DOKUZ EYLÜL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

AN INVESTIGATION ON SELF-HEALING BEHAVIOUR OF CEMENT-POZZOLAN BINDER MATRIX

by
Houtan VAHEDI

October, 2019
İZMİR

AN INVESTIGATION ON SELF-HEALING BEHAVIOUR OF CEMENT-POZZOLAN BINDER MATRIX

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M.Sc THESIS EXAMINATION RESULT FORM

We have read the thesis entitled "**AN INVESTIGATION ON SELF-HEALING BEHAVIOUR OF CEMENT-POZZOLAN BINDER MATRIX**" completed by **HOUTAN VAHEDI** under supervision of **PROF. DR. HALİT YAZICI** and we certify that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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AN INVESTIGATION ON SELF-HEALING BEHAVIOUR OF CEMENT-POZZOLAN BINDER MATRIX

ABSTRACT

Many concrete structures including infrastructures and all cementitious materials suffer from serious deteriorations such as cracking due to shrinkage and other factors. As a result, inspection and sustentation techniques for concrete structures have become of more attention. Even so, the implementation of inspection is difficult, especially in large-scale concrete structures. On the other hand, repair may be impossible because of existing conditions, such as untouchable damage locations and considerable large amounts of labor and funds required for repairing. Depending on the circumstances, self-healing in cementitious materials become more of significance.

The aim of this study is to investigate the efficiency factor of the various types of pozzolanic binders on the cementitious composite materials' healing characteristic. The investigation of self-healing on mixtures was undertaken with compressive strength, direct tensile, cylinder split-tensile and crack analysis test methods. The effect of water/binder and pozzolan /cement ratio on the self-healing ability were also determined.

Keywords: Self-healing, crack wide, cylinder, pozzolan, fiber, cement base material

ÇİMENTO – PUZOLAN BAĞLAYICI MATRİSİNDE KENDİ KENDİNE İYİLEŞME DAVRANIŞININ İNCELENMESİ

ÖZ

Altyapılar ve tüm çimento esaslı malzemeler dahil olmak üzere birçok beton yapı, büzülme ve diğer faktörler nedeniyle çatlama gibi ciddi bozulmalardan muzdariptir. Sonuç olarak, beton yapılar için inceleme ve kontrol teknikleri daha fazla önem kazanmıştır. Buna rağmen, denetimin uygulanması, özellikle büyük ölçekli beton yapılarda zordur. Öte yandan, dokunulmaz hasar yerleri ve onarım için gereken çok büyük miktarda işçilik ve maliyet gibi mevcut şartlar nedeniyle onarım yapmak mümkün olmayabilir. Koşullara bağlı olarak, çimento esaslı malzemelerdeki kendi kendini iyileştirme daha fazla önem kazanıyor.

Bu çalışmanın amacı, çeşitli pozzolanik bağlayıcı türlerinin çimentolu kompozit malzemelerin iyileştirme karakteristiği üzerindeki etkinliğini araştırmaktır. Karışımlarda kendi kendini iyileştirme araştırması, basınç dayanımı, doğrudan çekme, silindir ayırma ve çatlama analizi test yöntemleri ile yapılmıştır. Su / bağlayıcı ve puzolan / çimento oranının kendi kendini iyileştirme kabiliyeti üzerindeki etkisi de belirlenmiştir.

Anahtar Kelimeler: Kendi kendini iyileştirme, çatlak genişliği, silindir, pozzolan, elyaf, çimento bazlı malzeme

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CHAPTER ONE
INTRODUCTION

Concrete can easily be exposed to external factors such as stress under unconditional climate temperature during service life. Concrete shrinks and expands with fluctuations in moisture and temperature, which leads to the formation of cracks. Other factors that can affect concrete and its lifecycle include shrinkage, design mistakes, poor quality of construction material and man-made flaws (Mather, 1989). When cracks originate in concrete structures, a sequence of serious events begins to occur within those structures (Wu, Johannesson & Geiker, 2012). Not only do these cracks affect the functionality of the structure, but they also affect the durability and strength of the structure. The deterioration of concrete as a result of crack plays an important role in the serviceability limit. However, if it were possible to know the reason for the differing behavior of concrete structures in similar circumstances, it would be an important key for designing highly durable concrete structures (Breugel, 2007). Thus, to increase the service life of concrete structures, inspection and maintenance techniques for concrete structures have therefore become the focus of increasing attention. Nevertheless, the implementation of continuous inspections and maintenance is very difficult, especially in the case of large-scale structures such as infrastructures, owing to the considerable amount of labor and funds that are required. In some conditions, repair is much more difficult to be performed and, in some cases, even infeasible, because of substantial conditions such as when the exact location of damage in an affected structure (Wu, Johannesson & Geiker, 2012). Recently, numerous engineers from all across the world have reported the high costs of repairment and annual maintenance for concrete structures, for instance, the cost of reconstructing a bridge in the USA has been estimated at between 20 to 200 billion dollars. Additionally, the annual maintenance cost is estimated at 5.2 billion dollars (Breugel, 2007). Consequently self-healing cement-based materials become important in the market.

1.1 Aim and Scope

The purpose of this study is to assess the capacity of cementitious materials in self-healing and analysis the effect of pozzolan additives in cement base composite to determine the autogenous healing ability. The uniaxial direct tensile test method was used to pre-cracked samples up to acceptable crack wide. Respectively before and after expose samples to the conditioning recovery. Ultrasonic velocity (UPV) test was performed

1.2 Literature Review

This chapter summarizes the researches that have been done about self-healing in cementitious composites.

Many researchers have noticed self-healing or autogenous healing of cracks in fractured concretes (Wu, Johannesson & Geiker, 2012). The self-healing phenomena were also studied at the end of the nineteenth century (Hyde & Smith, 1998). Already at that time, a distinction was made between self-healing and self-sealing. In the first case, the original strength of concrete is completely recovered, while in the second case, leaking cracks will be closed but strength recovery does not happen. Jacobsen & Sellevold, (1996) studied self-healing in concrete specimens that had been exposed to freeze-thaw cycles. Hearn & Edvardsen, (1999) observed autogenous healing of leaking of cracks.

Schlangen, (2010) and Huang et al., (2015) base on significant experiment work done, it is believed that self-healing of cracks in cementitious material is a combination of chemical and physical processes. A schematic description the several possible causes of self-healing phenomena (see Figure 1.2).

Self-healing is an old phenomenon in concrete. Due to earlier damage which may cause cracks in concrete, the hydration of clinker and other minerals, or due to carbonation of calcium hydroxide, some cracks may be healed after a while. In 1994 C. Dry was the first who recommend the self-healing properties of concrete in the world. Figure 1.1 shows the history of self-healing research and the importance of this phenomena. Tittelboom & Belie, (2013) have mentioned that Malinskii, Ivanova, Kargin, & Prokopenka, (1970) first used the self-healing properties of polymeric material.

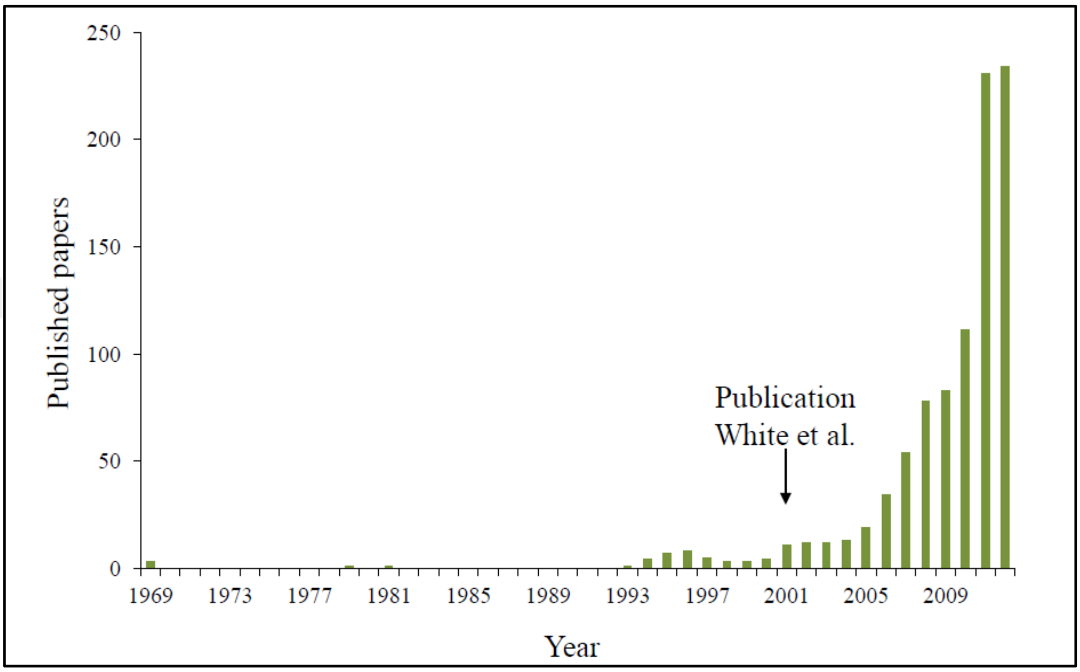


Figure 1.1 mentions the history of self-healing research and the importance of this phenomena between 1969 and 2012 (Tittelboom & Belie, 2013)

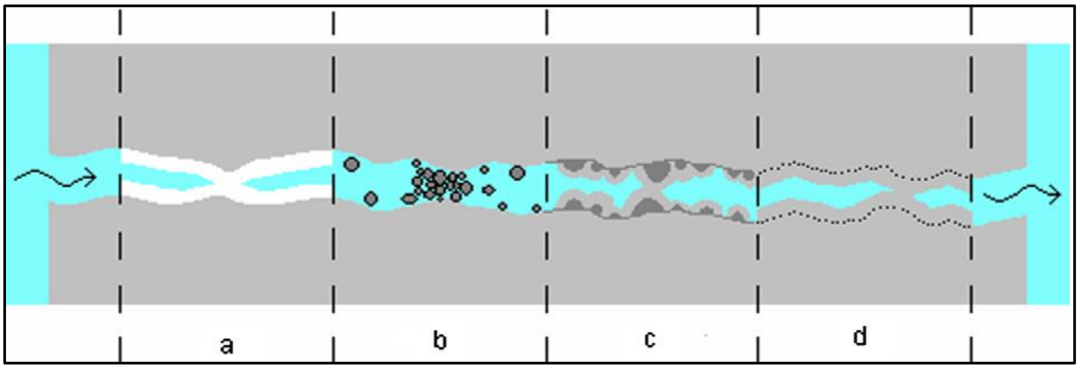


Figure 1.2 Feasible mechanisms for self-healing in cementitious materials (Heide, 2005)

In Figure 1.2, (Heide, 2005) describes the self-healing Steps:

(a) Formation of calcium hydroxide or calcium carbonate
(b) Possibility of blocking cracks by particles in impure water
(c) The reaction of unhydrate cement or cementitious materials
(d) The expansion of hydrated cement due to swelling of C-S-H

In the study of healed materials, Edvardsen, (1999) has noticed calcium carbonate ($CaCO_3$) in cracks after autogenous healing. Other research Yang et al., (2009) and Qien et al., (2009) also approved the existence of $CaCO_3$ in cracks. According to Edvardsen, (1999) research, after CO_2 dissolved in water, CO_3^{-2} ions released and diffused into the crack, the concentration of Ca^{+2} in concrete made the reaction is shown in Figure 1.3. However, Sisomphon, (2012), has mentioned the high value of CO_3^{-2} ions nearby the crack mouth (see Figure 1.3). As a result, primarily calcite is formed nearby the crack mouth. As self-healing proceeds, more CO_3^{-2} ions reach inside the crack. The composed portlandite inside the cracks will be carbonated and will lead to more calcite formation.

Palin, Wiktor & Jonkers, (2015) showed the self-healing capacity of cement base mortar specimens submerge in freshwater and sea. As it shows in Figure 1.4, the ordinary Portland cement-based mortar samples submerge in seawater has the maximum healing capacity. They underlined that the ions such as carbonate, magnesium, and calcium in seawater can sediment as minerals. (Palin et al., 2015) have stated that these settlements give cementitious materials in seawater a significant potential of healing compared to freshwater.

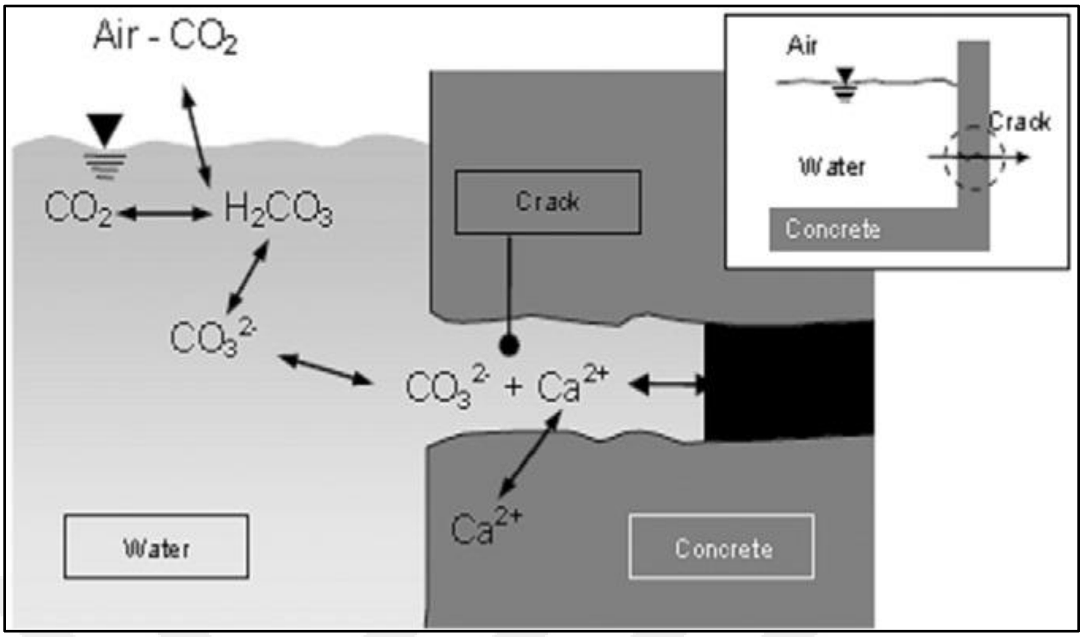


Figure 1.3 Self-healing due to reaction of calcium ions with dissolve ions of CO2 in water, (Mihashi & Nishiwaki, 2012)

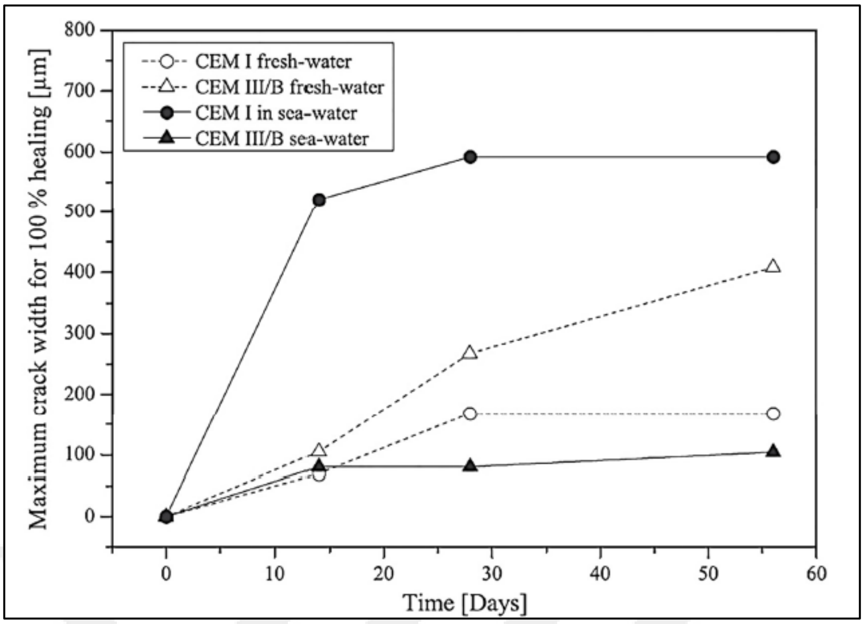


Figure 1.4 Self- healing capacity of cement-based mortar immersed in seawater (Palin et al., 2015)

Acccording to (Edvardsen, 1999) and (Neville, 1995), the chemical reaction process could be described as follows:

$$
\begin{array}{ll}
(1)\ H_2O + CO_2 \longleftrightarrow H_2CO_3 \longleftrightarrow H^+ + HCO_3^- \longleftrightarrow 2H^+ + CO_3^{-2} & \\
(2)\ Ca^{+2} + HCO_3^- \longleftrightarrow CaCO_3 \quad (pH_{water} > 8) & \\
(3)\ Ca^{+2} + HCO_3^- \longleftrightarrow CaCO_3 + H^+ \quad (7.5 < pH_{water}) &
\end{array}
\tag{1.1}
$$

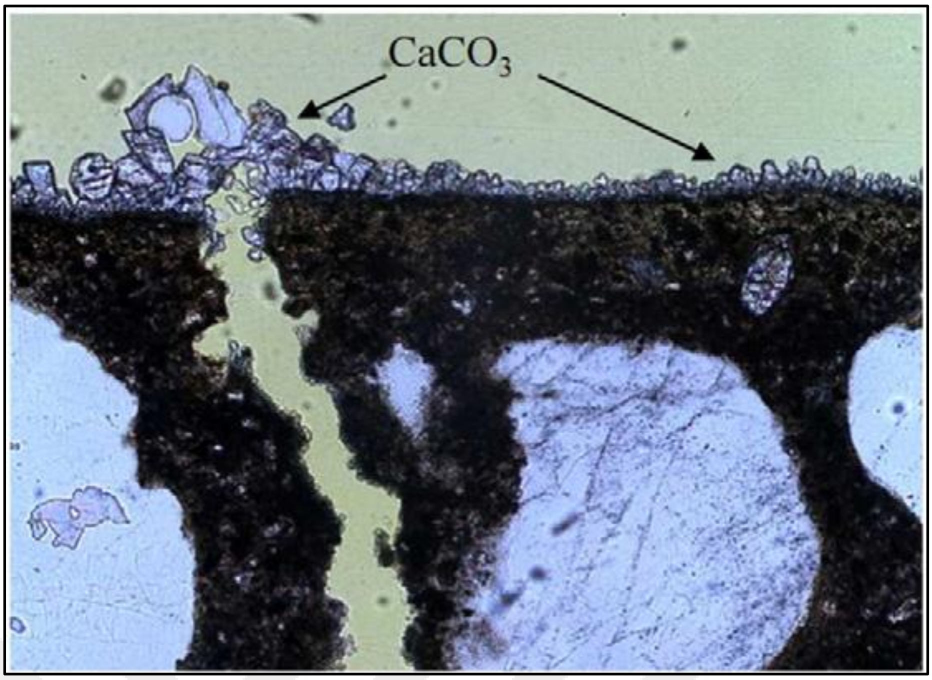


Figure 1.5 Experimental proof of calcium carbonate precipitation nearby cracking mouth, (Neville, 1995)

1.2.1 Healing due to SAP agents

Sisomphon & Copuroglu, (2011) have discussed the self-healing in different cementitious materials, by addition of crystalline additive and calcium-sulfoaluminate based expansive additive, the rapid self-healing was observed and is presented in Figure 1.4. The significant part of this experience is the crack range between 50 and 400 microns. The Figure 1.7 shows the effect of expansive cementitious material in crack healing in rush time.

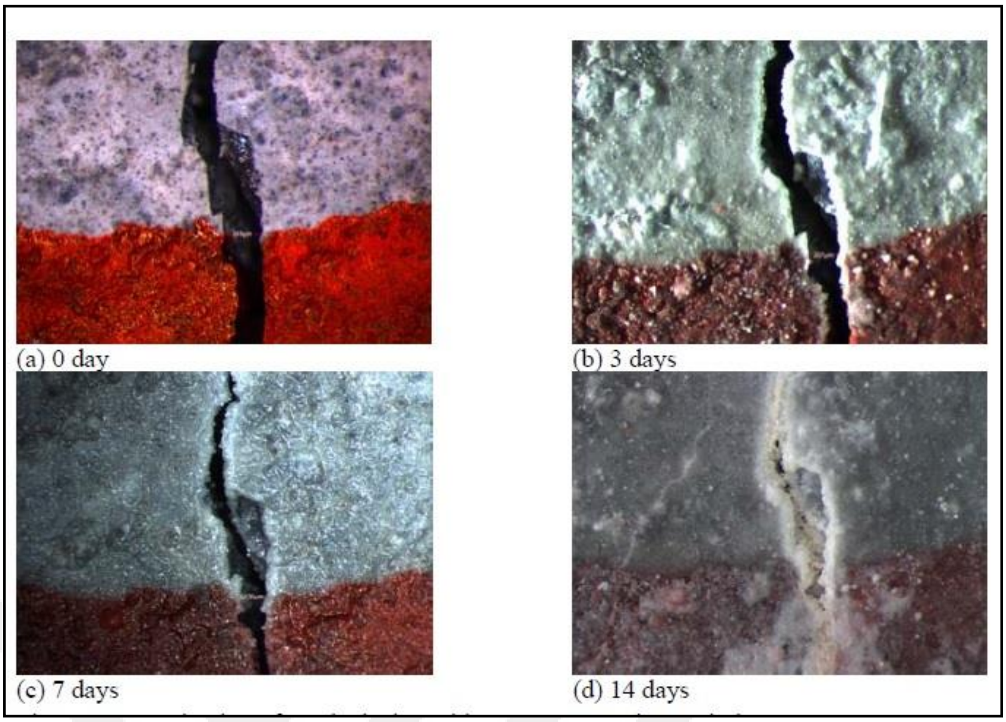


Figure 1.6 Crack closing due to expansion material in early time (Sisomphon & Copuroglu, 2011)

The expansion material improve the self-healing properties of mixtures by minimizing micro crack between matrix and aggregate transition zone.

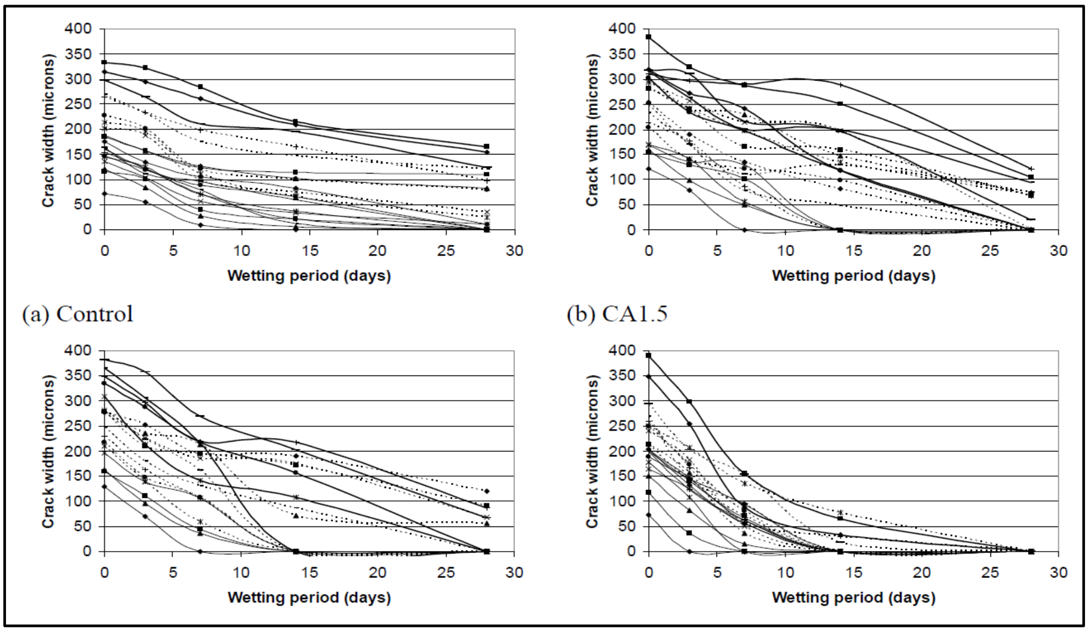


Figure 1.7 Show the effect of expansive cementitious material in crack healing in earlier time

Huang et al., (2014) have evaluated the effect of blast furnace slag on cementitious materials. The micro-crack healing was induced due to calcium hydroxide curing. The relation between concentration of calcium hydroxide and time in self-healing compare to filling fraction of cracks was shown in Figure 1.8.

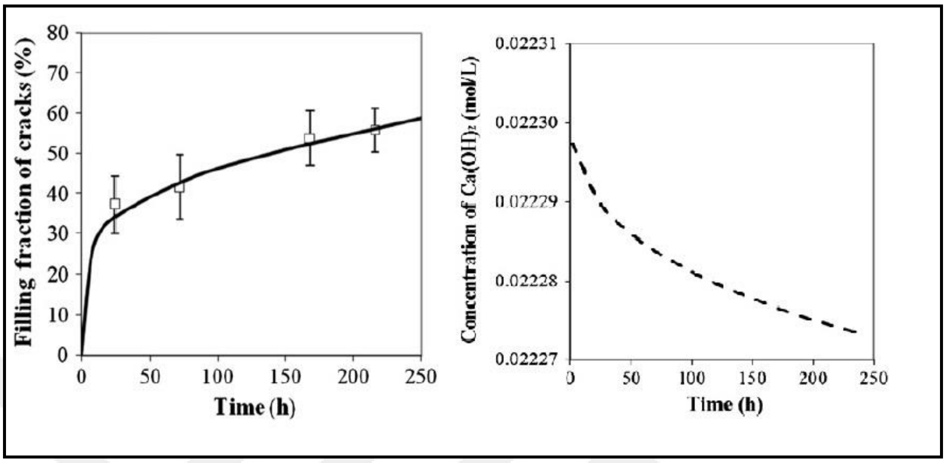


Figure 1.8 The relation between concentration of calcium hydroxide and time in self-healing compare to filling fraction of cracks (Huang et al., 2014)

The self-healing of micro-cracks in cementitious paste that cured in calcium hydroxide solution, upward up to 50h fast. Whereas after 50 h the healing kinetics rapidly decrease. As it shows in Figure 1.9, the major healing products formed in gaps are C-S-H. (Huang et al., 2014) focused on the reaction products, as it shown in Figure 1.9, the products are mainly formed as C-S-H, ettringite, and hydrotalcite.

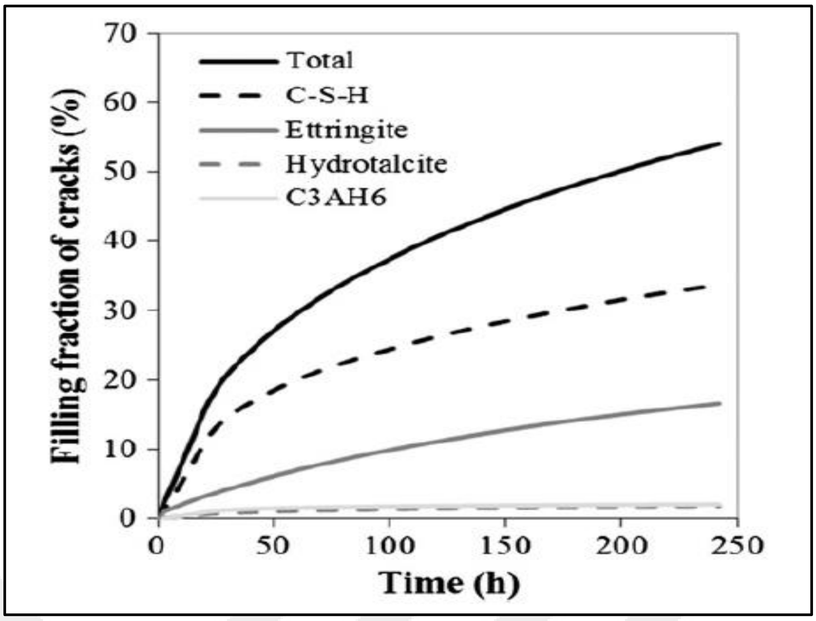


Figure 1.9 The Healing products that form in cracks (Huang et al., 2014)

Super absorbent polymer (SAP) is used as a new method for self- healing of cementitious material, whereas the efficiency of SAP on self-healing compare to disadvantage of this type of material has been mentioned in several studies. For instance the swelling and shrink of SAPs on different environmental condition cause severe damage, in one of the study the shrinking and swelling of SAP has exhibited high decrease on mechanical properties. Hu et al., (2019) has mentioned on high reduction of mechanical properties of concrete by adding SAP in cement. As a result, they prepared super absorbent CaAlg hydrogel, the benefit of this superabsorbent material was the restriction of swelling CaAlg in mixing process. The result of restriction was depend to high concertation of Ca^{2+} at mixing time (Hu et al., 2019).

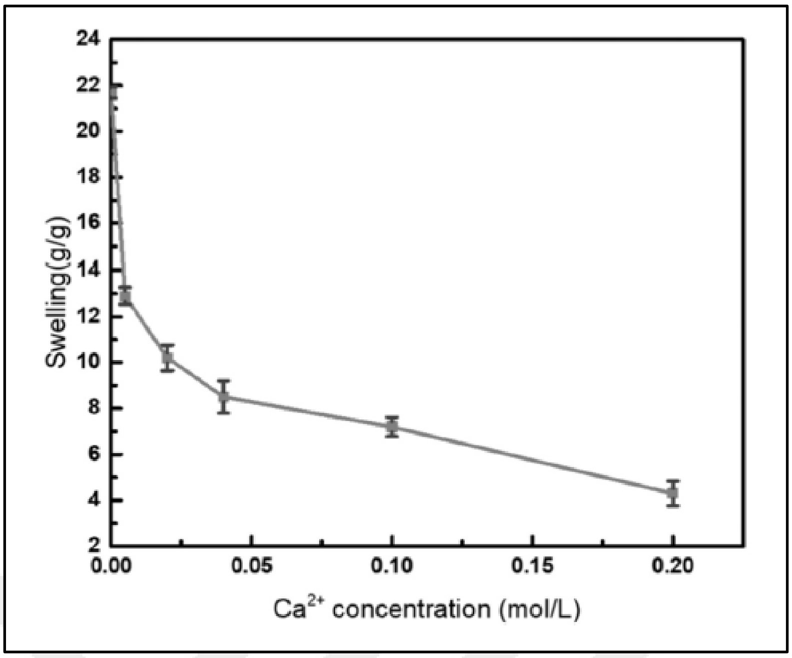


Figure 1.10 The Swelling restriction and capacity of CaAlg depend on Ca^{2+} concentration (Hu, 2019)

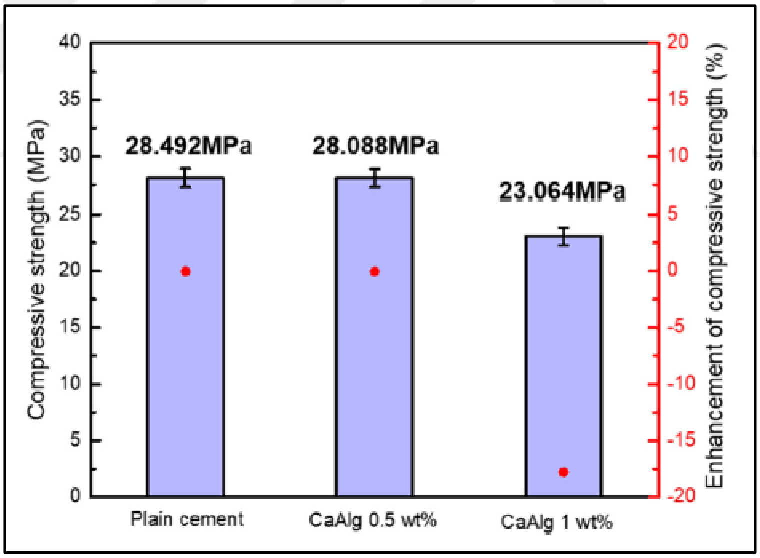


Figure 1.11 The reduction of compressive strength belongs to superabsorbent agents (Hu, 2019)

(Park & Choi, 2018), have investigated and evaluated the self-healing performance of cementitious materials by water absorption test method. In their study the have used research cement (RC), calcium sulfo-aluminate (CSA), Anhydrate and ground granulated blast-furnace slag (GGBFS). B. Park and Y. C. Choi are mentioned on irregular crack in concrete specimens that self-healing cannot obtain. As a result, in their study used the artificial crack method Haoliang et al., (2014) to obtain self-healing products. (Park & Choi, 2018) prepared slices specimens with dimension of 100×100×10 mm. Then the slice surface was polished to make a uniform surface. Finally, the surfaces were attached and fix with constant gap of 100μm (Haoliang et al., 2014; Park & Choi, 2018). See Figure 1.12.

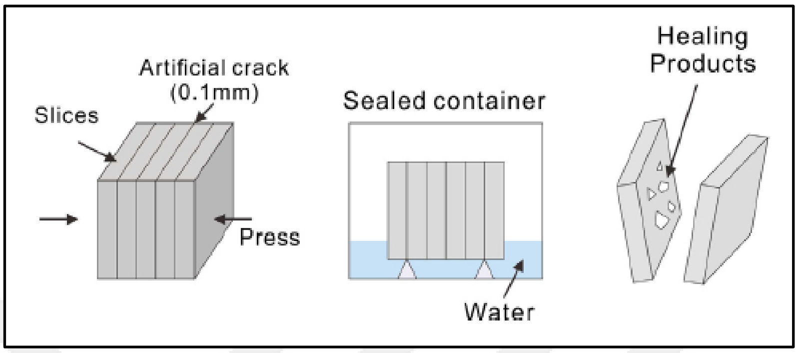


Figure 1.12 Self-healing products evaluating test method (Haoliang et al., 2014)

The rate of water absorption increased in crack more than uncracked specimens (Belleghem et al., 2016), the contact of water with crack surface made water absorbed into the cracks. The absorption ratio in first time was measured higher. After healing time the water absorption ratio began to decreased, the decreasing of the absorption ratio exhibit the healing of cracks. As it mentions on Figure 1.13 the blue area shows the amount of water immersed into the specimens from crack and increase the total mass of samples. The differential of crack without healing and self-healed cracked shows the healing ratio which Belleghem et al., (2016) were mentioned. Furthermore, calcite had a high effect on healing than other healing products.

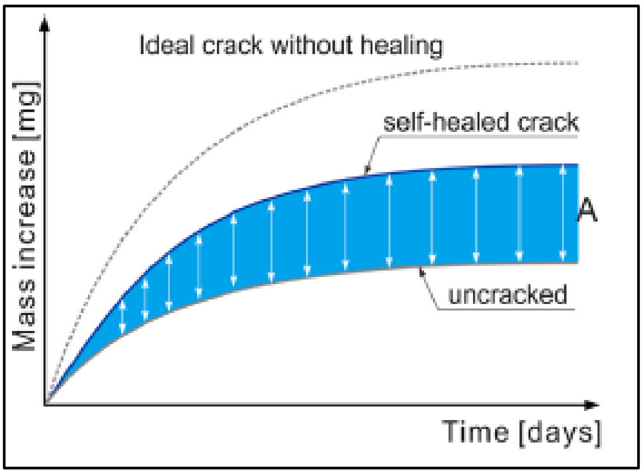


Figure 1.13 The evaluating of self-healing due to the mass increase of specimens through water absorption (Belleghem et al., 2016)

The adding of SAP agents has a significant effect on rapid self-healing in cementitious materials. Many researched has exhibited the rapid swelling of SAP that cause healing and closing cracks in early ages. Hong & Choi, (2017) were investigated the rapid healing of cracks due to SAP agents. They used water absorption test method for evaluating the rapid self-healing with contributing about 0.5% and 1.0% of SAP in cement binder. The result shows the cut off water absorption per unit time, respectively, 52% and 72% (Hong & Choi 2017). The water flow test was performed with novel method as it was presented at Figure 1.14 the amount of water on the surface of cracked specimens was fixed at 20mm. the amount of water passing through the crack was weighed with digital and sensitive resolution device and the water flow was measured depending on the timeout. The result shows the potential of rapid healing of SAP cementitious specimens due to rapid swelling in just 13 minutes.

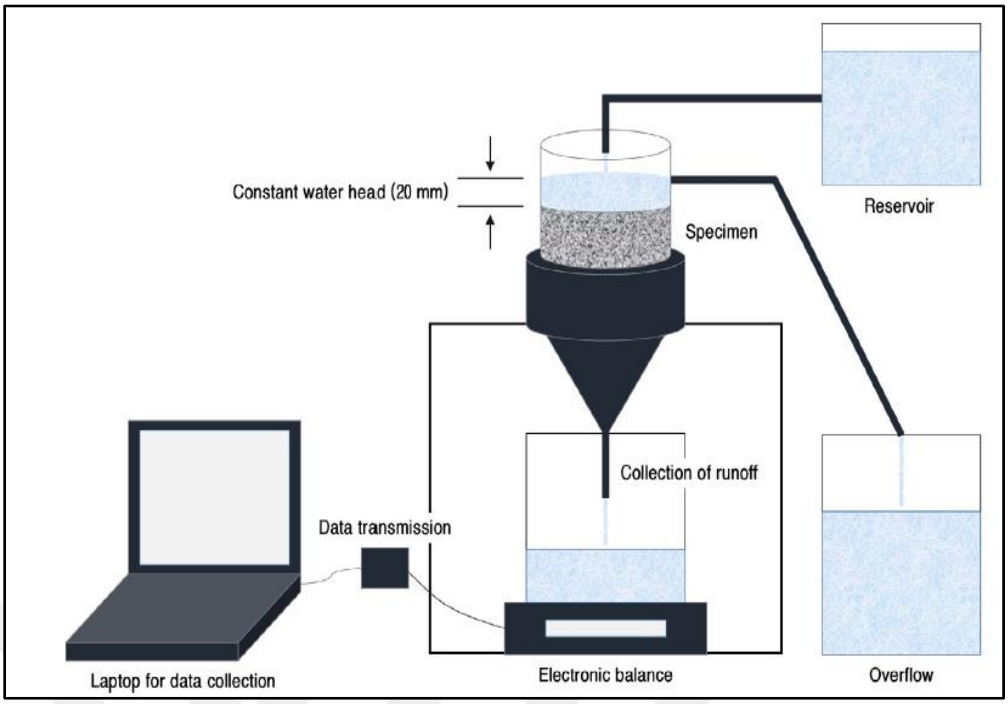


Figure 1.14 The novel method of water absorption for evaluating self-healing (Haoliang et al., 2014)

Many hydraulic structures are at high risk of damage due to cracking that are formed by environmental elements. Liu et al., (2017) were investigated the self-healing behavior of engineered cementitious composites (ECC) under mechanical loading. As they are mentioned in their researched, the penetrating of sulfate-chloride through cracks cause permanent damage on hydraulic structures. As they investigated, the mechanical properties of ECC under aggressive solution was not observed. Moreover, the formation of gypsum and ettringite due to sulfate ions are improved the healing process (Liu et al., 2017). Sahmaran & Li, (2008) were investigated that ECC can be healed under freezing and thawing cycles due to de-icing salt. Furthermore, Sahmaran and Victor Li (Sahmaran & Li, 2008; Li & V.C. Li, 2011) were proved that the cracked and pre-cracked ECC has a significant durability under chloride and alkaline environment.

1.2.2 Crack Restriction

Many researches mentioned on crack restriction, Li et al., (1998) recommended the use of fibers in engineering cementitious composite can limit the crack width. This recommend mention on the healing, in other word multiple micro cracks compare to single large crack have a significant healing. Li, V.C and coworkers in their research used polyethylene fibers, since later used poly vinyl alcohol, (Li, Wu & Wang, 2002; Li, V.C et al., 2011).

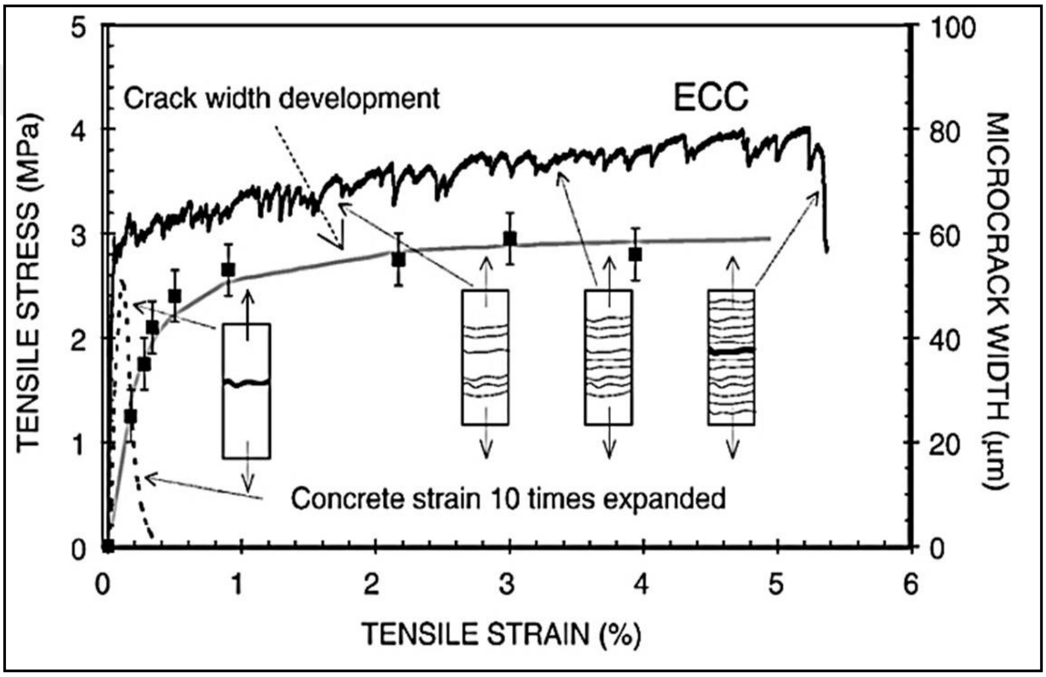


Figure 1.15 The curve of tensile stress compares to formations of cracks for cracks restriction

Beglarigale & Yazıcı, (2015) Fiber-matrix adherence is one of the most important factors affecting the performance of cement-based fibrous composites. Many parameters such as fiber type and geometry, matrix strength, fiber-matrix interface properties and curing conditions affect fiber-matrix adherence. Within the scope of the study, mixtures having various water / binder ratios were designed and fiber-matrix adherences were examined by pull-out experiment by applying different curing methods. The striking adherence increases after autoclave cure could be explained by internal structure analysis. In addition, the importance of micro fibers ratio has been examined extensively (see Figure 1.16).

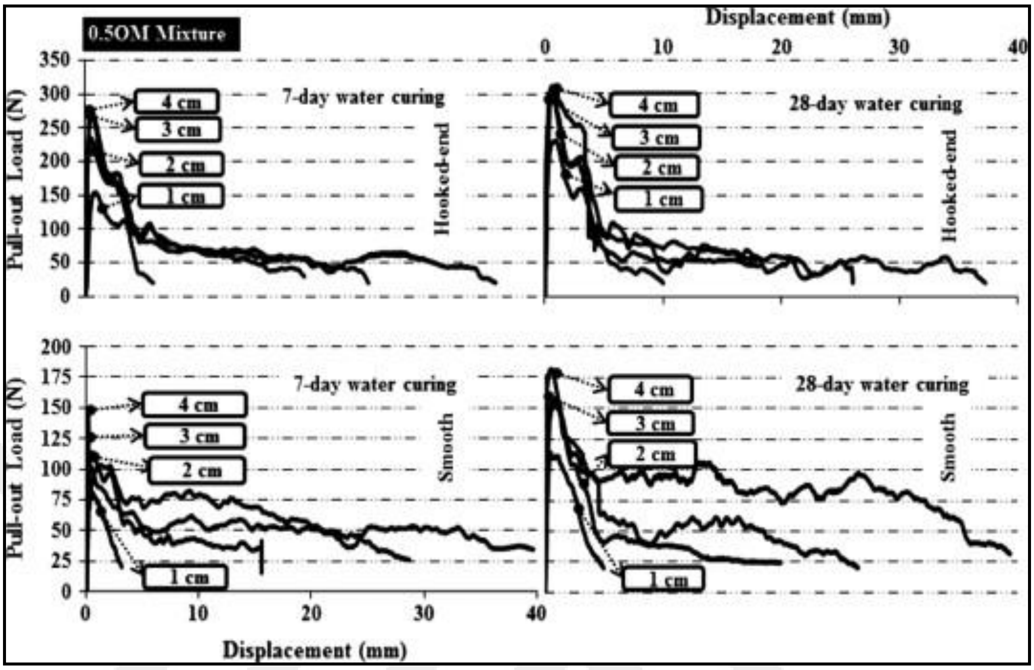


Figure 1.16 The pull-out test for measuring the adherence of fiber-matrix (Beglarigale & Yazıcı, 2015)

Beglarigale & Yazıcı, (2015) as mention on the adherence of fiber and cement base matrix the necessity of microfiber to restrict the crack width was became important. Kua et al., (2019) have research the effect of biochar- immobilized bacteria and superabsorbent polymers (SAP) on healing cracks on concrete surface. Two type of fiber were used as a crack restrictions respectively PVA and steel fibers, the main reason of this study was to found out which type of healing agent have best performance on damage concrete, whereas the Figure 1.17 shows the area where the high crack closing is happened in cracks with less than 300 micron.

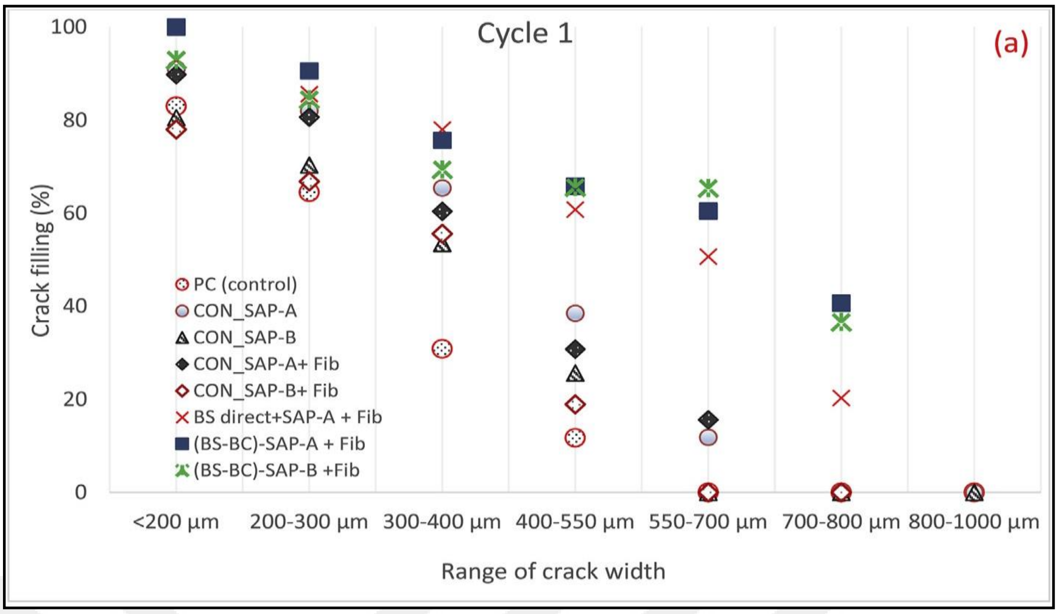


Figure 1.17 Crack filling of different material with different cracks width (Kua et al., 2019)

Kua et al., (2019) have also focused on the SAP (water supply agent) which can absorb a disproportional amount of liquid. Lee et al., (2010) also called SAP as hydrogel, which means, into the cementitious material, SAP role as water supplier. This SAP particle in cementitious base concrete structure is being dormant during service life, depend on unlikely service life condition, concrete can be cracked, whereas the crack made a bridge to moisture and fluid that ingress in the crack and made SAP be swelled and storage for dry period to release the absorbed liquid (see Figure 1.18).

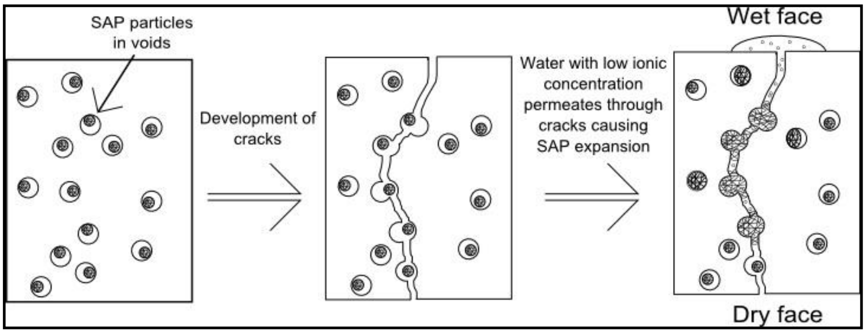


Figure 1.18 Illustrated the work mechanism of SAP (Lee et al., 2010)

CHAPTER TWO
MATERIALS AND METHODS

2.1 Traditional Mortar

In this section, the traditional mortar mixture and material used are described. Furthermore, the test methods used in this study to evaluate the self-healing properties of the mixture, are explained.

2.2 Material Used in Traditional Mortar

2.2.1 Supplementary Cementitious Materials (SCM) and Cement

CEM I 42.5 R Portland cement was supplied from Denizli cement. Cement properties are presented in Table 2.1. Two different class types of fly ashes are provided. The class F and C of fly ashes are supplied from thermal power planet of Soma and İzenerji and are called, respectively, FA1 and FA2. Furthermore, a ground granulated blast furnace slag (GGBFS) supplied from Iskenderun- Turkey iron and steel factory. The Chemical properties of supplementary cementations materials presented in Table 2.2.

Table 2.1 Chemical and Physical properties of cement

Chemical Properties and Composion (%)		**Physical Properties**	
SiO_2 (%)	19.1	Specific Surface (Blaine) (m^2/kg)	369
Al_2O_3 (%)	4.4	Initial setting time (min)	110
Fe_2O_3 (%)	3.96	Final setting time (min)	166
CaO (%)	61.85	Volume expansion (mm)	1.00
MgO (%)	2.05		
Na_2O (%)	0.27		
K_2O (%)	0.7	**Compressive Strength**	
SO_3 (%)	3.72	2 days (MPa)	27.1
Cl^- (%)	0.0004	7 days (MPa)	43.3
Loss on Ignition	1,82	28 days (MPa)	56

Table 2.2 Properties of Supplementary Cementitious materials

Chemical Composition (%)	**GGBFS**	**FA1**	**FA2**
SiO_2 (%)	39.95	41.96	48.95
Al_2O_3 (%)	11.46	19.55	28.68
Fe_2O_3 (%)	0.54	4.38	3.61
CaO (%)	34.07	26.18	8.44
MgO (%)	6.45	2.11	1.81
Na_2O (%)	0.3	0.38	0.54
K_2O (%)	1.02	1.46	1.06
SO_3 (%)	0.55	1.27	0.2
Cl^- (%)	0.0246	0.0181	0.0289
Loss on Ignition	2.95	1.69	2.93
Free CaO	0.13	1.66	1.01

2.2.2 Steel Fibers

The micro steel fibers used are brass coated steel fibers with a length of 6 mm and 13 mm. Some properties of the fibers used in the study are presented in Table 2.3. The images of the fibers used are presented in Figure 2.1.

Table 2.3 Properties of steel fibers

Fiber Type	**Diameter (mm)**	**Length (mm)**	**Aspect Ratio (L/d)**	**Specific Density gr/cm^3**	**Tensile Strength (MPa)**
F1	0.2	13	65	7.17	2750
F2	0.16	6	37.5	7.17	2000

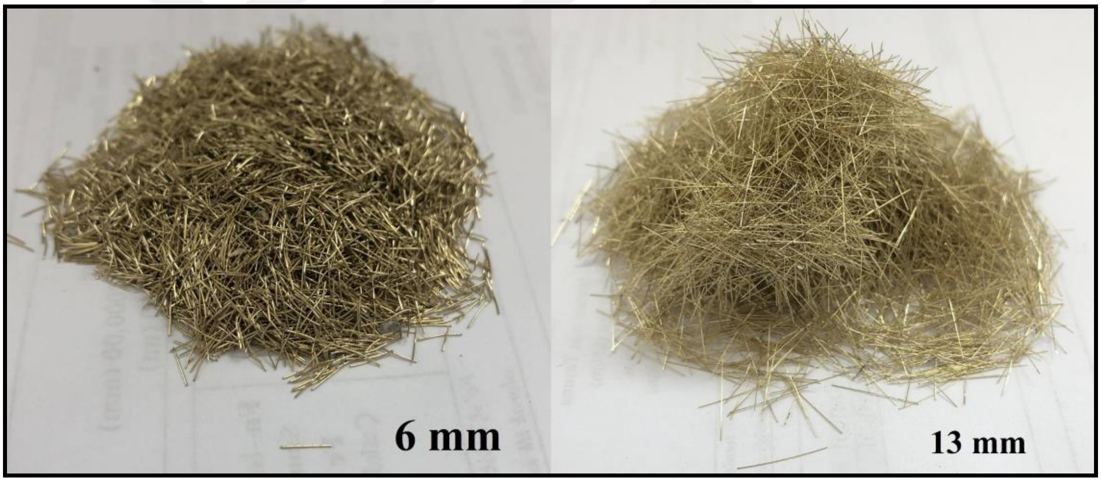


Figure 2.1 The micro-fibers used in this study (Personal archive, 2016)

2.2.3 Aggregate

During experimental study, crushed limestone with maximum grain diameter of 4 mm aggregate was used.

2.2.4 Chemical Admixture

A polycarboxylate ether polymer base superplasticizer with high range water reduction was used, the properties of admixture are presented in Table 2.4.

Table 2.4 Properties of superplasticizer

Properties	Explanation
Form	Liquid
Structure of mineral	Policarboxylic Ether Based
Color	Amber
Density	1.082- 1.142 kg/liter
Chloride Content (%)	<0.1
Alkaline Content (%)	<3

2.3 Mix design

2.3.1 Determination of the proper superplasticizer ratio of mixture.

In this study, traditional mortar mixture with 500 kg in cubic meter was used and two type of water-cement ratio was investigated. Before starting to batch the mixtures, the superplasticizer ratio for each mix was determined with using 2.0-liter laboratory mixer. As the ratio was determined, the dry ingredients of each mixture were weighted and premixed about 2 minutes for the homogenous mix. Then half of the mixing water was added to the mixture. Then the superplasticizer was added to the remaining water and was stirred. After stirring, the whole water was poured into the mixture and was mixed with a stable speed for about 5 minutes. The mixing process was implemented up to the intended workability that was controlled by mini-flow test (mini-slump) accordance to ASTM C1437 with 100 mm sub-diameter.

2.4 Compressive Strength Test

The traditional mortar mixture was cast into the 50×50×50 mm cubic molds. The compressive strength of specimens in 7 days and 28 days treatments were determined. Moreover, this test method was applied to determine the mechanical properties of two type of water-cement ratio mixtures that may have self-healing ability.

As the proper mixture was determined, this test method was used and applied on 18 cubic specimens that were prepared for each variable mixture for three different water curing treatment periods; 7 days, 28 days and 90 days. Six of the eighteen specimens of the 7-day treatment were pressed to measure their average maximum load. Then nine remaining specimens were pre-loaded up to the maximum load that was predetermined. In addition, loading was carefully applied without any further damage. Then two of the pre-loaded specimens were used to check for signs of damage ratio by being subjected to load. After the procedure was finished the eighteen samples were taken into the water as called healing period. After 30 days of healing period, all of the samples were loaded to determine the compressive strength for evaluating self-healing ratio. The same process was applied to the 28-day and 90-day treatments. Note that, the position of specimens in the aforementioned test should be arranged the same as pre-load time. (See Figure 2.2)

Figure 2.2 Mentioned that test must be applied on the same direction of specimens before and after healing (Personal archive, 2017)

2.5 Direct Tension Strength Test

The tension strength test method was carried out with a loading rate of 0.2 mm / min and used to evaluate the stress-strain curve of traditional mortar mixtures. Next, this test method was applied to the specimens that were cast as a shape like a dog bone, which is presented in Figure 2.3 for measuring and analyzing the performance of micro-fibers on crack width restriction of mixtures.

To evaluate the self-healing capacity of traditional mortar, twelve specimens were cast and cured in water for three different treatment periods; 7 days, 28 days and 90 days. Then all of the samples in each treatment were taken out of the water and were left to dry for one day. The samples in each treatment were categorized into 3 test groups (A, B and C). Three specimens in group A were used as control and were left in the water curing throughout the experiment. To obtain the stress-strain curve, three specimens in group B were subjected to a tensile strength test. The six remaining samples in group C were subjected to the tensile test until the 100μm ±10 width crack appeared. Then the pre-cracked specimens (group B) were immersed into the water for curing period of healing for 30 days. After curing period was finished the nine samples (group A and C) were subjected to the tensile test for obtaining the stress-strain curve to observe self-healing. The same process was applied to the 28-day and 90-day treatments. The position of specimens in the aforementioned test should be arranged the same as pre-load time (See Figure 2.4).

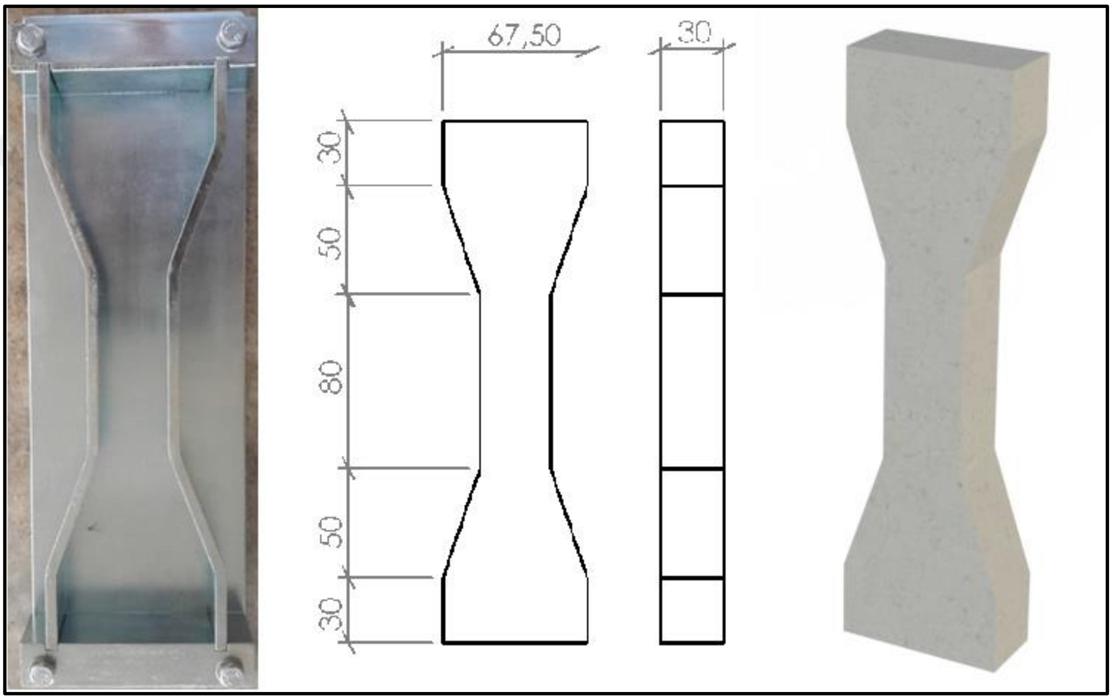


Figure 2.3 The detail of direct tensile test specimen (Personal archive, 2017)

Figure 2.4 Mentioned on the same direction of specimens before and after healing (Personal archive, 2017)

2.6 Crack Closing Test

The test was done to evaluate the self-healing capacity of the mixture containing supplementary cementitious materials. The fiber-reinforced traditional mortar and SCMs mixtures were cast into the 30 mm diameter and 10 mm height mold. Then Specimens were cured for 7 days, 28 days and 90 days treatment periods. Next, all of the samples were taken out of the curing water for being subjected to splitting in order to create crack with a wide range between 50 µm and 200 µm (See Figure 2.5). Then cracked specimens from each treatment were immersed into the water for 30 days as a healing period. The width and the crack area was measured by a digital optical microscope (See Figure 2.5), the measurement was applied three times. The first measurement was taken on the first day of splitting, then samples were immersed into the healing period up to 15 days for the second measurement. Afterwards, the same method was applied up to 30 days healing for the last measurement.

The self – healing capacity of this method was evaluated by using the following equation:

$$H = \frac{B-A}{B} \times 100 \qquad (2.1)$$

H: healing ratio percentage, A: the crack area after healing, B: the crack area before healing

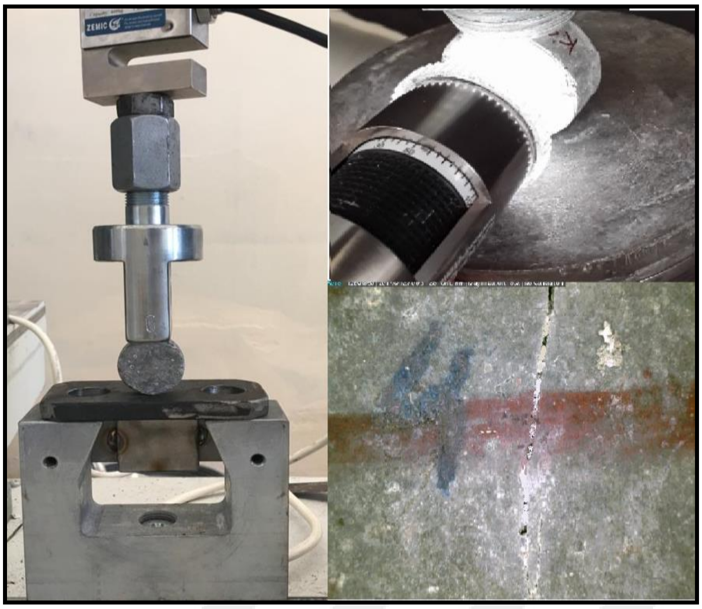


Figure 2.5 Crack Closing test method detail (Personal archive, 2016)

2.7 Self-Bonding Test

This novel test was designed in TÜBİTAK project (No: 215M783) to assess the healing capacity of mixtures by evaluating the bonding load that forms in splitting surfaces. This method used the same mechanism of direct tensile test. The high bonding load shows the high healing that was formed in surface. In this study, these methods were applied to the SCMs mixtures.

In this study, cylinder molds with 100 mm diameter, 100 mm height with a triangle bump in the middle of the inner surface (See Figure 2.6-f) were used. The bump in the mold was used as a splitting line. Two heads were designed to be mounted on the tensile strength measurement device for splitting specimens (See Figure 2.6-c). Furthermore, special pinholes designed at the top and bottom plates of the mold were mounted into the samples to be used as handles when pulling the samples apart for bond testing purposes (See Figure 2.6-b). The steel pins were chromium coated with 16 mm diameter and 75 mm length (See Figure 2.6-a).

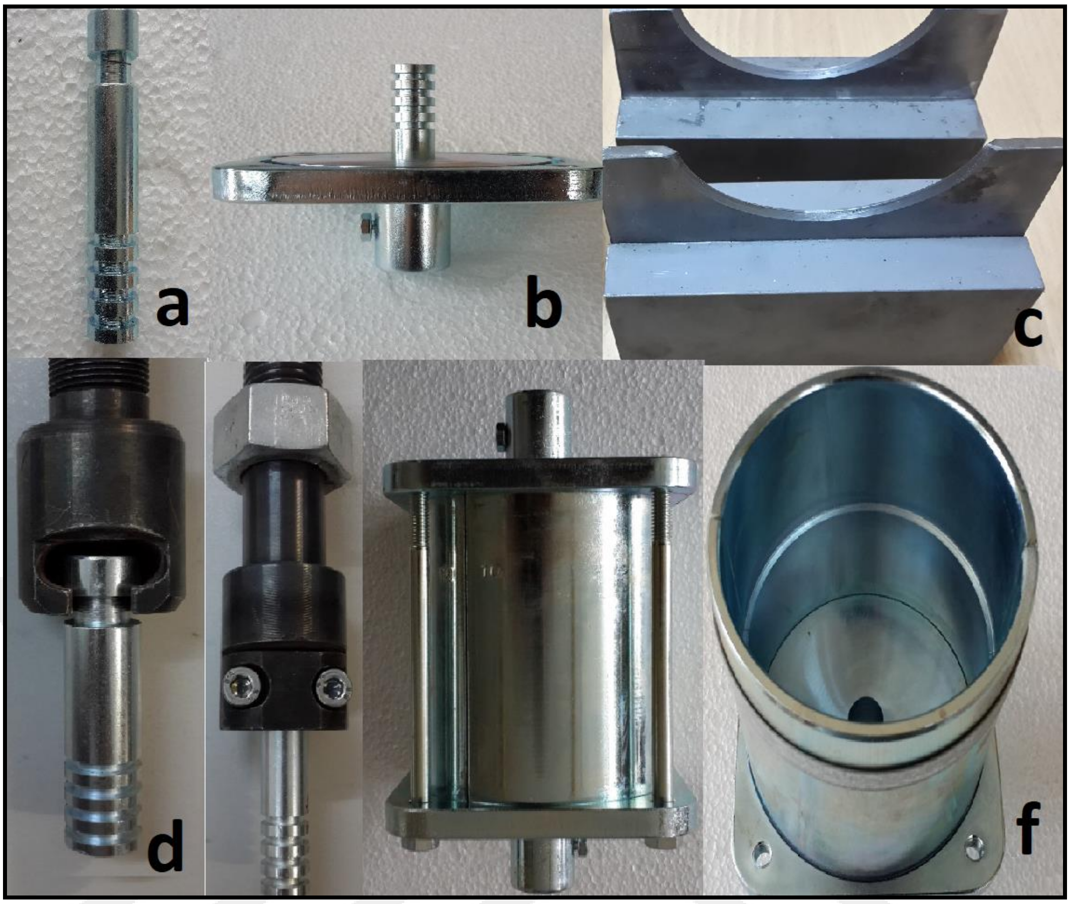


Figure 2.6 The mold details of bonding test method (Personal archive, 2016)

In this method, for each curing period, twelve's specimens were cast in a cylindrical shape. Note that, the necessity for numerous specimens is because of the precision purposes. Then specimens were cured in the water for 7 days, 28 days and 90 days as in the aforementioned study. Then all of the samples in each treatment were taken out of the water and next, the specimen were split (See Figure 2.7).

Figure 2.7 Shows the splitting steps (Personal archive, 2017)

After splitting steps, two parts of the specimen were precisely put together immediately. The attached apparatuses were used for connecting the splitting surfaces. For a homogenous test method, the torque wrench was set to 2.5 N.m and three bolts were tightened (See Figure 2.8), then samples were put into the water as healing period. (Beglarigale, Vahedi, Eyice ve Yazıcı, 2019)

Figure 2.8 Shows the attached apparatuses and bolts tightening (Tübitak, 2016)

After 30 days of healing period, the specimens were taken out of the water carefully, then the specimens were left to dry in a laboratory for 3 days. After samples were dried, the specimens were subjected to bonding tests (See Figure 2.9).

Figure 2.9 Self bonding test (Personal archive 2016)

CHAPTER THREE
RESULT AND DISCUSSION

A flowable mortar mixtures base on 500 kg / m3 cement dosage designed (See Table 3.1). The fresh and mechanical properties of two different fiberless mixtures studied. The optimum superplasticizer content for 220 mm free flow and a high compressive strength was selected as the main cementitious mix for this study. The two different mixtures were designed for considering the free flow value. The water-cement ratio of mixtures was adjusted to be 0.5 or 0.4. The workability of mixtures was measured and controlled by the mini flow test apparatus, the necessity amount of superplasticizer was determined.

3.1 Fresh properties and concrete design.

Table 3.1 Mortar mixture design

Mixtures	W/B	Cement	Water	Aggregate (0-5 mm)	SP
A	0.5	500	250	1481	2.5
B	0.4	500	200	1600	7

3.1.1 Compressive Strength Test Results

The compressive strength test is result about the average of six cubic samples (See 1.1). The compressive strength of mixture B at 7 and 28 days is higher than mixture A. As expected the differences in compressive strength belong to the water-cement ratio, was 20% (Figure 3.1).

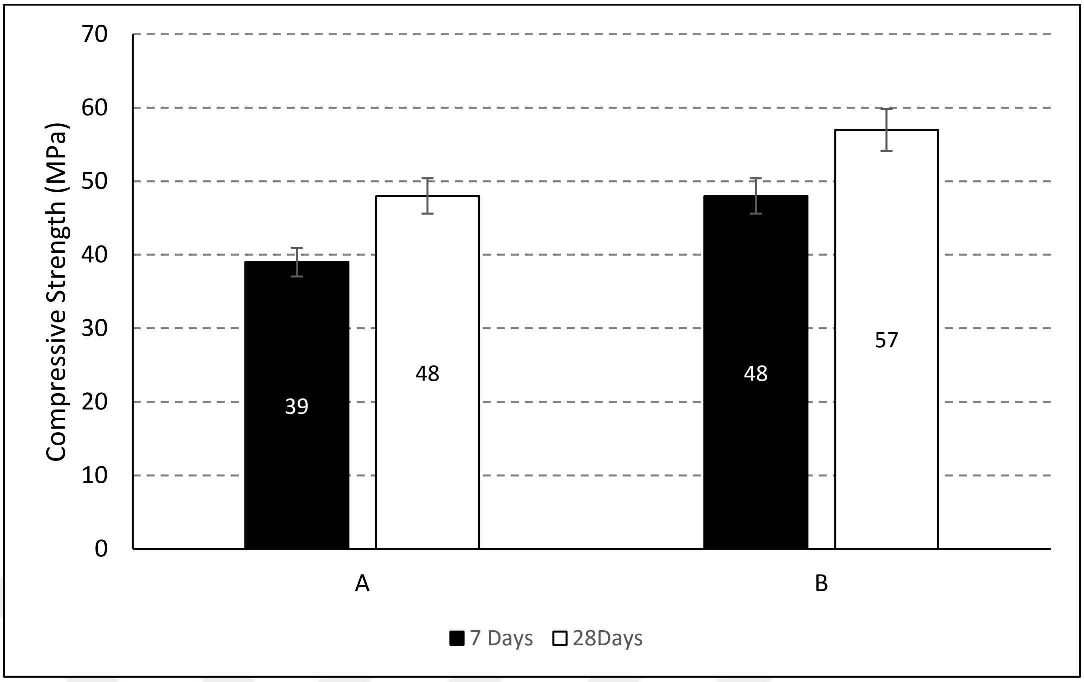


Figure 3.1 Compressive strength test result

3.1.2 Direct Tensile Test Results

The average of six tensile test specimens results show in Figure 3.2. The mixture B direct tensile strength is slightly higher than the mixture A. As we expected, superplasticizer has significant effect on the workability and consistency of mixture B. By considering the fresh properties, the mixture B was selected as the main mixture for this study.

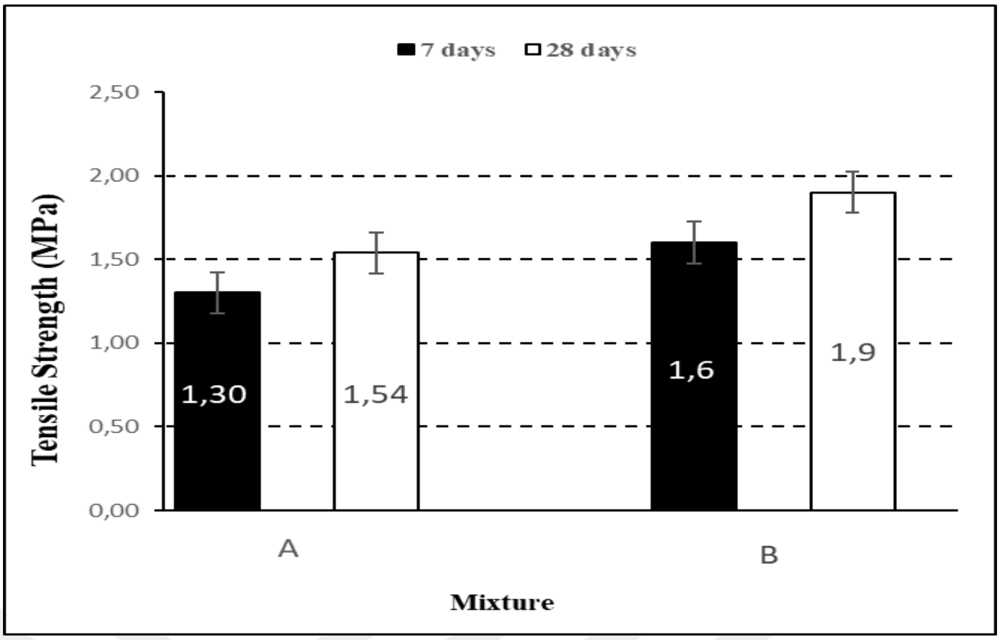


Figure 3.2 Direct tensile test result

3.2 Determined the Crack Restriction and Steel Fiber Ratio

The mixture B reinforced with two different kind of steel fibers for analyzing the restriction capacity of cracks.

3.2.1 Mixture Design, Mechanical and Fresh Properties

The mixture B were reinforced by two different fibers with 13 mm and 6 mm that named as F1 and F2, respectively. The mixtures with 0.5, 1.0, 1.5 and 2.0 volume percent of fibers were analyzed (Table 3.2).

Table 3.2 Mix design of fiber reinforced B mixture (kg/m3)

Mixtures	Fiber	Cement	Water	Aggregate (0-5 mm)	SP	Free Flow (mm)	
						F1	F2
B-0.0	-	500	200	1600	7	220	220
B-0.5	39	500	200	1585	6	217	200
B-1.0	78	500	200	1573	5	180	200
B-1.5	114	500	200	1560	5	155	190
B-2.0	152	500	200	1547	6	-	165

As a noticed, micro fiber with length of 6 mm (F2) versus micro fiber with 13 mm (F1) did not affect the ratio of superplasticizer very much as shown in Figure 3.3. While the F1 fiber greatly decrease the consistency and workability of mixture by using volume of 2% fiber as shown in Figure 3.3. Additionally, increasing of superplasticizer dosage did not improve the consistence and workability of this mixture. On the other hand with use of micro fibers at the volume fraction of 1%, 1.5% and 2%, a free flow value of 220 mm could not be achieved despite the high superplasticizer dosage. Furthermore, as can be seen the use of F1 fibers at a volume fraction of 2% has effected the flow of fresh mixture. Consequently, for this study the free flow of 155 mm was mentioned.

Figure 3.3 Consistency and workability of mixture (Personal archive, 2016)

3.2.2 Compressive Strength Result

The compressive strength test is result about the average of six cubic 50×50×50 mm samples. The compressive strength test was carried out on 7 days and 28 days water cure and the results shown in Figure 3.4. As the results shows, the addition of micro-fiber increased compressive strength. The compressive strength result of F1 and F2 micro-fibers are approximately close. Taking into consideration the plain mixture has a 7-day and 28-day compressive strength 48 MPa and 57 MPa respectively. The fraction of compressive strength improvement between BF-2.0 and BF-0.0 for 7-day and 28-day water curing is approximately 12%.

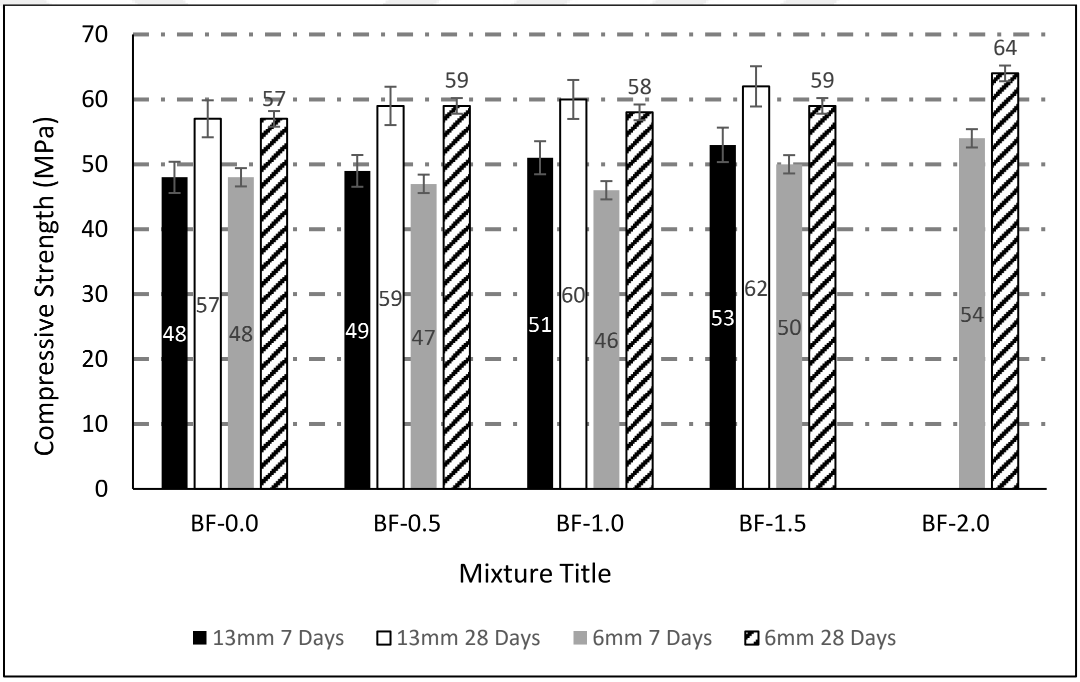


Figure 3.4 Compressive strength of 7 days water curing of BF mixtures

3.2.3 Direct Tensile Strength Test Result

Direct tensile test were performed by using two LVTD as shown in Figure 3.5 for determined the effect of micro-fibers used with different ratio on tensile strength and to find out, which has best performance in crack width restriction.

As it explained in section two, the average direct tensile strength of six specimens for each mixture were evaluated and presented in Figure 3.6. The maximum direct tensile strength with incorporation of F1 and F2 was for mixture BF-1.5 with corporation of F1 micro-fiber. As can be seen the mixture that containing 1.5% F1 fiber shows a best performance compare to other mixtures. Table 3.3 determined the crack width in direct tensile test.

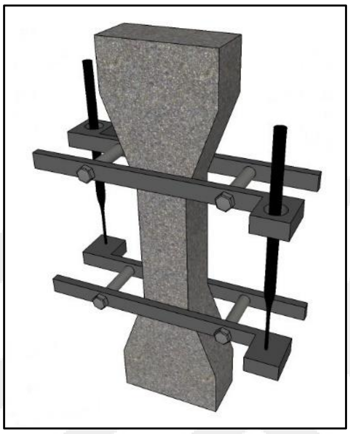


Figure 3.5 The Direct tensile test schematic

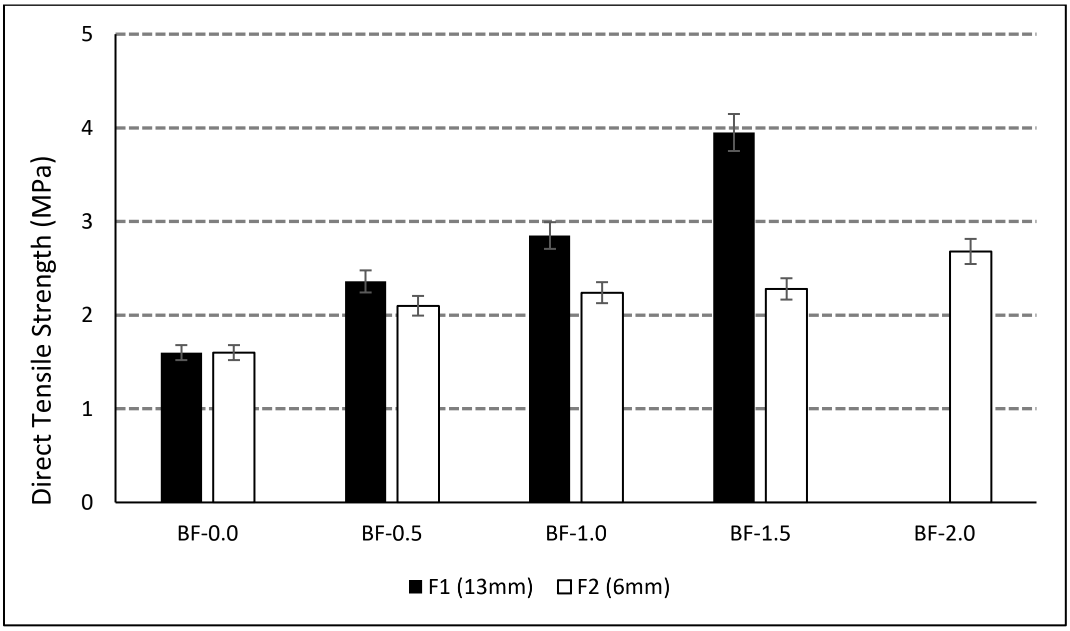


Figure 3.6 Direct tensile Strength with two different micro fiber and various volume

Table 3.3 Effect of fiber in crack width

Specimens	Fiber type (mm)	Fiber volume	Maximum Load (N)	Cracks Width Average (mm)	Minimum Cracks Width (mm)	Maxsimum Cracks Width (mm)
1	13	0.5%	987	0.707	0.184	1.324
2	13	0.5%	2239	0.113	0.036	0.159
3	13	0.5%	2004	0.037	0.022	0.048
1	6	0.5%	1988	0.062	0.014	0.119
3	6	0.5%	1799	0.037	0.007	0.064
1	13	1%	2080	0.298	0.056	1.197
2	13	1%	3059	0.069	0.02	0.113
3	13	1%	1692	0.012	0.007	0.019
1	6	1%	2939	0.054	0.017	0.097
2	6	1%	1715	0.077	0.047	0.116
3	6	1%	2319	0.066	0.037	0.123
1	13	1.5%	3170	0.034	0.005	0.072
2	13	1.5%	2614	0.371	0.072	0.754
3	13	1.5%	3943	0.036	0.018	0.068
1	6	1.5%	1932	0.05	0.023	0.083
2	6	1.5%	1689	0.023	0.009	0.05
3	6	1.5%	2174	0.036	0.009	0.091
1	6	2%	1623	0.019	0.006	0.03
2	6	2%	1291	0.1	0.025	0.196
3	6	2%	2413	0.06	0.014	0.113

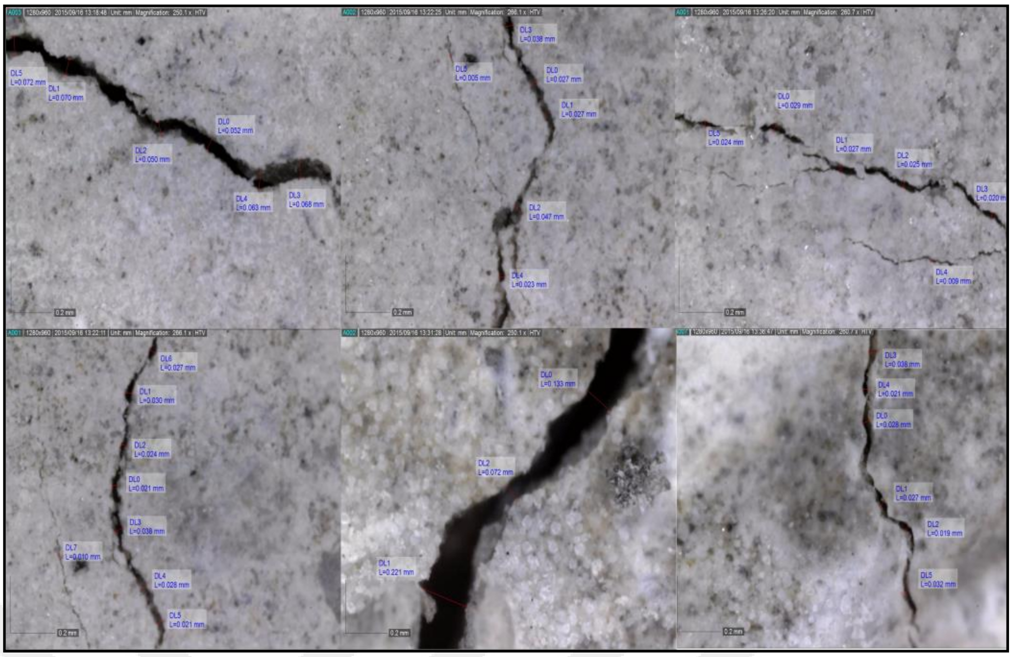


Figure 3.7 The effect of micro-fiber in gaps width restriction was mentioned

3.2.4 Discussion

The Effect of micro fibers were determined in this study. In this test method the load was conducted until the first crack was illustrated. Then the cracks width were measured by microscope. As it shown in Figure 3.7 the 13mm micro-fiber with 1.5% volume fraction has significant effect on crack restriction. As it seem in Table 3.3 the average crack width was 0.036 mm. The volume of discontinuous fibers has significantly affected the mechanical properties of mixture, the fiber –matrix bond transfer stress between fiber and matrix, in addition mechanical properties of component significantly improved. Beglarigale & Yazıcı, (2015) have discussed that the fiber-matrix bond improves by increasing volume of fibers.

As a result, the plain mixture was selected by determining the effect of water cement ratio, compressive strength, direct tensile strength and crack width restriction. Moreover the mixture B with F1-1.5 was selected as the self-healing research plain mixture.

3.3 Self-Healing Evaluation Tests

As previously noted, to improve the self-healing mechanism, the binder dosage of the selected mixture was increased to 750 kg / m3 by the adding of supplementary additional cementitious materials (SCM). The SCM materials were divided into two part, the first part was two kind of fly Ash (FA) and the last part was a ground granulated blast furnace slag (GGBFS). The self-healing capacity of seven mixtures was investigated by tests method that was told in chapter three as well as a crack healing test (See 2.7) This new test methods was referred by (Beglarigale, Vahedi & Yazıcı, 2018) to assess the healing capacity of products that form in very tight cracks, this method was called as soon as a self-bonding method. The self-healing capacity of cementitious material depend on high bonding capacity of binders. In addition, the compressive strength results of mixtures were assessed at 7 days and 28 days after discussion, for next step of study two SCM mixture were selected.

3.3.1 Mixture design

One of the binders used in the mixtures, GGBS was obtained from Iskenderun iron factory. In addition, FA was used in two different classes; the class C and F were obtaining from Thermal power plants of Soma (FA1) and Izenerji (FA2). The mixtures were divided into two groups except the Control mix, half of amount of cement as SCM binder was added to the mixture. As the second group, the amount of cement was reduced by half and the amount of SCM was increased by the amount of cement in the first group. It must be mentioned that for compressive strength, direct tensile strength and cylinder split test methods are prepared by using 13mm micro-fiber, whereas, the crack closing test method with small round shaped specimens were prepared with 6mm micro-fiber. It must be noted that the crack closing test method was carried out with two percent of 6mm micro fiber (F2). The differences of using the 6mm micro fiber is belong to the small size of crack closing test molds. More over the F2 micro fibers can easily spreads homogeneously into the mold. The cylinder specimens were casted with selected mixture in previous part for testing the self-bonding.

The mixtures are shown in Table 3.4, in the first group as called as GA, the cement amount contains 500 kg/m^3 and 250 kg/m^3 of SCM, in the second group as called as GB, the cement amount contain 250 kg/m^3 and 500 kg/m^3 of SCM. The superplasticizer amount for The self-healing capacity of seven mixture were investigated by compressive, direct tension, bonding and crack closing tests. Therefore, the performance of aforementioned methods were analyzed to evaluate self-healing. The workability of mixtures was measured and controlled by the mini flow test apparatus, the necessity amount of superplasticizer for free flow of 155mm was determined and show in Table 3.4.

Table 3.4 SCMs Mixture design

Mixtures	Fiber (F1)	Cement	SCM		Water	Aggregate (0-5 mm)	SP
			FA	GGBFS			
Control	114	500	-	-	200	1560	5
FA1-GA	114	500	250	-	300	1041	1.45
FA1-GB	114	250	500	-	300	990	1
FA2-GA	114	500	250	-	300	1041	1.85
FA2-GB	114	250	500	-	300	990	1.32
GGBFS-GA	114	500	-	250	300	1076	1.55
GGBFS-GB	114	250	-	500	300	1063	1.24

3.3.2 Compressive Strength

The compressive of SCM mixtures are shown in Figure 3.8. The 7 days compressive strength of second group mixture decreased compared to control mixture. Even thought, the cement amount of first group mixture had not reduced and the 7 days strength of SCMs mixtures decrease indistinctly. As expected, the rate of pozzolanic reaction of SCM during 7 days was lower, whereas the 28 days compressive strength of first group of SCMs are equal or higher than strength of control mixture. The 28 days is necessary for the reaction of siliceous or aluminous material (C-S-H) with

$Ca(OH)_2$ to produce and increased the compressive strength which is shown in Figure 3.8.

The 7 days compressive strength of second group are lower than control mixture since the amount of cement which was used in second group, was lower. The GGBS-GA 28-day compressive strength compare to GGBFS-GB, FA2-GB, FA2-GA and FA1-GB is higher since high pozzolanic reaction.

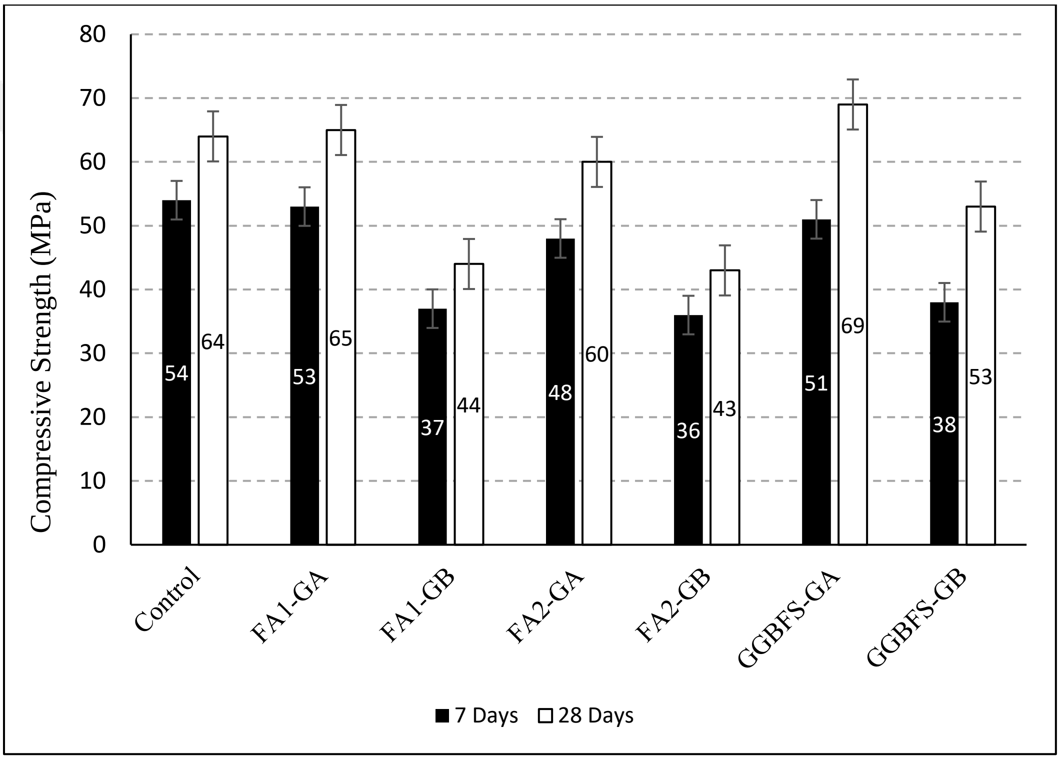


Figure 3.8 Compressive strength of SCMs mixture

The self-healing of seven mixture investigated by compressive test method was used and applied each variable mixture for three different water curing treatment periods; 7 days, 28 days and 90 days. Six of the eighteen specimens of the 7-day treatment were pressed to measure their average maximum load and named as original specimen which refers to specimens that are not pre-loaded. Immediately after preloading, half of the preloaded specimens were subjected to the compressive strength test to determine the damage ratio due to the preloading. Depending on the mixture type and the age of specimens, the damage were observed in specimens due to the preloading. Then nine remaining specimens were pre-loaded up to the maximum load that was predetermined and all the specimens immersed in the water. After 30 days of healing period, all of the samples were loaded to determine the compressive strength for evaluating self-healing ratio. Although the cement content of the carrier mixtures of SCMs was not reduced, their compressive strength of 7 days was slightly reduced (see test results of origin samples). It is well known that pozzolanic reactions of SCMs are limited for 7 days. The continuation of this section discusses the reduction in strength due to preload as well as the healing.

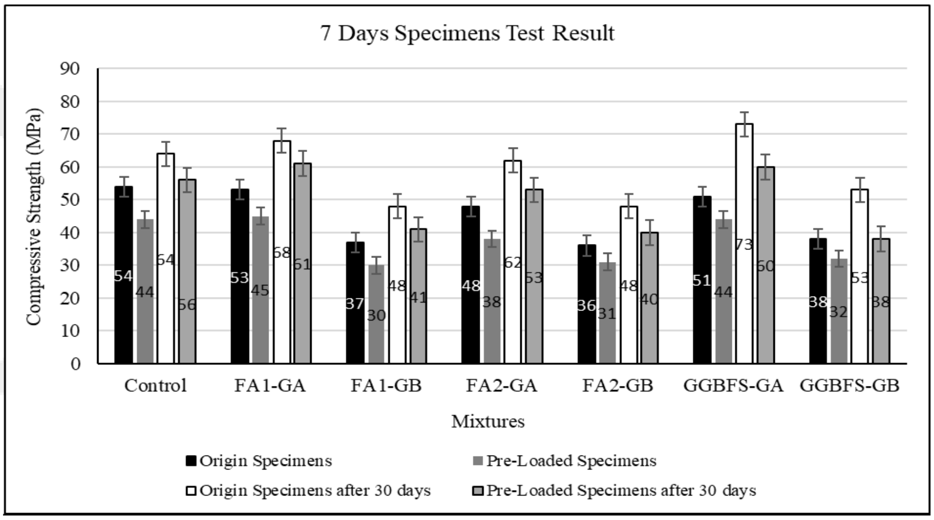


Figure 3.9 The compressive strength of 7-day of specimens before and after healing

The same process was applied to the 28-day and 90-day treatments the results are shown in Figure 3.10. The compressive strength of SCMs bearing mixtures are higher than the compressive strength of control mixture. The amount of C-S-H liberated by the pozzolanic reaction of SCMs with $Ca(OH)_2$ was enough as that it can enhance the 28-day and 90-day compressive strength of mixtures.

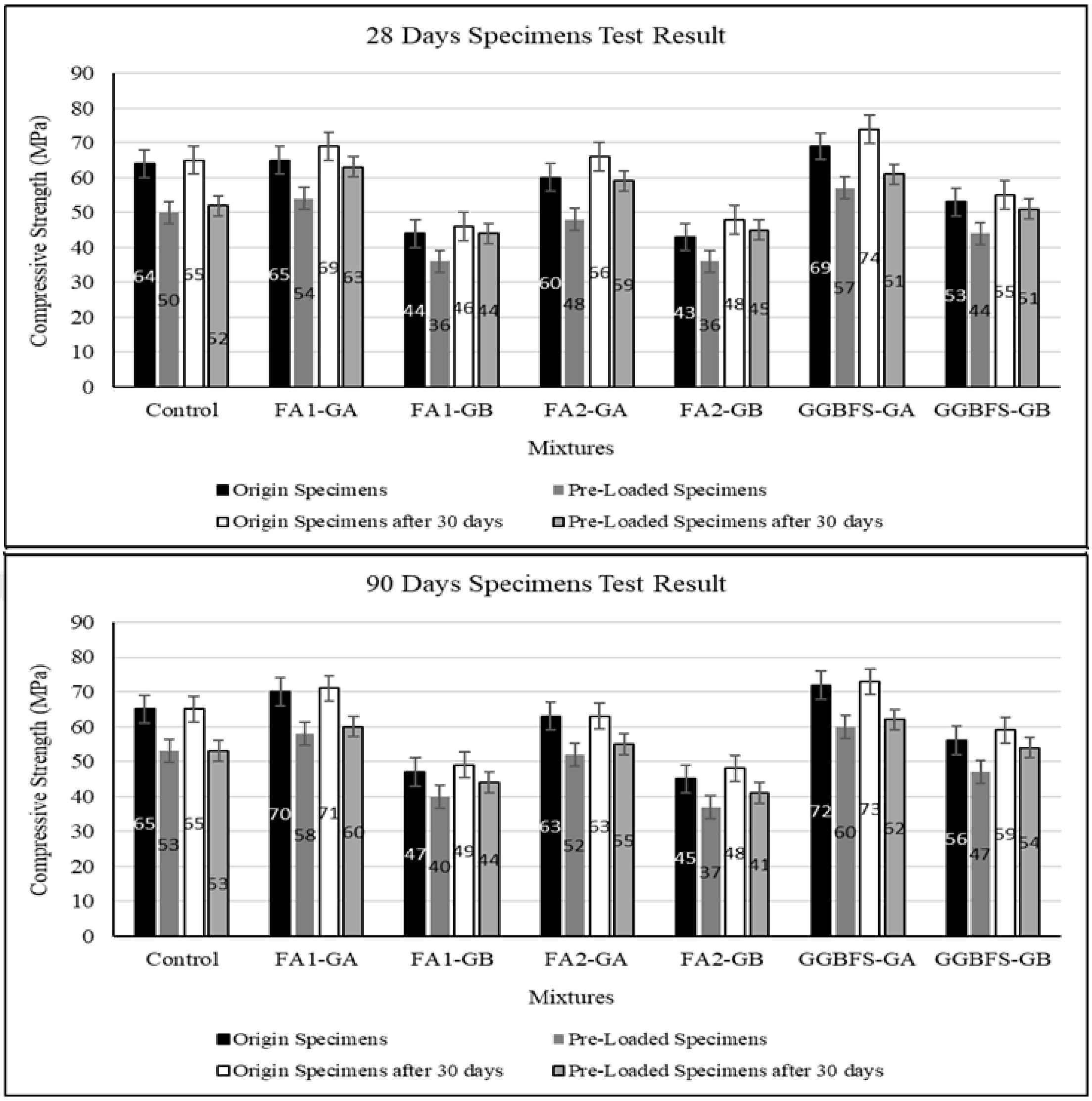


Figure 3.10 The compressive strength of 28-day and 90-day of specimens before and after healing

The compressive strength development of pre-loaded specimens and original specimens are presented at Figure 3.11 and Figure 3.12, respectively. When comparing the two figures it is detectible that original specimens, which were not pre-loaded, for example the control mixture has shown a 19% improvement in compressive strength over the 30 days healing time, however, the control mixture of pre-loaded specimens has demonstrated 27% improvement over the same curing period. Although, the result show that there is more healing in pre-loaded specimens, nevertheless, the realistic self- healing ratio of this improvement cannot be determined by simply subtracting the ratio. The percentage of regain compressive strength for first statement are express as below.

$$SR = \frac{a-b}{b} \times 100 \tag{3.1}$$

Where "SR" is the percentage of regain in strength, "a" and "b" are, respectively, the compressive strength of specimens after and before healing period (30 days). The average percentage of regain in strength are presented in Figure 3.11 and Figure 3.12. The healing ratio of FA2-GA (39%), FA1-GB (36%), FA1-GA (35%) and GGBFS-GA (36%) containing mixtures are higher than the control (27%) mixtures. This ratio were significantly decreased for 28-day and 90-day specimens. It must be noted that these healing ratio s are considerably depend on the specific damage level of each mixture.

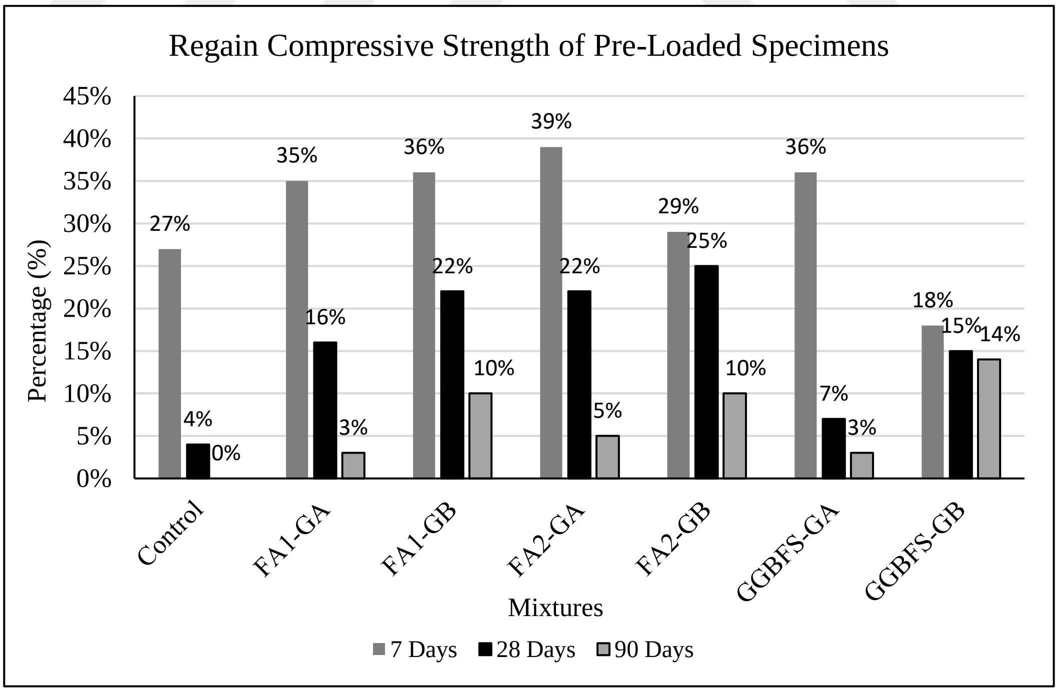


Figure 3.11 The pre-loaded specimens regain strength

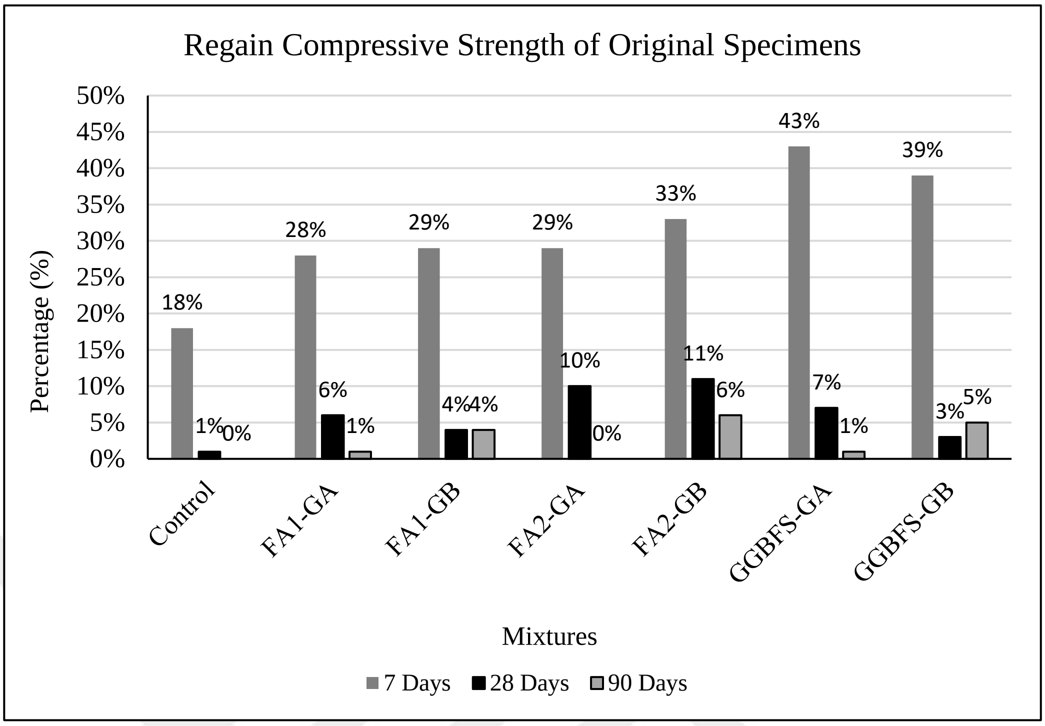


Figure 3.12 The original specimens regain strength

Second view of self-healing:

$$SR = \frac{\mathrm{d-b}}{\mathrm{c-b}} \times 100 \tag{3.2}$$

Where "SR" is the percentage of regain in strength, "d" and "b" are, respectively, the compressive strength of preloaded specimens after and before healing period (30 days), "c" the compressive strength of origin specimens after healing period.

The average percentage of regains in strength expressed with the second view are presented in Figure 3.13. As can be seen from Figure 3.13, a significant self-healing ratio was detected in the 7 days specimens of all mixtures. As expected, the self-healing ratio of FA2-GA (63%) and GGBFS-GA (66%) containing mixtures are higher than control (60%). whereas, in the 28 days specimens, the healing ratio increase significantly in pozzolanic mixtures. As can be expected, the healing ratio of control mixture decrease suddenly. As it shows in Figure 3.13 FA1-GA (60%), FA1-GB (80%), FA2-GA (61%), Fa2-GB (75%) and GGBFS-GB (63%) containing mixtures are higher than control (13%) and GGBFS-GA (24%). It must be noted that these healing ratios are considerably depend on the specific damage level of each mixture.

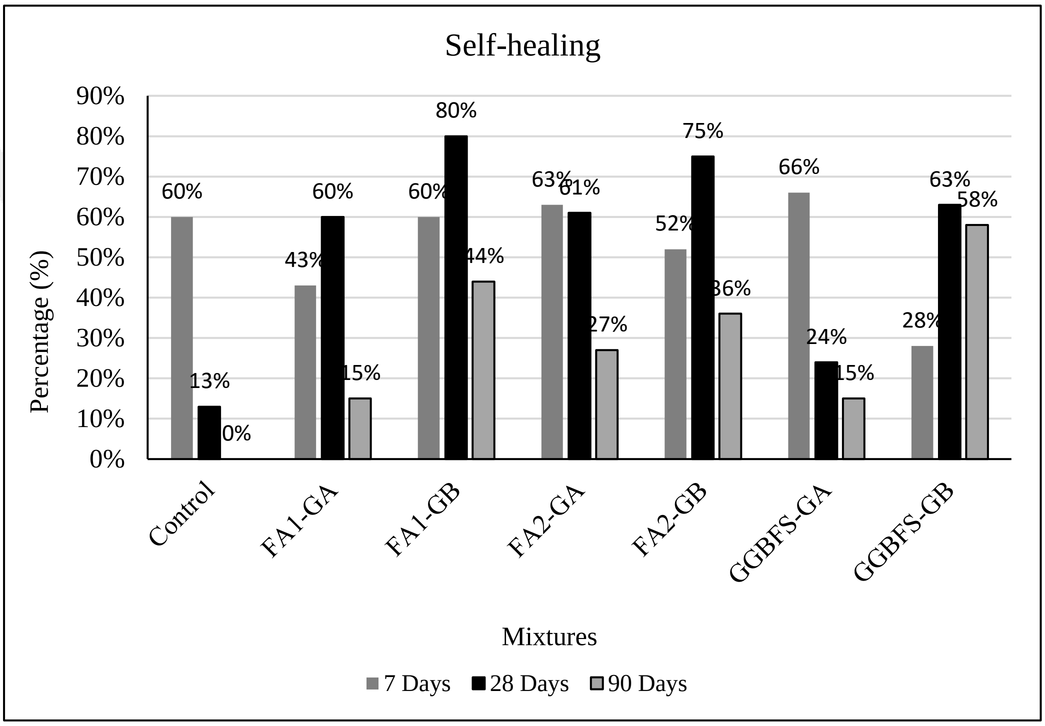


Figure 3.13 The self-healing calculating of 7-day, 28-day and 90-day of specimens

3.3.3 Bonding Test

In this part of study sixteen specimens were casted and been cured in water for 7 day, 28 days and 90 days. For each curing period specimens were split in two piece, then two piece were reattached fussily and been held in water as a healing period for 30 days. As can be seen the bond load due to bonding ability of healing products can be obtained in this novel test method. Huang, Ye and Damidot (2013) made an artificial crack in cement paste specimens to separate the healing products from the matrix. For doing this purpose, the specimens were cut in two pieces. Then the surface of each piece was sanded to achieve flat surfaces. Then the surfaces were reattached and healing for 200 hours. The microstructure analysis show that mainly healing product consist of calcium hydroxide (CH) crytals and calcium silicate hydrate gels (C-S-H) that can be formed by hydration of unhydrate cement particle in cracks. The self-bonding test result of 7 days cylinder specimens are shown in Figure 3.14. As expected, in the fracture surface of 7 days split specimens, the amount of unhydrated cement particle is higher, consequently the formation of healing products in fracture surface can be the main reason. Furthermore the bonding strength for FA1-GA compared to other mixtures is higher. The maximum load for detaching the attached surface was measured as 348 N. In other word the bonding strength was measured as 44 KPa.

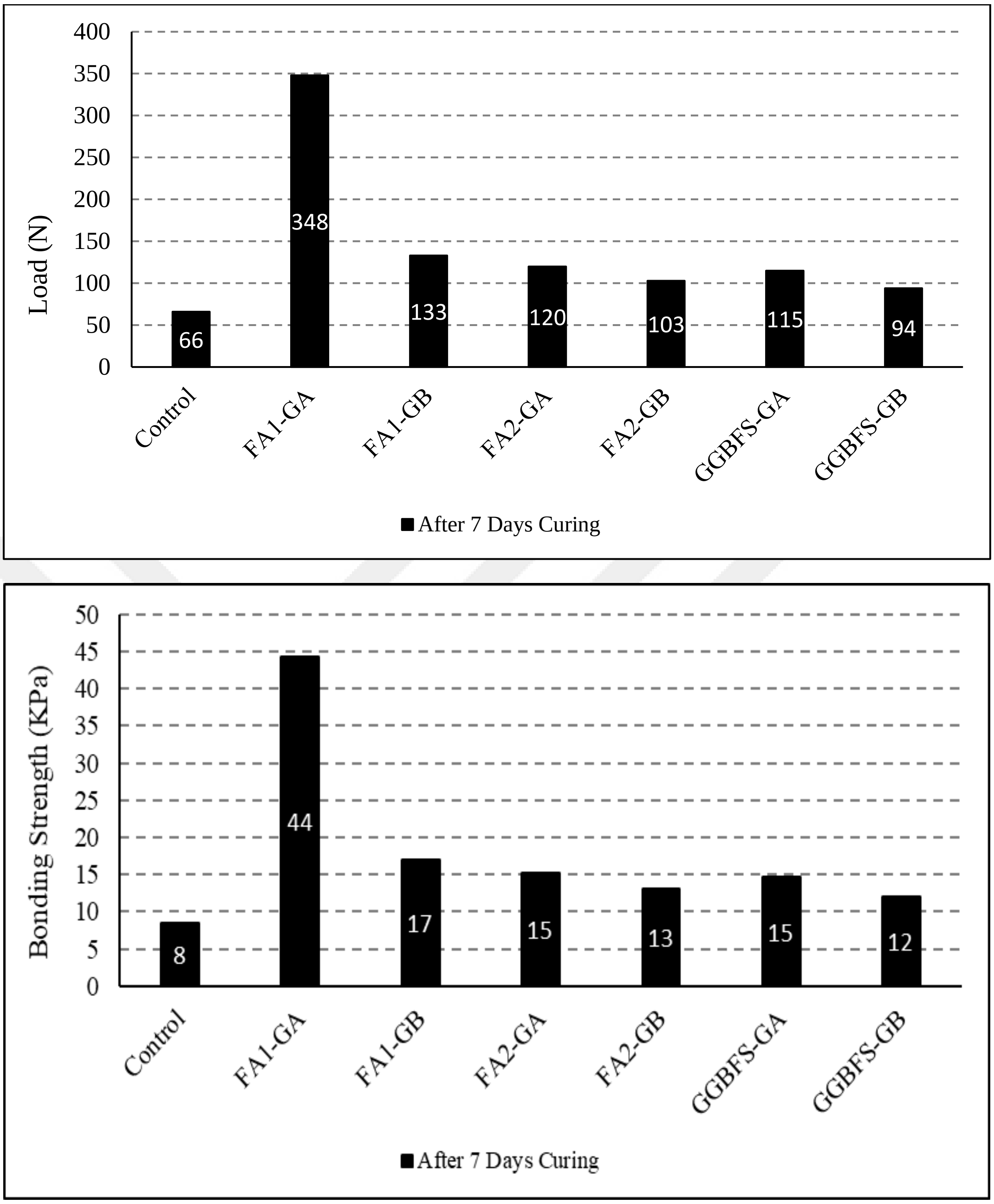


Figure 3.14 The 7 days cylinder specimens Self-bonding test result

The increase percentage of each mixtures are expressed by this formula:

$$Percentage = \frac{a-b}{b} \times 100 \tag{3.3}$$

Where “a” is the bonding load of SCMs mixtures and “b” is the bonding load of control mixture.

The reaction of pozzolanic binder calcium hydroxide in FA1-GA mixture sharply can be seen. As it was shown in Figure 3.135, the increase percentage of SCM mixture compare to control mixture, the CaO amount in FA1 is much higher than other SCMs material. In addition, the FA1-GA mixture binder increased by addition of 250 kg/m^3 FA1. As a result, the CaO percentage of FA1-GA mixture increased compare to control mixture.

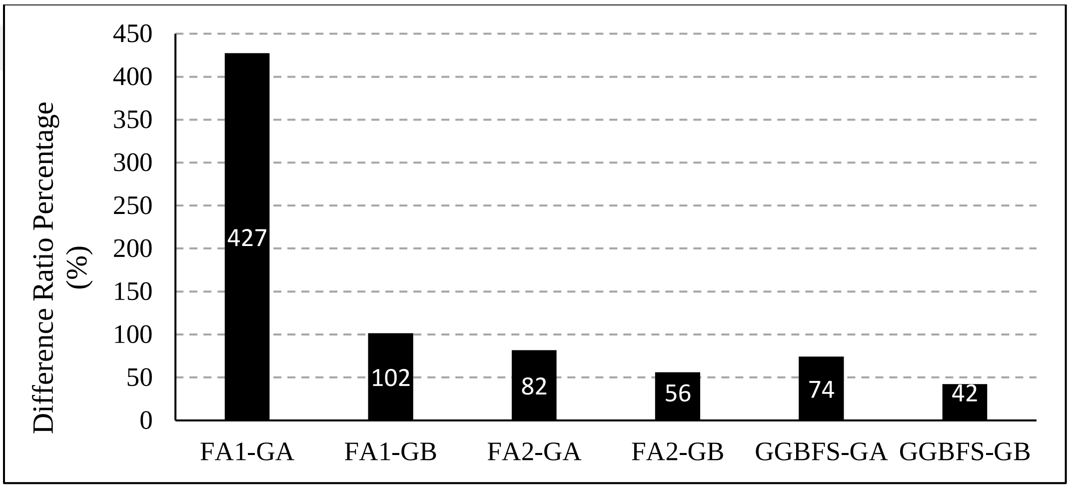
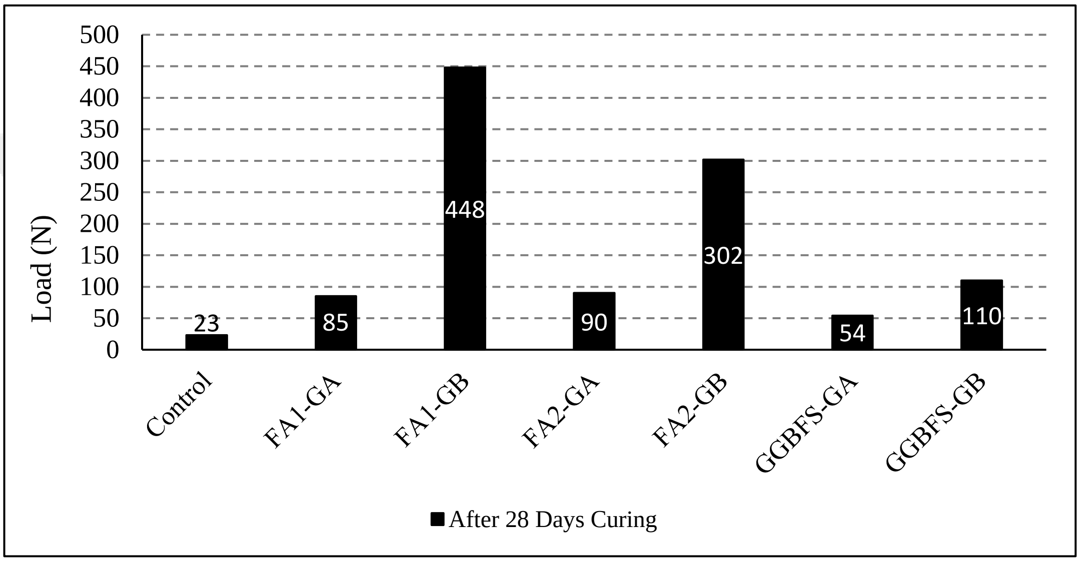


Figure 3.15 The 7 days increase percentage of SCM mixture compare to control mixture

The self-bonding test result of 28 days cylinder specimens are shown in Figure 3.16. As expected, in the fracture surface of 28 days split specimens, the amount of anhydrate cementitious particle of SCMs materials is higher. In other word, the bonding products mainly consist of hydration of un-hydrate cement particles with un-hydrate pozzolanic particle, which mainly consist calcium hydroxide crystals and calcium silicate hydrate, gels (C-S-H). Consequently, the bonding loads of FA1-GB and FA2-GB is much higher than other mixtures and represented as 448N and 302N, respectively. It means that the bonding strength for these two mixture are 57 KPa and 37 KPa, respectively. The second group of mixtures shows significant result, which is belong to high amount of SCMs. The increase and percentage of SCM mixture of 28 days compare to control mixture is shown Figure 3.17. As observed, the self-bonding properties of the second group of SCMs mixtures are superior to the control mixture. This remarkable increased can be explained as:

- The formation of calcium carbonate or calcium hydroxide.
- The reaction of C-S-H gel with calcium hydroxide.
- The high amount of binder.



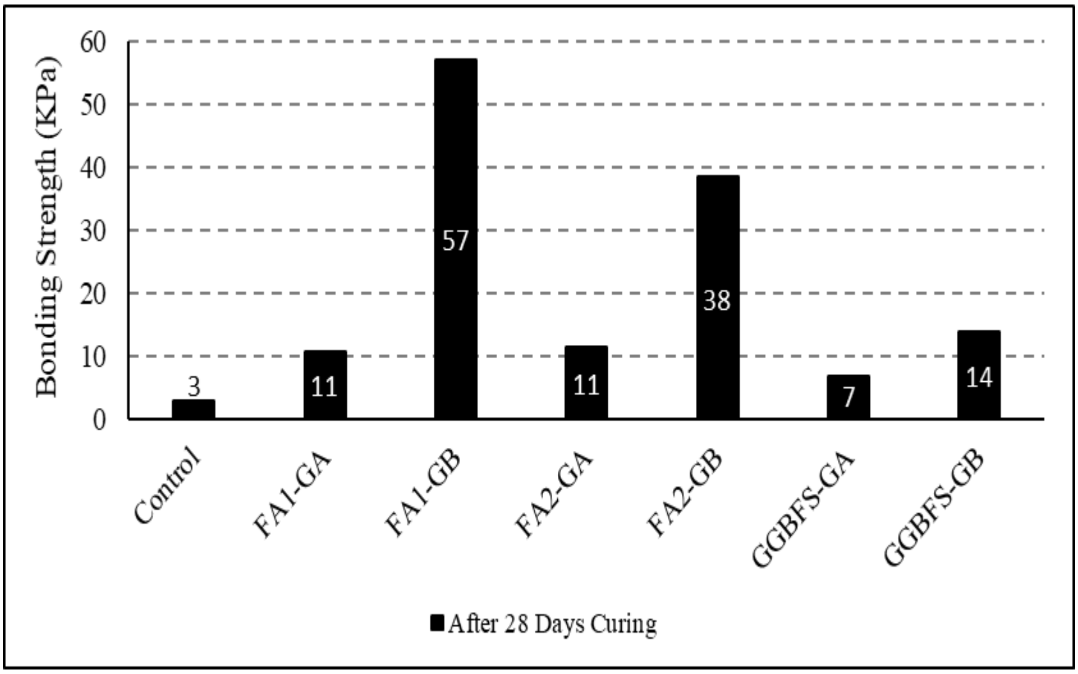


Figure 3.16 The 28 days cylinder specimens Self-bonding test result

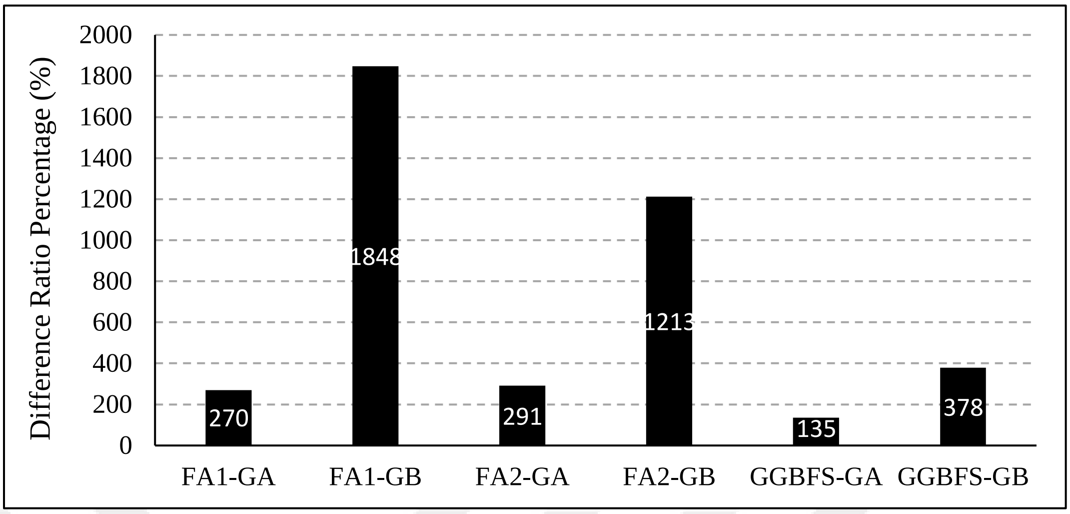


Figure 3.17 The 28 days increase percentage of SCM mixture compare to control mixture

The self-bonding test result of 90 days cylinder specimens are shown in

8. After 90-day curing, specimens were split and healed for 30 days. The bonding load were measured for each mixtures. The FA1-GA and FA1-GB have bonding load that show the healing on the surface of split parts. In other word it can be noted that 90-day specimens have self-healing properties. Whereas in many studies the concrete hydration time for 28-day were presented as a 90%. As it shows in Figure 3.18 control mixture specimens have 30 N bonding load that did not contain any SCMs binders. However, FA2-GA mixture bonding load has been exposed to a significant decrease compare to 7-day and 28-day. As we mentioned in the previous section, the bonding test method needs to be sensitive. In other word, the splitting must be done precisely. Meanwhile the FA2-GB has a considerable bonding load as 815 N with bonding strength of 104 KPa.

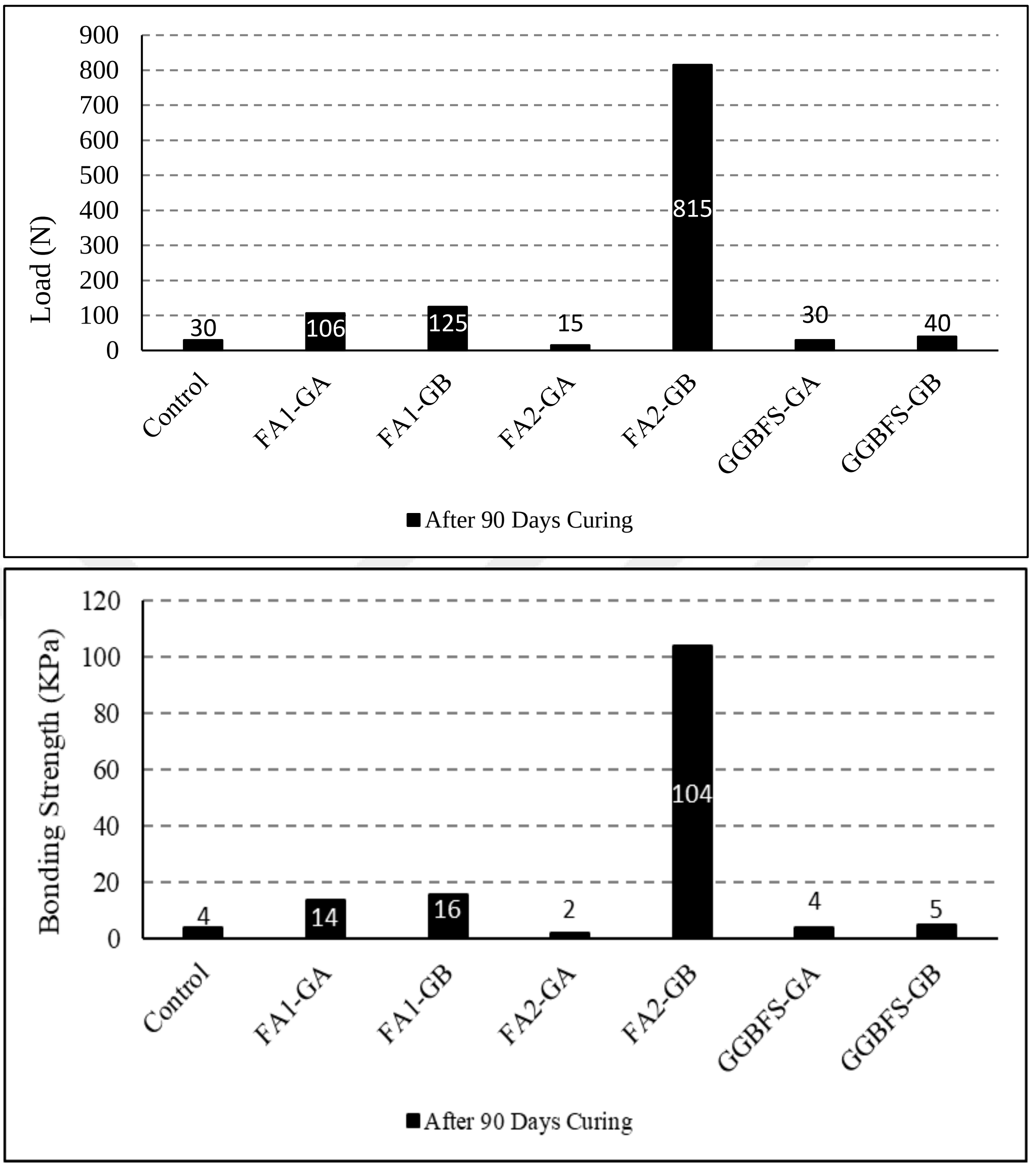


Figure 3.18 The 90 days cylinder specimens self-bonding test result

The increase and decrease percentage of SCMs mixtures compare to control mixture are shown in Figure 3.19. The FA2-GB mixture bond increment was 2617%. The remarkable increment can be explained as further pozzolanic reaction of unhydrated fly ash with $Ca(OH)_2$, high CaO content of fly ash.

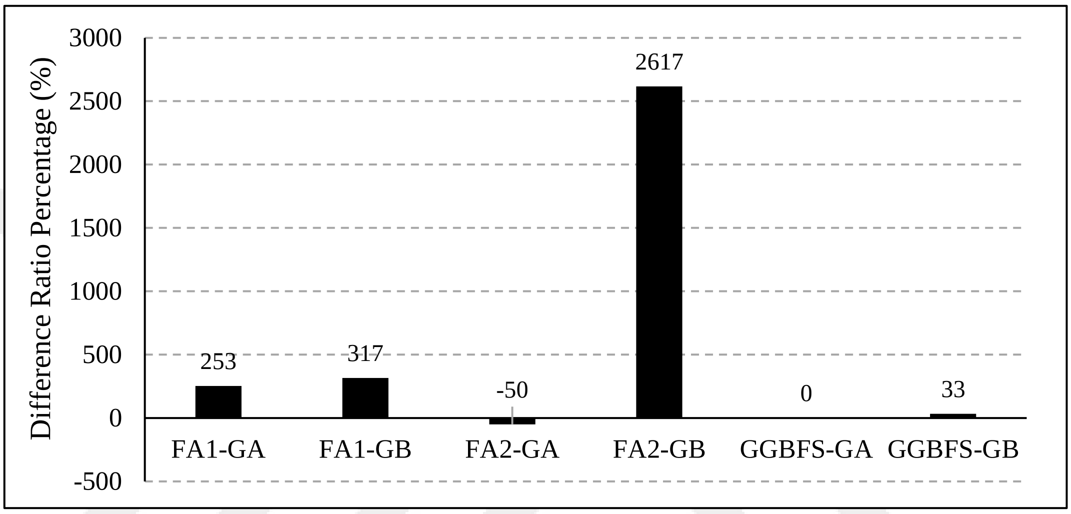


Figure 3.19 The 90 days increase and decrease percentage of SCM mixture compare to control mixture

3.3.4 Crack Closing Test Result

As described in the previous section, the SCMs specimens were casted into a 30 mm diameter and 10 mm high molds. The crack area before the healing cycle was measured with optical microscope (OM), then the same areas were premeasured in middle and after the healing cycle. The Figure 3.20 shows, the method of measuring of samples at before (a) and after (b) the healing cycle of cracks. The 7 days crack closure results are shown in Figure 3.21.

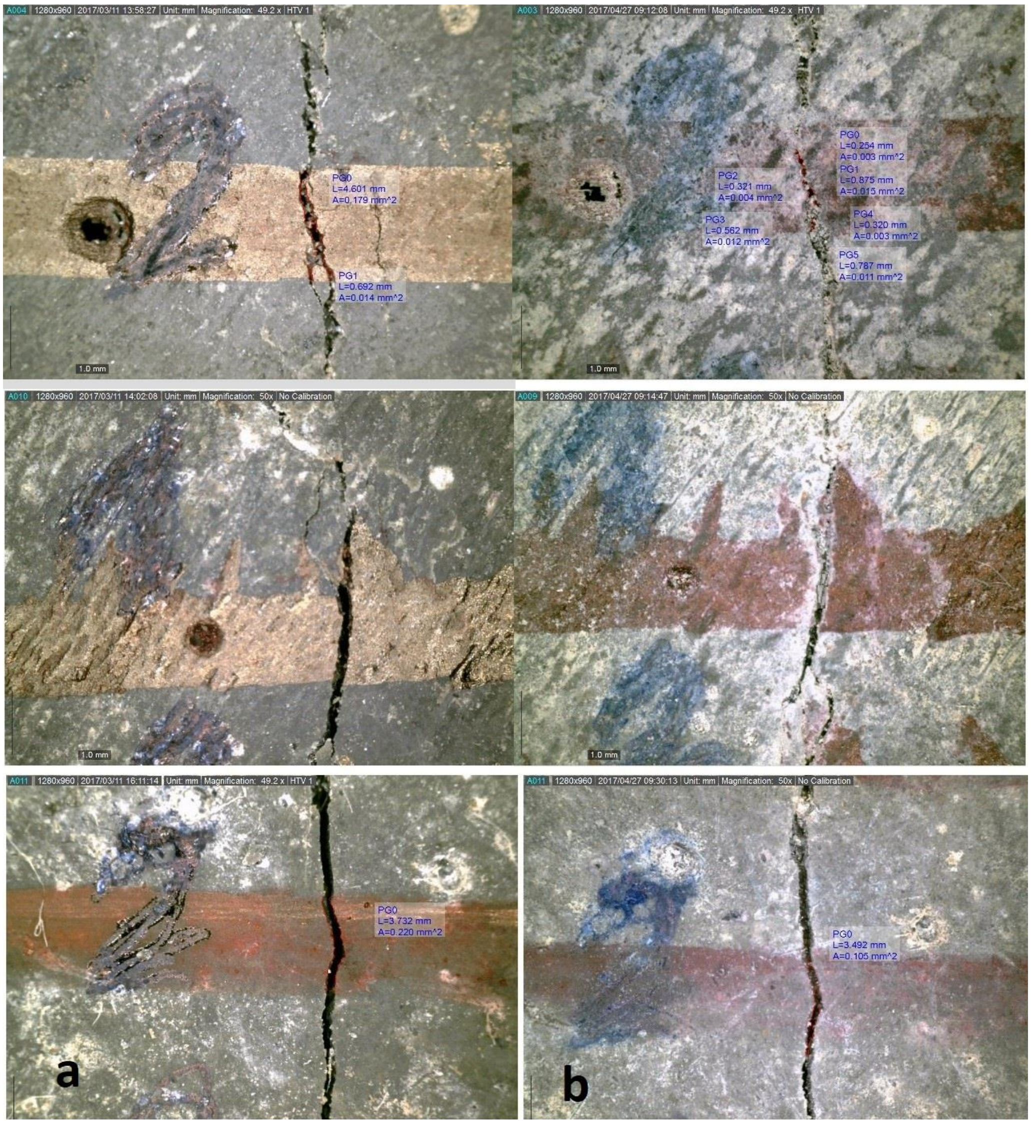


Figure 3.20 Before an after healing cycle cracks area (Personal archive, 2019)

As expected, the 7 days crack closing of SCM mixtures compared to control mixture were almost the same. The healing products on crack area can be mention as calcium hydroxide $Ca(OH)_2$ and $CaCO_3$ (Beglarigale et al, 2019). The lower healing mixture was FA2-GB. The maximum crack-closing ratio were measured in the FA1-GA and GGBFS-GA as shown in Figure 3.21. Sahmaran et al., (2013) have obtain that the formation of C-S-H was superior in healing crack that contain FA mixture, whereas in mixture which contains GGBFS, the main healing products was calcium carbonate.

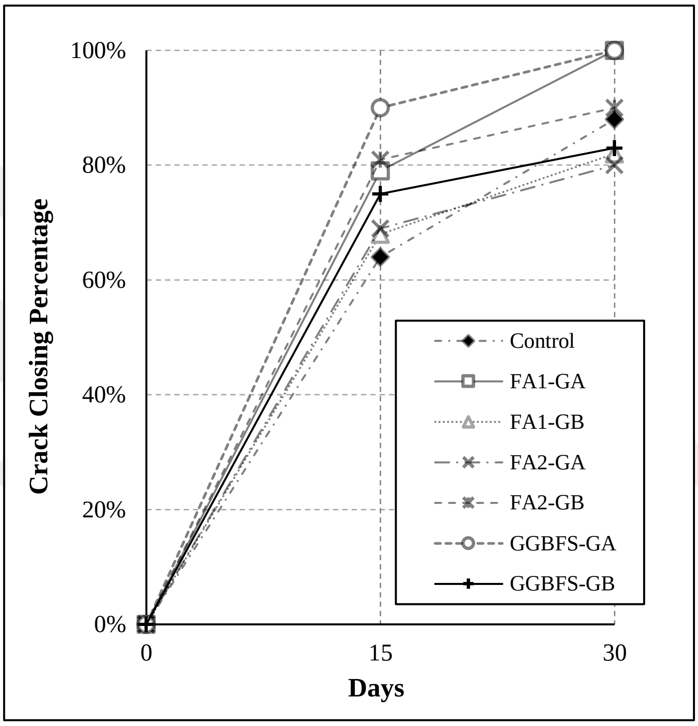


Figure 3.21 Crack closing test result of 7 days cured specimens

The 28 days cured specimens ability of closing cracks after 30 days healing period results are shown in Figure 3.22. The crack-closing ratio of FA2-GA and FA1-GA mixtures was 98% and 100%, respectively. The 15-day measurement values and behavior appear to be the same. As expected, the control mixture crack closing ratio decreased and it was observed that the closing ratio of the measurement period was the same. In other word the crack closing ratio for first 15-day was the same as the second period of 15-day.

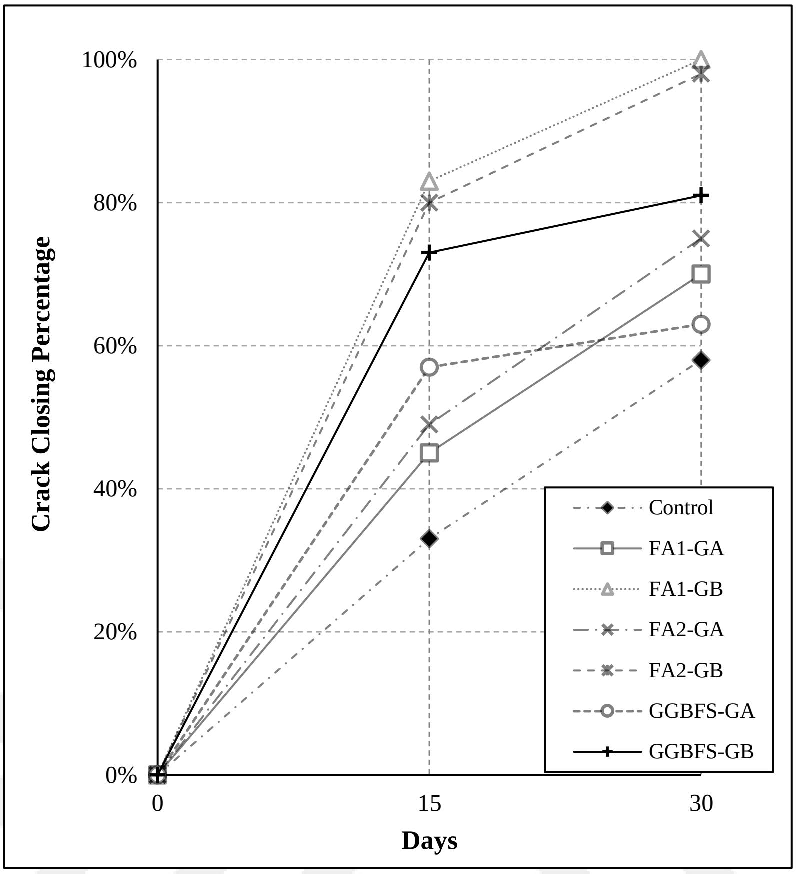


Figure 3.22 Crack closing test result of 28 days curing

The 90 days crack closure results are shown in Figure 3.23. The crack-closing ratio of FA2-GB mixtures was 62%. As expected, the control mixture crack closing ratio decreased and it was observed that the closing ratio of the measurement period was almost same. In other word the crack closing ratio for first 15-day was the same as the second period of 15-day. The 90 days crack closing result shows that the healing of binder material in cement and SCMs decrease significantly. The SCMs mixtures have great ability to close the gaps.

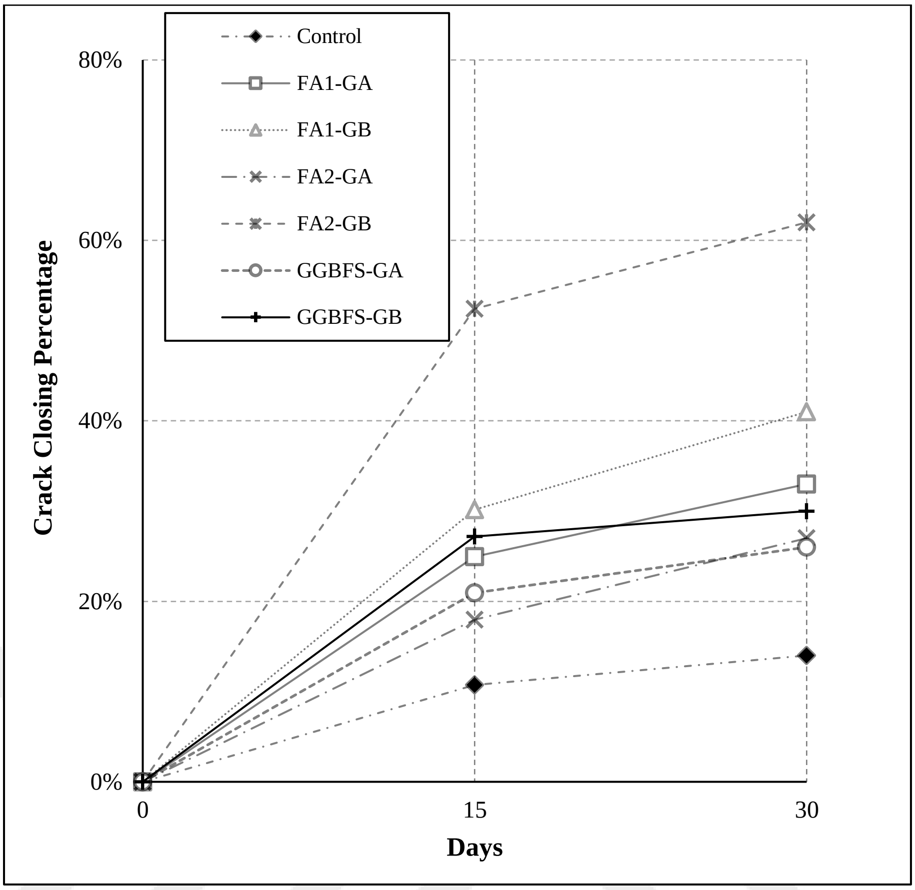


Figure 3.23 Crack closing test result of 90 days curing

As seen in crack closing test, the healing of crack depends on the age of the sample and the time of healing procedure. As it shows in Figure 3.21, the control and SCM mixtures specimens have significant healing. Whereas, in Figure 3.23 the self-healing decreases oddly. The formation of healing products are mentioned to be C-S-H, $Ca(OH)_2$ and $CaCO_3$. Sahmaran et al., (2013) have obtain that the formation of C-S-H was superior in healing crack that contain FA mixture, whereas in mixture, which contains GGBFS, the main healing products was calcium carbonate. Furthermore, (Huang et al.,2014) have demonstrated that in the mixtures which containing GGBFS, the self-healing products in cracks form C-S-H gel and have found a proportion between crack width and $CaCO_3$. The increasing in crack width increased the $CaCO_3$ ratio compare to C-S-H ratio.

3.3.5 Direct Tensile Strength Test

To evaluate the self-healing capacity of traditional mortar, twelve specimens were cast and cured in water for three different treatment periods, 7 days, 28 days and 90 days which are mention in section 2.5 Then all of the samples in each treatment were taken out of the water and were left to dry for one day. The samples in each treatment were categorized into 3 test groups (A, B and C). The group A specimens were used as control and were left in the 30-day healing. The group B specimens were subjected to a tensile strength test to obtain the stress-strain curve. The group C specimens were subjected to the tensile test until the 100µm ±10 width crack appeared, then immediately were immersed into the water for curing period of healing for 30 days. It must be noted, the crack width control under direct tensile loads can be achieved more easily with 1.5 % micro-fiber ratio. Nevertheless, it is obvious that the crack width control can be much easier and reliable in a composite that can exhibit deformation hardening behavior under direct tensile loads. The healing result are presented at Figure 3.24.

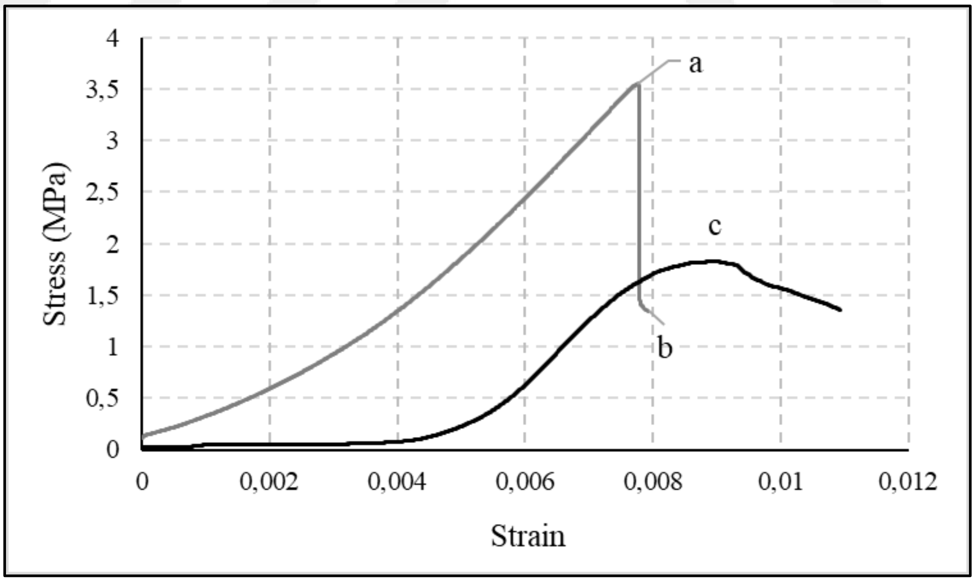


Figure 3.24 The tensile strength stress-strain curve detail for calculating healing ratio

As it presented in Figure 3.24 the (a) point is present the maximum tensile strength of each mixtures, furthermore, the (b) point is presented the load when the intended crack width happen. The (c) point is presented the maximum load of same specimen since after 30-day healing period. The ratio can be evaluated from the below formula.

$$\text{Healing ratio} = \frac{c-b}{a-b} \tag{3.1}$$

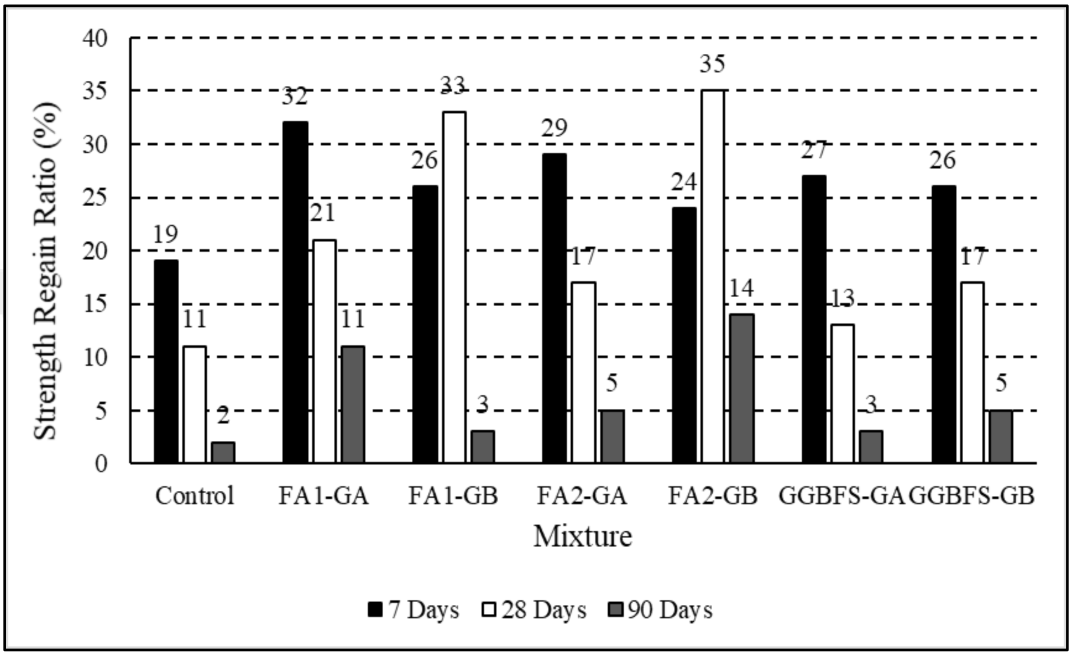


Figure 3.25 The self-healing ratio of mixtures

As can be seen in Figure 3.25 the SCMs have a significant effect on self-healing, in other word the control mixture strength improvement of 7-day is 19%, compare to the section 3.3.2, the results which indicated the 7 days and 28 days compressive strength of mixture improvement of virgin specimens of control mixture was 18%. As a result, the pre-loaded specimens have gain strength like virgin specimens and can be prove that healing can be observed. This improvement can be demonstrated in other mixtures. The FA2-GB mixture has a remarkable healing in three curing periods. It can be mentioned that the mainly healing product consist of calcium hydroxide (CH) crystals and calcium silicate hydrate gels (C-S-H) that can be formed by hydration of unhydrate cement particle in cracks that regained the tensile direct strength for 7-day, 28-day and 90-day specimens. In other word this regain can be mentioned by the filling of cracks and bonding the matrix with micro-fibers.

Mechanical properties of these composites are dramatically influenced by the fiber–matrix bond that provides the stress transferring between the fiber and matrix phases. Especially, the direct tensile strength of these composites is more dependent on the fiber-matrix bond strength. Beglarigale and Yazıcı (2015) have stated that the fiber-matrix bond characteristics improves as the embedment length of the fiber increases. They also reported that the effect of the embedment length of the fiber on the bond performance is much more noticeable for the smooth fibers. Therefore, the higher positive effect of micro fibers on direct tensile strength can be attributed to the higher length of the F1 fibers. It must be mentioned that the purpose of using fiber in this study was different. The efficiency of autogenous self-healing mechanism can be enhanced by restriction of the crack width. In other words, the narrower cracks are more likely to be closed by the autogenous self-healing mechanism (Van Tittelboom & De Belie, 2013). The use of different fibers is greatly contributing to the autogenous self-healing by restricting the crack width (Kunieda et al., 2012).

CHAPTER FOUR
CONCLUSION

Based on this study, the following can be concluded:

- This study mentions on self-healing properties of cementitious materials with high volume of binders in cement matrix. Two type of fly ash and one type of GGBFS are used to evaluate the healing properties.

- The performance of self-healing mechanism can be enriched by crack restriction. The crack width is the specific part of self-healing in cementitious composite. In order to restrict and control the crack width, two types of micro-fiber with 6 mm and 13 mm length were used. The micro-fibers have a significant effect on mortar mechanical properties but the workability and placement of fresh mortar were affected adversely. Depend on the mechanical performance, the 1.5% of 13mm micro fiber were used as a healing calculation.

- Compressive test, direct tensile test, self-bonding and crack closing methods. The damage rate is the main factor for compare the result to find out the healing for this reason the compressive test method is used. The damage ratio of 7-day, 28-day and 90-day, respectively, 18%, 28% and 22% measured. As it shows the optical damage ratio was proper to obtain the self-healing. Whereas in tensile test method the strength, drop suddenly and crack width control done hardly. In addition, the consistency and workability of mixture effected by fibers usage rate. Moreover the regain strength of the of FA2-GA (39%), FA1-GB (36%), FA1-GA (35%), GGBFS-GA (36%) and control (18%) mixtures were measured by first statement. Whereas with second statement calculation FA2-GA (63%), FA1-GB (60%), FA1-GA (43%), GGBFS-GA (66%) and control (60%) mixtures were measured for 7 days specimens.

- The high volume of binder has significantly affected the self-healing properties. According to the self-bonding test result at three different periods. The SCMs mixtures have a great performance on healing compare to control.

This can be mentioned on the high binder ratio. Whereas, if the SCMs mixture compares with each other, it is observed that the self-healing on FA1-GA mixture (44 KPa) was higher than others in 7 days. The group two of FA1 and FA2 healing ratio surpass other mixtures at 28 days with significant healing ratio of 57 KPa and 38 KPa, respectively. Furthermore, the astonishing healing was observed in 90-day for FA2-GB mixture. The average of maximum bonding load was 815 N (104 KPa). for this reason we can mention the bonding reason to the carbonation that cause the bonding on the split surfaces. The bonding test method is the novel test that leads this study to evaluate the healing. Evaluating healing ratio is the complicated part. Moreover, as many researches illustrated the mechanical properties of cementitious composite at the 28 days curing have gain at least 95%. Therefore, in early age testing methods, the comparison between pre-loading and origin specimens to keep tabs on any healing was the only way. However, self- bonding method can be one of the exacted methods to evaluating healing. As this research illustrated the result of self-bonding method.

- The self-bonding method target is to evaluate the healing of split surfaces of specimens. The split must undertake in low speed rate to restrict any harmful damage to the specimens. The healing in attached surface make a binding, which assesses by tensile test method.

- According to the test result of the novel self-bonding and crack closing methods, increasing the binder dosage of traditional mortar mixture from 500 kg/m3 cement content to 750 kg/m3 by addition of the class C and F fly ash and GGBFS enhanced remarkably the self-healing capacity of traditional mortar at early ages.

- The self-healing capacity of seven traditional mortar mixtures, which had been selected from section 3.2, were determined via various methods. It can be concluded from test results that it is only possible from a correct viewpoint to determine a realistic ratio of self-healing. Especially in the case of compressive strength test, if the undergoing hydration reactions and especially pozzolanic reactions during the healing period were not considered, over-optimistic self-healing ratios could be obtained. In addition, the efficiency of above-mentioned methods can be analyzed in terms of self-healing assessment. The compressive strength, crack-closing, self-bonding strength to assess the self-healing behavior of preloaded of traditional mortar specimens. The healing cause to filling the cracks with even low amount of products such as Ca(OH)2, CaCO3, C-S-H, and the particles and impurities suspended in the water can lead to obtain over-optimistic self-healing. In the case of direct tension test, as mentioned earlier, the sudden load drops in the direct tension test, which is a very sensitive and difficult experiment due to its nature, negatively affected the examination of the self-healing mechanism.

4.1 Recommendation

- The self-bonding method in this study is suggested and more precise test machines need to be designed.

- The Healing property requires support from mechanical engineers to obtain different methods and modeling.

- The crack closing test method only evaluating crack on surface. Furthermore, the healing in depths of cracks can be measured.

- The cement base materials are assessing with various methods in this study. Furthermore, the self-healing capacity can be evaluating under different conditions (salty water, sewage infrastructures, dry place and etc.).

- The long-term healing is the most important part of healing. As we know concrete in long term of service life are being deterioration by environmental factors.

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