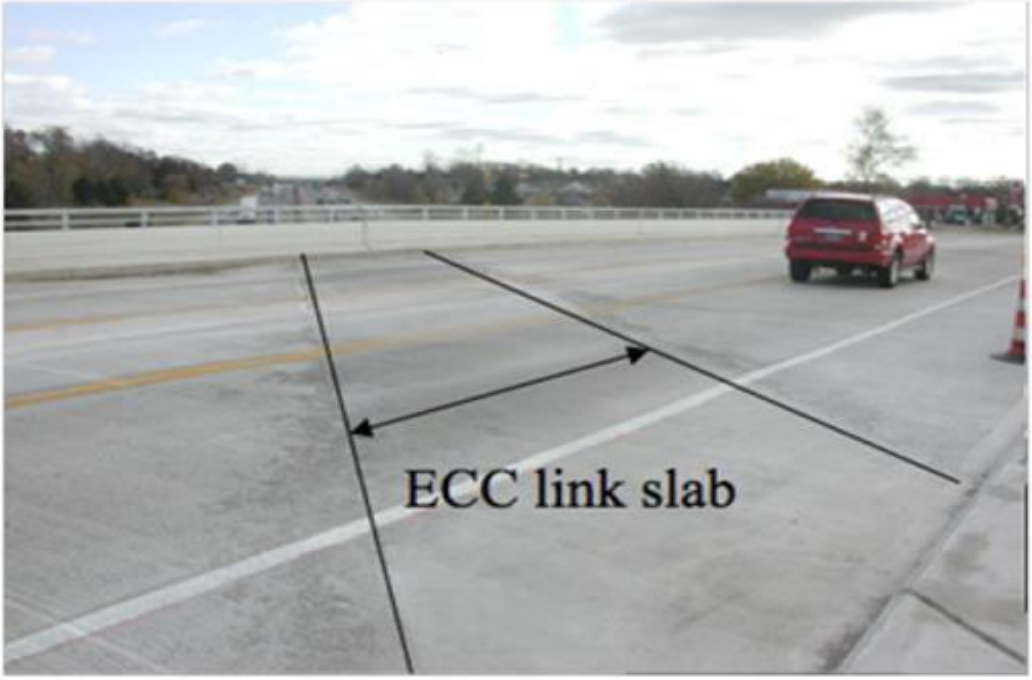


Figure 1: ECC Slab Used to Connect Bridge Deck as an Expansion Joint as a Replacement to Conventional Concrete (M. D. Lepech and V. C. Li, 2009)

Figure 2 demonstrates employing coupling beams made of ECC concrete for 60-story building in Osaka japan that experienced different humidity and precipitation rates and temperatures over a prolonged period. The significant ductility characteristic of ECC coupling beams improve the durability and safety of the buildings located in seismic zone due to its capacity to absorb the energy when an earthquake occurs (T. Kanda et al, 2011). The noticeable domain of applications of ECC indicates that this ductile and bendable concrete can be substantially used to reduce the maintenance cost and improve safety of the structures even under tough natural hazards.

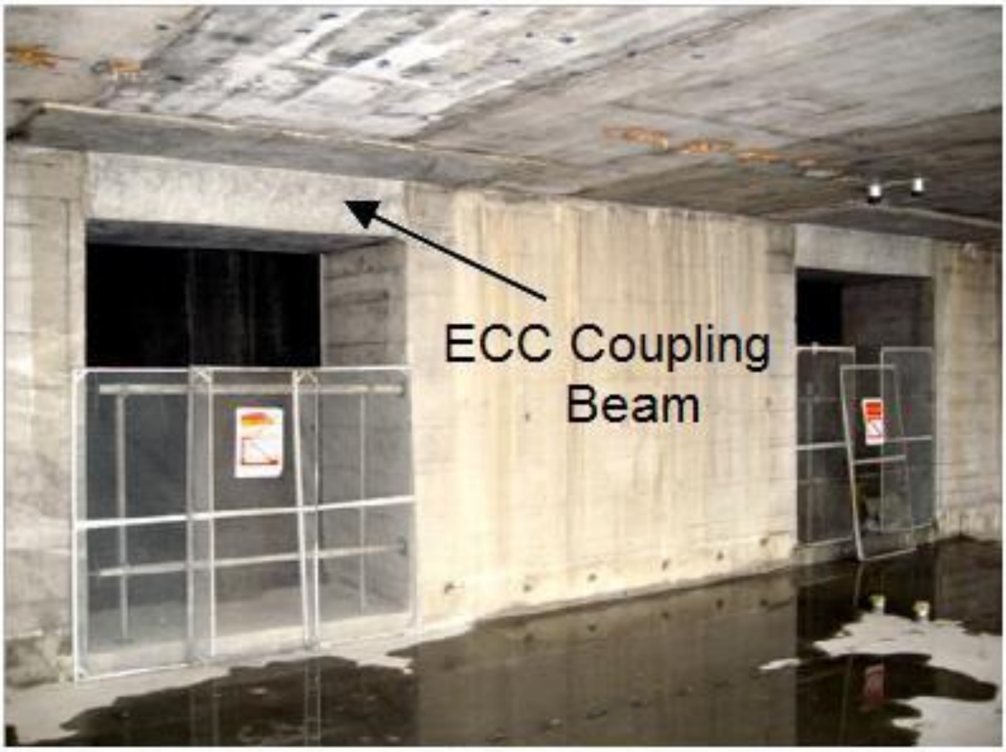


Figure 2: Beams Made of ECC Concrete Were Used in a Structural Building in Japan (T.Kanda et al, 2011)

2.4 Self-Healing of Engineered Cementitious Composite

Self-healing of ECC is a term called when ECC possesses an ability to heal itself when it comes in contact with air and water by producing calcium carbonate, lime in outer layer of concrete and C-S-H gels. Moreover, if concrete cracking happens, cement grains, which are not hydrated yet likely to be subjected to water ingress and in this case, crack can be sealed as a result of hydration product formation generated from continuing hydration processes. Hence, self-healing of ECC is resistant to crack formation, which keeps the crack very tight and protected from propagation. In addition, reinforcement can be protected from corrosion.

It has been illustrated that ECC possesses a great ability to heal under multiple detrimental conditions such as environments that contain highly harmful substances (alkaline and chloride). Moreover, self-healing of ECC can be achieved when it is subjected to numerous distinct conditions such as ongoing or infrequent water exposures with certain humidity and elevated different temperature rates (Yang et al., 2009).

Reduction of the stiffness and insufficient compaction are considered as the major restrictions that restrain self-healing efficiency. Therefore, controlled tight crack width is desired to be sealed effectively when self-healing compositions and extra hydration products are formed (Y. Yang et al, 2011).

It has been shown that the failure rate of concrete repairs cannot be ignored and undesirable, which is attributed to remarkable insufficiency in the early age performance and lack in durability. Therefore, it is imperative and fundamentally important to produce a novel type of concrete with self-healing capability and sustainable characteristics, which may assist to address severe concrete problems and prevent aggressive substances to cause deterioration to existing concrete (Mather and Warner, 2003).

2.5 Mechanisms used for Self-Healing

In order to improve functionality and durability of any concrete structure, controlled cracks and reduction in the deflections of the members should be achieved. It has been shown that the mechanism type, which is used to seal the crack, depends heavily on the surrounding conditions (Ter Heide, N, 2005). Moreover, concrete age is considered as a significant parameter that can determine the amount of contribution of the mechanism utilized for crack sealing. Remaining cement particles, which are not hydrated, yet bring about continues hydration in case the cracks happen at an early age, whilst calcium carbonate products are formed to be the appropriate mechanism for healing the cracks that occur at a later time. The presence of water is indispensable for healing the cracks and functioning effectively both mechanisms mentioned above (W. Ramm and M. Biscoping 1998).

N. Ter Heide (2005) stated that the specimen age when the cracks occur, possesses an important impact on the strength recovered during sealing the cracks. A large percentage of the strength is significantly regained if the cracks take a place in the specimens at an early age, while strength regain is very small when the specimens cracked at later ages.

The majority of researchers have agreed that ongoing reaction of dehydrated cement particles and formation of calcium carbonate namely carbonation are the prominent and fundamental mechanisms for healing the cracks (Edvardsen, 1999)**.**

Distinctive self-healing mechanisms are as follows:

- Chemical precipitation of calcium carbonate;
- Further reaction of dehydrated cement
- Formation of C-S-H gels.

2.5.1 Formation of Calcium Carbonate

Calcium carbonate crystallization is produced due to the reaction between carbon dioxide CO_2 (dissolved in water or existed in the atmosphere) and calcium ions with presence of water *(Mihashi et al, 2004)*. This is the chemical equation that represents forming $CaCO_{3:}$

$Ca^{2+} + CO_2 + H_2O \leftrightarrow CaCO_3$

It has been proven that calcium hydroxide generated from cement hydration and CO_2 gas, which is produced as a result of water entering the cracks, can react together to create calcium carbonate products as can be explained in this chemical formula.

$CO_2 + Ca(OH)_2 \leftrightarrow CaCO_3 + H_2O$

Edvardsen (1999**)** conducted many studies that illustrated the healing in the cracks can be completely achieved due to sole formation of calcium carbonate. Slits width and the quantity of CO_2 compound significantly affect the development rate and the amount of $CaCO_3$ crystallization.

2.5.2 Extended Reaction of Dehydrated Cement

The process of hydration, as recognized, generally happens through premature ages of concrete. Relying on different factors such as cement/binder and water (cement + binder) ratios, as well as environmental conditions particularly ingress of water or presence of moisture. It has been shown that incorporating binder particles such as fly ash and slag will expand the hydration process. Forming the cracks is very essential, for the continuation of cement hydration as it gives a great opportunity for the dehydrated cement particles to be exposed to water and moisture and thus crack healing will be conducted and promoted (N. Ter Heide, 2005).

Neville et al (2002) ascribed significance of the ongoing cement hydration only when damage happens at the early age of concrete. However, at later ages, the Calcium carbonate crystallization mechanism assists considerably to seal the cracks.

Water/ (cement + binder) ratio parameter affects significantly the rate of hydration and the time of self-healing occurrence: many cement particles are not exposed to hydration because of using low Water/ (cement + binder) ratio, which enhances the reaction of un-hydrated cement over time and thus the crack sealing is developed (P. Termkhajornkit et al, 2009).

2.5.3 Formation of C-S-H gels

C-S-H gel is considered one of the significant self-healing products that considerably contributes to seal the formed cracks, which may occur as a result of transformation of calcium hydroxide $Ca(OH)_2$ that produced throughout the hydration reaction. Moreover, prolonging hydration and pozzolanic properties of SCMs contribute to produce C-S-H gels, which are more likely formed from combinations of calcium, silicon and carbon (Herbert et al, 2013).

2.6 Parameters Influencing Self-Healing Efficiency

The effectiveness of each mechanism mentioned before relies heavily on the different factors that will be explained in more detail in the following paragraphs.

2.6.1 Mix Ingredients

It has been shown based on the investigations conducted in the self-healing that features and magnitudes of different constituents of the concrete not only define the mechanical properties of concrete, but also significantly affect the probability of self-sealing occurrence (Yooa D.Y., 2003, Reinhardt H-W, 2003).

Using SCMs including fly ash and silica fume influences the structure of the pores, width of the crack generated and, early and advanced hydration level. The size of the pores determines the effectiveness of self-healing processes happened to the cracks and they assist in later phases to improve further hydration reactions that fundamentally crucial to form sealing or healing substances. Moreover, an increasing the quantity of cement leads to increase the available cement particles required for ongoing hydration especially when the cracks take place inside the concrete due to higher amount of potential unreacted cement particles (Ferrara et al, 2014).

2.6.2 Existence and Pressure of Water

Water is considered as the most intrinsic parameter that promotes self-healing mechanisms to occur, the absence of water and moisture that is required for the cracks to be healed results in deactivation of self-healing mechanisms. (Edvardsen, C. 1999)

Lauer et al (1956) reported that, if the humidity is relatively lower than 95%, the degree of crack healing will be obviously decreased and production of crystallization products will not follow regular pattern. The best demonstration for this phenomenon is that CO_2 is displayed on water superficial side and thus, formation of calcium carbonate becomes delayed, therefore, surfaces of specimens must be completely immersed in water in order to motivate autogenous healing process.

Self-healing in the cracks happens slowly if the water flow is rapid through the cracks. Edvardsen (1999) conducted many studies in order to confirm the impact of water flow rate on the formed cracks width, she demonstrated that lower pressure rate resulted in clogging entirely the cracks, while 25% of the cracks were sealed when the higher pressure rate was utilized. Both pressures were used for seven weeks to measure the crack width.

2.6.3 Crack Width and its Stability

The width of the crack is a very crucial parameter, which defines the volume required to be sealed by products of hydration and the amount of water that will flow through the cracks. Durability can be characterized depending on the proportion of water parameter used to assist the cracks to be filled by different products. It has been proved that the permeability of concrete gradually reduced due to water ingress to the cracks. Furthermore, durability function can be adversely affected due to flowing deleterious agents such as acids inside the concrete (Ferrara L et al, 2013).

It has been pointed out that the tolerable crack width is the most significant parameter that should be taken into consideration to completely perform healing of the cracks. Larger cracks display slower healing rate than smaller cracks and the healing capacity is not completed in case when larger cracks occur (Reinhardt et al, 2003).

In general, the amount of the products produced during cement hydration is not adequate to seal the big cracks. Some studies on ECC have investigated that crack width ranging from 50 μm to 100 μm is sufficient to recover the transport and mechanical properties of concrete. Whereas in cracks with width from 100 μm up to 200 μm, recovery in both properties can be partially achieved (Y. Yang et al, 2009)

In order to induce the sealing products to fill the gaps created in the cracks, propagation and movement of the cracks should be prohibited. As a matter of fact, stabilization of the cracks relies on the stress that happens along the cracks. Healed cracks will be deteriorated again when the crack is moveable or dynamic. However, the active cracks with tiny width can be clogged completely due to autogenously healing process.

2.6.4 Mechanical Properties of Healed Cracks

The majority of the experiments conducted regarding to the efficiency of concrete to be autogenously healed were concentrated on permeability properties of healed samples. Whereas, recovery percentage observed in mechanical properties of concrete was not the focus for many researchers.

Mustafa Sahmaran et al (2008) conducted a study to examine the degree of the recovery in terms of compressive strength after distinguishing the cracks with different cracking patterns. The specimens were preloaded up to 90% of their compressive strength to form the cracks. He demonstrated that pre-damaged specimens, after water curing for further 30 days displayed a significant healing, as the decrease in the strength was only 7% compared to virgin samples.

Yang, Li et al (2009) implemented an experimental study in order to investigate the recovery level of mechanical characteristics of ECC specimens after healing the cracks. Uniaxial tension test was conducted on specimens after three days of water curing. Cracks were generated by loading the specimens up to certain levels of tensile strain. Different curing conditions (water curing, air curing, and wet/dry cycles) were utilized in this study. It has been proven that using water as a curing condition shows healing improvement compared to other curing conditions. Exposing the specimens for longer time improves the strength recovery level and increases the quantity of healing products formed. However, formation of calcium carbonate can be limited as a result of exposing the specimens to short time of curing regardless to the type of curing (Y. Yang et al, 2009).

2.7 Influence of SCM on Self-Healing Capacity and Mechanical Properties of ECC

2.7.1 Limestone Powder

Jian Zhou et al (2010) conducted an investigation to observe the effect of distinctive percentages of limestone powder (LSP) as a substitution to sand and blast furnace slag (BFS) replaced to Portland cement on the development of ECC. They showed that using LSP and BFS as alternative materials to sand and cement respectively improved concrete greenness and decreased the cost needed to prepare concrete; this is due to the fact that these materials decreased quantities of CO_2 emissions produced from cement and consume less energy compared to Portland cement. Moreover, they proved that workability and durability of concrete could be remarkably improved in case the limestone powder and slag were used (Jian Zhou et al, 2010).

The increase in limestone powder and BFS proportions resulted in forming smaller crack width, all ECC mixtures exhibited tight cracks with width less than 100 μm. The mix proportion that had limestone and BFS as high as 85% of total amount of powder generated tight crack with width of 57 μm. Furthermore, it has been demonstrated that this mix displayed elevated capacity level of tensile strain of around 3.3 % and a reasonable value of compressive strength of around 40 MPa. This mix possessed impermeable characteristic and thus led to significantly improve the durability (Jian Zhou et al, 2010).

The decrease in compressive strength was noticed as long as there was an increase in percentage of limestone powder used. However, the lowest value was 38 MPa, which can be utilized to meet the requirement of the most engineering projects. Flexural deflection level and magnitude of tensile strain increased with certain increase of limestone powder content and then decreased. The study also demonstrated that increasing BFS from 50 to 70 %, with using the same amount of limestone powder, improved the capacity of both tensile strain and flexural deflection (Jian Zhou et al, 2010).

Limestone powder acts as inert filler material and toughness of the matrix can be reduced as a result of increasing limestone powder content. Therefore, adding limestone powder to the matrix leads to decrease the tensile strength. On the other hand, the interface between fibers and matrix becomes very weak in case the limestone powder is too much added (more than 20%) and thus strain-hardening behavior of ECC can be negatively influenced (Li VC, 2003).

Hocine Siad et al (2015) conducted a study to investigate effect of the partial replacement of 5%, 10% and 20% of LSP with cement and FA on mechanical and self-healing properties of ECC. A previous study revealed that self-healing efficiency and tensile ductility could be considerably enhanced due to reducing cement to FA or slag ratio (Y. Zhu et al, 2014). They reported in their study that compressive strength of the mixture having 5% limestone powder as a replacement to fly ash, when the later age is taken into consideration, displayed the optimized value due to the reaction that happens between Aluminates from FA and calcium Carbonate from LSP resulting in decreasing the porosity and improving the strength. The decrease in compressive strength with replacing FA with LSP up to 20% is due to the reduction in the amount of FA, which is considered as a pozzolanic material. The reaction between silica from FA and calcium hydroxide from cement assists to produce supplemental C-S-H gels, which augment compressive strength. The flexural strength displayed the same pattern of compressive strength.

The modulus of rupture (MOR) recovery after preloading the specimens decreased with increased LSP replacement due to the lack of the quantity of dehydrated materials needed for additional reaction and the reduction in pozzolanic reaction after replacing FA with LSP, which is required to produce C-S-H gels that are considered the distinguished products accountable for mechanical properties recovery. Flexural deflection capacity exhibited a direct proportion with the LSP replacement degree. Augmenting LSP percentage enhanced the recovery level of deflection. Healing up to 27% was observed after water curing for ECC incorporating 20% LSP, whereas ECC mix without LSP showed 17% recovery. The ductility recovery can be enhanced with increased LSP replacement as LSP is featured by its outstanding filling capacity comparison with FA.

It has been observed based on the microstructural analysis of sealed cracks that cracks for both mixtures either with 20% LP or without LSP were completely clogged after 90 days of healing. LSP- included ECC mixtures demonstrated that the formation of calcite is the healing product, while the mixture without LSP generated C-S-H gels are responsible for self-healing. Moreover, monocarboaluminate product was produced on the surface of the healed cracks, which confirmed the interaction between aluminates from fly ash and calcite from limestone. (Hocine Siad et al, 2015)

S. Qian et al (2009) conducted a study about self-healing of ECC comprising blast furnace slag as a supplementary cementitious material and LSP as an alternative material to sand, the experiments conducted at distinct water/ binder ratios. It has been observed that after preloading the samples at 28 days of curing and comparing the results to the control specimen, deflection capacity of preloaded specimens displayed recovery of around 65–105% of the sound specimen after prolonging the water curing to additional 28 days, whereas after 28 days of air curing, the recovery was just around half of the sound specimen. They showed that the water curing condition is the ideal one to heal the cracks and to recover the mechanical properties of concrete. It was revealed that calcium carbonate was the prominent product that considerably contributed to heal and clog the cracks.

2.7.2 Slag

Chern, J et al (2012) conducted a study to investigate the consequences of using slag on ECC. Slag was used as an alternative material to cement, 20 and 40% of cement were substituted with slag. They stated that not only the quantity of slag affects the mechanical properties of ECC but also slag fineness. Increasing the amount of slag and its fineness level results in increasing the compressive strength. Slag grade 100 fineness needs more time to achieve the same compressive strength that slag 120 does. ECC possesses lower elastic modulus compared to the conventional concrete displaying the same strength. The reason for this was found to be the absence of course aggregate. Moreover, the polymer fibers don’t have any effect on modulus of elasticity. As regards to flexural strength, the relation between the modulus of rupture, which indicates the flexural strength level and the percentage of slag replacement is directly proportional and therefore, expectedly, material shows remarkable ductile properties with increasing slag replacement proportions to cement. The same pattern was observed for tensile strength.

They came up with that substituting slag not only improves the strength, but also produces superior fiber bridging characteristic, which can play a significant role to suppress the propagation of cracks and thus the failure will not happen immediately after first crack. Moreover, this superior property displays a great improvement in ECC ductility.

Kim JK et al (2009) illustrated that adding BFS improves the ability of concrete to resist sulfate attack and decreases the deleterious effect of the penetration of chloride ion. Besides, addition of BFS assists to homogeneously distribute the fibers due to the driving force caused by BFS particle that can disperse fibers, accordingly, BFS diminishes the cost of concrete production and grant considerable contributions in term of workability and durability of ECC. BFS-incorporated concrete is considered more tolerable and effective to resist freeze-thaw weathering as compared to concrete not having slag. The porosity of the material can be reduced and the hydration rate is decelerated due to pozzolanic properties of slag used, which means that sufficient amount of dehydrated cement will continue hydration resulting in healing the concrete.

Mustafa Sahmaran et al (2013) conducted an investigation to study the self-healing capability of ECC incorporating slag and fly ash class F and C. They found that compressive strength of the specimens with slag is substantially higher than what was observed for the mixtures casted with fly ash. This is due to the fact that the slag has larger specific surface area than that of fly ash; therefore, the slag exhibits more effective hydration and is more advanced in pozzolanic reactions than fly ash, which contributes to improve the strength. However, ECC with fly ash (class F) revealed the highest deflection capacity and thus the most remarkable ductility compared to other supplementary cementitious material. The advancement of the deflection level showed in the middle of the beam by utilizing fly ash class F might be ascribed to the tendency of fly ash to maximize the chemical bond of matrix interface with PVA fiber while augmenting the interface frictional bond. Therefore, obtaining a high tensile strain capacity can be remarkably observed for FA-F category.

Mustafa sahmaran et al (2013) showed that after preloading the specimens under splitting tensile test up to certain level of splitting deformation (1.25mm). ECC specimens incorporating FA displayed crack having tighter width than S-ECC samples. Reduction in the width of the cracks indicates that water curing has significantly induced the self-healing efficiency and consequently the chloride ion transference property was improved after generating cracks.

Mustafa sahmaran et al (2013) revealed that although FA-ECC specimens possess more un-hydrated cementitious materials, S-ECC displayed healing product, which improved the capacity and efficiency of self-healing of ECC. They stated by using microscope observation taken for pre-loaded ECC samples that the permissible crack widths are not the only parameter that can influence the self-healing capability but also the type of cementitious material should be taken into consideration. S-ECC sample with a width slightly above 100 μm can be completely healed after 60 days of curing, whereas, crack width of 50 μm was healed for FA-ECC. It has been shown that calcite and C–S–H gels are the main products utilized to seal the cracks for FA-ECC, while carbonation product C–S–H gels were reasonable about self-healing process for S-ECC. High quantity of CaO in the slag material, which can be used to precipitate the calcite, might be the reason that leaded to attain substantial amount of self-healing of S-ECC.

2.7.3 Glass Powder

Du and Tan (2015) stated that a 30% GP replacement level with cement displayed higher strength, better transport properties, remarkable carbonation products and great ability to resist sulfate attack. On the other hand, it was shown that 60% of cement could be substituted with GP resulting in decreasing chloride penetration and better sorptivity performance.

Hocine Said et al (2017) conducted a study to examine the influence of distinct substitution levels of FA with GP on the mechanical and self-healing behaviors of ECC. Fly ash was partially substituted with 25, 50, 70, and 100% of GP. They showed that at early curing ages, the increase in compressive and flexural strengths happened with the increase in replacing FA with GP due to high level of GP alkalinity. Elevated alkali ion concentration can accelerate early age hydration in cement pastes by reducing the solubility of Ca^{++}ions and increasing the formation of ettringite (hydrous calcium aluminiumsulfate mineral), which leads to create C-(N,A)-S-H products and thus, increased the early strength of concrete.

On the other hand, at later age, the decrement in compressive and flexural strength was observed with increasing the level of GP replacement to fly ash, which was attributed to high alkali products of GP resulting in reducing reaction rates over time which limited forming C-S-H gels and made the matrix more heterogeneous and thus, decreased ECC strength. Moreover, the reason behind that was the reduction in portlandite that corresponded with increasing GP replacement levels and the pozzolanic reaction levels of GP particles. However, ECC incorporating 25% GP content displayed higher results than what was observed for control ECC, moreover, ECC with 50% of GP had approximately the same result compared to ECC not having GP, which might be due to high fineness level of GP particles, hence acting like a filler material which contributed to fill the voids and pores and increased the strength of ECC. In addition, a C-S-H formation, which is responsible about increasing the strength of concrete, increased as a result of the reaction between carbon hydroxide from cement and silica from GP material. Although the crack widths were found to be increased with GP, the ductility of the specimens was considerably decreased with increased GP replacement level. However, increasing the percentage of GP replacement up to 25% displayed roughly the similar crack characteristic compared to control ECC incorporating 100% FA.

Hocine said et al (2017) demonstrated that using both ECC Mixes with FA and GP produce C-S-H products that contribute to clog the cracks. Moreover, calcium carbonation product was significantly responsible for self-healing micro cracks of ECC- ref and ECC with 100% GP. However, the effect of the self-healing products that generated to seal the cracks was found to be less pronounced in ECC 100% GP compared to control ECC. This is attributed to the larger crack widths occurred in ECC-100GP specimens than ECC-FA crack widths. The best self-healing performance was observed for the mixture having 25% GP replacement to fly ash.

2.7.4 Fly Ash

Yang et al (2007) demonstrated that fly ash quantity possesses a significant influence on ECC mechanical characteristics. Fly ash is deemed as a useful constituent in terms of improving the long-term strength as a result of its remarkable pozzolanic properties. Incorporating fly ash leads to decelerate the hydration rate for the later stages, which results in enhancing microstructure behavior of concrete and displays improvement in self-healing process. Moreover, width of the cracks produced in ECC can be diminished with increasing the volume of fly ash material and thus high content of fly ash confirms the significant enhancement of self-healing capacity of ECC as a result of producing tighter crack width.

Zhigang Zhang et al (2014) conducted a study about the effect of different proportions of fly ash on the different properties and self-healing performance rate of ECC. Ratios of FA/cement are 1.2, 2.2 and 4. Increase in fly ash shows a decrement in compressive strength of ECC specimens, which can be attributed to the reduction in the cement proportion, which has the dominant hydration products that affect considerably on compressive strength. The same pattern was illustrated for first cracking strength and flexural strength. Whereas the increase in deflection capacity of ECC was clear with increasing the content of fly ash. Regarding the features of produced cracks in ECC, the crack number increases while the crack width decreases and becomes tighter with increasing the proportion of FA replacement to cement. And hence, increasing proportion of fly ash improves the permeability of ECC and accordingly the durability of ECC will be increased. However, the higher fly ash content more than a certain level make matrix more porous, which can be reduced due to self-healing occurrence after water curing. These remarkable characteristics exhibit concrete that is more durable and possesses beneficial self-healing behavior.

It has been demonstrated that calcium carbonate and C–S–H products are formed by the existence of fly ash and they are responsible for healing the cracks. Energy dispersive spectroscopy (EDS) was utilized in order to detect formed healing products.

Wang & Li (2007) and Yang et al (2007) stated that decreasing the percentage of fly ash improves the effect of chemical bonding, reducing fiber-matrix frictional force at the same time. The weakness that happened in the interface between fiber and matrix brings about a decrease in strain-hardening behavior of ECC and hence decreasing crack number and deflection capacity, while results in increasing crack width.

Mustafa Sahmaran et al (2008) conducted a study to prove that not only the width of the cracks has the effect on self-healing but also the type of supplementary cementitious material plays a significant role on self-healing capacity. The same proportions of Class-F fly ash, Class-C fly ash and slag were used.

The specimens that contain slag displayed higher compressive strength than what was observed for the mixtures incorporating both classes of fly ash for the first 7 days. Nevertheless, the recovery of compressive strength for fly ash was remarkably higher compared to S-ECC specimens at 28 days and 60 days due to the fact that using fly ash exhibited more dehydrated particles (Mustafa Sahmaran et al, 2008)

The increase in compressive strength of slag rather than fly ash was attributed to the fact that the slag has more hydration and pozzolanic reactions compared to FA-ECC as a result of its high available surface area comparison with what was observed for fly ashes, in this case, OH^- ions and alkalis in addition to more nucleating sites can flow into the pores, which can provide compressive strength improvement. Moreover the fineness level of slag due to smaller particle size increased the compressive strength value.

In the first 7 days, compressive strength of F_ECC and C_ECC specimens were the same, while C-ECC displayed more compressive strength than F_ECC due to not only the smaller fineness level of C-ECC but also due to chemical compositions of FA (C class), which represented more lime than C-ECC (Mustafa Sahmaran et al, 2008).

Flexural strength as well as ductility characteristic of different ECCs were investigated based on Four-point bending test. The flexural strength measured for S-ECC was not significantly higher than FA-ECC. The reason behind that is in fact due to characteristics of these cementitious materials, which are not easily predictable, such as capacity level of tensile strain, maximum value of tensile strength and tensile strength of first cracking formation.

In order to measure the ductility for all mixes, ultimate mid-span defection was conducted. Over time, the defection level of all specimens slightly decreased due to the progression and advancement of the interface between matrix and fibers. The most deflection capacity and the best ductility were observed for F_ECC. Class-F FA tends to diminish interface chemical bond between fibers and matrix and reduce toughness capacity but increasing the friction level of interface between the matrix bonds (Mustafa Sahmaran et al, 2008).

In order to generate cracks, all specimens were conducted under splitting tensile test up to different certain levels and up to failure as well. Microscope was utilized to measure the width and the number of the cracks. It has been shown that the crack width of the FA-ECC was tighter when compared to S-ECC. Although S-ECC possesses less dehydrated cementitious materials compared to FA-ECC, the cracks formed in S-ECC were sealed and displayed better self-healing capacity than FA-ECC. It can be observed that the crack width up to 100μm for S-ECC was healed during 60 days of water curing, while self-healing happened for the cracks with width of about 30 μm for F_ECC and 50 μm for C_ECC.

By conducting Microstructural analysis, it was found that the fundamental self-healing products that contributed to seal the cracks generated in FA-ECC mixtures were calcium carbonate and C–S–H gels, while the main product for self-healing purpose in S_ECC specimens was calcite. The reason behind the enormous amount of self-healing capacity that occurred in S_ECC samples is attributed to the fact that this concrete possesses more CaO compound due to using slag material, which significantly contributed to form calcium carbonate.

2.8 Influence of Polypropylene Fibers (PP) on Engineered Cementitious Composite (ECC)

Ms. E. Ramya et al (2015) conducted an investigation to study the effect of different percentages of polypropylene fibers (0%, 0.1%, 0.2%, 0.3%, 0.4%, 0.5 %) incorporated with silica fume on mechanical properties of ECC. Here, compressive strength showed increment with increasing the percentage of PP fiber up to 0.2%. However, increasing polypropylene fiber quantity obstructed and limited crack formation.

Yaw ChiaHwan and Han JianBo (2014) conducted an experimental study that proved the possibility of using polypropylene fiber (PP) instead of polyvinyl alcoholic fiber (PVA). It has been shown that the cost of PP is five times lower than the cost of PVA, which is economically better. Moreover, PP ECC displayed astonishing and improved properties such as structural strength, ductility, durability and it can resist freeze-thaw effect.

They stated that utilizing PP fiber improved ductility and toughness of ECC, absorbed more energy which gives significant contribution to relieve the effect of earthquake in seismic zones and displayed good impact resistance.

When earthquake occurs, the structure should be designed to resist seismic forces without decreasing significantly the strength. Using PP fiber in ECC showed that energy dispersal capacity and loading bearing ability could be improved and thus, the performance of structural buildings exposed to reverse cyclic loading is improved.

Chapter 3

RESEARCH METHODOLOGY

3.1 Introduction

The main objective of this study is to determine and investigate the effect of SCMs on workability, mechanical properties, microcracking behavior and autogenous self-healing efficiency of ECC. Therefore, different proportions of SCMs were utilized.

In this chapter, materials used in the production of eight different ECC mixtures, listed and defined in terms of physical and chemical properties. Moreover; appropriate test methods and (ASTM) and (BS EN) standard codes used for mix design and determination of mix proportions were selected and explained in this chapter of this thesis.

3.2 Materials

The materials that were employed for this experimental investigation can be summarized as follows.

Cement: Portland slag cement CEM II/B-M (S-L) (32.5 grade) that is prepared by grinding clinker, with blast furnace granulated slag and gypsum, was utilized in this study. Using this type of cement improves workability of concrete, highly resists deleterious chemical reactions such as sulfate attack and contributes to delay and decelerate hydration rate.

Slag (S): Slag was produced as the main SCM of ECC. Slag has particle size less than 150 μm as can be shown in Figure 3. The chemical composition of slag is given in Table 1.

Glass Powder (GP): Glass powder was produced by grinding the glass bottles to a certain level, which is substituted for slag. The particle size of glass powder used in this study was less than 90 μm in diameter as can be demonstrated in Figure 3. The chemical composition of glass powder is presented in Table 1.

Limestone Powder (LSP): Limestone powder (LSP) having a maximum particle size of 150 μm as can be shown in the figure 3, was utilized in ECC mixtures as a replacement for slag. Its chemical composition is given in Table 1.

Fly Ash (FA): Fly ash class-C having particle size passing sieve# 200 (75μm) as can be seen in figure 3, was used in this study instead of slag. The chemical compositions of fly ash are summarized in Table 1.

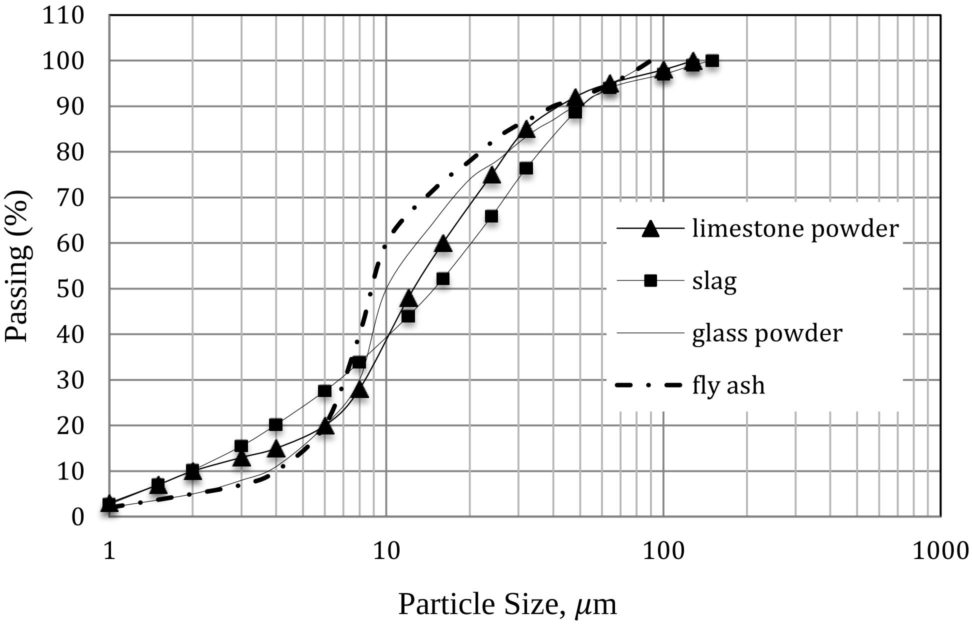


Figure 3: Particle Size Distribution of SCMs

Table 1: Chemical Composition of Cement, Slag, Fly Ash, Limestone and Glass Powders.

Chemical compositions	Cement (CEM)	Slag (S)	Fly ash (FA)	Limestone powder (LSP)	Glass powder (GP)
$CaCO_3$	-	-	-	94.0	-
CaO	60.4	35.1	15.5	-	13.7
SiO_2	18.7	37.5	47.0	0.5	74.6
Al_2O_3	4	10.5	11.9	-	2.2
Fe_2O_3	2.6	0.3	7.9	-	0.4
MgO	2	7.9	6.6	2.5	1.4
SO_3	2.2	2.9	3.4	-	-
K_2O	1.2	1.1	3.2	-	0.7
Na_2O	-	0.3	2.4	-	7.3

Mixing Water: Tap water that is free from harmful substances such as, acids, alkalis and organic materials was used for all ECC mixes and for curing process.

Fine Aggregate: Sand that was sieved on sieve that has diameter of 300 μm was used in order to reduce the pore size and the crack width and therefore the durability will be expected to increase as a result of decreasing permeability. Moreover, decreasing the particle size of fine aggregate keeps the homogeneity between the particles of the mix and decreases toughness of the specimens and thus ductility will be improved. Fine aggregate is produced from crushed limestone.

Superplasticizer: type F polycarboxulic high range water- reducing (HRWR) admixture was used in order to maintain high workability and compensate the small amount of water added acoording to BS EN 934-1 (2008)

Polypropylene Fiber: The fiber used in this study was the Polypropylene fiber with a length of 18 mm and a diameter of 40 μm. The tensile strength of the Polypropylene fiber is 1200 MPa and the density is 910 kg/m^3

3.3 Mix Proportions

ECC is a new category of ultra-ductile fiber reinforced concrete depending on the theory of micromechanics design firstly originated in the beginning of 1990s at Michigan University (Li VC, 1993).

In order to achieve the objective of this study, four distinguished supplementary cementitious materials were used to produce three different ECC groups, which can be explained in the following sections.

The first group represented four distinct ECC mixtures, which were formulated with and without LSP, as summarized in Table 2. One ECC mixture without LSP was designed and utilized as a reference; the reference mix was prepared with 100% of slag material (ECC-Ref). For ECC-incorporated LSP (ECC-LSP5, ECC-LSP20 and ECC-LSP40), Slag was systemically substituted with LSP at 5, 20 and 40% by mass. Constant mineral admixtures to cement [CEM/(S+LSP)], water to cementitious materials [W/ (CEM+LSP+S)], and sand to cement (SA/CEM) ratios of 1.2, 0.29 and 0.8 respectively were utilized to produce ECC mixtures. Super-plasticizer quantity was 3% of cement amount. Polypropylene fiber (PP) content was 1% by volume.

The second group displayed four distinctive ECC mixtures that were prepared in which glass powder was replaced for slag material in different proportions, as demonstrated in Table 3. Slag was methodically substituted with GP at 20, 40 and 60% by mass. The ratios that used for this group were the same utilized in the first group.

The third group consists of fly ash as a supplementary cementitious material instead of using slag with the same ratios used before as shown in Table 4.the last group was in order to compare between them in terms of mechanical properties and self-healing efficiency.

Table 2: The Mixture Proportions of ECC Specimens (LSP)

Mixes	CEM Kg/m^3	S Kg/m^3	LSP Kg/m^3	Sand Kg/m^3	W Kg/m^3	SP Kg/m^3	PP fiber Kg/m^3
ECC-Ref	578	693	0	463	370	17.3	9.1
ECC-LSP5	578	554	139	463	370	17.3	9.1
ECC-LSP20	578	416	277	463	370	17.3	9.1
ECC-LSP40	578	277	416	463	370	17.3	9.1

CEM: cement W: water S: slag
SP: Superplasticizer, PP: polypropylene fiber LSP: limestone powder,

Table 3: The Mixture Proportions of ECC Specimens (GP)

Mixes	CEM Kg/m^3	S Kg/m^3	GP Kg/m^3	Sand Kg/m^3	W Kg/m^3	SP Kg/m^3	PP fiber Kg/m^3
ECC-Ref	578	693	0	463	370	17.3	9.1
ECC-GP20	578	416	277	463	370	17.3	9.1
ECC-GP40	578	277	416	463	370	17.3	9.1
ECC-GP60	578	277	416	463	370	17.3	9.1

CEM: cement; S: slag; GP: glass powder
W: water SP: Superplasticizer, PP: polypropylene fiber

Table 4: The Mixture Proportions of ECC Specimens (FA)

Mixes	CEM Kg/m^3	FA Kg/m^3	Sand Kg/m^3	W Kg/m^3	SP Kg/m^3	PP fiber Kg/m^3
ECC-FA	578	693	463	370	17.3	9.1

CEM: cement; FA: fly ash W: water
SP: Superplasticizer, PP: polypropylene fiber

3.4 Pozzolanic Activity Index

The pozzolanic activity index of FA, GP and S were measured according to ASTM C311 (ASTM2013b). In order to conduct this experiment, 20% of cement material was substituted with FA or GP or S. The water-to-cement ratio for the control mixture and for mortars including GP, FA and S was 0.485. The ratio between the compressive strength of the mortar containing 20% of pozzolanic material and the strength of the equivalent control mortar was tested on 7th and 28th day of age.

The pozzolanic activity index of glass powder, fly ash and slag after 7 and 28 days of curing are illustrated in Figure 4. Flay ash displayed more pozzolanic activity index than glass powder and slag. For instance, at 28 days, the differences between FA and GP and between FA and S are 5.3% and 8.7% respectively. However, pozzolanic activity index value of S after 28 days largely satisfied the pozzolanic materials requirement of 75% specified in ASTM C618.

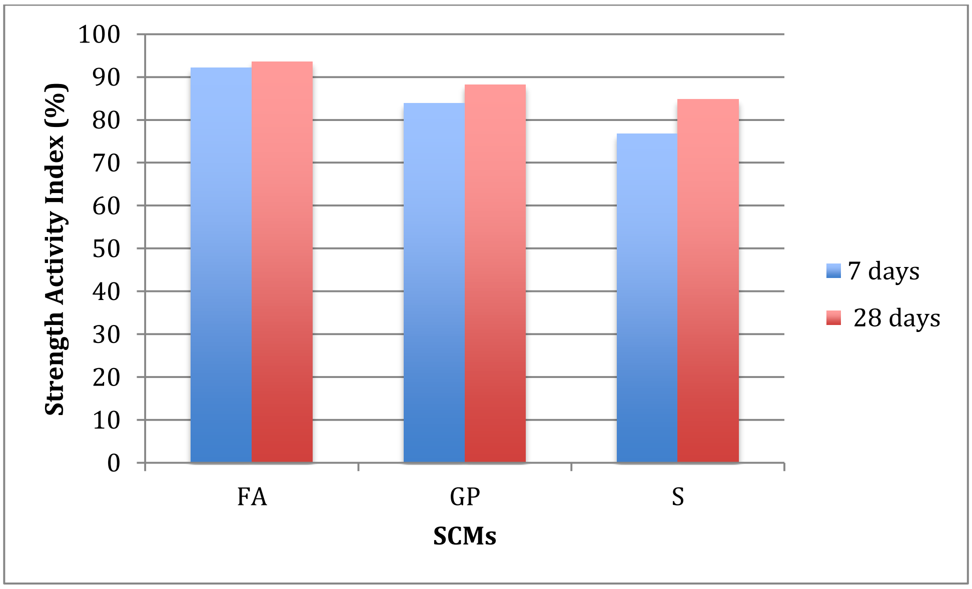


Figure 4: Pozzolanic Activity Index of GP, FA and S at 7 and 28 Days

3.5 ECC Mixing Procedure

All ECC specimens were supposed to be produced using a planetary-type mixer with a 50-L or 25-L capacity. Due to the absence of this mixer, a blender was purchased to prepare all ECC samples. All solid components except fibers (cement, FA, GP, LSP, S and fine aggregate) were mixed for one minute. Water then was added to the dry mixture for an extra two minutes. After that, HRWR admixture was added to the mixture and mixed until it was noticed that the mixture became workable, consistent and had fluidity characteristic, that period was around two minutes. Polypropylene fibers were gradually added to the mixture for additional three minutes in order to not only prevent sticking between the materials of ECC matrices but also to obtain the desired workability.

3.6 Specimen Preparations and Curing

In this study, twelve 100-mm cubic specimens from each ECC mixture were cast to determine the mechanical properties of ECC and to investigate the capacity and efficiency of distinct ECCs in terms of their self-healing ability. After casting, specimens were kept inside the molds, which were carefully cleaned and oiled in order to ease demolding samples, and then the molds were saved in the curing room for 24 h at 99% relative humidity (RH). After that, the samples were transferred to water curing tank with a normal temperature of around 25 C for 28 days before conducting the experiments as can be illustrated in Figure 5.

Figure 5: Water Curing Tank

3.7 Experiment on Fresh ECC

3.7.1. Slump Flow Test

The ECC mixtures should possess creamy texture and workable characteristics. Therefore, a standard concrete slump cone as can be shown in the Figure 6, was filled with fresh ECC material and placed on the flat surface. The ECC was then allowed to flow by lifting up the cone. Two orthogonal diameters of this “pancake” were measured and a characteristic deformability factor, denoted by Γ.

$$\Gamma = \frac{D1 - D0}{D0}$$

Where D_1 is the average of two orthogonal diameter measurements after slump cone removal, and D_0 is the diameter of the bottom of the slump cone (200mm). For good self- consolidation, Γ should have a minimum value of 2.75 (Lepech and Li, 2007).

Figure 6: Slump Cone Test

3.8 Experiments on Hardened ECC

3.8.1 Compressive Strength

In order to investigate the compressive strength of all ECCs and to demonstrate the effect of supplementary cementitious materials on compressive strength values, three 100-mm cubic samples for each ECC mixture were prepared and cured for 28 days before subjecting them to the compressive strength test according to BS EN 12390-3:2009 standard specifications as illustrated in Figure 7. The average of three measurements was calculated. The ultimate capacity of the universal compressive strength-testing machine is 3000 KN. The specimens were tested using a constant strain rate of 0.002 mm/s.

Figure 7: Compressive Strength Testing Equipment

3.8.2 Stress-Strain Test

In order to observe the influence of SCMs on microcracking behavior of ECC, stress-strain diagrams were plotted for each ECC mixture by conducting compressive strength test for 100-mm cubic specimens using the same strain rate utilized for compressive strength test. Moreover, the test was conducted to measure the percentages of compressive strength to be loaded relative to the ultimate for self-healing objectives.

3.8.3 Splitting Tensile Strength Test

Splitting tensile strength test was conducted according to BS EN 12390-6:2009 standard specifications in order to measure the tensile strength for each ECC mixture and to evaluate the influence of different proportions of supplementary cementitious materials used in this study on the value of tensile strength. To conduct this test, three cubes (100mm) were prepared and tested after 28 days of curing under 0.2 KN/S loading rate as illustrated in Figure 8.

Figure 8: Splitting Tensile Test Apparatus

3.8.4 Tensile Strength Loss (TSL) Test

100-mm cubic specimens were preloaded up to 90% of ultimate compressive strength and then tested under splitting tensile strength test up to ultimate to measure the loss in tensile strength, which is referred to it as damage. The value of tensile strength loss gives a primary indication about the microcracking behavior. It has been founded that TSL is strictly related to the quantity of cracks and pores (Tulin Akçaoğlu,2005).

3.9 Pre-Cracking and Self-Healing Testing

3.9.1 Compressive Strength after Preloading ECC Samples to Evaluate Self-Healing Efficiency

Since the type and volume of mineral admixtures have a high influence on the cracking behavior and self-healing ability of ECC, this study assessed the rate of recovery of pre-cracked ECCs. At the age of 28 days, three new cubes were preloaded up to 85% of their ultimate compressive strength values and then left to recover in water tanks in order to evaluate the compressive strength recovery. The healing efficiency of ECC mixtures was evaluated by reloading the preloaded specimens to failure after 30 days of moist curing. Compressive strength values of preloaded specimens were recorded and compared to those of sound specimens.

3.9.2 Using Stereo Microscope for Self-Healing Purpose

To gain further insight into the effects of incorporating supplementary cementitious materials on self-healing efficiency of ECC, it was supposed to preload three specimens up to certain levels of ultimate splitting tensile strength such as 60%, 80% and even 90%, but it was observed that there was no any crack generated at those levels. Therefore, in order to generate cracks on the surface of the specimens and to observe the healing rate after water curing, it has been decided to pre-crack the cubic specimens by loading them up to ultimate values of tensile strength under splitting tensile test at a loading rate of 0.2KN/S and then, the specimens were kept inside the water tank for farther curing. The microscope shown in the Figure 9, was used in order to detect the cracks generated, to measure the crack width for each ECC mixture and to investigate the self-healing capacity and efficiency that occurred in the cracks at 0, 7, 30 and 45 days.

Figure 9: Ultrasonic Test

3.9.3 Ultrasonic Test

Based on ASTM C597-09 standard specifications, ultrasonic test was conducted on two cubic specimens (100*100*100mm) from each mix proportion after generating the cracks under splitting tensile test, the transducers are positioned on the center of two opposite sides of the specimens as shown in the Figure 10. The travel time was measured for each ECC immediately after causing the cracks before healing and then the specimens were kept in water tank for one month of healing before measuring the travel time again. The difference between travel time for each ECC before healing and after healing was measured. The small travel time is an indicator of a good quality of concrete, which means that concrete comprises not many cracks and voids. Moreover, the higher the difference between the travel time after and before healing is, the better self-healing rate. This indicates that more cracks are healed.

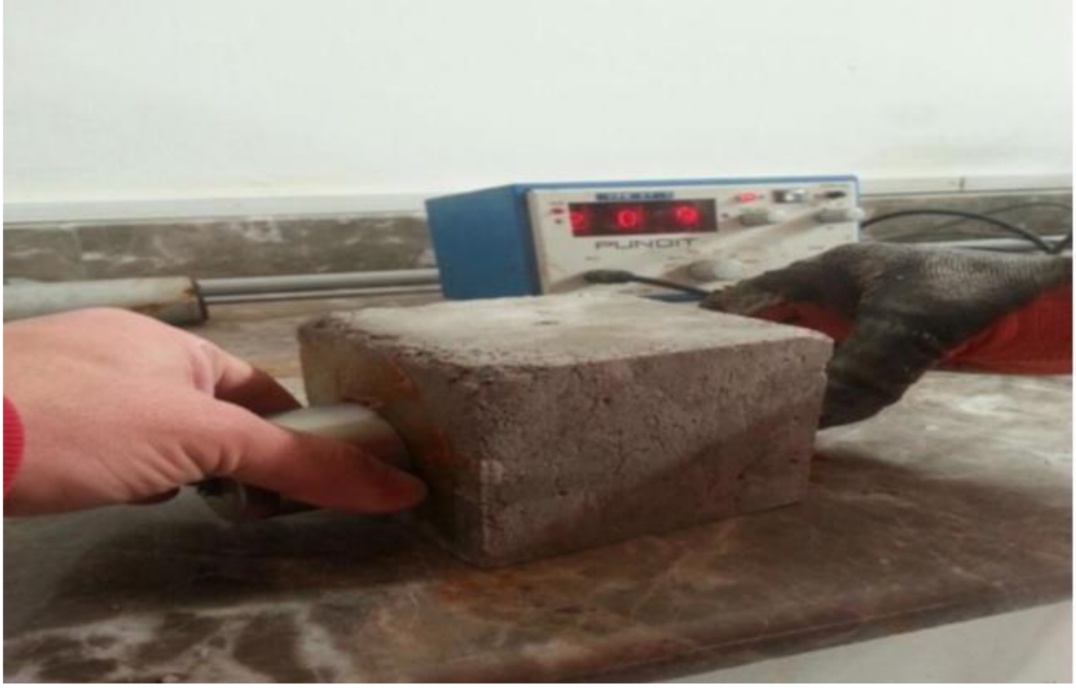


Figure 10: Ultrasonic Test

Chapter 4

RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter comprises the results of the experimental study and discussions of all conducted experiments for all ECC mixtures that were done by using the tabulated tables and drawn figures.

4.2 Effect of SCMs on Workability of ECC Concrete

Flow properties of all ECC mixtures are given in Table 5. The flowability of ECC is presented in terms of flowability index (Γ), which was calculated by using the following equation

$$\Gamma = \frac{\mathrm{D1} - \mathrm{D0}}{\mathrm{D0}}$$

Where: D1 is the average of two orthogonal diameter measurements after slump cone removal, and D0 is the diameter of the bottom of the slump cone. For good self-consolidation, Γ should have a minimum value of 2.75 (Lepech and Li, 2007).

Table 5: Workability Test Results of All ECC Mixtures

Mixtures	D1 (mm)	Γ
ECC-Ref	850	3.25
ECC-LSP5	860	3.30
ECC-LSP20	890	3.45
ECC-LSP40	780	2.90
ECC-GP20	865	3.33
ECC-GP40	765	2.83
ECC-GP60	755	2.78
ECC-FA	880	3.40

It can be shown from the Table 5 that increasing slag replacement with GP up to 20% led to increase slump flow. After that, the workability started to decrease with increasing the glass powder proportions up to 40 and 60%.

The results of slump cone test revealed that the replacement of LSP up to 20% showed a beneficial effect on flow properties of ECC. The slump flow of mixtures ECC-LSP5 and ECC-LSP20 were 3.30 and 3.45 respectively. However, the decrease of slump flow of ECC-LSP40 (2.9) was observed.

It was observed from the Table 5 that ECC-Ref displayed less slump flow than that in ECC-FA mixture. Flowability index (Γ) of ECC-Ref and ECC-FA were 3.25 and 3.40 respectively.

The higher gain in workability for ECC-LSP5 and ECC-LSP20 over ECC-Ref can be attributed to that the use of LSP up to that level, contributed to improve the particle distribution of the powder, and therefore, resulted in diminishing inter-particle friction. Moreover, The particle size of limestone powder in the binder stage of a mixture enhanced particle packing effectiveness, which resulted in improving blocking process of capillary pores and that led to reduce penetrability. Accordingly, the water demand was decreased as a result of water bleeding reduction, thereby improving workability. On the other hand, the decrease of slump flow of ECC-LSP40 might be associated with increasing LSP up to that level caused agglomeration of LSP particle, thus, increased voids between agglomerated LSP particles. Furthermore, using more LSP replacement reduced uniformity and distribution of fibers and as a result decreased the workability.

The increase in workability for ECC-GP20 can be due to the fact that the smaller particle size of GP compared to slag created less friction against the flow of ECC. Then Using GP particles up to 20% reduced the water demand needed for chemical reaction activities. Moreover, GP can behave as a filler material resulting in filling the voids of concrete, which increased the amount of free water, subsequently, improved the workability of the concrete. The decrement in slump flow with the increasing proportion of GP substitutions to slag was brought about by some reasons. Firstly, increasing GP replacement percentage to slag created more voids and pores as a result of agglomeration of GP particles. Thus, more water was consumed which results in reducing the workability. Moreover, adding GP material more than 40% as a replacement, bond the fibers together, which limited uniform distribution of fibers through the whole mix, thus, decreased workability.

The increase in slump flow of ECC-FA over ECC-Ref was attributed to that the spherical shape of fly ash particles can considerably increase the workability of fresh concrete which acted as bearing ball that decreased friction forces between cementitious materials or matrix particles. . In addition, mineral admixtures like fly ash acted as filler, which can improve the workability. The smaller particle size of FA compared to slag created less friction against the flow of ECC and reduced the water demand needed for chemical reaction activities and thus improved workability.

4.3 Effect of Different Type and Proportion of SCMs on 28-days Compressive Strength of ECCs

Compressive strength values of all ECCs containing both 100% of slag and different proportions of LSP are demonstrated in Figure 11, which illustrates that by increasing the proportion of the slag replacement with LSP, the compressive strength results decreased. The compressive strength of ECC-LSP5, ECC-LSP20 and ECC-LSP40 were 59.75,57.5 and 50 MPa respectively. However, ECC-LSP5 and ECC-LSP20 displayed higher compressive strength than ECC-Ref, while the compressive strength of ECC-LSP40 mixture was slightly lower than what was observed for reference mixture.

The remarkable strength gain at 28 days for ECC-LSP5 and ECC-LSP20 over ECC-Ref was attributed to the fact that the limestone powder possessed filling effect as it behaved as inert material. Consequently, the voids can be decreased and hence LSP improved the compressive strength. Moreover, the potential carboaluminate formation that might be produced as a result of the reaction between Aluminates from slag and calcium carbonate from LPS diminished the porosity and encouraged LSP to fill the pores and accordingly improved the strength. The slight decrease of

ECC-LSP40 compressive strength when compared to the reference mixture was in fact due to the pozzolanic reactivity of S in ECC-Ref that made a difference at this age. Moreover, during the pozzolanic reactions, the reaction between calcium hydroxide from cement and silica from the slag material occurred, which led to produce extra C–S–H gels that significantly resulted in increasing compressive strength. The decrease in compressive strength with replacing slag with LSP up to 40 % can be possibly attributed to inadequate cement paste to coat that amount of limestone powder.

The compressive strength results of four distinct ECCs with and without glass powder at 28 days are illustrated in Figure 11. ECCs incorporating 20% and 40% of GP materials displayed higher compressive strength values than ECC-Ref mixture. In contrast, compressive strength of ECC-GP60 revealed lower value than ECC-Ref. compressive strength of ECC-Ref, ECC-GP20, ECC-GP40 and ECC-GP60 were 53.2, 55, 58.1 and 51.8 respectively.

The greater strength gain in ECC-GP20 and ECC-GP40 mixtures over ECC-Ref at 28 days can be attributed to the higher pozzolanic reactivity of GP than that is in slag material. In addition, GP particles exhibited higher Blaine fineness compared to slag particles, hence, glass powder possessed higher possibility to improve paste matrix compactness through filling effect up to 40% replacement level, thus increasing compressive strength. It can be said that high amount of silica from GP significantly contributed to improve compressive strength due to forming C-S-H gels after reacting with calcium hydroxide from cement during pozzolanic reactions. Nevertheless, ECC-GP60 revealed lower compressive strength than the reference mixture, which can be associated with high alkali content of GP resulted in occurring heterogeneity in microstructure of C–S–H and reducing reaction rates and thus reduced the strength of the matrix. Moreover, augmenting amount of GP substitution led to continuous decrease in portlandite quantity as a result of the cement diminished production of C-S-H gels throughout pozzolanic reaction process. Moreover, Agglomeration in GP particles increased the pores and voids and thus decreased compressive strength.

It can be shown from the Figure 11 that ECC-FA (69.5MPa) displayed a significant improvement in compressive strength compared to ECC-Ref (53.2MPa). This result can be explained by relying on particle size of the material, pozzolanic reactions and cementitious activity of the material. According to the particle size distribution Figure, fly ash possessed smaller particle size than slag, therefore, fly ash assisted to seal the spaces between cement particles and distributed homogenously the cementitious particles and that was resulting in establishing denser microstructure and higher compressive strength. Moreover, the improvements in hydration and pozzolanic reactions of the fly ash due to its high specific surface area compared to that of slag significantly contributed to improve compressive strength. It has been proven that the presence of lime substance in fly ash with its high pozzolanic reactivity played an effective role in improving strength of ECC.

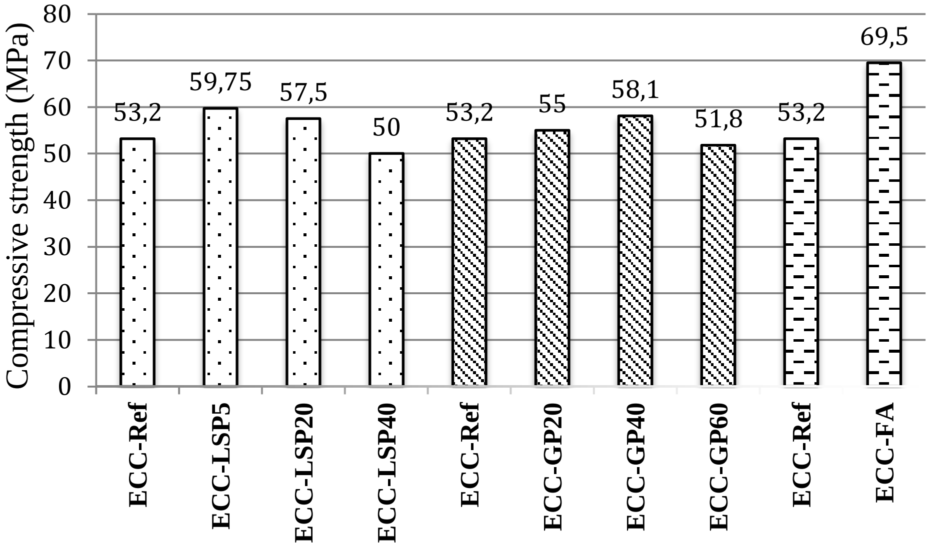


Figure 11: Effect of SCMs on 28-days Compressive Strength of ECCs.

4.4 Effect of Different Type and Proportions of SCMs on Microcracking Behavior of ECC under Compressive Loading

Stress-strain curves for all ECCs were plotted by conducting compressive strength test for all specimens under deformation control with a loading speed of 0.002 mm/s. it can be observed from the Figure 12 that GP and LSP replacement proportions in addition to fly ash and slag materials displayed an effect on microcracking behavior. For each ECC, the stress-strain curve showed linear pattern up to a certain stress level, before becoming non-linear. The non-linearity indicated that formation and propagation of cracks started at that point when non-linearity started. In order to define microcracking behavior for each ECC, the changes in the slopes of stress-strain curve were classified according to suitable tangent lines as can be tabulated in Table 6. Moreover, the objective of determining the tangent points was to choose the percentages of compressive strength to be loaded relative to the ultimate compressive strength for self-healing purposes.

The Table 6 illustrated that the first tangent line values slightly increased with increasing the proportions of LSP replacement to slag, which means that higher stress levels required for the cracks to start formation and propagation. The same pattern was observed for the GP replacement that means that increasing GP proportion required more stress value in order to generate cracks. Moreover, using fly ash instead of slag increased the first tangent line value, which means that formation and propagation of the cracks in ECC-Ref started earlier compared to ECC-FA.

Table 6: Effect of Different Type and Proportion of SCMs on Microcracking Behavior of ECC

Stress levels with respect to ultimate compressive strength (%)			
Mixtures ID	First tangent line	Second tangent line	Third tangent line
ECC-Ref	70.3	83.8	92.5
ECC-LSP5	71.6	83.3	93.3
ECC-LSP20	72.7	81.8	90.1
ECC-LSP40	74.0	84.0	93.0
ECC-GP20	71.8	85.4	92.7
ECC-GP40	72.5	84.2	92.9
ECC-GP60	73.7	85.8	93.5
ECC-FA	71.2	84.9	93.2

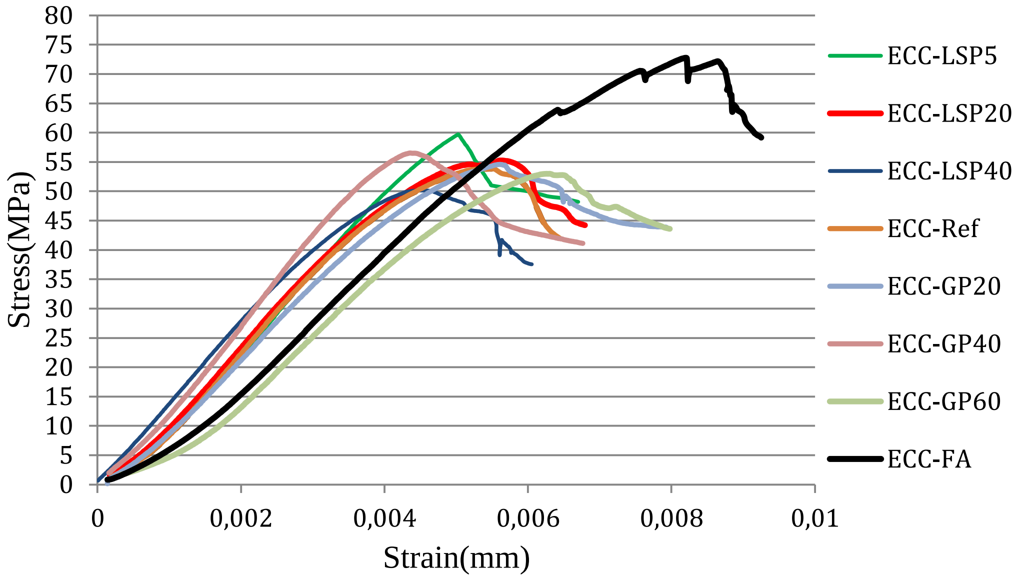


Figure 12: Effect of SCMs on Microcracking Behavior of ECC under Compressive Loading.

4.5 Effect of Different Type and Proportions of SCMs on Tensile Strength of ECCs

Tensile strength results of LSP-based ECCs and the reference mixture are presented in Figure 13, which illustrated that increasing the limestone powder proportion as a replacement to slag brought about decreasing in tensile strength value. Tensile strength values of ECC-LSP5, ECC-LSP20 and ECC-LSP40 were 5.8, 5.1 and 4.37MPa respectively. However, it was observed from the Figure that increasing slag replacement with LSP up to 5% caused an increase in tensile strength compared to ECC-Ref.

The reason for the decrease in tensile strength occurred with the increase in the quantity of LSP was due to the fact that limestone powder is considered as inert filler materials not as a pozzolanic material and limestone powder displayed low hardness and thereby, limestone powder particles can be easily exposed to breaking and thus reduced the matrix toughness. Moreover, adding more quantities of limestone powder (>5%) resulted in weakening the interface between matrix and fibers and thus, decreased tensile strength. The greater tensile strength gain in ECC-LSP5 over ECC-Ref can be attributed to the fact that replacing slag with LSP up to 5% enhanced fiber-matrix interface and displayed a reasonably denser structure. Moreover, the limestone powder actually acted as fillers, which helped to fill the voids and pores and thus increased the matrix density and tensile strength.

The tensile strength for different mixtures containing different proportions of GP (0,20,40 and 60%) at curing age of 28 days can be found in Figure 13. Increasing GP replacement percentage up to 20% increased tensile strength compared to ECC-Ref mixture. Tensile strength of ECC-GP20 and ECC-Ref were 5.85 and 5MPa respectively. It can be also demonstrated from the Figure that replacing glass powder after 20% resulted in decreasing tensile strength of ECCs. However, each GP-based ECC exhibited higher tensile strength value than reference mixture.

The decrement in the tensile strength with increasing the glass powder proportions can be attributed to the higher alkali content that GP possesses, which reduced the rate of hydration reaction and caused potential heterogeneity between the particles of matrix. Moreover, agglomerating glass powder occurred and that resulted in producing more voids and pores, thus producing less dense concrete and tensile strength. The higher achievement of tensile strength of GP-incorporated ECCs and particularly ECC-GP20 over ECC-Ref was associated with the fact that GP can produce better chemical bonds between fibers and surrounding matrix than slag material. In addition, the optimal tensile strength of ECC-GP20 was attributed to the angular shape of GP that significantly prevented the concrete from splitting.

The Figure 13 revealed that ECC containing completely slag material as a SCM displayed higher tensile strength compared to ECC-FA mixture. Tensile strength of ECC-Ref and ECC-FA were 5 and 4.5MPa respectively.

This result was attributed to that S improved the fiber-matrix interface. Furthermore, using slag led to produce C-S-H gels, which improved the matrix density and friction bond with the fiber. On the other hand, the lower value of ECC-FA tensile strength due to the FA in ECC diminished the blend toughness and the chemical bond of interface between the fiber and adjacent matrix.

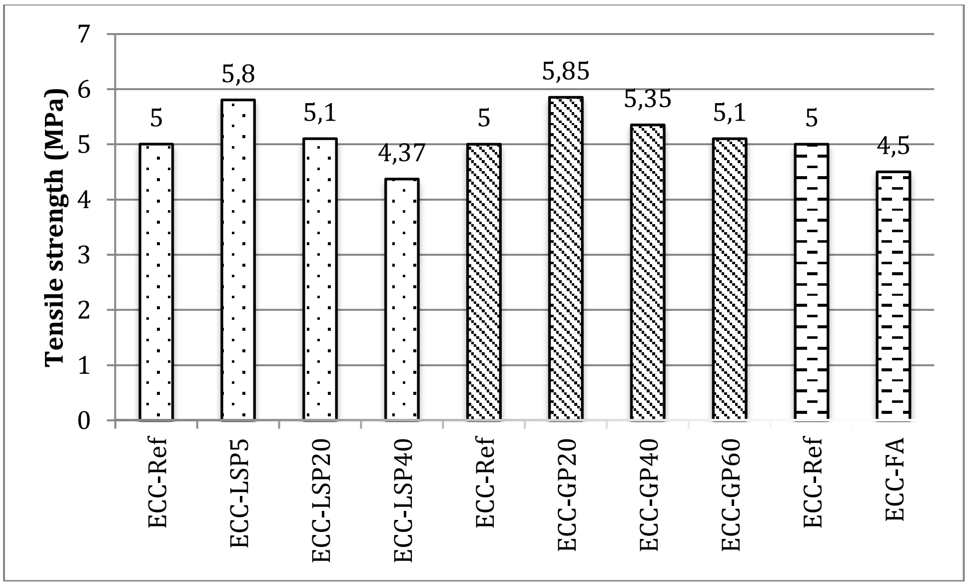


Figure 13: Effect of SCMs on 28-days Tensile Strength of ECCs

4.6 Effect of Different Type and Proportions of SCMs on Tensile Strength Loss (TSL) of ECCs

The results of TSL that were demonstrated in Figure 14 showed that increasing LSP replacement quantity to slag up to 20% resulted in decreasing the TSL compared to ECC-Ref. After that, the TSL started to increase with 40% slag replacement with LSP. TSL in ECC-Ref, ECC-LSP5, ECC-LSP20 and ECC-LSP40 were 25, 20.8, 18.6 and 21.4% respectively.

The decrement in TSL with increasing the LSP replacement up to 20% can be due to at first, the larger particle size of slag compared to LSP created more voids and cracks and thus the TSL of ECC-Ref was higher. At second, the limestone powder acted as a filler material, which resulted in filling pores and voids and accordingly the cracks were smaller and TSL was improved. However, using 40% of LSP as a replacement to slag increased the TSL again, which might be attributed to that at this replacement level, the matrix-fiber interface was weakened and thus more voids and cracks were generated, hence, resulting in increase in TSL.

According to the Figure 14, the TSL of ECCs incorporating different proportions of PG displayed the same pattern as was observed for LSP-incorporated ECC. TSL was decreased with increasing the amount of GP substituted up to 40%. TSL in ECC-Ref, ECC-GP20 and ECC-GP40 were 25, 21.8 and 18.8% respectively, following that, ECC-GP60 displayed higher TSL than what was observed for ECC-GP40, TSL of ECC-GP60 and ECC-GP40 were 24.4 and 18.8% respectively.

The decreasing in TSL with increasing slag substitution with GP up to 40% can be attributed to the higher pozzolanic reactivity of GP than that was in slag material. In addition, GP particles exhibited higher Blaine fineness (smaller particle size) compared to S particles, which means that glass powder possessed higher possibility to improve paste matrix compactness through filling effect up to 40% replacement level and thus, the voids and cracks were reduced, which resulted in improving in TSL, in other words decreasing the TSL. Nevertheless, using 60% slag replacement with GP resulted in agglomerating glass powder and producing more voids and pores and thus producing less dense concrete and more cracks, which increased TSL.

It can be shown from the Figure 14 that the loss in tensile strength of ECC-FA was higher than TSL of ECC-Ref. TSL of ECC-FA and ECC-Ref were 32.3 and 25% respectively.

This result can be attributed to that S improved the fiber-matrix interface. Furthermore, using slag led to produce C-S-H gels, which improved the matrix density and friction bond with the fibers, which resulted in reducing the voids and cracks produced, hence, decreased TSL. On the other hand, the higher value of ECC-FA TSL was due to the FA in ECC diminished the blend toughness and the chemical bond of interface between the fiber and adjacent matrix and that created more cracks and voids and accordingly increased TSL.

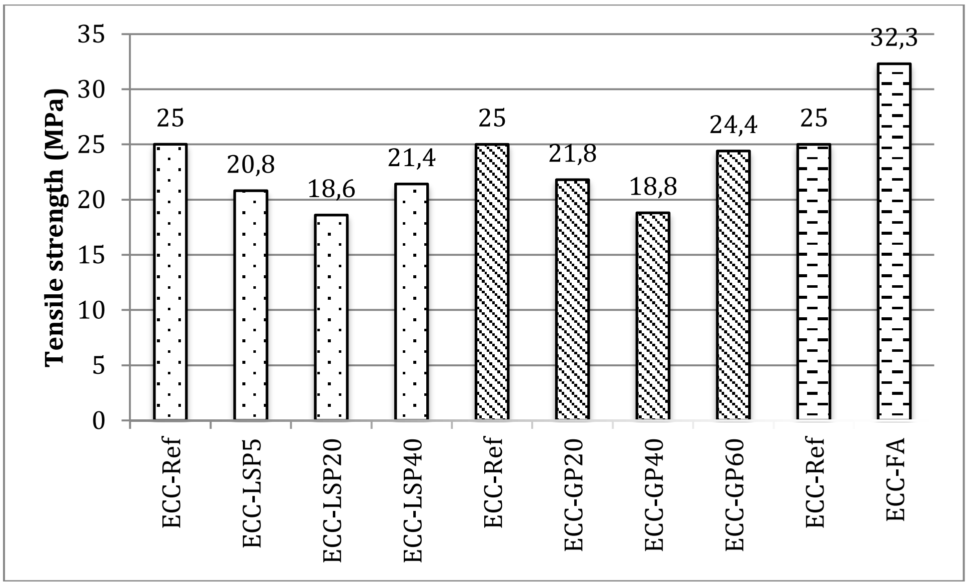


Figure 14: Effect of Different Type and Proportions of SCMs on TSL of ECCs

4.7 Effect of SCMs on Self-Healing of ECCs

Self-healing performance of ECC mixtures was assessed in terms of compressive strength recovery, Ultrasound Readings before and after self-healing process and microstructural analysis by using microscope for detecting the healed cracks

4.7.1 Effect of SCMs on Compressive Strength Recovery after Self-Healing of ECCs

At the age of 28 days, three 100-mm cubic specimens from each composition were preloaded up to ultimate under compressive strength test. Thereafter, two new cubes used to evaluate the self-healing rate were preloaded up to 85% of their ultimate compressive strength values and left to recover in water tanks for 30 days of healing. The healing efficiency of ECC mixtures was evaluated by reloading the healed specimens to failure after 30 days of moist curing. Compressive strength values of preloaded (healed) specimens were recorded and compared to those of sound specimens.

It can be shown from the Figure 15 that compressive strength of preloaded (healed) specimens incorporating different proportions of LSP displayed the same pattern of sound specimens. The compressive strength increased with increasing LSP replacement to slag up to 20%, after that, the decrease of compressive strength was observed for ECC-LSP40. These results can be attributable to the same reasons mentioned in section 4.2.1. On the other hand, Figure 16 illustrated that compressive strength recovery decreased with increasing slag substitution quantities with LSP.

Compressive strength recovery (gain) of ECC-Ref, ECC-LSP5, ECC-LSP20 and ECC-LSP40 were 31.5, 29.2, 27.1 and 25% respectively. These results might be attributable to that increasing replacement of S with LSP resulted in reducing un-hydrated cementitious required for further reaction and pozzolanic reaction and thus decreased the rate of compressive strength gain. Moreover, C-S-H gels, which are considered as the most outstanding products that responsible for compressive strength recovery, were reduced not only because of the absence of the pozzolanic reaction in LSP, but also due to decreasing the amount of pozzolanic reaction of slag with increasing LSP replacement levels.

According to the Figure 15, the compressive strength of healed ECCs-incorporated GP represented the same trend observed for sound specimens, increasing GP replacement level to slag up to 40% increased the compressive strength value. However, compressive strength was reduced for ECC-GP60. The reasons behind these results were explained in section 4.2.2. Figure 16 showed that although the results revealed small decrements in recovery with GP content, the maximum difference of <7% (noted for ECC-GP60) proved the good self-healing capability of

GP-contained ECCs. The greater compressive strength gain for ECC-Ref and ECC-GP20 over ECC-GP40 and ECC-GP40 might be attributable to the continued pozzolanic reaction of the plentiful un-hydrated cementitious particles remained for GP and particularly for slag. Moreover, slag and glass powder materials possessed substantial amount of silica, which was highly responsible for the pozzolanic reactions at later ages and significantly contributed to form C-S-H gels that played a fundamental role in increasing compressive strength. The decrement in compressive strength recovery with increasing quantity of GP replacement to slag can be associated with the high alkali content of GP, which resulted in occurring heterogeneity in microstructure of C–S–H gels and reducing reaction rates and thus reduced the strength gain or recovery rate.

Preloaded specimens of ECC-FA displayed more Compressive strength than preloaded samples of ECC-Ref as can be observed in Figure 15. The same pattern exhibited in sound specimens of both ECCs. The explanations of this result were mentioned in section 4.3.2. However, Figure 16 revealed that compressive strength recovery rate of ECC-Ref was slightly higher than what was measured for ECC-FA, which might be attributed to that at later ages, pozzolanic reaction of slag remarkably started to occur and that increased compressive strength rate. Furthermore, the reaction between silica that slag possessed and calcium hydroxide from cement substantially contributed to produce more C-S-H gels and thus significantly improved compressive strength gain rate. The slight difference in compressive strength recovery between ECC-Ref and ECC-FA was due to the high pozzolanic activity of fly ash and its high specific surface area and thus, FA helped to seal and fill the voids and pores and increased the compressive strength recovery.

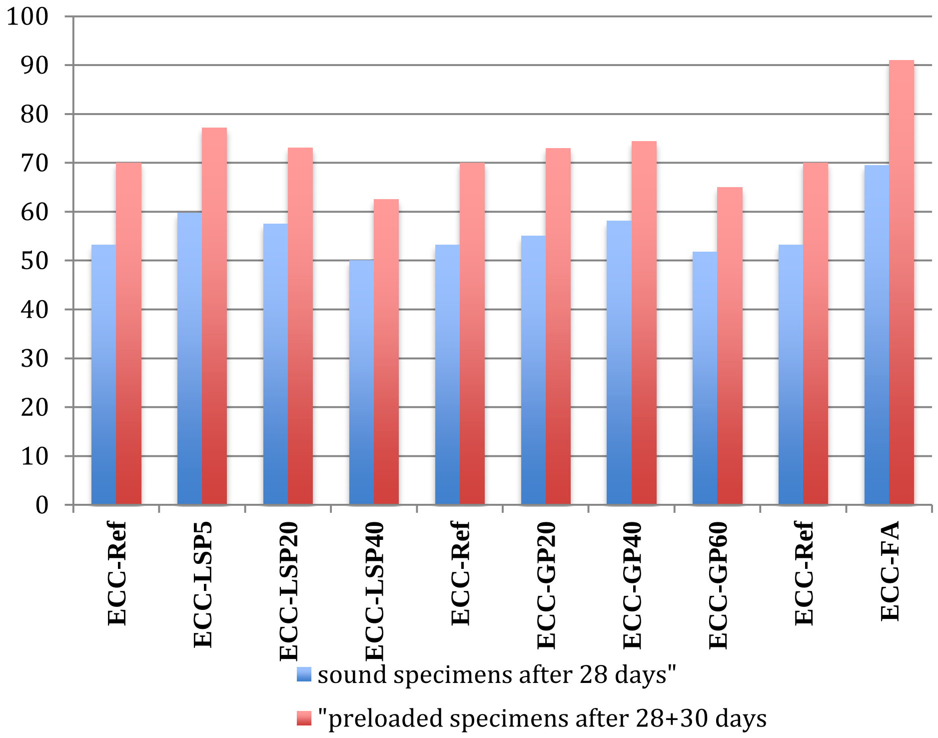


Figure 15: Compressive Strength of Sound and Preloaded ECC Specimens

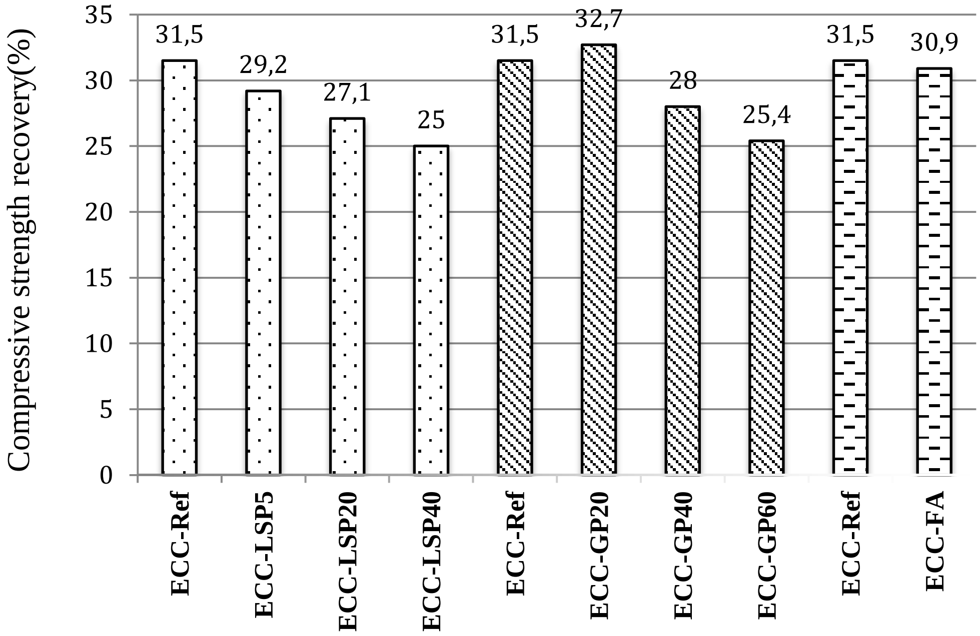


Figure 16: Effect of SCMs on Compressive Strength Recovery of ECCs after 30 days of Healing

4.7.2 Effect of SCMs on Ultrasound Readings before and after Self-Healing of ECCs

In addition to using compressive strength recovery measurements in order to determine the effect of SCMs on self-healing of ECC, ultrasonic test was performed to evaluate self-healing efficiency of different ECCs. The travel time was measured for each ECC immediately after causing the cracks under ultimate splitting tensile strength (T_0) before healing and then the specimens were kept in water tank for one month of healing before measuring the travel time again (T_1). The difference between travel time for each ECC before healing and after healing was measured and shown in the Table 7.

Table 7: Ultrasonic Test Results of ECC Specimens

MIX ID	T_0 microsecond	T_1 microsecond	(T_0- T_1) microsecond
ECC-Ref	26.5	23.3	3.2
ECC-LSP5	26.2	23.4	2.8
ECC-LSP20	25.8	23.4	2.4
ECC-LSP40	25.5	23.7	1.8
ECC-GP20	26.6	23.6	3.0
ECC-GP40	26.9	24.5	2.4
ECC-GP60	27.5	25.4	2.1
ECC-FA	24.8	23	1.8

Table 7 showed that the travel time measured for each ECC after one month of healing (T_1) was lower than the time measured for all ECC specimens loaded under splitting tensile test up to ultimate and accordingly, healing improvement occurred for the cracks generated.

It can be seen from the Table 7 that the difference between T_0 and T_1 decreased with increasing the LSP replacement proportion to slag material, which indicated that the decrease in self-healing rate happened. This result was attributable to that increasing replacement of S with LSP resulted in reducing un-hydrated cementitious required for further reaction and pozzolanic reaction as a result of absence the pozzolanic reactivity of LSP and thus decreased the rate of crack healing. Moreover, C-S-H gels, which are considered as the most outstanding products that responsible self-healing improvement, were reduced not only because of the absence of the pozzolanic reaction in LSP, but also due to decreasing the amount of pozzolanic reaction of slag with increasing LSP replacement proportions.

The same pattern was observed when GP substitutions were used. (T_0- T_1) value was decreased when GP replacement quantity was increased. The decrement in (T_0- T_1) value with increasing slag replacement level with GP can be attributed to the higher alkali content of GP, which reduced the rate of hydration reaction and caused slight potential heterogeneity between the particles of matrix and accordingly, reduced healing crack rate. Moreover, agglomerating glass powder can occur and that resulted in producing more voids and pores, thus producing less dense concrete and rate of cracks healing. Replacing slag with GP increased the crack width and thus cracks needed more time to be healed.

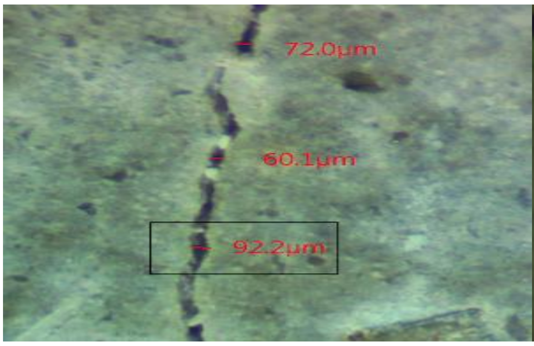
4.7.3 Effect of SCMs on Largest Crack Width before Self-Healing of ECCs

In order to generate cracks on the surface of ECC specimens, three 100-mm cubic specimens were loaded up to ultimate tensile strength under splitting tensile test at 28 curing day. Stereomicroscope immediately was used to observe the cracks and measure the width of the cracks.

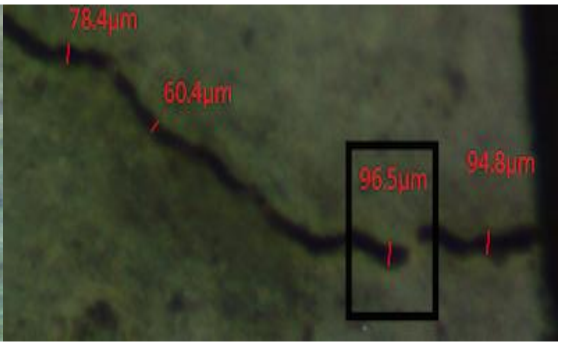
The results of Table 8 and Figure 17 revealed that crack width with less than 105 μm was displayed for each ECC mixture. Increasing GP replacement proportion to slag resulted in increasing slightly the crack width. The crack width of ECC-Ref, ECC-GP20, ECC-GP40 and ECC-GP60 were 92.2, 96.5, 98.6 and 104.1μm respectively. This increment in crack width might be attributable to that increasing GP substitution level to slag increased the chemical bond between the matrix interface and fiber, moreover, augmented packing density and accordingly, increased the width of the crack formed. Moreover, it might be associated with less uniform distribution of the fibers throughout the matrix. On the other hand, increasing slag replacement proportion with LSP made the crack tighter, which means that crack width was decreased. This result could be associated with that LSP possessed filling characteristic resulting in an improved packing at the fiber-matrix interface and thus reduced the width of the crack.it can be noticed from the Table 8 and the Figure 17 that crack width of ECC-AF specimen was smaller than the width of the crack generated in ECC-Ref specimen. Crack width of ECC-FA and ECC-Ref were 54.3 and 92.2 μm respectively. This result was due to ability of FA to distribute the fiber uniformly and its smooth spherical shape, which limited the chemical bond interaction between the matrix and fibers and thus reduced the crack width.

Table 8: Crack Width (μm) of ECC Specimens.

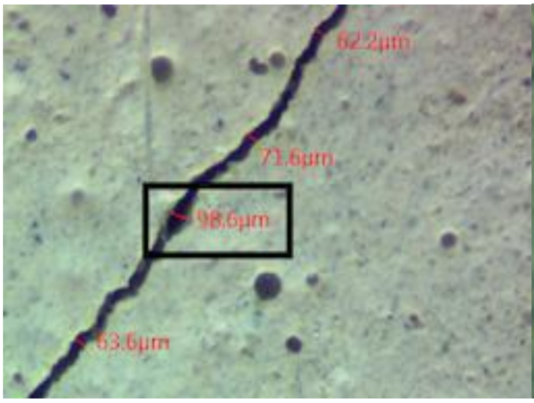
Mixture ID	Width average (μm)	Max width (μm)
ECC-Ref	61.3	92.2
ECC-GP20	65.5	96.5
ECC-GP40	67.6	98.6
ECC-GP60	70.9	104.1
ECC-LSP5	57.1	88.7
ECC-LSP20	53.8	82.6
ECC-LSP40	50.7	76.3
ECC-FA	35.5	54.3



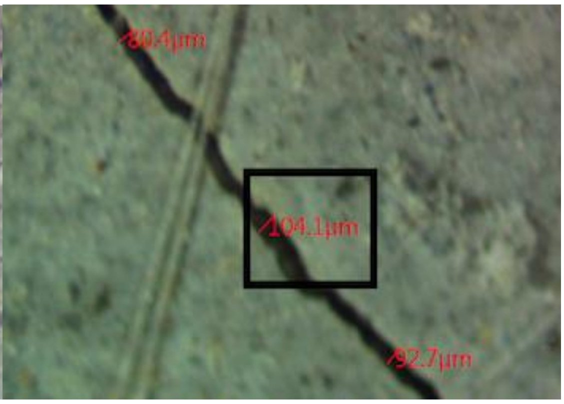
ECC-Ref



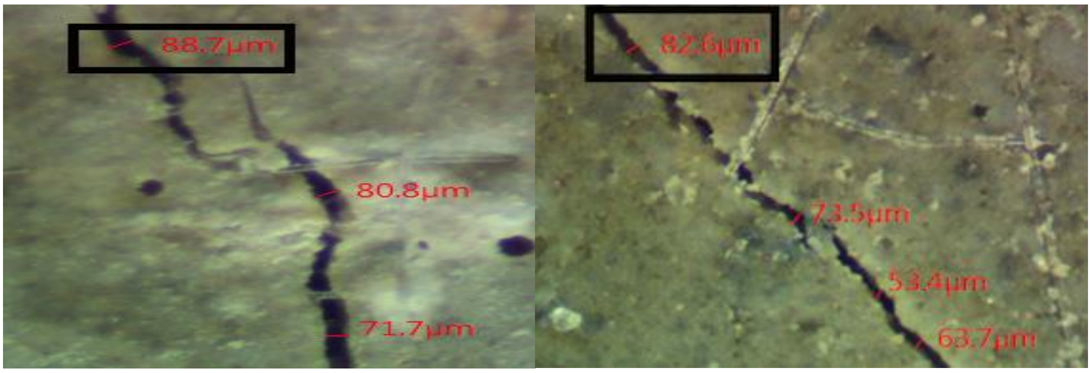
ECC-GP20



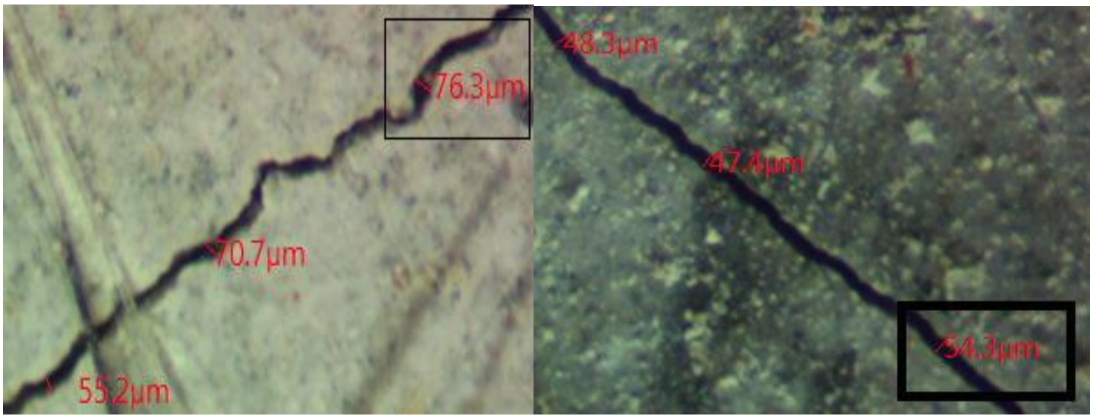
ECC-GP40



ECC-GP60



ECC-LSP5 ECC-LSP20



ECC-LSP40 ECC-FA

Figure 17: Maximum Crack Width for each ECC Observed by Microscope.

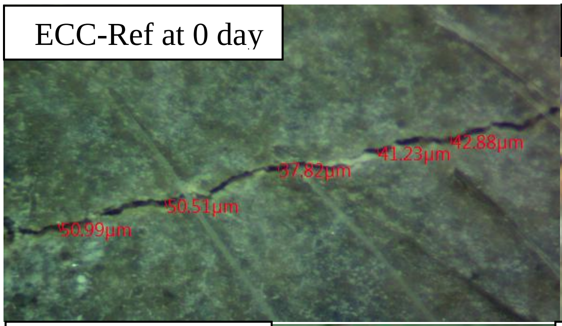
4.7.4 Effect of SCMs on Crack Width of ECCs after 45-days of Self-Healing

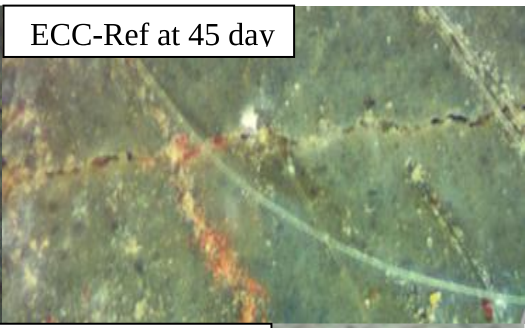
In order to observe self-healing rate and healing products formed in the cracks, three specimens from each ECC were loaded up to ultimate tensile strength under splitting tensile test in order to generate cracks on the surface and then they were kept in the water tank for 45 days of healing. After that, microscope was used in order to measure the crack width and to observe any change or healing product over the period of healing (45 days).

The results of Figure 18 revealed that all ECC mixes displayed different levels of self-healing rate as a result of different crack width formation and SCMs type. Water curing significantly encouraged the self-healing efficiency and rate by reducing crack width and forming healing products and accordingly, this type of curing could be considered as an ideal curing condition to improve durability of ECC.

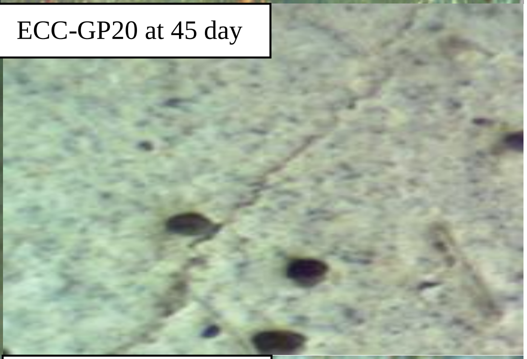
It can be seen from Figure 18 that produced crack with width up to 50μm in ECC-Ref was almost completely healed. However, for ECC-GP20, complete healing was observed when the crack width did not exceed 36 μm. increasing GP replacement proportion for slag promoted more healing products to be formed in the crack. Crack width up to 41.5 μm and 67 μm for ECC-GP40 and ECC-GP60 were entirely healed respectively.

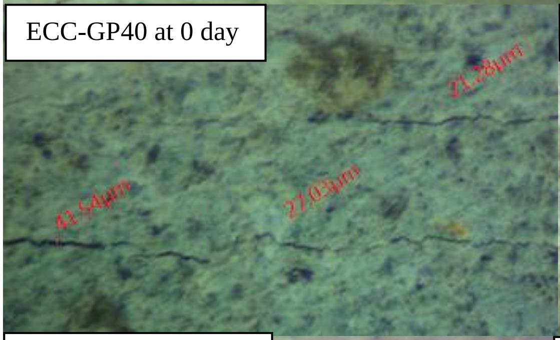
Crack of ECC-LSP5 mixture was observed to be partially healed when the width of the crack was almost bigger than 40 μm; however, crack-having width under 40 μm was completely sealed and clogged with healing products. Regarding to ECC-LSP20, Complete healing occurred in the crack width of less than 32 μm. while the healing was partially observed in the crack of ECC-LSP40. Using FA demonstrated lower healing rate than what was observed when the slag was used. Crack width for complete self-healing to take place was about 34 μm for ECC-FA, while around 50 μm for ECC-Ref. crack of ECC-FA was observed to be partially healed when its width was between 48 μm and around 53 μm.

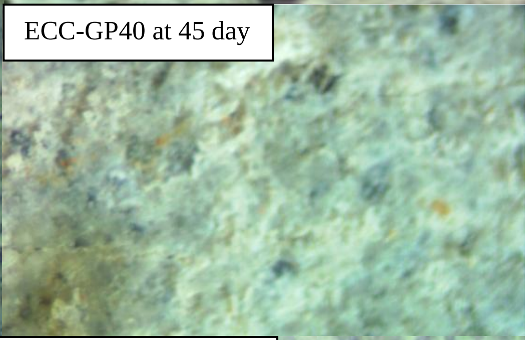

ECC-Ref at 0 day
50.99μm
50.51μm
57.82μm
41.23μm
42.88μm

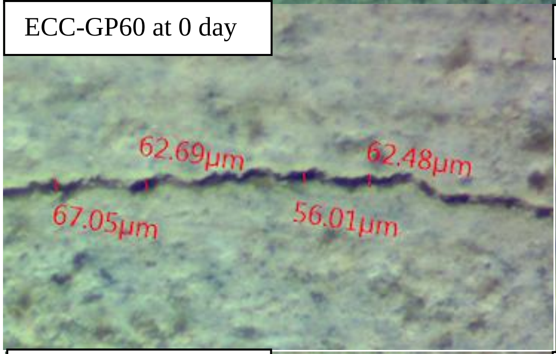

ECC-Ref at 45 day

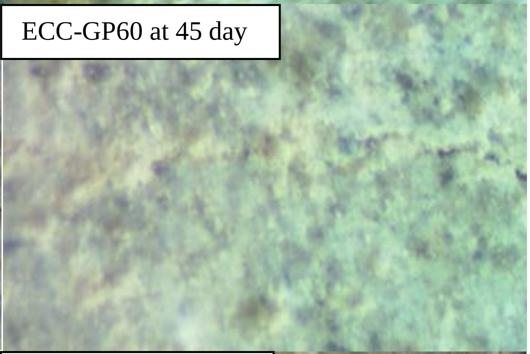

ECC-GP20 at 0 day
36.09μm
23.64μm

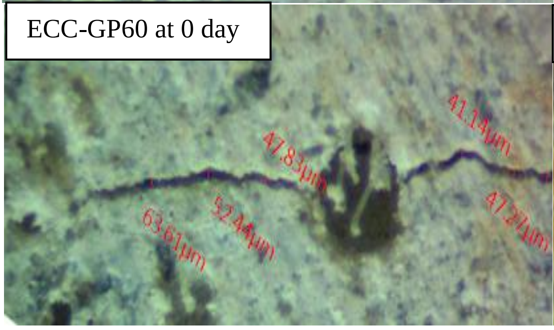

ECC-GP20 at 45 day

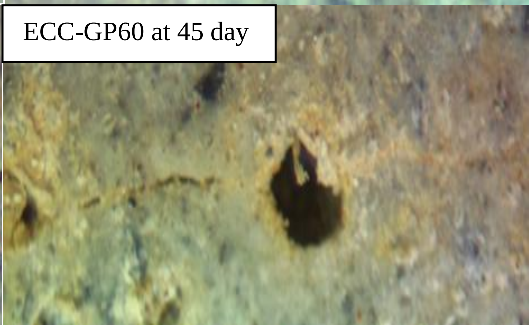

ECC-GP40 at 0 day
21.28μm
41.54μm
27.03μm


ECC-GP40 at 45 day


ECC-GP60 at 0 day
62.69μm
62.48μm
67.05μm
56.01μm


ECC-GP60 at 45 day


ECC-GP60 at 0 day
47.83μm
41.14μm
52.44μm
63.61μm
47.27μm


ECC-GP60 at 45 day

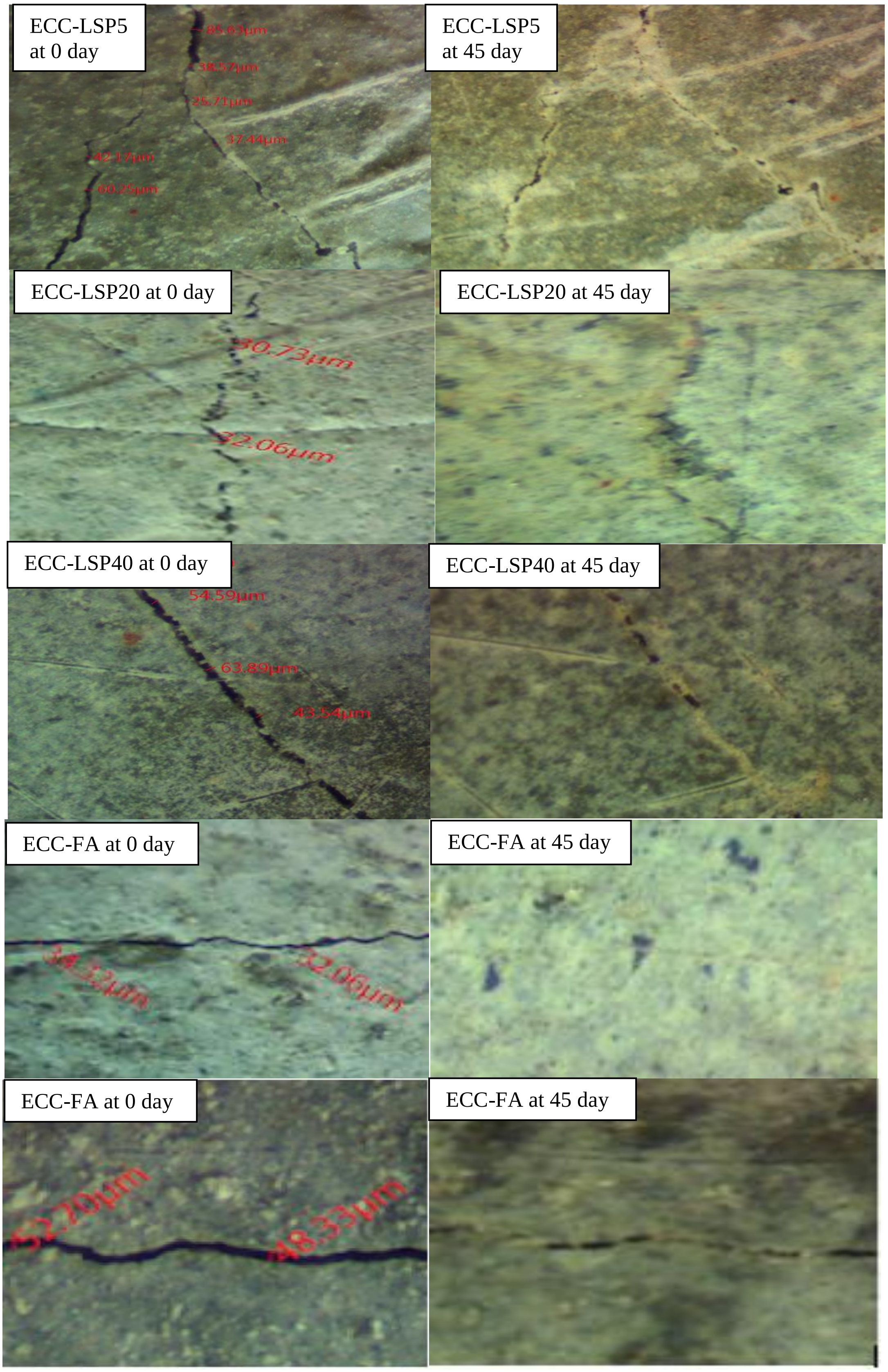


Figure 18: Self-Healing in ECC Microcracks before Exposure to Water Curing and after 45 days of Healing

4.8 Analysis of Healing Products Formed in the Healed Cracks of ECCs

According to what Mustafa Sahmaran et al (2012) stated regards to healing products of ECC-Ref, it is expected that formation of calcium carbonate in the crack can be significantly observed, which is attributable to high quantity of CaO that slag possesses. Moreover, it can be said according to chemical compositions of slag that a relative low SiO_2 content compared to FA, leads to remain sufficient amount of CH unconsumed and thus, PH value of pore solution is kept high, which is resulting in increasing precipitation rate of $CaCO_3$. These are the reasons why calcite precipitation is more observed for ECC-Ref compared to ECC-FA.

Although $CaCO_3$ is the prominent healing product formed in the crack of ECC-Ref, presence of quartz (SiO_2) in slag indicates that C-S-H gels can be also partially formed in order to heal the cracks generated in ECC-Ref specimens.

Cracks generated in ECC-FA specimens are partly healed with calcite products and mainly healed with C-S-H gels formation. The presence of SiO_2 and CaO compounds in FA contribute to produce the healing products already mentioned.

Although replacing slag with LSP decreased the efficiency of self-healing process, results show that there are healing products formed in the healed cracks of LSP-incorporated ECCs. It is predictable that calcium carbonate is the most outstanding healing product due to high $CaCO_3$ content of LSP. Moreover, the presence of aluminates from slag might react with calcium carbonate from LSP; hence, monocarboaluminate products can be produced in the healed cracks, which can be considered as a healing product beside calcite precipitations. On the other hand, C-S-H gels produced from pozzolanic reaction might be limited as a result of decreasing the content of SiO2 with increasing replacement level of LSP to slag. These results were confirmed by Hocine Siad et al (2015).

Based on the results obtained by Hocine Siad et al (2017), it can be suggested that presence of excessive quantity of silica from GP dissolution results in consuming more CH during pozzolanic reaction and thus decreases the value of PH, which leads to limit the calcium carbonate capacity in healed cracks compared to reference mixture (100% of slag). However, the higher intensity of SiO2 content in GP significantly contributes to form C-S-H gels, which indicates that C-S-H formation in the healed cracks dominates the other healing products.

4.9 A General Comparison about the Effects of SCM on Workability, Mechanical Properties and Self-Healing Efficiency of ECC.

4.9.1 Comparison Between the Same Replacement Levels of GP and LSP

It can be illustrated from the Table 5 that although the particle size of GP is smaller than the particle size of LSP, ECC-LSP20 displayed more flowability than ECC-GP20, which can be attributed to the potential agglomeration that might occur when GP was used and accordingly decreased the workability. Replacing slag with LSP and GP up to 40% decreased the workability. The decrement was more obvious in the mix containing GP than the mixture with LSP, which can be attributed to that using GP resulted in agglomerating the particles together. Moreover limited uniform distributions of fibers through the whole mix more than when the LSP replacement was utilized and thus decreased the workability.

It can be shown from the figure 11 that replacing slag with LSP up to 20% displayed improvement in compressive strength more than ECC-GP20. The compressive strength gain of ECC-LSP20 over ECC-GP20 was attributed to the filling characteristics of LSP and the carboaluminate formation produced from the reaction between Aluminates from slag and calcium carbonate from LPS that reduced the porosity and filled the voids. However, the compressive strength of ECC-GP40 was significantly higher than the compressive strength of ECC-LSP40, this was due to the high pozzolanic reactivity of GP and forming C-S-H gels by reacting calcium hydroxide from cement with silica from GP, which significantly contributed to improve the compressive strength.

It can be revealed from figure 13 that tensile strength of the mixtures incorporating GP up to 20% and 40% replacement levels to slag were higher than tensile strength of the mixtures having the same replacement level of LSP. The increment of tensile strength of ECC-GP20 and ECC-GP40 over ECC-LSP20 and ECC-LSP40 was associated with the fact that limestone powder is considered as inert filler materials not as a pozzolanic material and limestone powder displayed low hardness and thereby it can be easily exposed to breaking and thus reduced tensile strength. Moreover, using GP improved the interface between matrix and fibers and thus, increased tensile strength. In addition, angular shape of GP significantly prevented the concrete from splitting.

Figure 14 illustrated that TSL of ECC-GP20 was higher than the TSL of ECC-LSP20, which was due to agglomerating glass powder and producing more voids and pores and thus producing less dense concrete and more cracks, which increased TSL. Moreover, that level of limestone powder acted as a filler material, which resulted in filling pores and voids and accordingly the cracks were smaller and TSL was improved. However, replacing slag with GP up to 40% displayed less TSL than when 40% of slag was replaced with LSP, which might be attributed to that at this replacement level of LSP, the matrix-fiber interface was weakened and thus more voids and cracks were generated, hence, resulting in increase in TSL compared to GP. Although there was agglomeration in GP, but its smaller particle size compared to LSP contributed to reduce the voids and thus decreased TSL.

It can be demonstrated from figure 16 that compressive strength recovery of ECC-GP20 and ECC-GP40 was remarkably higher than the compressive strength recovery of ECC-LSP20 and ECC-LSP40. This result was due to the substantial amount of silica in GP, which was highly responsible for the pozzolanic reactions at later ages and significantly contributed to form C-S-H gels that played a fundamental role in increasing compressive strength recovery. Moreover, increasing replacement of S with LSP resulted in reducing un-hydrated cementitious required for further reaction and pozzolanic reaction and thus decreased the rate of compressive strength recovery. The same pattern was observed regarding Ultrasonic Test Results for both replacement levels of GP and LSP.

The results of Table 8 and Figure 17 revealed that ECC-LSP20 and ECC-LSP40 displayed tighter crack width than ECC-GP20 and ECC-GP40, which might be attributable to that increasing GP substitution level to slag increased the chemical bond between the matrix interface and fiber and accordingly, increased the width of the crack formed. However, LSP possessed filling characteristic resulting in improved packing at the fiber-matrix interface reduced the voids and pores, thus reduced the width of the crack.

Self-healing efficiency of ECC-GP20 and ECC-GP40 was more obvious than self-healing occurred in ECC-LSP20 and ECC-LSP40 as can be shown in figure 18. This might be attributed to the absence of pozzolanic reactivity of LSP and decreasing the pozzolanic reaction of slag with increasing slag replacement level with LSP. While the high pozzolanic reactivity of GP significantly contributed to heal the cracks. Moreover, the higher intensity of SiO_2 content in GP significantly contributed to form C-S-H gels, which played fundamental role in improving Self-healing efficiency.

4.9.2 Correlation Results for ECC Produced With LSP and GP

As it can be seen in Figure 19, there is a good correlation between compressive and tensile strength values of ECC produced either with LSP or GP.

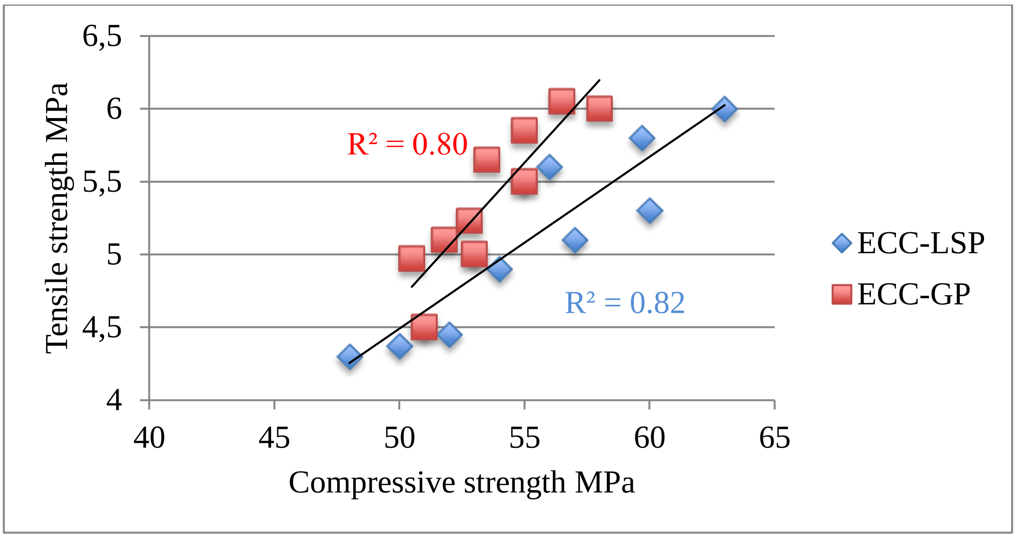


Figure 19: Relationship Between Compressive Strength and Tensile Strength for ECC Produced With LSP and GP

Figure 20, represents the relationship between compressive strength recovery and the ultrasound travel time difference (T0-T1) for both types of ECC incorporating either GP or LSP after 45 days of self-healing. As it can be read from the above figure, there is a perfect correlation between compressive strength recovery and crack healing rate, which was obtained from ultrasound reading differences before and after healing.

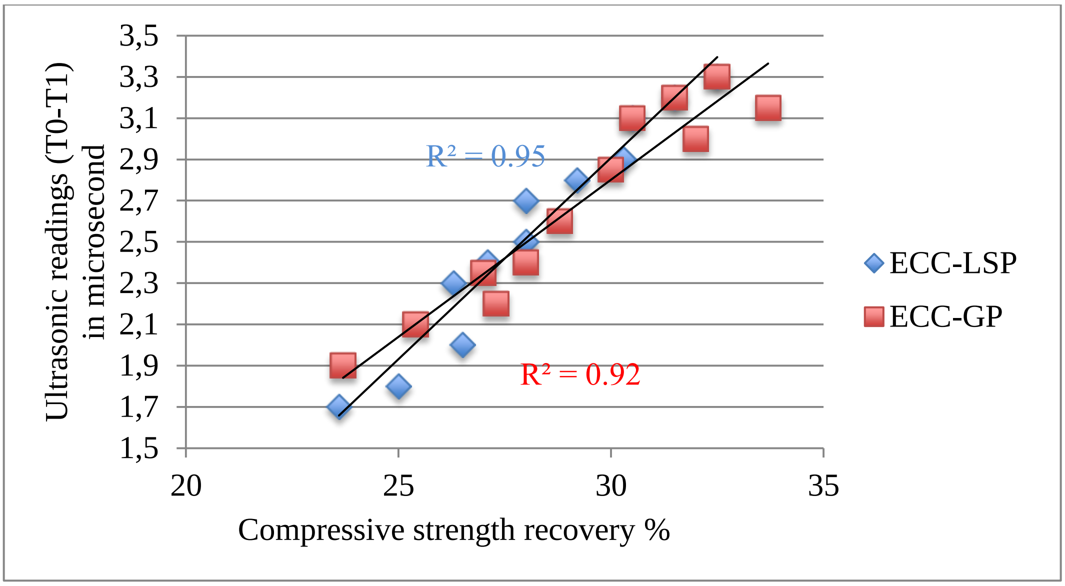


Figure 20: Relationship Between Compressive Strength Recovery and Ultrasound Readings for ECC Produced With LSP and GP

Chapter 5

CONCLUSION

5.1 Conclusion

This experimental study investigates the possibility of utilizing distinct proportions of limestone powder and glass powder as slag replacement in engineered cementitious composites (ECC) in order to improve its fresh and mechanical properties. The effect of using different levels of SCMs on self-healing efficiency of ECC was also examined by measuring the recovery level after preloading specimens from each ECC up to a certain level of ultimate compressive strength at the age of 28 days and then kept in the water-curing tank for 30 days of healing. Moreover, for more self-healing investigation, cracks were produced on the surface of samples after loading them up to ultimate splitting tensile strength and then left under water curing up to 45 days of healing. The obtained results resulted in drawing the following conclusions:

1. Using LSP and GP as a slag replacement up to 20% improved workability of ECC, while increasing replacement levels of slag with GP and LSP more than 20% led to reduce ECC flowability. However, all ECCs displayed flowability index (Γ) more than 2.75 and accordingly, achieved desired workability.

2. Incorporating LSP and GP as a slag replacement significantly improved compressive strength results at advanced curing age. GP and LSP materials displayed compressive strength improvements up to 40% and 20% of slag substitution respectively.

3. Using different proportions of GP and LSP replacement for slag showed slightly positive effect on microcracking behavior. However, Results of stress-strain curves revealed that in order to start crack formation and propagation for each ECC, stress value should be at least 70% of ultimate stress.

4. Replacing slag with LSP up to 5% increased tensile strength, while; the optimal improvement in tensile strength among GP-incorporated ECCs was observed when 20% of slag was substituted with GP.

5. Self-healing efficiency of ECC mixtures was measured in terms of compressive strength recovery. Self-healing recovery decreased with increasing LSP replacement quantities to slag, which was mainly attributed to the absence of LSP pozzolanic reactivity. Although the results revealed small decrements in recovery with GP content, the maximum difference of <7% (noted for ECC-GP60) proved the good self-healing capability of GP-contained ECCs.

6. According to ultrasonic test results, replacing slag with LSP and GP decreased self-healing rate and efficiency, which was attributable to that increasing replacement of S with LSP resulted in reducing un-hydrated cementitious required for further reaction and pozzolanic reaction and thus decreased the rate of crack healing and to the higher alkali content of GP, which reduces the rate of hydration reaction and

causes slight potential heterogeneity between the particles of matrix and accordingly, reduced healing crack rate.

7. Increasing the proportion of LSP replacement to slag resulting in decreasing the width of the crack, while replacing slag with GP increased the crack width. However, all ECC specimens displayed an average crack width with less than 105 μm. In addition, crack width of ECC-GP20 and ECC-LSP5 was observed to be comparative to ECC-Ref.

8. ECC mixes displayed different levels of self-healing efficiency as a result of different crack width formation and SCMs type. Replacing slag with LSP decreased the self-healing capacity compared to ECC-Ref. On the other hand, substituting slag with GP up to 60% showed improvement in self-healing capacity compared to the reference mixture. Crack width up to 67 μm and 50 μm were completely healed for ECC-GP60 and ECC-Ref respectively.

9. Replacing slag with fly ash increased compressive strength while slightly decreased tensile strength. Comparative self-healing efficiency was observed for both ECCs in terms of compressive strength recovery. ECC-FA displayed tighter crack width than ECC-Ref. However, complete healing was observed more remarkably in ECC-Ref rather than ECC-FA.

10. Overall, replacing slag with LSP up to 5% and with GP up to 20% displayed significant improvement on mechanical properties of ECC and a high relative comparative of self-healing efficiency with ECC-Ref.

5.2 Recommendations for Further Investigations

1. Utilizing more accurate proportions of limestone and glass powders as a slag substitution material in order to determine the optimal replacement level that significantly improves self-healing efficiency and mechanical properties of ECC.

2. Increasing the pozzolanic reactivity of slag by increasing the fineness level of it, which might contribute to reveal better self-healing capacity and mechanical properties of ECC.

3. Using combination between slag and fly ash materials, which might improve self-healing behavior of ECC.

4. Conducting more tests such as X-ray diffraction (XRD) and scanning electron microscope (SEM), which can be utilized in order to detect the self-healing products formed in the crack. Thereby, the contribution of each SCM on self-healing efficiency by investigating the healing products produced by these SCMs can be determined.

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