EFFECT OF FLY ASH AND BOTTOM ASH ON SELF-HEALING ABILITY OF EXPANSIVE MORTARS

BY

MR. NGHIA DAI TRAN

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE (ENGINEERING AND TECHNOLOGY)

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ENTITLED

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ABSTRACT

In this study, the effect of fly ash (FA) and bottom ash (BA) on the self-healing (S-H) behavior of expansive mortar was experimentally investigated by using crack closing ratio and water flow rate as indicators of self-healing ability. To overcome cracking that may be caused by shrinkage, various types of fly ash (high CaO-SO_3-free lime fly ash – FAB, high CaO fly ash – FAA, and low CaO fly ash – FAR), and one type of expansive additive were used as the partial binder replacement. Pre-soaked bottom ash with high water retainability was used as a water-providing agent for the internal curing (IC) technique in order to reduce shrinkage and increase hydration in long term. A pre-cracked mortar samples were prepared in a disc shape with a fixed crack width of 0.1mm. The crack width ratio and water flow rate were monitored every 7 days for 3 months. It was observed that expansive mortars with the use of 30% fly ash showed significant improvement in self-healing ability when compared to the non-fly ash mortar. In addition, it was demonstrated that the use of 10% bottom ash can reduce the risk of cracking in concrete. Moreover, the combined use of both fly ash and bottom ash showed significant effectiveness for the self-healing behavior.

Keywords: Self-healing, Internal curing, Expansive additive, Fly ash, Bottom ash, Crack width, Crack closing ratio, Water permeability.

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LIST OF SYMBOLS/ABBREVIATIONS

Symbols/Abbreviations	Terms
SIIT	Sirindhorn International Institute of Technology
TU	Thammasat University
S-H	Self-healing
EC	Expansive concrete
EA	Expansive additive
FA	Fly ash
BA	Bottom ash
LP	Limestone powder
OPC1	Ordinary Portland cement type 1
FL	Free lime
LW	Lightweight
LWA	Lightweight aggregate
IC	Internal curing
CS	Curing sensitivity
CCR	Crack closing rate
WFR	Water flow rate
SCC	Self-compacting concrete

CHAPTER 1
INTRODUCTION

1.1 General

Concrete is one of the primary commonly used construction materials. Concrete structures usually have a long service life but some particular structures are vulnerable to several factors that cause deterioration, which may result in weakening both mechanical properties and durability.

Shrinkage (autogenous and drying shrinkage) is a common property of concrete. Tension can occur in the concrete, especially under a restraining condition on the shrinkage. In addition, tensile strength of concrete is not very high. Therefore, under restraint, normal concrete often cracks when the tensile stress caused by restrained shrinkage exceeds its tensile strength. These cracks lead to the reduction of durability and structural integrity. Also, the service lifetime is significantly decreased, the structure is difficult to be repaired, and the repairing cost is high. Then, shrinkage cracks are considered an unfavorable phenomenon of concrete structures.

There are many applicable methods to solve this problem, such as to select an appropriate mix proportion; to use a alkali silica solution, to use $Ca(NO_2)_2$ solution, to use bacterial solution, etc. However, some of these methods are extremely expensive, while some of them are required high technic or difficult to apply. Therefore, applying mineral admixture, especially expansive additive is an effective solution to compensate shrinkage of concrete (ACI Committee 223, 2010).

By using expansive additive, self-healing - one of the most positive abilities of concrete can be generated. With this ability, concrete can heal damages, mainly cracks, by itself without any external course of action. Therefore, properties, functionality, and durability of the concrete can be recovered and improved (Sisomphon, Copuroglu & Koenders, 2012). In addition, using expansive additive can generate self-healing ability with acceptable cost. This method is simple to apply to many types of structures, especially structures that are extremely difficult to be repaired whenever cracks occur such as underground and water retaining structures. Self-healing ability is especially necessary for these structures.

Expansive concrete is the concrete which shows expansion during its hardening. By using an expansive additive in concrete, expansive concrete is generated. One of the most important benefits of expansive concrete is self-healing ability (Hosoda, Kishi, Arita & Takakuwa, 2007).

In this study, fly ashes were used to promote the self-healing ability as well as to reduce the dosage of expansive additive then reduce the cost for expansive mortar. In addition, bottom ash was used as an internal curing material that provides water from inside the structure for hydration acceleration. As a result of this solution, the self-healing ability of the structure is improved, and shrinkage cracks can be eliminated.

1.2 Statement of problems

Concrete has been widely applied in construction because of many advantageous abilities. However, the problems caused by autogenous shrinkage and drying shrinkage are very common nowadays. The shrinkage cracking of concrete may lead to loss of strength, decrease of durability, and deterioration of concrete structures. Furthermore, sometimes deterioration happens in main structures that may lead to severe damages. For structures that are difficult to maintain and repair such as underground structures and water retaining structures, this phenomenon is more serious.

Recently, new materials and advanced technologies are studied and applied to solve these problems and enhance the properties of concrete. Using a material with self-healing ability such as expansive concrete is one of the most effective solutions. However, the price of expansive additive (EA) is still very high that leads to the limited use of expansive concrete and so this needs to be optimized. In this study, fly ash (FA) was used to reduce the amount of expansive additive in order to reduce the cost and improve the self-healing ability. Moreover, the internal curing (IC) technique was applied to provide continued hydration and increase self-healing products by time. Bottom ash with high water retainability was used as the internal curing material.

However, the effect of fly ash with different contents of calcium oxide, free lime, and sulfur trioxide and bottom ash on the self-healing behavior of cement-based materials has not been clarified. The objective of this study is to investigate the effectiveness of fly ash and bottom ash on the self-healing ability of expansive mortars. Moreover, this study aims to support the practical application of bottom ash as an internal curing agent, and the use of fly ash in order to eliminate the shrinkage cracking and promote the self-healing ability of concrete, especially expansive concrete. Therefore, the self-healing ability of mortars with two types of expansive additive and three types of fly ash together with a type of bottom ash was studied. Surface crack closing rate and water flow rate were used as the evaluation criteria for degree of self-healing ability.

1.3 Objectives of study

The aim of this study is to enhance the self-healing performance of expansive mortars. Various types of fly ash were applied in this research to partially replace cement. Bottom ash was used to partially replace fine aggregate as the internal curing material.

The main objectives of this study are divided into 3 points:

1) Study the self-healing behavior of expansive mortars using fly ash.
2) Study the self-healing behavior of expansive mortars using bottom ash.
3) Determine the self-healing behavior of expansive mortar with the combined use of both fly ash and bottom ash.

1.4 Scope of study

In this study, the effect of fly ash and bottom ash on the self-healing behavior of expansive mortars was investigated by conducting crack closing rate test and water permeability test as the indicators. Therefore, the scopes of materials, mixtures, and tests are limited as shown in Figure 1.1.

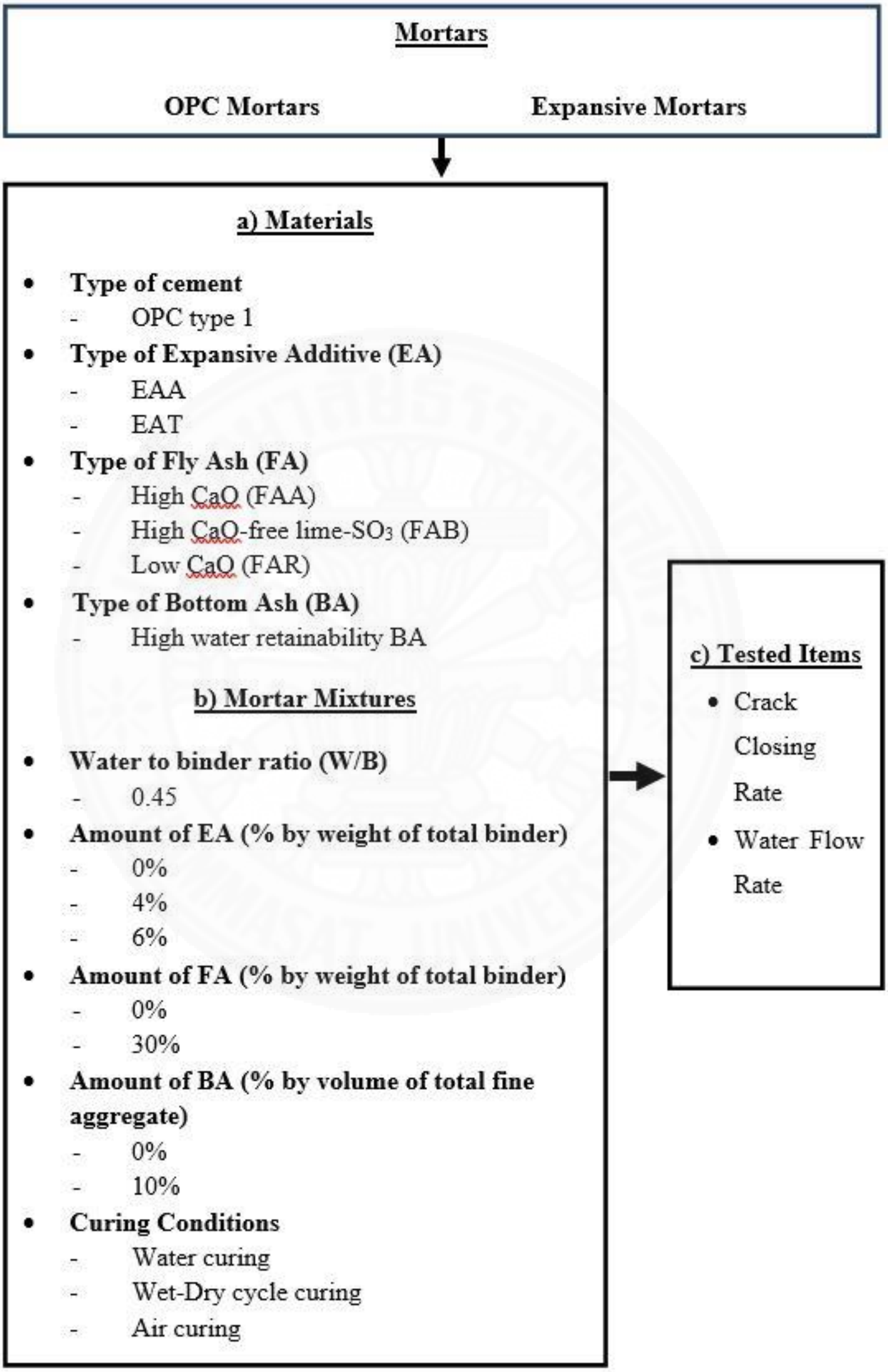


Figure 1.1 Experimental plan.

CHAPTER 2
REVIEW OF LITERATURE

2.1 Self-healing ability of concrete

Self-healing (S-H) is one of the most favorite ability of concrete that help eliminate shrinkage cracking. When concrete cracks, the crack can recover itself, the crack will be healed without any intervention. With this ability, cracks can be self-closed, and also the properties of concrete can be recovered. Therefore, the service life, functionality and durability of structures can be maintained or even improved. Self-healing was defined in many reports (Morita, Koide, Ahn and Kishi, 2010; Igarashi, Kunieda and Nishiwaki, 2009; De Rooij, Van Tittelboom, De Belie and Schlangen, 2013; Mihashi and Nishiwaki, 2012).

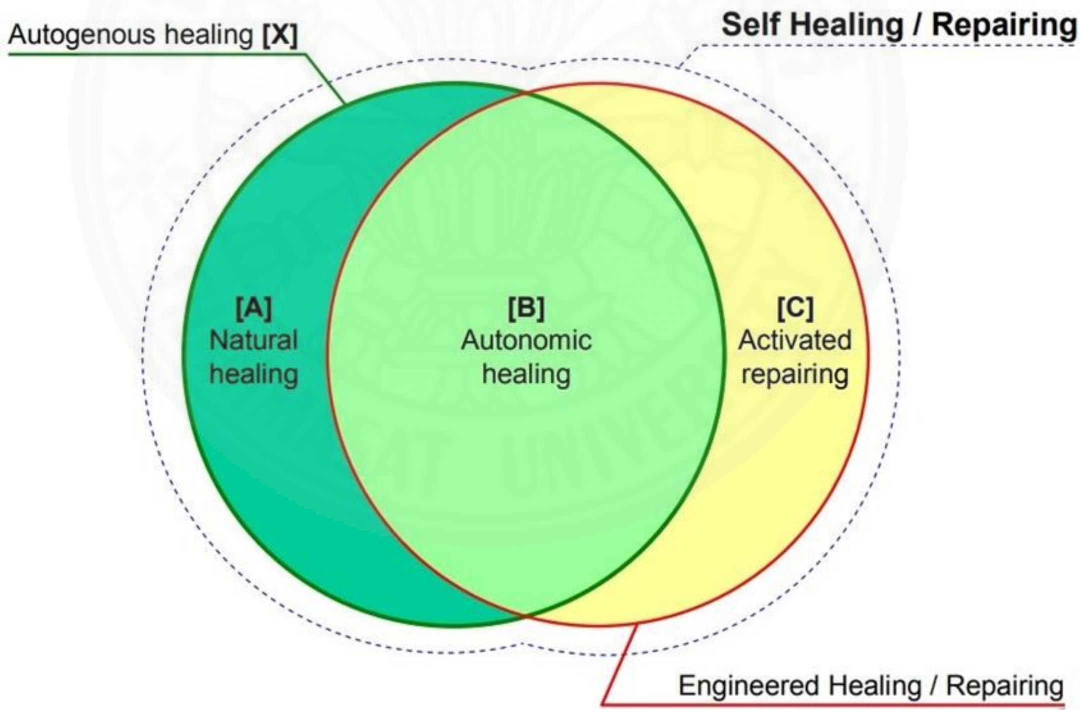


Figure 2.1 Definition of self-healing/self-repairing concrete (JCI 2009; Morita, Koide, Ahn and Kishi, 2010; Igarashi, Kunieda & Nishiwaki, 2009).

According to previous studies, there are two categories of self-healing: Natural healing (Autogenous healing) and Engineered healing (Activated repairing).

Engineered healing is generated when some additives are used in concrete to promote the self-healing process, while Natural healing occurs in concrete without adding any specific material (see Figure 2.1).

In many researches, the S-H ability of concrete was studied and mentioned. Recently, the effect of different substituted cementitious materials was investigated in order to clarify the self-healing ability of concrete.

Clear (1985) reported that the water flow rate reduces by time, and it depends on the crack width and concrete composition. The study concentrated on the effect of autogenous healing by testing the water leakage of various crack widths (0.1 mm, 0.2 mm, and 0.3 mm) in different concrete. It was confirmed that the calcite precipitation was filled in the crack. However, previously the author believed that the decreasing of water flow rate was a result of the particles losing in the crack.

Edvardsen (1999) measured the behavior of the water flow rate of pre-cracked concrete samples. It was indicated that water flow rate and crack closing rate can be used as indicators to observe the crack healing. The crack healing depends on the water pressure and the crack width. The author confirmed that calcite formation was the main mechanism of crack healing. Especially, the time-dependent and the formation of calcite carbonate was investigated through the water flow rate test.

Reinhardt and Jooss (2003) investigated the S-H capability of high strength concrete in terms of flowing water with various levels of temperature (20, 50 and 80 ^{o}C) and crack width from 0.05 mm - 0.2 mm. It was confirmed that the water flow rate depends on the temperature and the width of cracks. When the temperature is higher and the crack width is smaller, the decrease in flow rate is faster.

The effectiveness of crack width and water pressures on the S-H ability of concrete were investigated (Anura, 2003; Yi, Hyun & Kim, 2011). It was said that under the same pressure gradient there was a relationship between S-H time and crack width (liquid height/ crack width). Moreover, the crack width value should not be over the critical value in order for the S-H process to happen. The relationship between the hydraulic gradient and critical crack width was determined by Breugel (1984) and shown in Table 2.1. The water permeability of concrete is trivial when crack width is too small (< 50 μm). But the water permeability of concrete will dramatically increase with the larger crack width and higher pressure.

Table 2.1 Relationship between hydraulic gradient and critical crack width (Breugel, 1984).

Hydraulic gradient (Liquid height/ crack length)	Critical crack width (mm)
< 2.5	< 0.2
< 5	< 0.15
< 10	< 0.1
< 20	< 0.05

The S-H behavior of early age crack was studied under the effect of forces, type of binder, relative humidity, and age of crack creating of concrete (Heide, 2005). The results were concluded in Figure 2.2. However, this study confirmed that crack can be completely healed if the crack was generated at an early age (not over 3 days, even within 24 hours after casting). The crack caused by drying shrinkage happens very lately, especially when concrete is not under water, the healing is very difficult and limited.

Zhong and Yao (2008) used the UPV (ultrasonic pulse velocity) to measure the effect of damage level on the S-H ability of concrete. Specimens were cured in room condition after generated micro cracks by compression load in both normal and high strength concrete. The healing level was indicated by comparing the pulse velocity before and after curing. It was shown that both normal concrete and high-strength concrete had "damage threshold" when the damaged level is smaller than "threshold value", the S-H ratio of concrete will be higher when the damage is higher. However, when the damaged level is larger than the threshold value, the S-H ratio will reduce with higher damage.

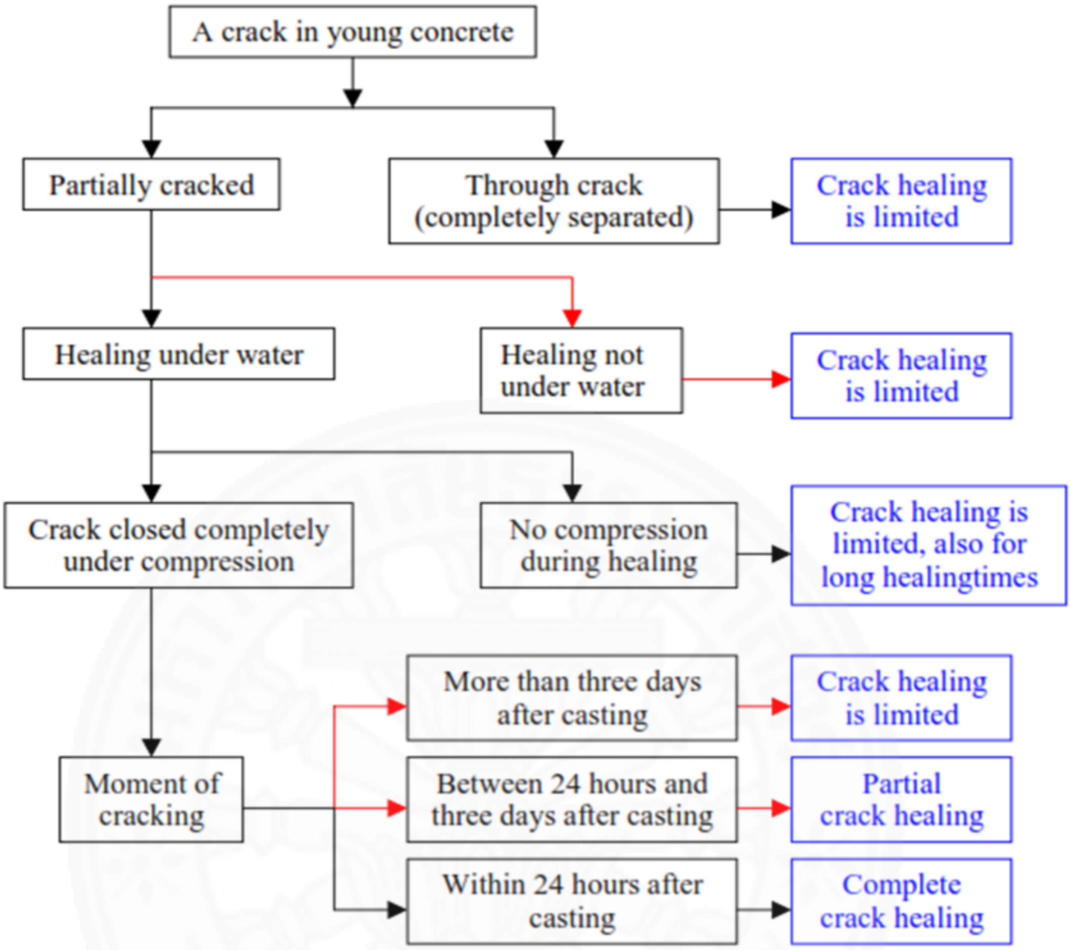


Figure 2.2 Crack healing in hydrating concrete (Heide, 2005).

Lin (2005) studied the effectiveness of various particle sizes to the S-H ability of concrete. Li, Zhou, Xu, and Yu (2011) and Li (2012) also researched the influence of particle size and the content of binder on the S-H behavior. By using the ultrasonic method, the result showed that coarse particles of binder promoted the S-H ability of concrete. However, this study should be investigated more to improve, because it leads to bleeding of fresh concrete and the reducing of the strength of concrete at early age.

Moreover, favorable conditions for self-healing of concrete were carried out. Qian, Zhou, de Rooij, Schlangen & van Breugel (2009) confirmed that to submerge samples in water is the better method to improve the self-healing capability of concrete when compared to air curing conditions. The behavior of water flow rate of pre-cracked concrete samples was measured (Edvardsen, 1999). It was observed that water flow rate of pre-crack concrete samples decreased with time meaning that concrete cracks were healed by themselves.

Reinhardt and Joose (2003) investigated the correlation between self-healing behavior and water permeability of cracked concrete. Their results showed that flow rate decreased and self-healing proficiency depended on the width of cracks and temperature. It is reported that S-H preferably occurs in low water to binder ratio (W/B) concrete, and only small crack width (not over 0.1 mm). The smaller crack width and higher temperature, the faster flow rate reduction.

Mihashi and Nishiwaki (2012) said that there are two main types of self-healing:

- Natural self-healing: The self-healing that is generated in concrete without adding specific materials.
- Engineered self-healing: The S-H that occurs in concrete caused by some admixtures to enhance the healing process.

Possible causes of self-healing were reported by De Rooij, Van Tittelboom, De Belie and Schalangen (2013) that are shown in Figure 2.3.

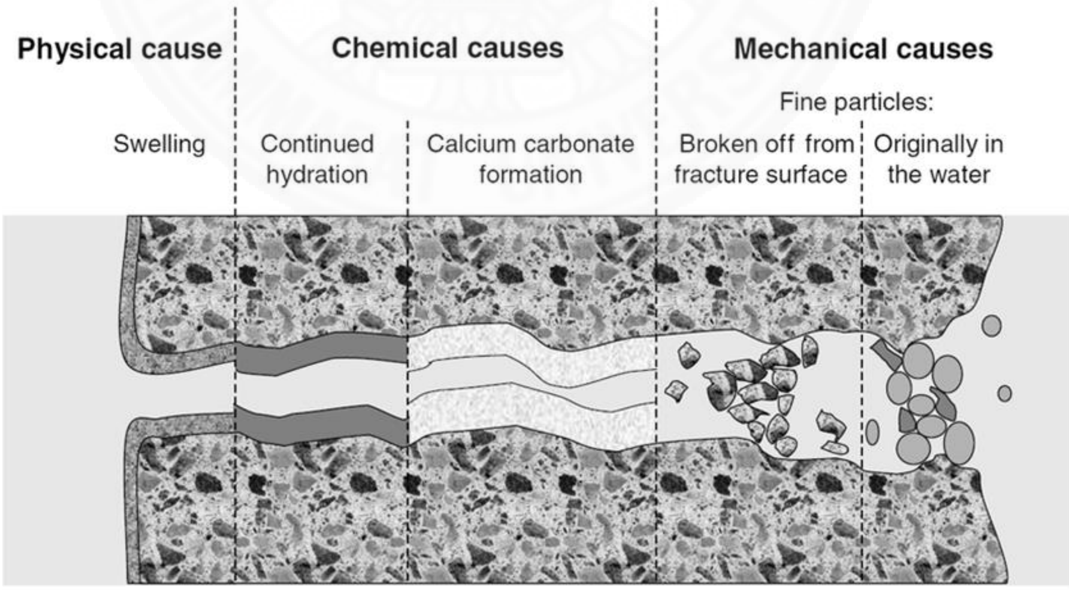


Figure 2.3 Possible causes of autogenous crack healing in concrete (De Rooij, Van Tittelboom, De Belie, & Schalangen, 2013).

Figure 2.3 shows 3 major causes of the self-healing in concrete these are physical cause, chemical causes, and mechanical causes. Physical cause includes swelling of the cement matrix; chemical causes are generated by the continued hydration and the calcium carbonate formation; and the sedimentation of particles is the mechanical cause of the self-healing ability.

- *Swelling of the cement matrix (De Rooij, Van Tittelboom, De Belie & Schalangen, 2013):*

  The crack can be smaller when concrete is kept in saturated condition. Cement matrix can lead to the closing of small crack or can enhance other reasons for self-healing (S-H) in wider crack. However, the crack still will be wider when samples are in dry condition again.

- *Continuing hydration (De Rooij, Van Tittelboom, De Belie & Schalangen, 2013):*

  When concrete gets hardened, there are many partials of cement that have not been hydrated. In the long term, these partials of cement might continuously hydrate and generate hydration products that can fill cracks.

- *Formation of calcium hydroxide or calcium carbonate (De Rooij, Van Tittelboom, De Belie & Schalangen, 2013):*

  The hydration process generates calcium hydroxide, this hydration product distributes around the crack areas will dissolve in water inside the crack and also precipitate at the surface of crack.

  $$Ca(OH)_2 \leftrightarrow Ca_2^{+} + 2OH^{-}$$

  CO_3^{2-} comes from the CO^{-} containing water:

  $$CO_2 + H_2O \leftrightarrow CO_3^{2-} + 2H^{+}$$

  => The dissolved carbon dioxide has contained in water inside the crack:

  $$Ca^{2+} + CO_3^{2-} \leftrightarrow CaCO_3$$

- *Sedimentation of particles (De Rooij, Van Tittelboom, De Belie & Schalangen, 2013):*

  It is high potential that cracks are closed by the filling of the particles. These particles can come from the liquid flowing through the crack, they can be the particles of cement loosened from the surface of crack then flow carry them through the crack until they are stuck in the small part of the crack.

Another issue is the quality of crack-closing. Ideally, the crack-healing should recover both the mechanical properties (strength, toughness, etc.) and water tightness properties (stop the water leak, crack closing, etc.). However, the healing requirement can be different, and it depends on the structural functionality. For instance, in the water retaining or underground structures, the water tightness ability of concrete is the most important ability (e.g. water leakage should be stopped, and crack should be fully closed, but the strength might be unrecovered). Besides, structures that are built for standing the load, the recovery of both mechanical and water tightness ability is necessary.

2.2 Expansive concrete and expansive additives

2.2.1 Expansive concrete

ACI 223R-10 (2010) defines that shrinkage-compensating concrete is made with an expansive cement or component system, in which the expansion, if properly restrained, induces compressive stresses that approximately offset tensile stresses caused by shrinkage.

JSCE (1994) defined that an expansive concrete is a concrete made using an expansive admixture.

According to the Japan Society of Civil Engineers - JSCE (1994), expansive concrete is classified based on the size of expansive force as shrinkage compensating concrete and chemical pre-stressing concrete. It could be classified into shrinkage compensated concrete and chemical pre-stressed concrete when considered based on the function. The qualification of maximum uniaxial expansion rate within 7 days according to the test method A of JIS 6202 (0.95% of restraining steel ratio) was set following the point of expansive force as follows:

- The standard range of uniaxial expansion rate for shrinkage compensating concrete was 150×10^{-6} to 200×10^{-6}.

- The standard range of uniaxial expansion rate for chemical pre-stressing concrete was $200x10^{-6}$ to $700x10^{-6}$.
- The standard range of uniaxial expansion rate for chemical pre-stressing concrete used for factory products was $200x10^{-6}$ to $1000x10^{-6}$.

2.2.2 Expansive additives

2.2.2.1 Definition and classification of expansive additives

In the "Recommended practice for expansive concrete" of Concrete Library of JSCE No.23, June 1994, it is stated that "expansive admixture is an admixture having the action of expanding concrete by producing ettringite or calcium hydroxide due to hydration reaction when mixed with cement and water".

Tablet 2.2 Classification of expansive cement and expansive additive according to ACI 223R-10 (2010).

Type	Expansive additive	Expansive cement
K	- Calcium sulfoaluminate (CSA) - Calcium sulfate ($CaSO_4$)	- Portland cement - Anhydrous tetracalcium trialuminate sulfate - Calcium sulfate - Lime
M	- Calcium aluminate cement - Calcium sulfate	- Portland cement - Calcium aluminate cement - Calcium sulfate
S	- Tricalcium aluminate cement - Calcium sulfate	Portland cement containing high amount of: - Tricalcium aluminate - Calcium sulfate
G	- Calcium dioxide - Aluminum dioxide	

Based on the principal constituents that form ettringite and calcium hydroxide, ACI 223R-10 (2010) classifies the expansive additives and expansive cement as shown in Table 2.2. There are 4 types of expansive additives (EA type K, type M, type S, and type G) and 3 types of expansive cements.

2.2.2.2 Effect of expansive additive on concrete properties

Hosoda, Kishi, Arita and Takakuwa (2007) used high amount of EA in low W/B concrete to investigate the water permeability and the crack closing of expansive concrete. The initial crack width was 0.22 mm, it was healed after one month cured in water. The S-H ability was the result of the additional expansion and new products such as ettringite and calcium hydroxide. However, the disadvantage when used this method is the very high cost due to the huge dosage of EA. Moreover, with this method specimens are necessary to be kept under restrained conditions.

In order to solve these disadvantages, some admixtures were applied to replace a part of EA with higher W/B ratios. In this way, a new product such as $CaCO_3$ was generated and filled the cracks.

Ahn and Kishi (2010) used cement with EA and geo-materials to enhance the S-H ability of concrete in terms of swelling, expansion, and carbonate formation. Within 33 days of curing in water, crack width of 0.27 mm was completely healed. However, some phenomena should be studied more for improvement, such as the decrease of S-H capacity and the workability as the result of the reactions between the admixture and water during casting time and hardening period of concrete.

The S-H ability of concrete under the effect of EA and different crystalline additives was studied (Hosoda, Kishi, Arita & Takakuwa, 2007; Sisomphon, Copuroglu, & Koenders, 2012). The S-H of cement-based with calcium sulfo-aluminate (CS) based crystalline additive and EA was investigated (Sisomphon, Copuroglu & Koenders, 2012). Surface cracks were created at the age of 28 days with crack width from 100 to 400 μm, before curing samples in water. It was shown that CS can enhance the S-H ability of concrete. By using blended cement with 10% and 15% by weight of CS, within 28 days, the crack of 400 μm in width was fully closed, and the water flow rate decreased to zero. It was indicated that samples using CS released more Ca^{2+} than the control samples. This is the main cause of the crack healing of concrete due to the healing product that was generated from Ca^{2+}.

Lam, Sahamitmongkol and Tangtermsirikul (2008) researched the durability properties of expansive concrete under the effect of EA content, FA content, and W/B ratio. Some durability properties such as carbonation resistance, sulfate resistance, chloride penetration, and chloride binding capacity were investigated. The authors found that with the use of EA at 30 kg/m^3, the carbonation resistance was improved but diminished when EA content was higher than 30 kg/m^3. The use of EA showed a positive effect on chloride permeability but negative one on sulfate resistance and chloride binding level of concrete.

Under the restrained condition, the capability of EA in controlling shrinkage cracking of an early age high strength concrete was investigated (Sun-Gyu, Maruyama, Jeong-Jin & Noguchi, 2003). This study confirmed that the use of EA leads to a significant reduction of tensile stress and autogenous shrinkage of concrete.

Nagataki and Gomi (1998) said that the more use of EA (not in excess of 30 kg/m^3), the larger the expansion of expansive concrete. When the used quantity of EA is not over 30 kg/m^3 of concrete, strength, creep, and Young's Modulus of expansive concrete are similar to those of normal concrete. Expansive concrete that was cured in water for a long time showed the unchanged values of shrinkage and expansion when they met the settled value. The samples that were kept in air after cured in water showed some shrinkage but still smaller than the normal concrete. Both concrete with and without EA showed the same results for the wear resistance and sulfate resistance.

The compressive strength and expansion of the concrete with EA were investigated (Lam, Sahamitmongkol & Tangtermsirikul, 2008). The authors studied the influences of the EA amount, W/B ratio, curing temperature, restraining ratios, curing condition, and types of binder. The results showed that the higher amount of expansive additive in use, the higher the expansion in concrete (see Figure 2.4). In detail, the graph shows that the free expansion of sample with the use of 20% of expansive additive (FA0HEA20) is higher than that of the sample with 15% of expansive additive (FA0HEA15). Moreover, it indicated that the free and restrained expansions of concrete used both FA and EA were higher than those of concrete with EA only. Samples cured in water showed the highest expansion when compared to other curing methods. In expansive concrete, the higher W/B ratio, the higher the free expansion, and this behavior are clearer in expansive concrete with FA. Concrete incorporating FA with high amount of EA showed problem with the loss of strength under free condition, but no problem under restrained condition.

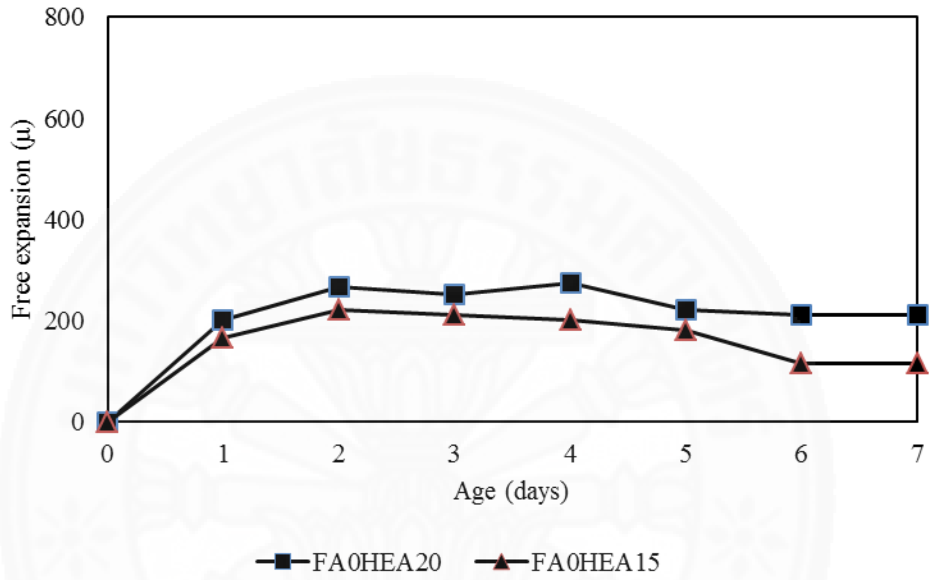


Figure 2.4 Expansion behaviors and prediction of net expansion of concrete with hyper-expansive additive (Lam, Sahamitmongkol & Tangtermsirikul, 2008).

Table 2.3 Performance of expansive concrete and normal concrete (Rachot, 2018)

Properties	Performances of EA mixtures
Setting time	Faster
Free expansion	Increase
Autogenous shrinkage	Decrease
Total shrinkage	Decrease
Unconfined compressive strength	Increase
Confined compressive strength	Increase

Based on previous studies, the comparison between the performance of expansive concrete and normal concrete shown in Table 2.3. It was clearly confirmed that expansive concrete is an advanced material with the improvement of many properties when compared to normal concrete.

2.2.2.3 Mechanism of expansive additive in concrete

a) General

Generally, ordinary concrete will shrink in the drying process; under restrained condition, tensile stress occurs that leads to cracks in concrete. In expansive concrete, the same process happens, but the formation of expansive products in concrete is created that leads to expansion. Then it compensates the shrinkage cracks.

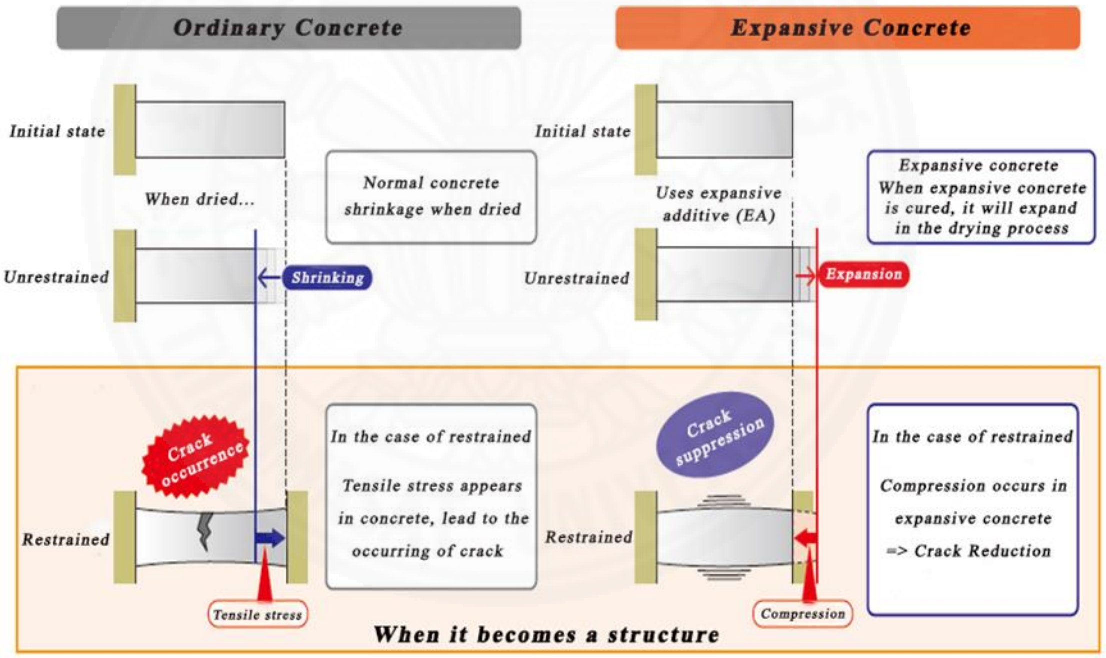


Figure 2.5 Mechanism of shrinkage cracking and crack suppression in expansive concrete (DENKA, Denki Kagaku Kogyo, 2008)

The behavior of concrete with and without expansive additive is described in Figure 2.5. In detail, when normal concrete gets dry, it will shrink. After that, when concrete becomes a structure, under restrained condition, tensile stress appears in concrete that leads to cracking. In case of expansive concrete, the same phenomenon happens. However, by using expansive additive, the shrinkage is compensated by the expansion as the result of expansive products formation. Therefore, cracks are suppressed and reduced.

ACI 223R10 (2010) points out 2 major types of expansive additives such as expansive additive with ettringite - based system (expansive additive type K, type M, and type S) and expansive additive with calcium hydroxide – based system (expansive additive type G).

b) Mechanism of expansive additive with ettringite based system:

Expansive additive with ettringite - based system includes three types of expansive additives (expansive additives type K, type M, and type S) (ACI 223R-10, 2010):

- Type K => $C_4A_3S + 8CS + 6CH + 90H \rightarrow 3C_6AS_3H_{32}$
- Type M => $CA + 3CS + 2CH + 30H \rightarrow C_6AS_3H_{32}$
- Type S => $C_3A + 3CS + 32H \rightarrow C_6AS_3H_{32}$

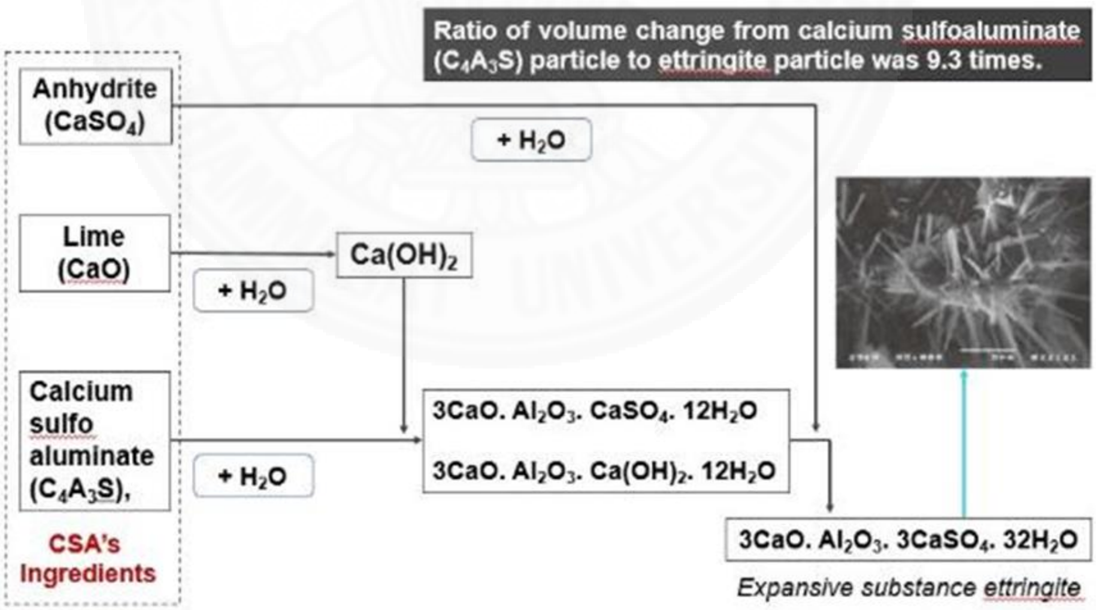


Figure 2.6 Mechanism of expansive additive with ettringite based system (DENKA, Denki Kagaku Kogyo, 2008).

C-S-A type expansive additive contains mainly calcium sulfoaluminate ($3CaO \cdot 3Al_2O_3 \cdot CaSO_4$). It causes the transformation of a mixture of calcium sulfoaluminate (C_4A_3S), lime (CaO) and anhydrite ($CaSO_4$) into ettringite that leads to expansion in concrete. The mechanism is clearly illustrated in Figure 2.6. Mechanism of expansive additive with ettringite based system is shown in this figure (DENKA, Denki Kagaku Kogyo, 2008). Crystal form of expansive substance ettringite is also illustrated. Ratio of volume change from calcium sulfoaluminate (C_4A_3S) particle to ettringite particle is 9.3 times.

c) Mechanism of expansive additive with calcium hydroxide-based system:

CaO type (Calcium hydroxide base system) contains mainly free calcium oxide (CaO). It causes the expansion of concrete due to the transformation of calcium hydroxide from the lime.

Type G => $CaO + H_2O \rightarrow Ca(OH)_2$

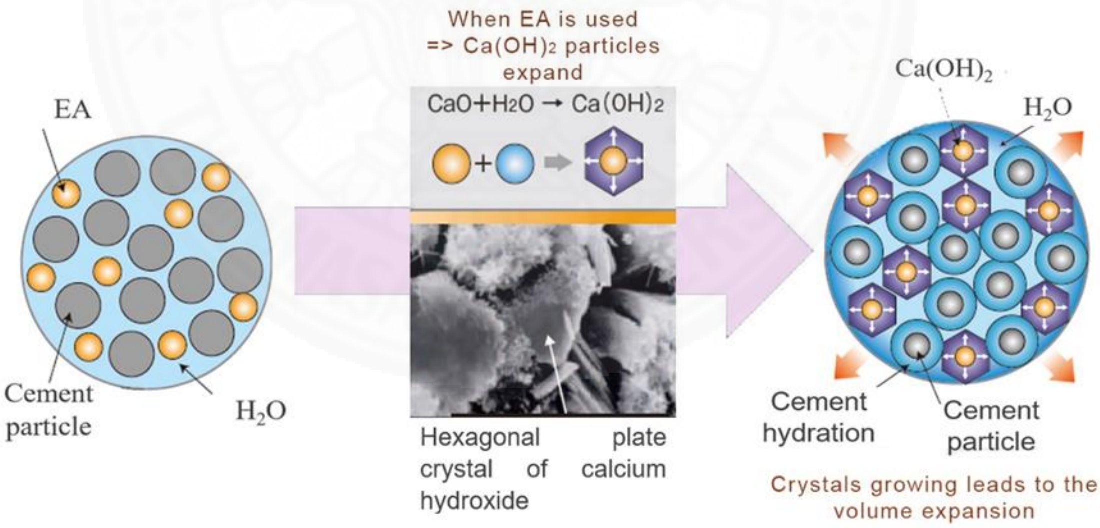


Figure 2.7 Mechanism of expansive additive with calcium hydroxide – based system (Catalogue of Taiheiyo Materials, 2008).

The mechanism of expansive additive with calcium hydroxide-based system is shown in Figure 2.7. When expansive concrete/ mortar is cast, the hydration reaction between cement and water occurs. Moreover, EA also reacts with water to form calcium hydroxide with the hexagonal plate crystal. Then crystals growing leads to the volume expansion of concrete/ mortar.

2.3 Effect of fly ash on concrete properties

Recently, been supplementary cementitious materials (fly ash, silica fume, blast furnace slag) have practically applied to enhance the mechanical properties and durability properties of concrete with reasonable cost (Lothenbach, Scrivener & Hooton, 2011). It was confirmed that these materials that contain pozzolanic substances also meliorate the S-H ability of concrete (Pipat, Dechkhachorn, Ishikawa, ICSHM2013).

Termkhajornkit, Nawa, Yamashiro and Saito (2009) confirmed that cementitious materials with pozzolanic substances can enhance the S-H ability. Namely, the use of fly ash in fly ash-cement paste leads to the improvement of self-healing ability. The self-healing ability is higher when fly ash is replaced cement with larger ratios (from 0% to 50% by volume). However, the use of fly ash can lead to the negative result for compressive strength as confirmed in previous studies.

The S-H behavior of cementitious based materials through the effect of calcium sulfo-aluminate based materials was researched (Sisomphon, Copuroglu & Koenders, 2012; Hosada, Kishi, Arita & Takakuwa, 2007; Ahn & Kishi, 2010). Jaroenratanapirom and Sahamitmongkol (2010) used fly ash, silica fume, and crystalline admixtures as different cement replacement materials to study the self-healing potential of mortars.

Kaewmanee, Krammart, Sumranwanich, Choktaweekarn, and Tangtermsirikul (2013) studied the behavior of free lime (FL) in FA, and the properties of mortars and cement pastes containing FA with various amount of FL. By adding various amount of FL to FA, the effect of FL to FA was clarified. It was found that most of FL particles were distributed outside of the FA particles. A minor dosage of FL was found inside the FA particles in the FA formation process that led to a similar effect on the properties of FA mixtures of the original high free lime FA and the added free lime FA. Moreover, the physical properties of mortars and cement pastes were not affected by FL. The use of 4.51% of FL led to the faster setting time, compressive strength improvement, and higher autoclave expansion. The added FL showed a trivial effect on the shrinkage, chloride resistance, carbonation depth, and sulfate resistance. Figure 2.8 illustrates the autoclave expansion of samples with FA containing different amounts of total free lime. There were 6 types of FA with different amounts of total free lime that were used (see Table 2.4), and fly ash was used as cement replacement at 20%. The result shows that the higher amount of free lime in FA, the higher the autoclave expansion.

Table 2.4 Total free lime content of different tested types of fly ash (Kaewmanee, Krammart, Sumranwanich, Choktaweekarn & Tangtermsirikul, 2013).

Fly ash	A	B	C	D	E	F
Total free lime content (%)	0.83	2.43	3.81	2.11	3.11	4.51

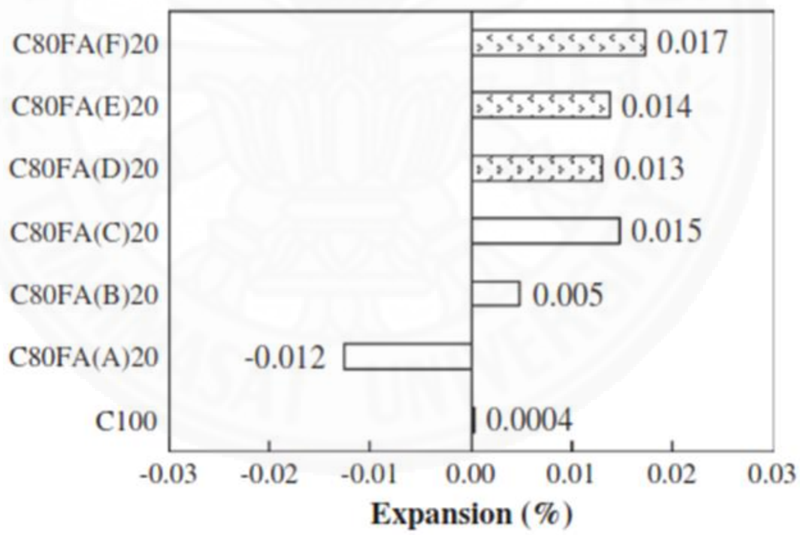


Figure 2.8 Effect of free lime content on autoclave expansion of cement – fly ash mixtures (Kaewmanee, Krammart, Sumranwanich, Choktaweekarn & Tangtermsirikul, 2013).

Atis, Kilic and Sevim (2004) studied the effect of different amounts (0%, 10%, 20%, 30%, and 40%) of non-standard FA contained high amount of CaO and SO_3 on the compressive strength, drying shrinkage, and flexural strength of mortars. It showed that both compressive and flexural strength were improved when using non-standard FA, and the shrinkage decreased with the increasing of FA (with 40% of FA, sample showed an expansion). The authors confirmed that the FA was effective to eliminate drying shrinkage of cement-based materials.

Nawaz, Julnipitawong, Krammart and Tangtermsirikul (2016) added FL to various types of FA until the total amount of FL were 5%, 7%, and 10% to study the limitation of free lime (FL) amount added to FA. The results indicated that when FL content in FA increased, some properties were changed (such as setting time is faster, water requirement, autoclave expansion, and compressive strength are higher). The authors also confirmed that high free lime fly ash can be partly used as the cement replacement when it contains low amount of SO_3 and vice versa.

Nguyen, Sahamitmongkol, Le, Tongaroonsri and Tangtermsirikul (2010) studied the shrinkage and expansion mechanism of expansive concrete, the effectiveness of FA content on properties of expansive concrete was also investigated. The compressive and tensile strengths in both free and restrained conditions and cracking strain were studied. The effect of FA on the expansion of expansive concrete is shown in Figure 2.9 and Figure 2.10. Figure 2.9 indicates that expansive concrete with FA shows larger expansion than cement-only expansive concrete. The free expansion of expansive concrete with different FA content is shown in Figure 2.10. It confirms that the more amount of fly ash, the larger expansion in the expansive concrete.

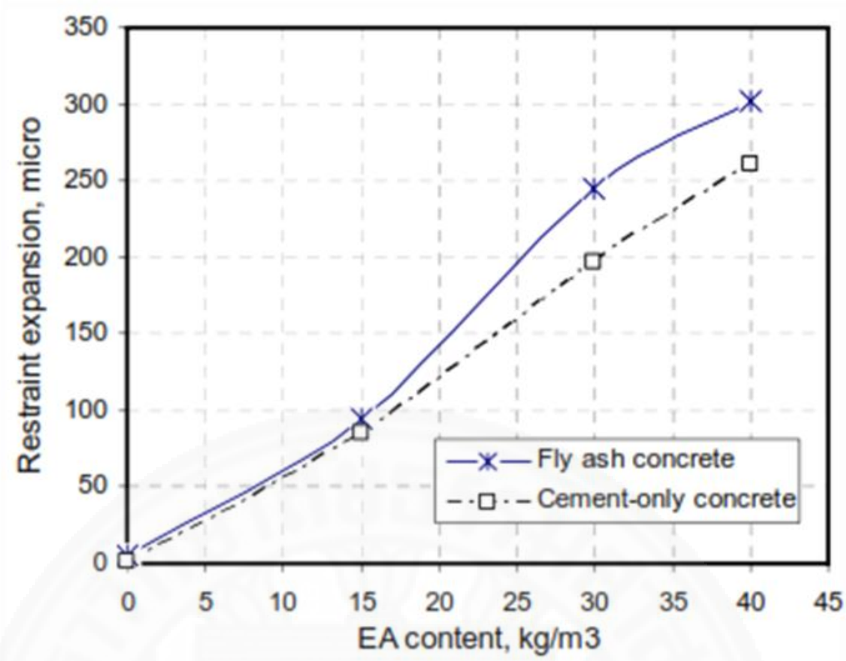


Figure 2.9 Relationship between EA content and restrained expansion of concrete at 28 days (Nguyen, Sahamitmongkol, Le, Tongaroonsri & Tangtermsirikul, 2010).

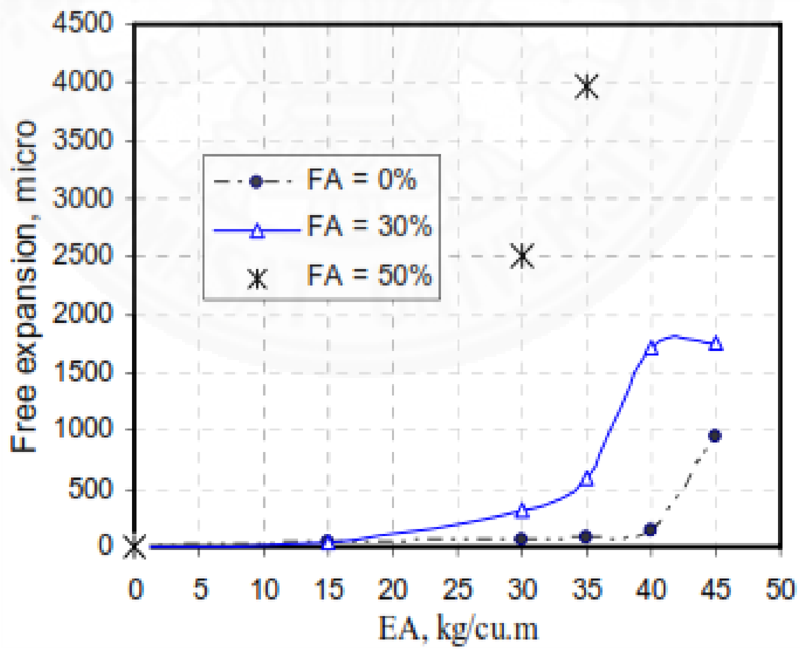


Figure 2.10 Relationship between the free expansion of concrete containing different FA content and amount of EA at 3 days age (Nguyen, Sahamitmongkol, Le, Tongaroonsri & Tangtermsirikul, 2010).

A type of high-calcium nonstandard fly ash was used as a fine aggregate replacement to investigate tensile, compressive strength and drying shrinkage properties (Atis, Kilic & Sevim, 2004). FA was used to replace cement with different ratios ot 10%, 20%, 30%, and 40%. The results indicated that with the replacement of 40%, FA reduced drying shrinkage of mortar, and FA made the mortar expanded in volume. Therefore, the authors confirmed that FA could be used to compensate drying shrinkage and to reduce shrinkage of cement-based materials. Figure 2.11 describes the relationship between the amount of FA and the length change of mortar (Atis, Kilic & Sevim, 2004). In Figure 2.11, FA was used as a partial replacement of cement with 0% (M0 mixture), 10% (M1 mixture), 20% (M2 mixture), 30% (M3 mixture), and 40% (M4 mixture). The results showed that the higher amount of high-calcium (51.29%) and high-SO_3 (12.06%) FA replacement, lower shrinkage and higher expansion of the samples were obtained.

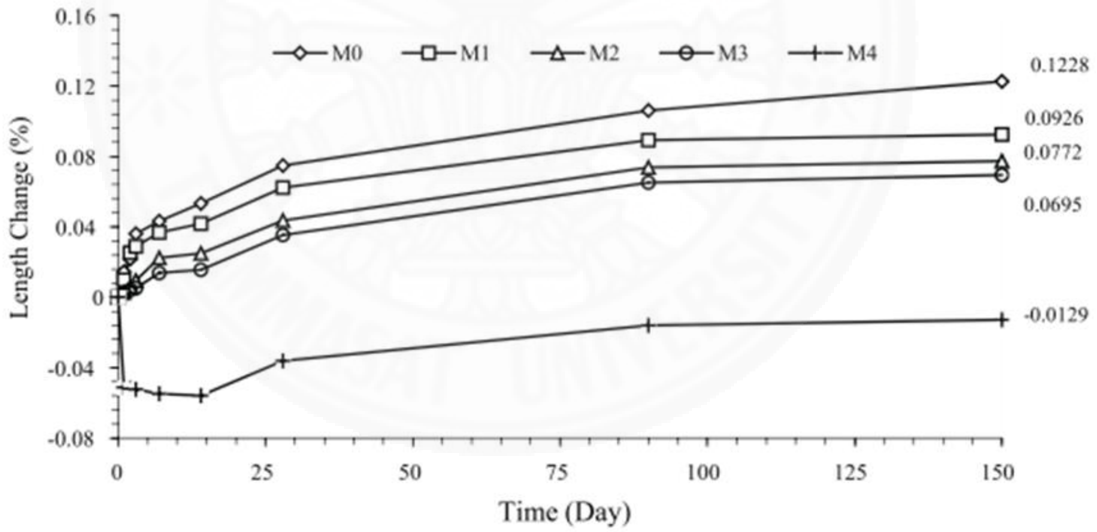


Figure 2.11 Relationship between length change and the amount of FA (Atis, Kilic & Sevim, 2004).

Chatchawan (2017) confirmed that various properties of expansive concrete containing fly ash are ameliorated. In detail, free expansion was increased, shrinkage was decreased, and amount of ettringite was higher. Three types of fly ash were used in this study that are low CaO fly ash, high CaO fly ash, and high free lime and SO_3

fly ash. The high free lime and SO_3 fly ash is the most effective when used in expansive concrete.

Nguyen, Chatchawan, Saengsoy, Tangtermsirikul and Sugiyama (2019) also studied the performance of expansive concrete using various types of fly ash. The authors stated that properties of expansive concrete such as the compressive strength, pore structure, and expansion (both free expansion and restrained expansion) are improved when using fly ashes especially fly ash that contains high CaO, free lime, and SO_3.

2.4 Effect of internal curing on concrete properties

Internal curing (IC) is an active curing method to promote the self-healing capability of concrete. Internal curing is defined as "supplying water throughout a freshly placed cementitious mixture using internal reservoirs, via pre-wetted lightweight aggregates, that readily release water as needed for hydration or to replace moisture loss through evaporation or self-desiccation" (ACI 308-213R, 2013).

By using internal curing, shrinkage can be reduced, strength and some durability properties of concrete can be enhanced. A variety of materials can be used for internal curing, including superabsorbent polymers, pre-wetted lightweight aggregates, and pre-wetted wood fibers.

Bottom ash (BA) is a type of porous material that can be used as an IC material by a partial replacement of fine aggregate with very low cost and high water-retaining properties.

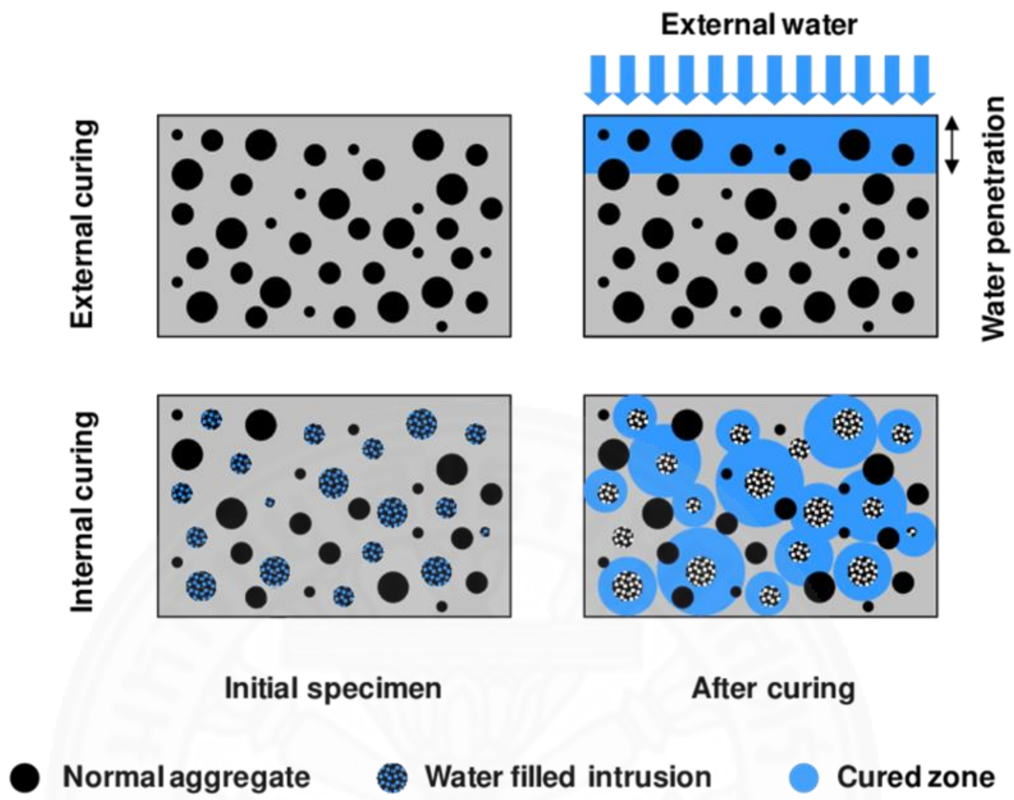


Figure 2.12 Illustration of the external and internal curing (De la Varga, Castro, Bentz, & Weiss, 2012).

De la Varga, Castro, Bentz and Weiss (2012) describe the mechanism of external curing and internal curing of concrete in Figure 2.12. When applying the external curing, water is absorbed from the surface contacting with water (curing surface). This process depends on various factors such as amount of curing water, time, temperature, and depth of structure. It is confirmed that this curing method is low effective, especially with the structures are unable to cure or big structures because water is unable to reach the inner part deeply.

On the other hand, internal curing is more effective when compared to external curing. In this way, water is absorbed into the aggregate which is equally distributed to all position of the structure at long term. When concrete dries, water contains inside the aggregate will be provided at all location inside structure for the continuously hydration reaction. Therefore, the curing effectiveness is higher and concrete properties are better as well.

Kasemchaisiri and Tangtermsirikul (2008) investigated the properties of self-compacting concrete (SCC) in-corporating BA as a fine aggregate partial replacement. The authors used various amounts of BA (0%, 10%, 20% and 30% by weight) to partially replace normal sand. The effect of bottom ash on the performance of self-compacting concrete is presented in Table 2.5 and Figures 2.13 – Figure 2.16. The results showed that slump flow and L-box passing ability of SCC with BA decreased, while 500 mm slump flow time was longer with the higher amount of BA. The increase of BA content resulted in decrease of compressive strength (see Figure 2.14) and caused the increase of porosity of hardened concrete (see Figure 2.13). However, the properties of SCC in the long term were meliorated with the replacement of 10% fine aggregate. With 10% of BA to replace fine aggregate, the durability such as drying shrinkage, carbonation depth, chloride penetration were larger than non-BA SCC because of the higher porosity (see Figure 2.15 and Figure 2.16). In addition, the more BA content, the higher the sodium sulfate resistance. It was confirmed that 10% of BA was the optimum replacement for fine aggregate.

Table 2.5: Effect of bottom as on the performance of self-compacting concrete (Kasemchaisirin & Tangtermsirikul, 2008).

Properties	Performances			
	Mixture with 0% BA	Mixture with 10% BA	Mixture with 20% BA	Mixture with 30% BA
Slump flow	****	***	**	*
Compressive strength	****	***	**	*
500 mm slump flow time	*	**	***	****
Porosity	*	**	***	****
Sodium sulfate resistance	*	**	***	****
Remark: The performance level is represented by the numbers of symbol (*).				

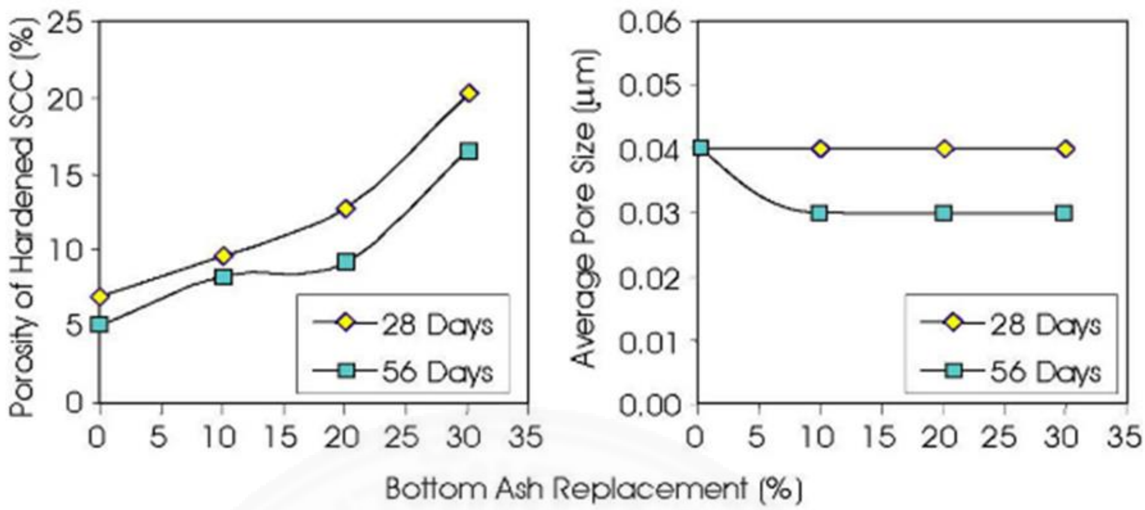


Figure 2.13 Porosity and average pore size of the mixtures with BA of 0 %, 10 %, 20 % and 30 % at the ages of 28 and 56 days (Kasemchaisirin & Tangtermsirikul, 2008).

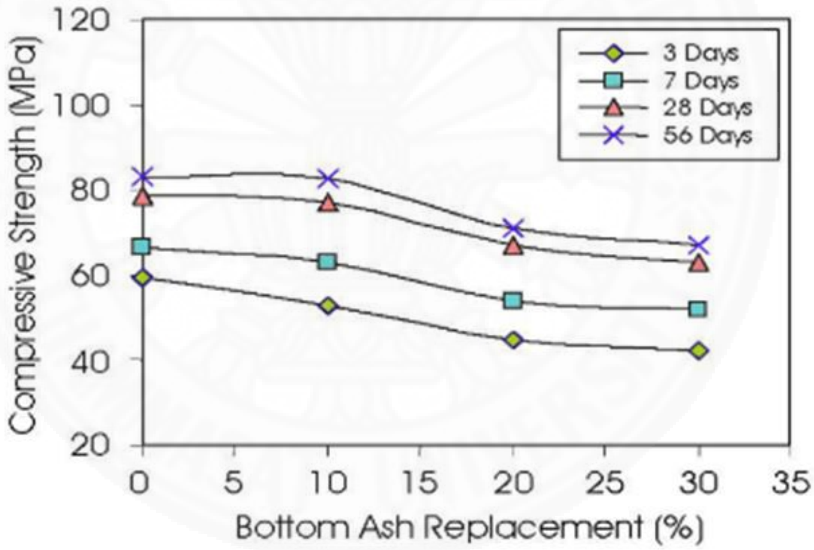


Figure 2.14 Compressive strength of the mixtures with BA of 0 %, 10 %, 20 % and 30 % (Kasemchaisirin & Tangtermsirikul, 2008).

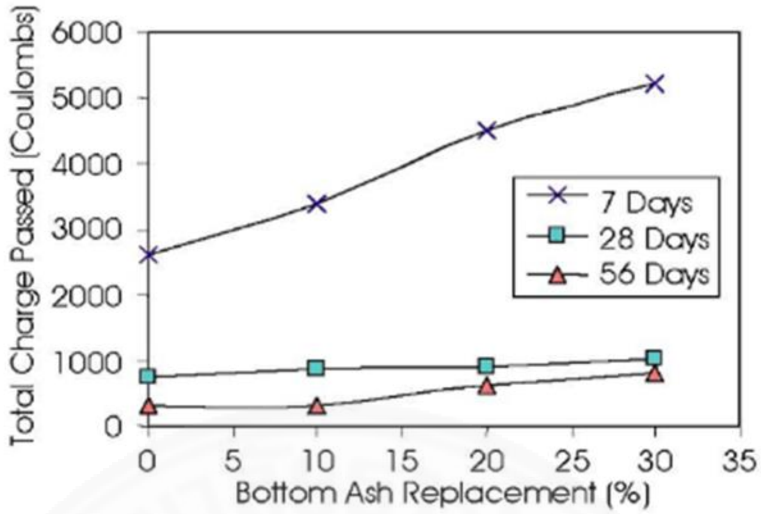


Figure 2.15 Rapid chloride permeability of the mixtures with bottom ash of 0 %, 10 %, 20 % and 30 % (Kasemchaisirin & Tangtermsirikul, 2008).

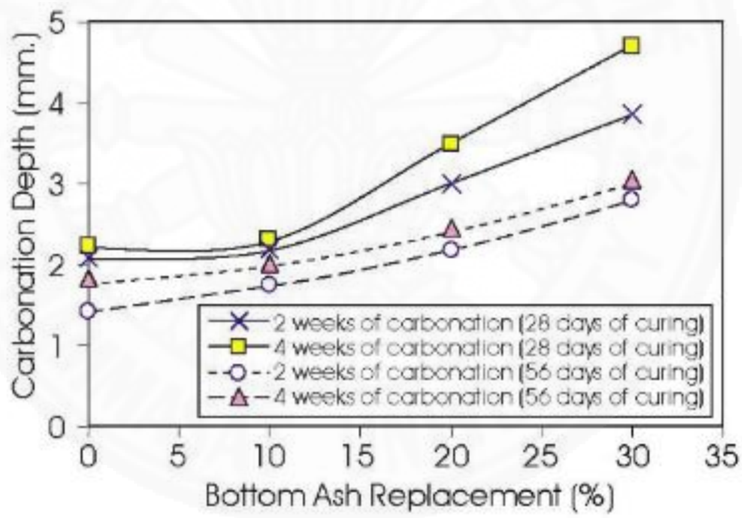


Figure 2.16 Carbonation depth of the mixtures with bottom ash of 0 %, 10 %, 20 %, and 30 % tested after 28 and 56 days of curing (Kasemchaisirin & Tangtermsirikul, 2008).

Hussain, Choktaweekarn and Tangtermsirikul (2011) studied the curing sensitivity (CS) of concrete with various types of cement, fly ash (FA), and limestone powder (LP). In this report, BA was applied as an IC agent and a partial replacement of fine aggregate. Samples were cured in two curing conditions (in water and in air).

The carbonation depth and compressive strength were used to calculate the CS index. The test results showed that the use of 10% of BA in fly ash concrete cured in air resulted in an enhancement of compressive strength. BA increased the carbonation depth, but sample with 10% BA and cured in air showed almost the same carbonation depth as the non-BA sample. The compressive strength based CSI indicated that FA increased the CS, while limestone powder decreased the CS of OPC concrete and FA concrete. BA reduced the CS of concrete due to the IC effect, especially at a low W/B ratio.

Wyrzykowski et al. (2016) applied various types of high open porosity and high water retain-ability (10-20% by mass) BA as the IC materials in terms of internal RH and autogenous deformation observation in high performance mortar. The use of BA eliminated autogenous shrinkage and slightly enhanced the mechanical properties of mortars. Moreover, because of the high interconnected pores, the BA absorbed water quickly, then it can be used to dry the surplus water and avoiding the saturated step to save time and cost.

Sutthiwarre, Sahamitongkol and Tangtermsirikul (2015) investigated the effect of IC on the expansive concrete (EC) behavior by using BA to partially replace fine aggregate. Three tests were carried out to evaluate the length change of EC sample. The early age expansion behavior of EC was evaluated by free and restraint expansions before testing the free shrinkage to clarify subsequent shrinkage of sample exposed in drying environment. It was confirmed that BA increased expansion of EC with and without FA in both free condition and also restrained condition. However, the increase of the subsequent shrinkage was also recorded. BA can be practically applied as the IC material in order to improve the EC properties, but it is important to carefully consider the balance between the subsequent drying shrinkage and the improved expansion.

Different levels of IC were used to study the effect of IC on autogenous shrinkage cracking in high-performance concrete. Cusson and Hoogeveen (2008) replaced a part of regular sand by a pre-soaked LWA (lightweight aggregate) to supply water for IC. Four pairs of specimens under restrained and free shrinkage conditions were tested. It was found that HPC with 178 kg/m^3 of saturated LWA can provide 27 kg/m^3 of IC water, the elimination of tensile stress was found, while the elastic modulus and early-age strength were not reduced. Autogenous expansion can dramatically decrease the risk of cracking when creep and elastic strains start to develop compression that leads to the increase of tensile strength before the presence of tensile stresses happens later.

Kumar, Gupta and Ram (2014) replaced a part of fine aggregate by different amounts of BA (10%, 20%, 30%, 40% and 50%) in order to investigate the compressive and flexural strength of concrete at different ages (7, 14, 28, and 56 days). The results showed that the compressive and flexural strength of concrete were increased with a higher amount of BA (under 40%) to replace fine aggregate. The authors confirmed that BA can be used to reduce cost, enhance speed of construction, increase strength of concrete.

Nguyen, Saengsoy and tangtermsirikul (2019) researched the effect of bottom ashes with different water retainabilities on the performance of expansive mortar and expansive concrete. By using bottom ash as an internal curing material, workability of the expansive mortar was improved. Autogenous shrinkage, total shrinkage, and CSI_f the of expansive mortar decreased. However, high porosity led to the decrease of compressive strength of the expansive mortar. Meanwhile the use of bottom ash in the expansive concrete caused the increase of compressive strength and slightly decrease of total shrinkage at long time.

CHAPTER 3
RESEARCH METHODOLOGY

3.1 Materials

3.1.1 Cement

The study was performed with the use of Ordinary Portland cement type 1 (OPC type 1) as the main binder for all mixtures. The chemical compositions and physical properties of the OPC type 1 are shown in Table 3.1.

3.1.2 Expansive additive

One type of commercial expansive additive denoted as EAT and one type of domestically manufactured expansive additive denoted as EAA were used as OPC type 1 partial replacement. The physical properties and chemical compositions of expansive additives are shown in Table 3.1.

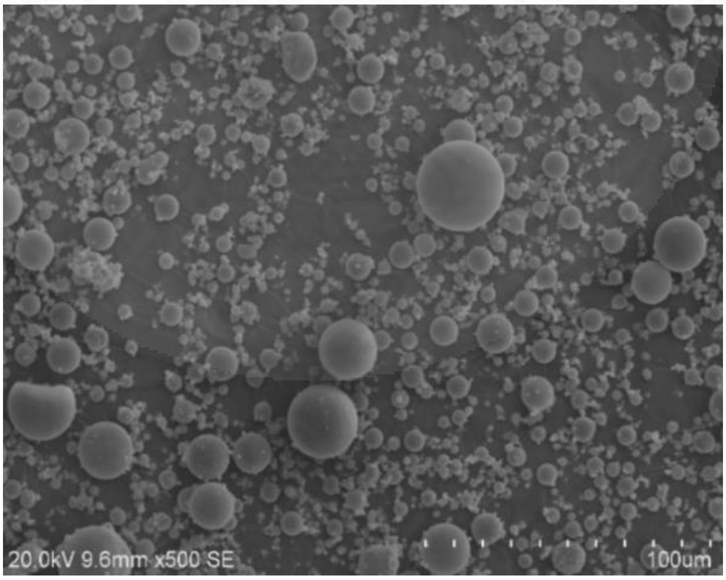
Figure 3.1 Binder samples (OPC1, EAA, EAT).

Figure 3.1 shows the binder samples that were used in this study. From left to right respectively are OPC, EAA and EAT where OPC type 1 was used as the main binder, while EAA and EAT were used as the partial replacement of cement OPC type 1.

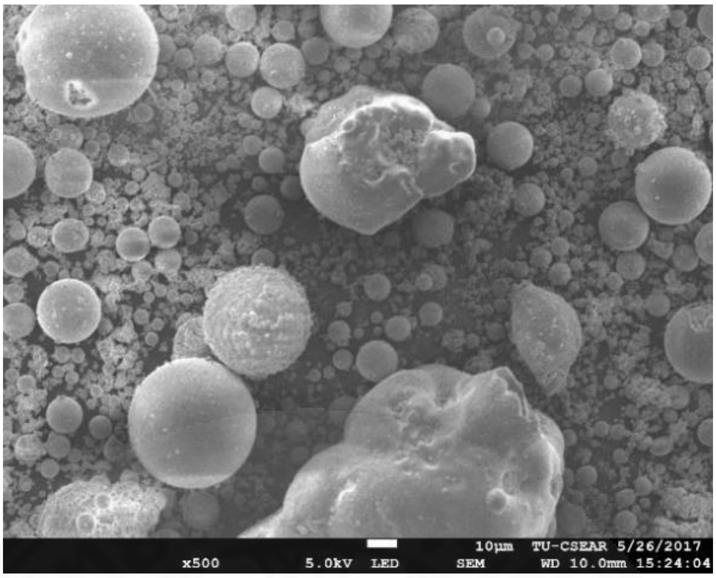
3.1.3 Fly ash

Three types of fly ash (FA) denoted as FAA, FAB, and FAR were used as cement partial replacement by weight of the binder. Three types of fly ash (FAA, FAB, and FAR) were used in the study are shown in Figure 3.2.

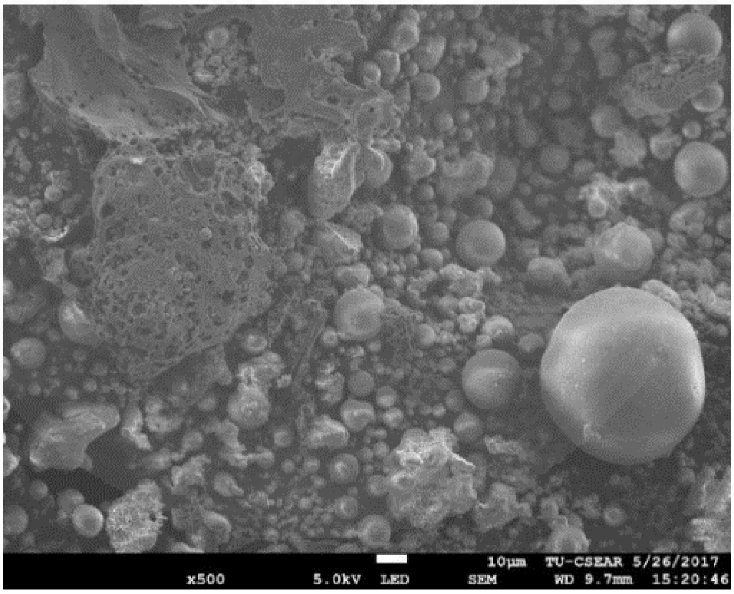
Figure 3.2 Three types of fly ash.



a) FAA (Mae Moh power plant).



b) FAB (Mae Moh power plant).



c) FAR (BLCP power plant).

Figure 3.3 SEM images of FAA, FAB, and FAR fly ashes.

FAA and FAB were fly ashes from Mae Moh power plant while FAR was a fly ash from the BLCP power plant. Fly ash type A (FAA) contains high CaO content, fly ash type B (FAB) contains high CaO, high SO_3 and high free lime content, fly ash type R (FAR) contains low CaO content. FAA and FAR were classified as class 2b and class 2a fly ash, respectively, according to TIS 2135 standard classification. However, according to BS EN 450 standard, FAB was classified as the non-standard fly ash due to the free lime content which was higher than 2.5%. The SEM images of FAA, FAB, and FAR were illustrated in Figure 3.3, and their chemical compositions and physical properties are shown in Table 3.1.

Table 3.1: Properties of binders used.

Chemical composition and Physical property (% by weight)	OPC	Fly Ash			Expansive additive	
		FAA	FAB	FAR	EAA	EAT
SiO_2	19.70	37.09	26.61	61.09	2.12	9.04
Al_2O_3	5.19	19.69	13.60	20.35	4.75	2.53
Fe_2O_3	3.34	14.18	18.34	5.2	0.14	1.47
CaO	64.80	19.36	24.97	2.32	61.19	68.60
MgO	1.20	2.32	2.33	1.35	0.73	0.78
SO_3	2.54	3.24	8.53	0.28	26.46	12.19
Na_2O	0.16	0.35	1.75	0.79	-	0.23
K_2O	0.44	2.92	1.77	1.36	-	0.13
TiO_2	-	-	-	-	-	0.14
P_2O_5	-	-	-	-	-	0.10
MnO	-	-	-	-	-	0.03
SrO	-	-	-	-	0.11	0.03
ZnO	-	-	-	-	-	0.02
CuO	-	-	-	-	-	0.02
Free lime	0.87	2.36	3.93	0.03	28.94	27.98
LOI	2.10	0.01	0.53	5.68	4.48	4.65
Specific gravity (cm^2/g)	3.15	2.20	2.57	2.11	2.94	3.02
Blaine fineness (cm^2/g)	3100	2790	2820	3400	-	-

3.1.4 Aggregate

River sand with a specific gravity of 2.58 g/cm^3 at SSD condition and a water absorption of 1.07% was used as the fine aggregate. Bottom ash (BA) with a specific gravity of 1.83 at g/cm^3 at a water retainability (WR) of 36.61% was used as the partial fine aggregate-replacing material. Fine aggregates (sand and bottom ash) are shown in Figure 3.4. The water retainability test of BA developed by Nguyen, Saengsoy, and Tangtermsirikul (2019) was conducted in the same way as that proposed by Lathsoulina, Sancharoen, and Tangtermsirikul (2015) but with a longer vibrating time. BA that passed sieve No. 4, was immerged in water for 72 hours before moving out of the water and drained for 30 minutes to remove free water. After that, 3 layers of BA was filled into a PVC pipe, each layer was compacted 30 times by a rubber hammer. After covering the top of the PVC pipe to prevent the evaporation of water, the PVC pipe was vibrated for 60 minutes. By the test method, the effect of gravity force, the excess water of BA was removed while the retained water was still kept by the particles. The water retainability of BA was determined by the moisture content of the BA sample collected from the top 3cm of the depth of the sample.

Figure 3.4 Fine aggregate samples.

The sieve analysis result and physical properties of river sand and bottom ash are shown in Figure 3.5 and Table 3.2

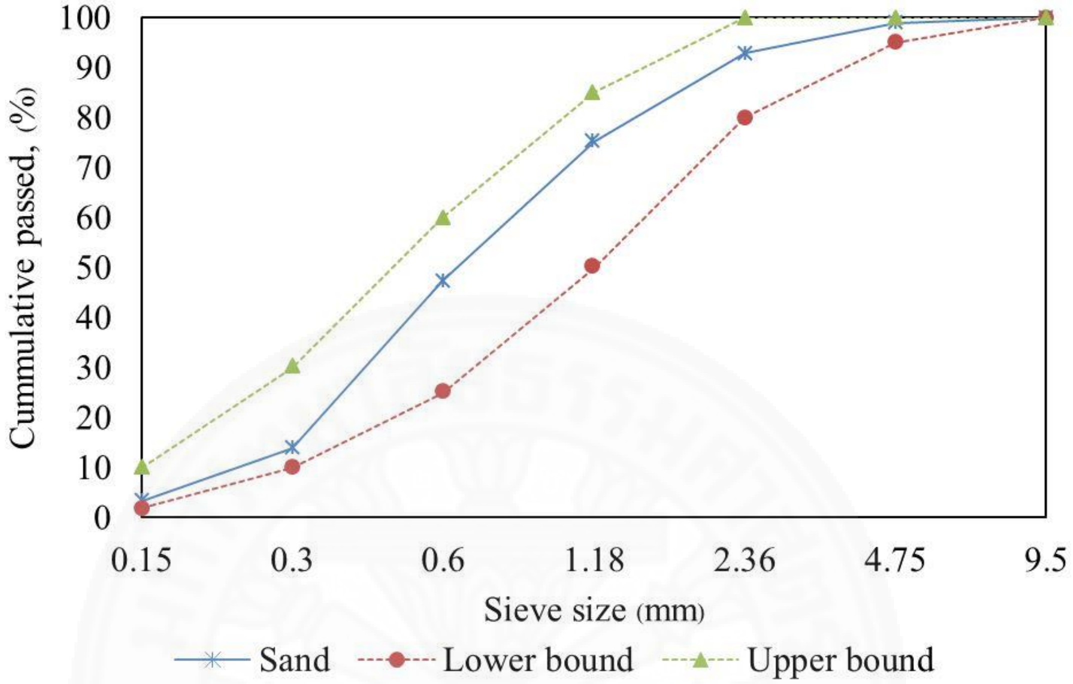


Figure 3.5 Sieve analysis results of fine aggregate (sand)

Table 3.2 Physical properties of fine aggregate and bottom ash.

Properties	River sand	Bottom ash
Absorption (%)	1.07	-
Water retainability (%)	-	36.61
Specific gravity (g/cm^3)	2.58 (SSD)	2.55 (SSD)

3.2 Mix proportions

In this study, various mixtures were designed and prepared to study the self-healing ability of expansive mortars with the use of fly ash and bottom ash (see Table 3.3). There are 12 mixtures which were prepared and grouped in order to investigate the effects of type and amount of expansive additive, fly ash content, type of fly ash as well as bottom ash on the self-healing behavior of expansive mortar.

Table 3.3 Mix proportions of mortars.

Mix ID	OPC 1, by weight (kg/m^3)	Expansive additive (kg/m^3)		Fly ashes (kg/m^3)			Fine aggregates (kg/m^3)		Water (kg/m^3)
		EAA	EAT	FAA	FAB	FAR	River sand	Bottom ash	
OPC	534.54	-	-	-	-	-	1469.99	-	240.54
OPCBA10	534.54	-	-	-	-	-	1322.99	145.29	240.54
EA4	512.90	21.37	-	-	-	-	1469.26	-	240.42
EA6	502.10	32.05	-	-	-	-	1468.90	-	240.36
ET6	502.24	-	32.06	-	-	-	1469.33	-	240.44
EA6BA10	502.10	32.05	-	-	-	-	1322.01	145.18	240.36
FAA30	366.09	-	-	156.9	-	-	1438.21	-	235.34
FAB30	369.84	-	-	-	158.50	-	1452.95	-	237.76
FAR30	364.84	-	-	-	-	156.36	1433.29	-	234.54
FAB30BA10	369.84	-	-	-	158.50	-	1307.66	143.61	237.76
EA6FAB30	337.89	31.68	-	-	158.39	-	1451.89	-	237.58
EA6FAB30BA10	337.89	31.68	-	-	158.39	-	1306.70	143.50	237.58

3.3 Experimental method

3.3.1 Experimental parameters

In this study, various parameters were applied to study the behavior of self-healing ability of expansive mortars with the presence of fly ashes and bottom ash. Some major parameters are illustrated in Figure 3.6 such as mix proportion, curing condition, crack width, age at crack introducing, and pressure.

Samples were prepared with various mixtures such as mortar with and without expansive additive, mortar with different amount of expansive additive, mortar with and without fly ash, mortar with and without bottom ash, expansive mortar with and without fly ash, expansive mortar with and without bottom ash, OPC mortar and expansive mortar with and without the combination of fly ash and bottom ash.

Crack with of 0.1 mm in width was created at 3 days of age before curing in different conditions (air cure, wet-dry cycle, and water cure). After that, samples were tested for crack closing rate and water permeability. With water permeability test, a constant pressure of 0.5 Bar was applied to all the samples.

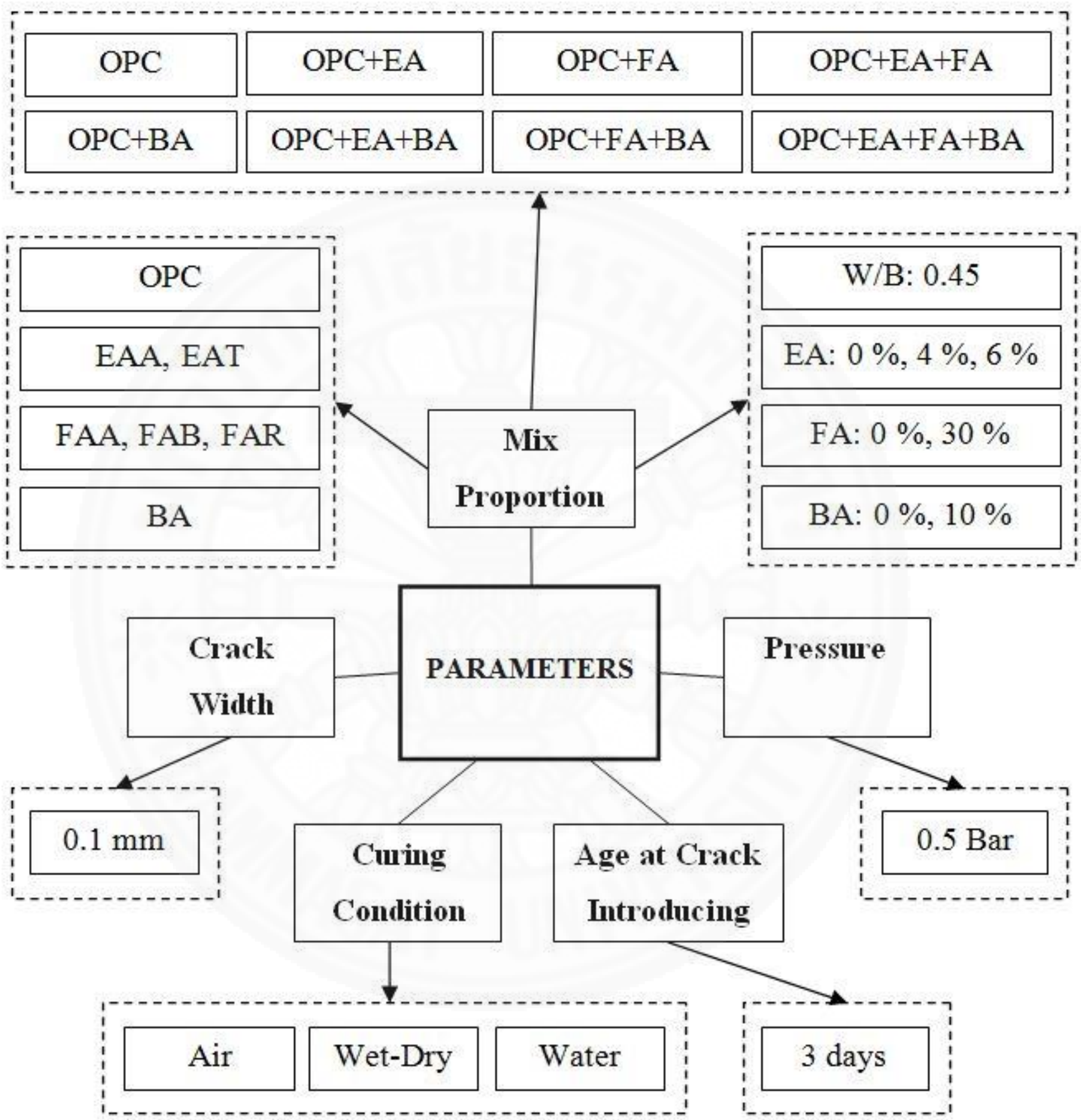


Figure 3.6 Research parameters.

3.3.2 Crack closing rate measurement

Mortar specimens were prepared in a disc shape having a 100 mm diameter and a 25 mm thickness (see Figure 3.7a). Figure 3.7 illustrates a photo, drawings, and dimension of specimens for the surface crack closing rate test. Specimens were prepared for all mix proportions. A crack of 0.1 mm in width was introduced to each specimen at 3 days of age. A crack was created on each mortar specimen by the splitting method (see Figure 3.7b) at the age of 3 days after casting. Stainless rings (see in Figure 3.8) were used to control a constant surface crack width at 0.1 mm before the curing step (see Figure 3.7c). A digital microscope with computer software was applied to observe and measure the surface crack width. Five locations were marked along the crack line (the distance between two marked locations was 20 mm) to fix the order and direction of the crack width measurement as illustrated in Figure 3.7b and Figure 3.7c. Crack widths were measured at 20 points along the crack line to obtain high accuracy of crack width measurement (see in Figure 3.7d).

By using a digital microscope with a computer software (see in Figure 3.9), initial crack widths were measured at 3 days of age, then time-dependent crack widths were measured at 7, 14, 21, 28, 35, 42, 49, 56, 63, 70, 77, 84, and 91 days of age. After having recorded the crack width of samples at each age, the crack-closing rate was computed using equation (3.1). The crack-closing ratio quantitatively indicates the decrease of crack width of the sample at a specific age. The surface crack-closing ratio $\beta_{(t)}$ quantitatively demonstrates the changing in the surface crack width at a specific time t, $\beta_{(t)}$ is defined as:

$$\beta_{(t)} = \left(1 - \frac{CW_{(t)}}{CW_i}\right) \cdot 100\% \tag{3.1}$$

where CW_i is initial crack width (mm) and $CW_{(t)}$ is crack width after t days of age (mm). Therefore, $\beta_{(t)}$ = 0% means no healing while $\beta_{(t)}$ = 100% means complete healing.

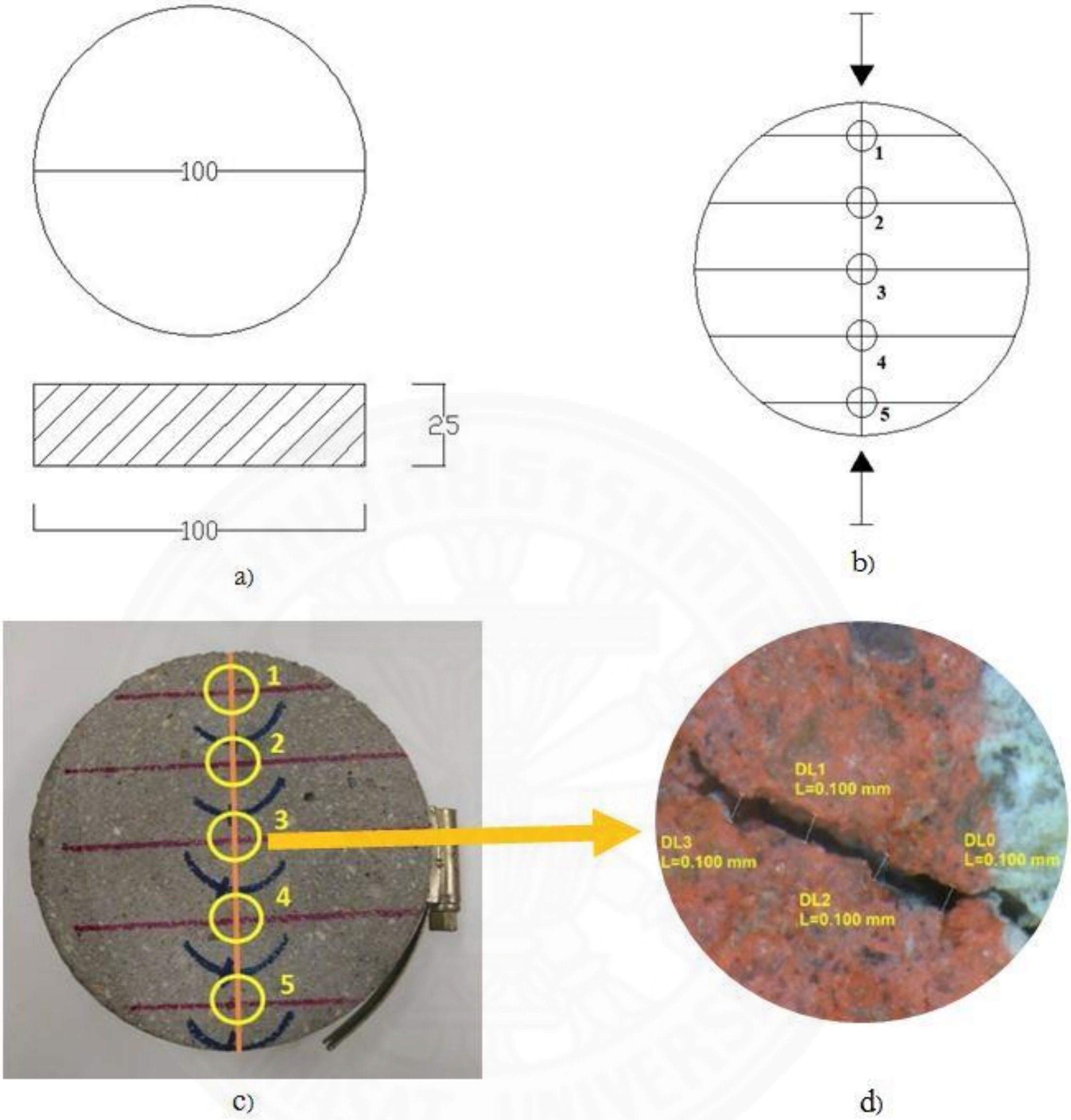


Figure 3.7 Mortar specimen for crack closing rate measurement, (a) Dimension of the specimen (mm), (b) Creating and marking crack, (c) Specimen with a stainless ring clamp, (d) Four minor points in one marked major point

Figure 3.8 Stainless ring clamp.

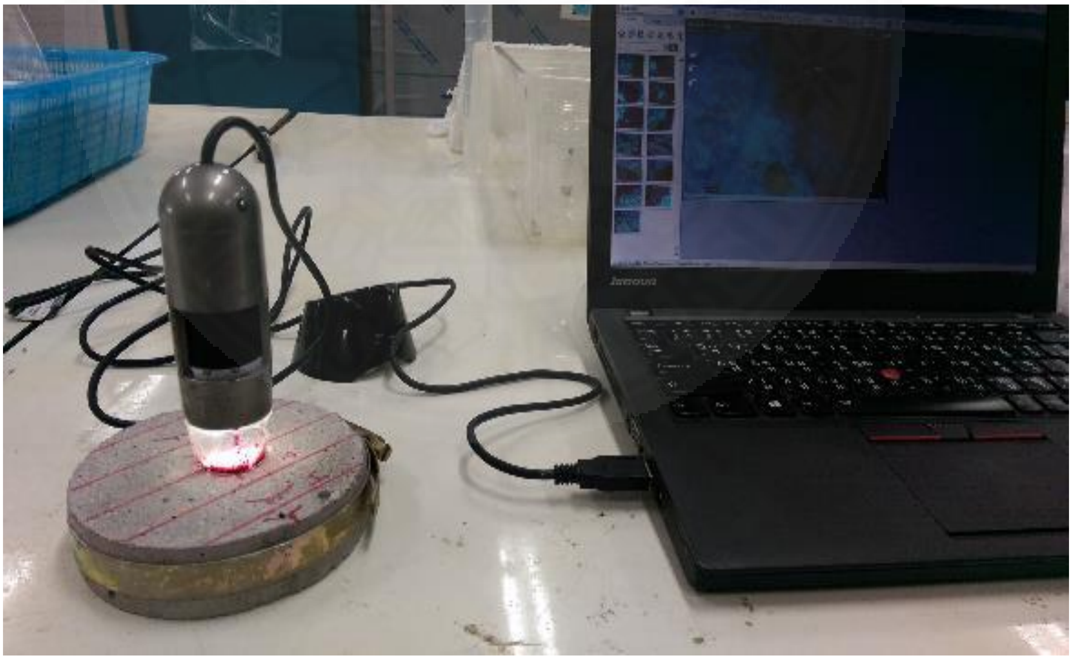


Figure 3.9 Using digital microscope with computer software to measure crack width.

3.3.3 Water permeability test

In order to confirm the self-healing performance of mortars, the water permeability test was also carried out simultaneously with the surface crack closing rate test. The water permeability test was carried out according to Western Australia WA625.1 standard (based on DIN 1048). Samples were prepared in the same fashion as those of the crack closing rate test. After introducing the crack width of 0.1 mm, they were coated with an epoxy layer of 25 mm in thickness and 25 mm in width on the side surface (Figure 3.10), then cured in air or water. Samples were immersed in water to obtain a saturated condition before installation to the permeability cell (see Figure 3.11) and applying water pressure (see Figures 3.13 - 3.18). After that, a water cylinder column (1500 mm in height, 8 mm in diameter) was installed (see Figure 3.15) and 0.5 Bar of water pressure was constantly applied to each specimen (see Figures 3.16 – 3.18) until the height of water in the column dropped from 50 cm point to 0 cm point. Time for the water column drop from the 50 cm point to 0 cm point was recorded in order to calculate the flow rate of water following the equation (3.2). The water-permeability testing device is shown in Figure 3.12.

After conducting the experiment, time for 50 cm of water column to run out was recorded and used to calculate the water flow rate by equation (3.2).

$$Q_{(t)} = \frac{V_w}{\Delta_t} \quad (3.2)$$

Where:

$Q_{(t)}$ = Water flow rate (m^3/s).

V_w = The volume of 50 cm of water column (m^3) flowing through the sample within a period of time, Δ_t (s).

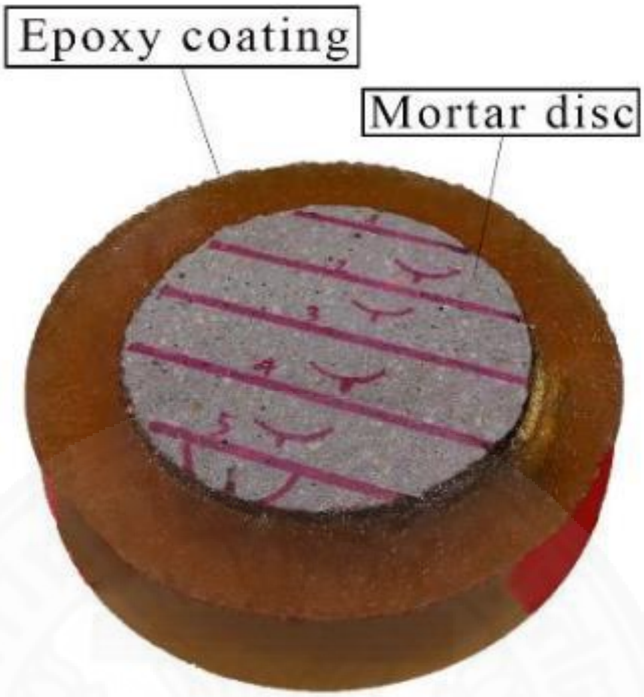


Figure 3.10 Water permeability specimen.

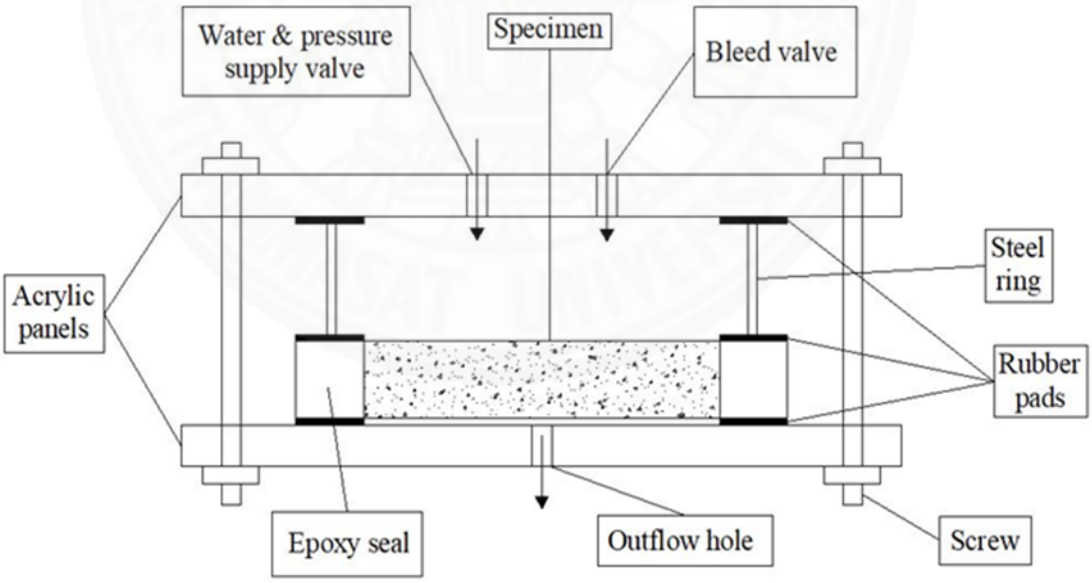


Figure 3.11 Water permeability cell.

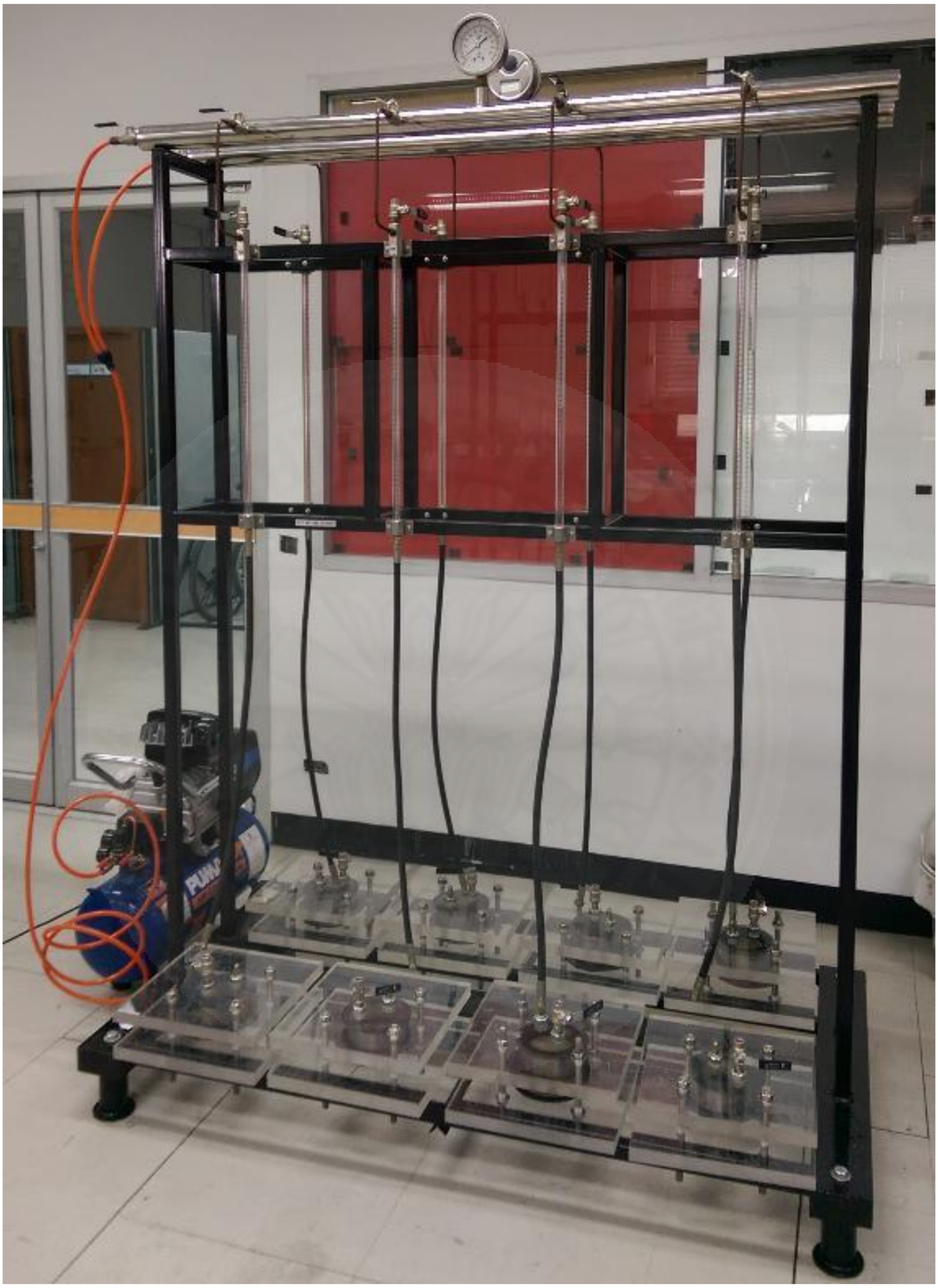


Figure 3.12 Water permeability testing device.

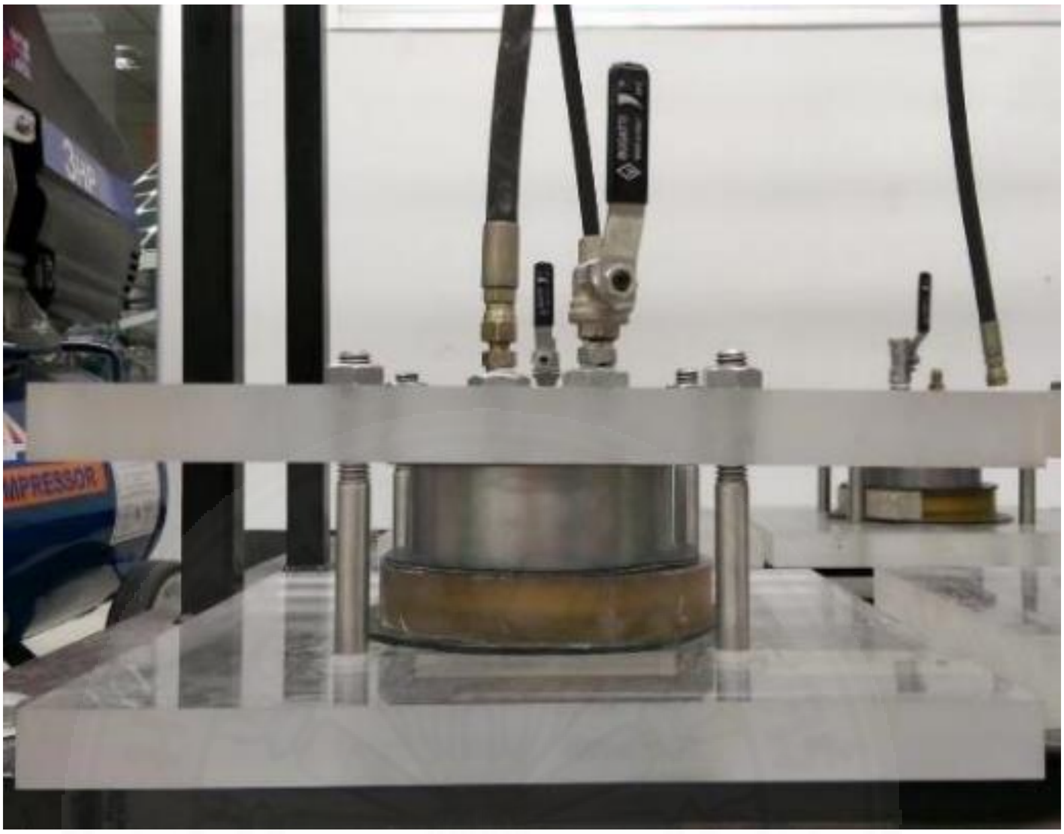


Figure 3.13 Install sample into water permeability cell.

Figure 3.14 Fully fill water into the water permeability cell.

Figure 3.15 Fully fill water into the water level measurement pipe.

Figure 3.16 Pressure regulator.

Figure 3.17 Pressure gauge.

Figure 3.18 Pressure lock.

CHAPTER 4
RESULTS AND DISCUSSIONS

According to the previous researches, there are many factors that may affect the self-healing ability of mortars which were investigated and reported. However, the studies on effect of various types of fly ash and bottom ash on the self-healing ability of mortars are still lacking and it needs to be studied. Therefore, this study focuses on investigating the effect of fly ash and bottom ash on the self-healing ability of mortars in many aspects.

The effects of many factors, such as type and amount of expansive additive, curing conditions, types of fly ash, and bottom ash, were clarified in terms of self-healing behavior of mortars. The effects of these kinds of factor were described by surface crack closing ratio and water flow rate that were used as indicators of self-healing ability.

4.1 Effect of expansive additive on self-healing ability of mortar

4.1.1 Effect of expansive additive type on self-healing ability of mortar

The effect of two types of expansive additive (EA and ET) with the same dosage (6%) was investigated. The results are shown in Figure 4.1. The results show that with different types of expansive additive, the self-healing behavior of mortar is different.

Figure 4.1 indicates that the surface crack closing ratio of EA6 sample, with EAA expansive additive, is higher than that of ET6, with EAT expansive additive, at all ages. The surface crack of EA6 sample was completely closed in 35 days, while the surface crack of ET6 sample needs 49 days to be fully closed. Chatchawan (2018) reported that the free expansion of mortar with EAA is higher than that of mortar with EAT. His research used the same materials, and the mix proportions were mostly similar to the tested mixes in this study except for the smaller amount of expansive additive (5% of EA was used in his study instead of 6% used in this study). He also stated that with the same level of expansion, mixtures with EAT produced a larger amount of $Ca(OH)_2$ while mixtures with EAA generated a much larger amount of ettringite. Therefore, mixtures with EAA showed more expansion with the larger amount of ettringite than mixtures with EAT did, which resulted in the better self-healing ability of mortars with EAA when compared with mortar with EAT.

On the other hand, this result could be related to the difference of total free lime content and total SO_3 content in EA6 and ET6 mixtures. The total free lime content and the total SO_3 content in EA6 mixture (13.64 % of free lime, 21.23 % of SO_3), with EAA expansive additive mixtures, are higher than those in ET6 mixtures (13.34 % of free lime, 16.67 % of SO_3), with EAA expansive additive. The higher amount of total free lime content and total SO_3 content can result in the larger the expansive products to fill cracks. Therefore, the crack closing ratio of the EA6 mixture is higher than that of the ET6 mixture. The correlation between total free lime content as well as total SO_3 content and the crack closing ratio is confirmed in Figure 4.8a and Figure 4.8b. The higher the total free lime content and total SO_3 content, the higher the crack closing ratio. Then, expansive additive type A (EAA) is more effective on the crack self-closing ability of mortar than EAT.

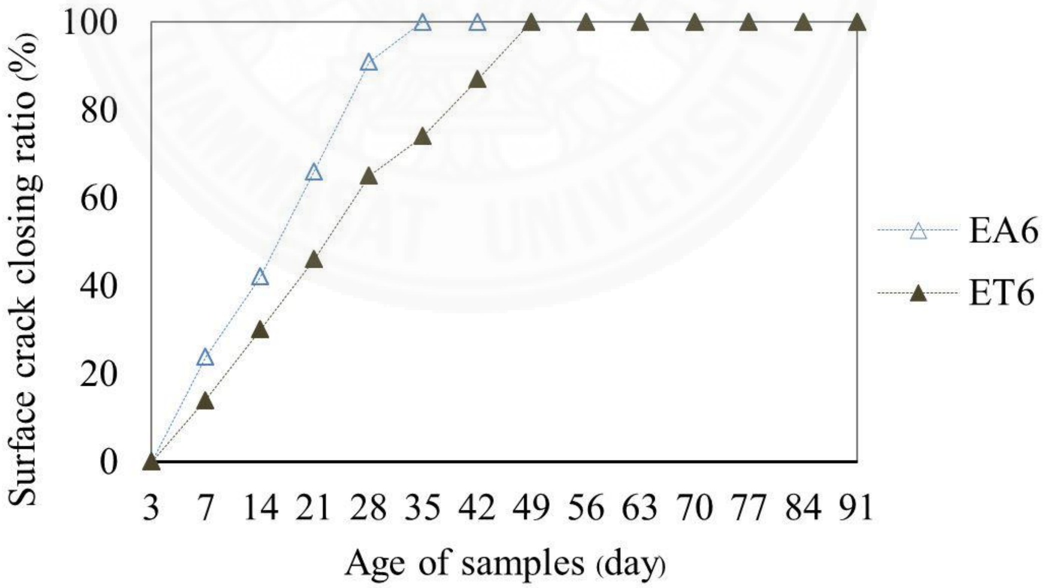


Figure 4.1 Surface crack closing ratio of mortar samples with different types of EA.

4.1.2 Effect of expansive additive amount on self-healing ability of mortar

With different dosages of the expansive agent (0%, 4%, and 6%), pre-cracked mortars with 0.1 mm of initial surface crack width were prepared to evaluate the surface crack closing ratio.

The surface crack closing ratio results are illustrated in Figure 4.2. It is clearly observed that the higher the amount of expansive additive, the faster closing of the crack. The surface crack closing ratio of the mortar sample without an expansive agent at the age of 91 days is 67.8%, which means the crack healing process slowly happens. Meanwhile, the surface crack closing ratio (SCCR) of mortar sample with 4% of expansive additive reaches 100% at the age of 56 days, and mortar sample with 6% of expansive agent completely healed crack even faster in 35 days. This is the result of the relationship between the expansive dosage and the expansive products especially ettringite. The higher the dosage of expansive additive, the larger the amount of ettringite amount is formed. Therefore, the self-healing ability of mortar is improved with the increase of expansive additive dosage.

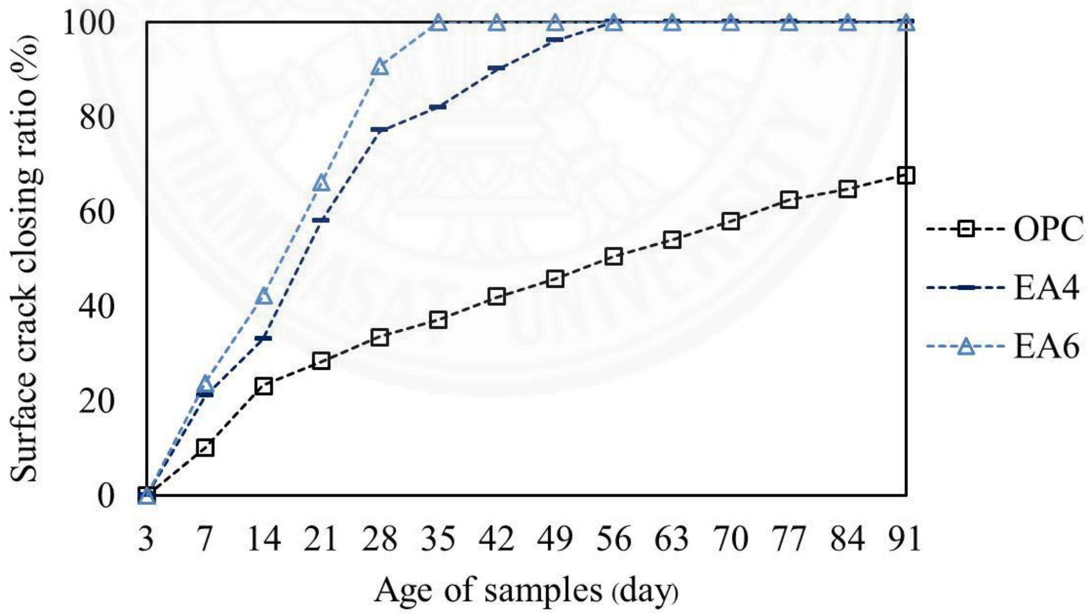


Figure 4.2 Surface crack closing behavior of mortars with different dosages of expansive additive.

4.2 Effect of curing condition on self-healing ability of mortar

Among various factors, curing condition is one of the major factors that strongly influences the self-healing of mortars. Three curing conditions were applied to observe the self-healing behavior i. e. water curing, air curing, and wet-dry curing. In Figure 4.3, the results show that with the same mixture, samples cured in water were completely closed in 35 days. Surface crack closing ratio of sample cured with the wet-dry cycle method reaches 93% at the age of 91 days, while the surface crack closing ratio of the sample kept in air achieved only 55% at the same age. The results confirm that submerging in water is the most effective curing method, followed by the wet-dry cycle, and the least effective method is air curing.

Water is required to promote the reaction that leads to the higher speed of surface crack closing ratio of mortar causing the products to be generated faster than other curing conditions. In addition, crack can be smaller when mortar sample is kept in saturated condition because of swelling of the cement matrix. Moreover, particles can come from the liquid flowing through the cracks, they can be the particles of cement loosened from the surface of crack then flow carry them through the crack until they are stuck in the small part of the crack.

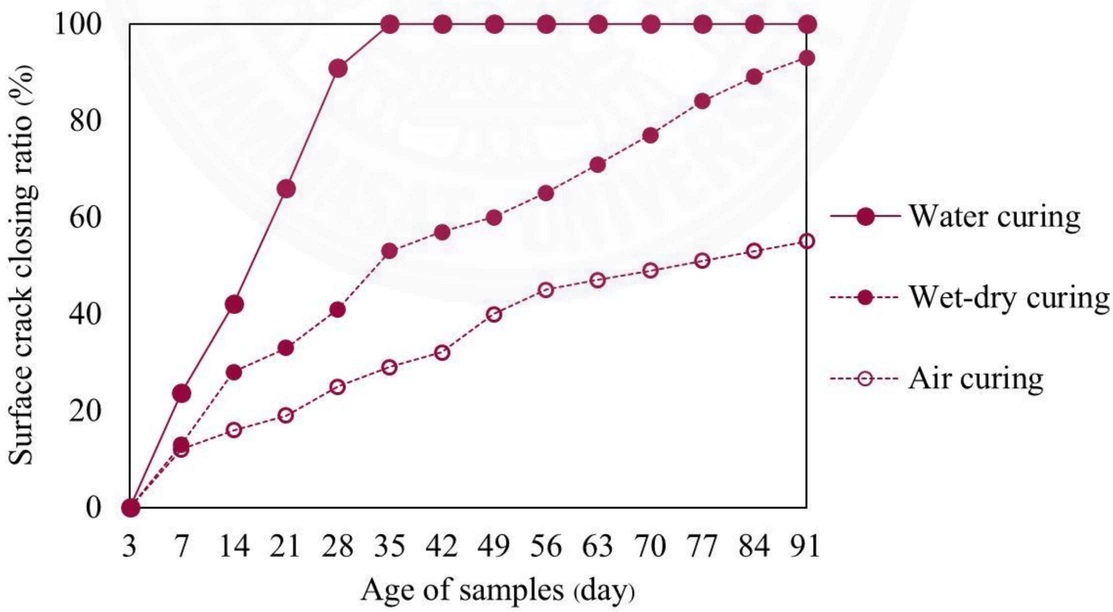


Figure 4.3 Surface crack closing ratio of expansive mortar samples cured in different conditions.

4.3 Effect of fly ash on self-healing ability of mortar

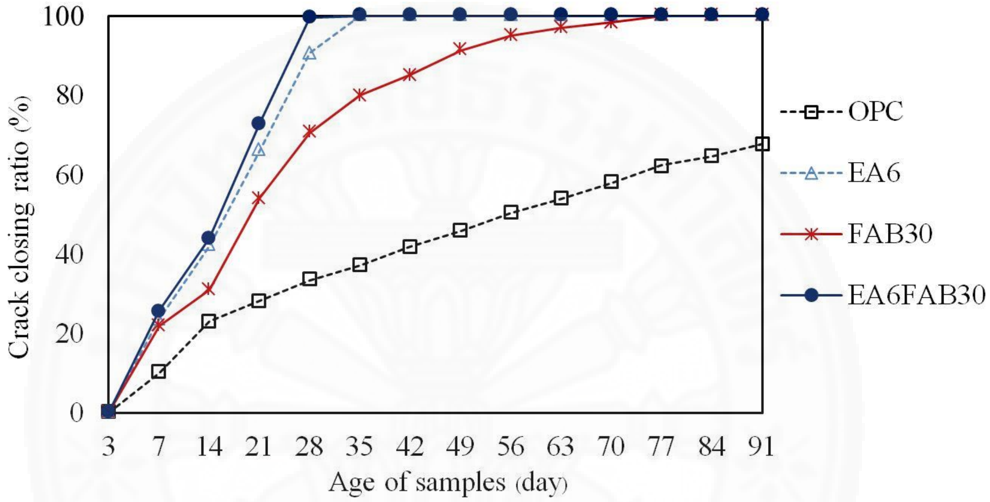
The effectiveness of fly ashes is experimentally studied by using surface crack closing ratio (SCCR) and water flow rate (WFR) as the indicators of self-healing ability. The results are demonstrated in Figure 4.4a and Figure 4.5a. The amount of CaO, SO_3, and free lime are the highest in FAB, followed by FAA. The amount of these chemical compositions are the smallest in FAR. The presence of different types of FA leads to different self-healing ability of mortars.

The surface crack closing ratios (SCCR) of pre-cracked mortars including each of the three types (FAA, FAB and FAR) of fly ash, with the initial surface crack width of 0.1 mm, are shown in Figure 4.4a. The figure indicates that the FAB30 mixture fully heals the surface crack in 77 days, followed by FAA30 mixture that completely closes surface crack at the age of 91 days. FAR30 mixture shows the lowest speed of surface crack closing where the crack healing does not complete within 91 days, and the SCCR at the age of 91 days only reaches 67.8%. Moreover, the surface crack closing ratio of the OPC mixture is much smaller than that of the fly ash mixtures, which means fly ashes can enhance the self-healing ability of mortars.

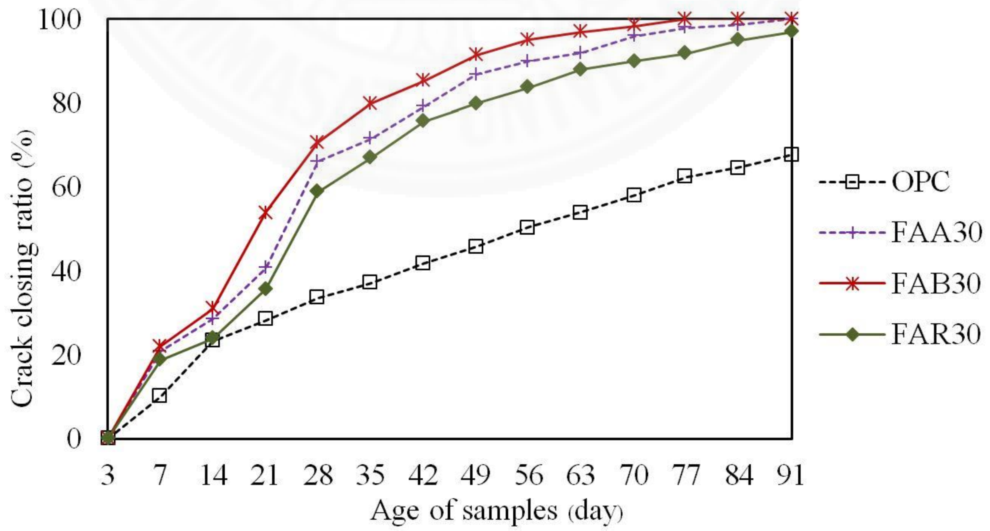
Besides that, the self-healing abilities of samples with the three types of fly ash are presented using the change of water flow rate of pre-cracked samples. The tests were conducted according to the Western Australia WA625.1 standard (based on DIN 1048). In Figure 4.5a, the water flow rate (WFR) changes over time of OPC mixture and fly ash mixtures are described. Similar to the surface crack-closing ratio, the water flow rate results of the FAB30 mixture shows the shortest healing time followed by FAA30 and then FAR30 mixtures.

Figure 4.4b and Figure 4.5b illustrate the self-healing ability with the presence of expansive additive and fly ash. Figure 4.4b shows the crack-closing ratio of OPC mortar and expansive mortar with and without fly ash. It indicates that the crack-closing ratio of mortar remarkably increases by using the expansive additive. When using both expansive agent and fly ash in the mortar, the crack-closing ratio becomes much higher than that of the mortar with EA only. The results clearly demonstrate that EA6 sample completed crack closing at the age of 35 days, and EA6FAB30 sample did it in only 28 days. Figure 4.5b describes the same tendency of self-healing behavior under the effect of expansive agent and fly ashes. In this figure, the EA6FAB30 mixture shows the smallest flow rate, followed by EA6 mixture, while the flow rate of the OPC mixture is the largest.

This study proposed a hypothesis for the mechanism of self-healing ability of mortars. It is believed that ettringite product fully filled crack area and this is the main mechanism of the self-healing ability. Generally, hydration products such as calcium hydroxide, ettringite and others can be found inside the concrete/ mortar samples. If these products occur inside the crack area, they will fill the crack as shown in Figure 4.6. In this research, ettringite is reported as a product formed by the reaction of fly ash, especially fly ash that contains high free lime and SO_3. For regular fly ashes which contain low amount of free lime an SO_3, cracks can be filled by the pozzolanic products formed by the pozzolanic reaction that progresses in long term. In different circumstances, for cracks introduced at the long term, expansive additive may not help because its reaction stops after only 3-7 days. This phenomenon is clearer in the expansive mortar without fly ash. By using image analysis (RGB method), Hosoda, Higuchi, Eguchi, Yoshida and Aoki (2012) confirmed that 100 μm and 200 μm cracks of 34-year precast concrete poles were filled by calcite and ettringite. Ettringite was found along the cracks and at the interface area of the cracks and the matrix. When cracks present in the matrix, the cement paste matrix is the source providing major constituent elements of the ettringite such as Ca^{2+}, Al^{3+}, and SO_4^{2-} to formed ettringite. In addition, from the study of Danner, Jakobsen, and Geiker (2019), ettringite formation was denoted as one of two main mechanisms of the self-healing ability of cracks in 25-year concrete exposed in marine condition. A mineralogical investigation was conducted on paste samples at an age of 7 days by XRD analysis method by (Chatchawan, 2018) (see Figure 4.7). Nguyen, Chatchawan, Saengsoy, Tangtermsirikul, and Sugiyama (2019) used the same materials (OPC cement, expansive additive, and fly ash) as this study, but the mixes are slightly different. They addressed the presence of ettringite in an expansive paste, a fly ash paste, and an expansive paste with fly ash. This is the explanation for the self-healing ability of expansive mortar that the use of fly ash helps to significantly improve the self-healing ability of expansive mortars and OPC mortar. As mentioned, their mix proportions were mostly similar to the mixes tested in this study except for the amount of expansive additive used in EA mortar with fly ash (5% of expansive additive was used in their study instead of 6% used in this study), however the tendency of the result is expected to be similar excepts that the amount of ettringite product of EA6 mix should be higher than that in EA5 mix. The S-H enhancement of expansive mortar mixture containing FA due to the amount of expansive products. FAA and FAR show the similar effect as the FAB does, however, FAB is the most effective one.

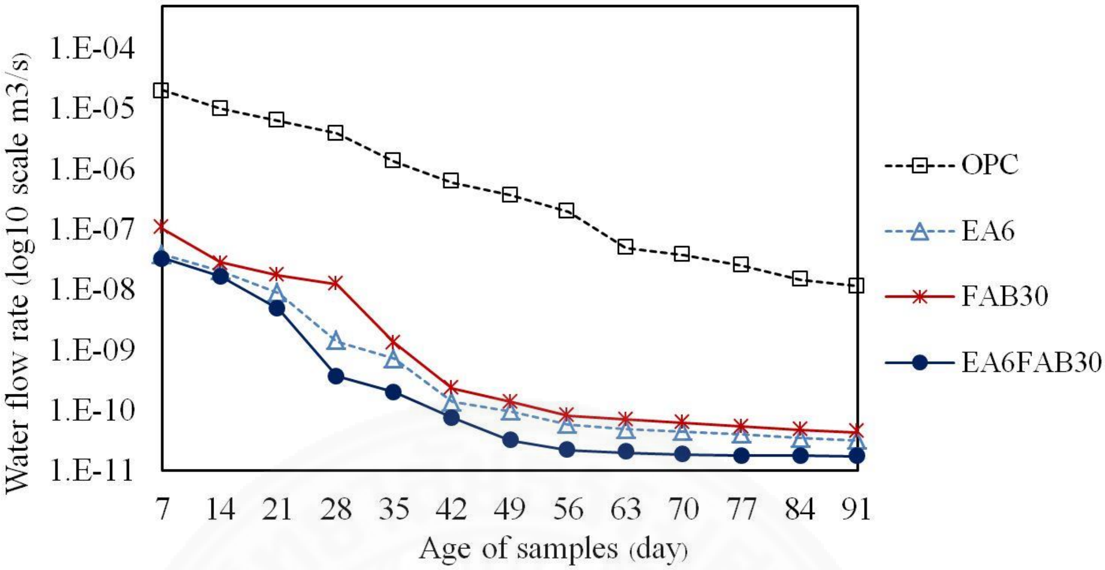


a) OPC mortar and expansive mortars with and without fly ash.

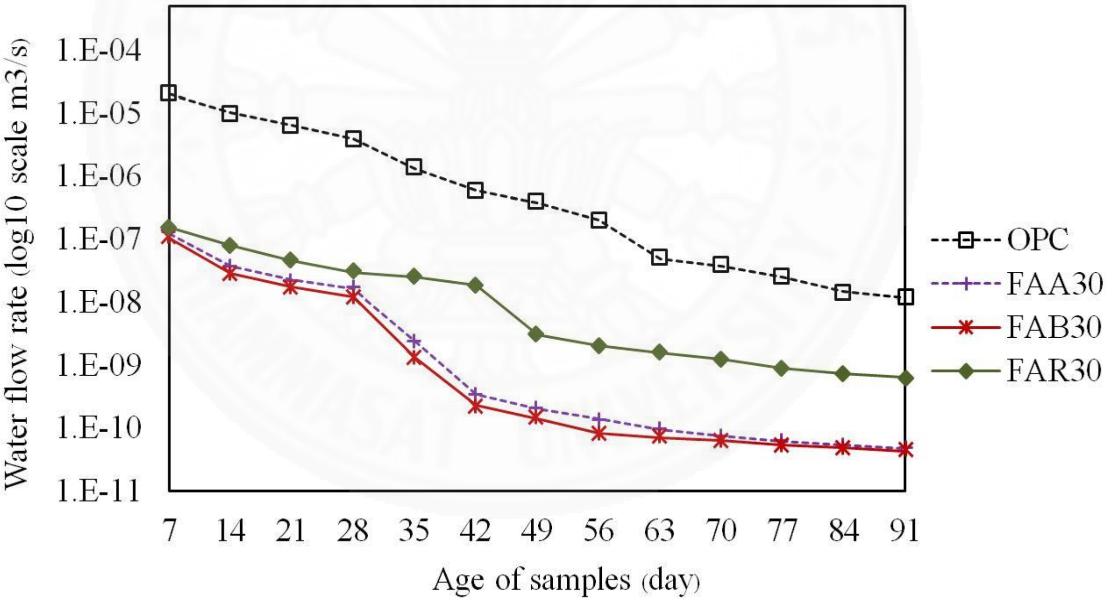


b) OPC mortar and mortar with different types of fly ash.

Figure 4.4 Crack closing ratio of mortars (in water curing condition).



a) OPC mortar and expansive mortars with and without fly ash.



b) OPC mortar and mortars with different types of fly ash.

Figure 4.5 Water flow rate of mortars (in water curing condition).

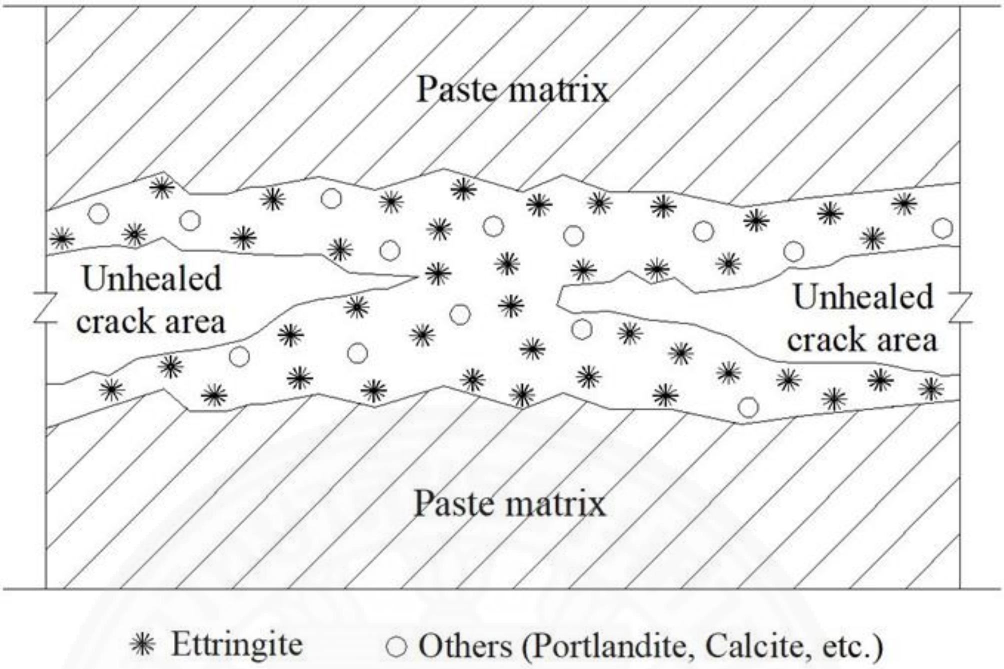


Figure 4. 6 Self-closing of crack due to the filling of expansive products.

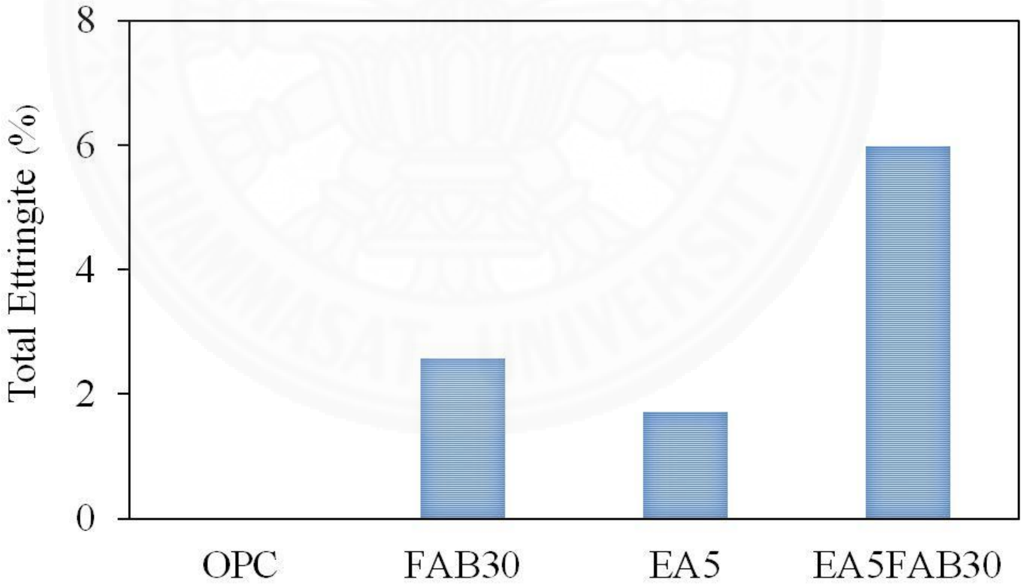


Figure 4.7 Results of ettringite amount in different mixtures (Chatchawan, 2018).

The CaO, free lime, and SO_3 contents in mortar mixtures were calculated to study the relationship between them and the self-healing behavior. The calculation was carried out following equations (3), (4), and (5).

$$F = \frac{(\%F_C \times W_C) + (\%F_e \times W_e) + (\%F_f \times W_f)}{100} \tag{3}$$

$$S = \frac{(\%S_C \times W_C) + (\%S_e \times W_e) + (\%S_f \times W_f)}{100} \tag{4}$$

$$C = \frac{(\%C_C \times W_C) + (\%C_e \times W_e) + (\%C_f \times W_f)}{100} \tag{5}$$

Where: F – Total free lime content in the mixture (kg/m^3)

$\%F_C$, $\%F_e$, $\%F_f$ – Free lime content in OPC I, EA and FA, respectively (%by weight)

S – Total sulfur trioxide content in the mixture (kg/m^3)

$\%S_C$, $\%S_e$, $\%S_f$ – Sulfur trioxide content in OPC I, EA and FA, respectively (%by weight)

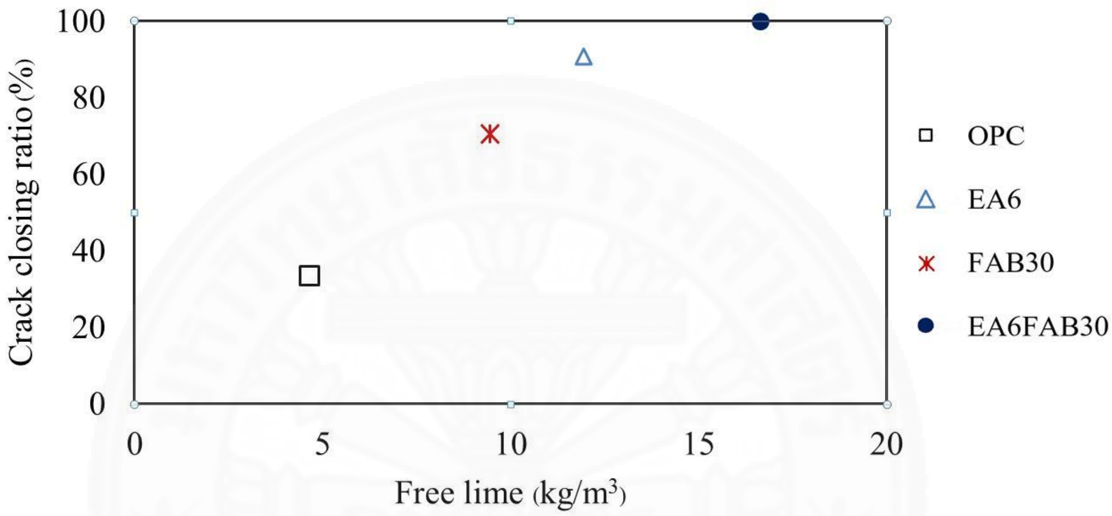
C – Total calcium oxide content in the mixture (kg/m^3)

$\%C_C$, $\%C_e$, $\%C_f$ – Calcium oxide content in OPC I, EA and FA, respectively (%by weight)

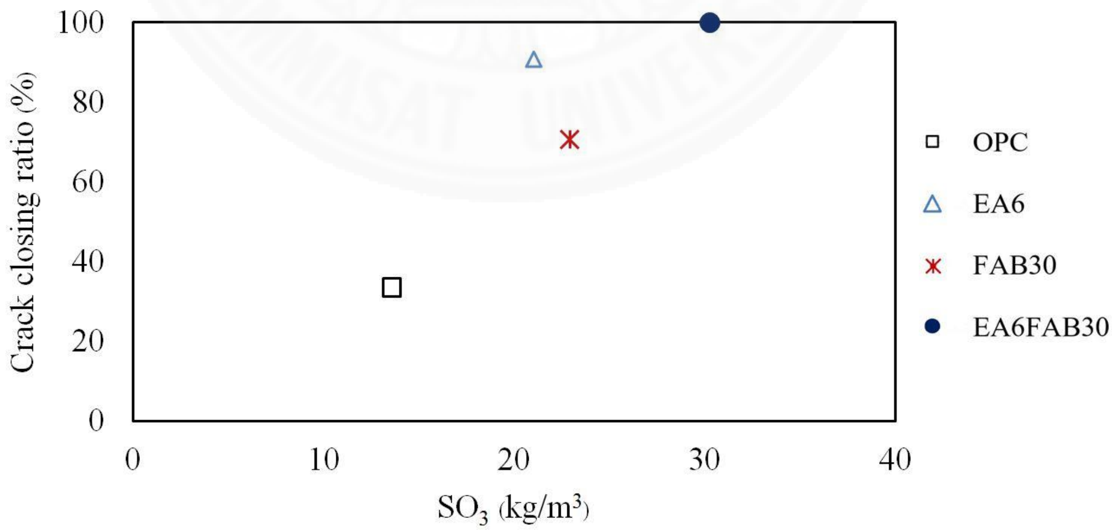
W_C, W_e, W_f – Weight of OPC I, EA, FA in 1 m^3 of mortar, respectively (kg/m^3)

Figure 4.8 illustrates the correlation between the CCR of samples at the age of 28 days and the total chemical compositions (SO_3, CaO, and free lime). In Figure 4.8a, the relationship between the CCR and the total free lime content is described. It is observed that the CCR increases with the larger free lime content. Figure 4.8b shows the correlation between the CCR and total SO_3 content. It indicates a similar trend. The higher SO_3 content leads to the improvement of crack healing. The CCR of EA6 mixture is higher than that of FAB30 mixture though SO_3 content in EA6 is smaller than that in FAB30 (1.89 kg/m^3), because free lime content in EA6 is higher than that in FAB30 (2.5 kg/m^3) and both free lime and SO_3 content affect the CCR of mortar.

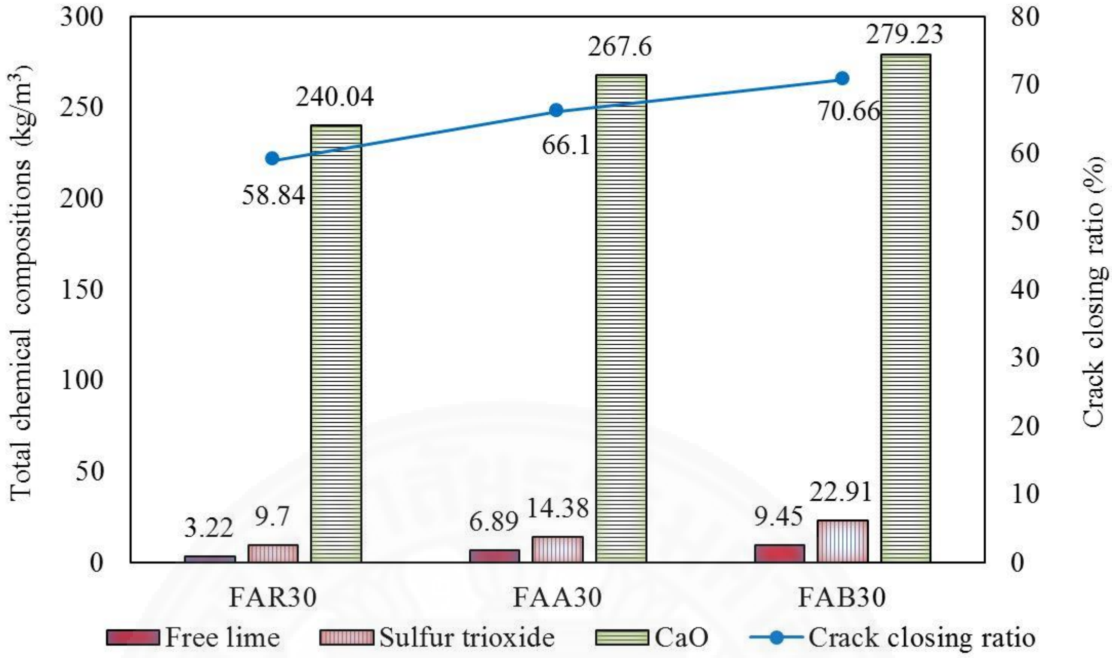
Nawaz, Julnipitawong, Krammart, and Tangtermsirikul (2016) investigated the behavior of the expansion of concrete with different types of FA, and the consideration about effect of CaO, free lime, and SO_3 content was denoted. They confirmed that fly ashes containing higher amount of SO_3 showed the higher expansion due to the internal sulfate effect by ettringite formation. The correlation between the CCR of fly ash mixtures and the total CaO content, total SO_3 content and total free lime content obtained in this study is demonstrated in Figure 4.8c. Fly ash mixtures perform a similar tendency that the higher total free-lime and total SO_3 content and also the higher total CaO content, the higher the crack closing ratio. Therefore, it can be confirmed that high CaO fly ashes which contain higher amount of SO_3 and free lime are more effective to enhance the self-healing ability of mortars.



a) Relationship between free lime contents and crack closing ratios of the tested mixtures.



b) Relationship between SO_3 contents and crack closing ratios of the tested mixtures.

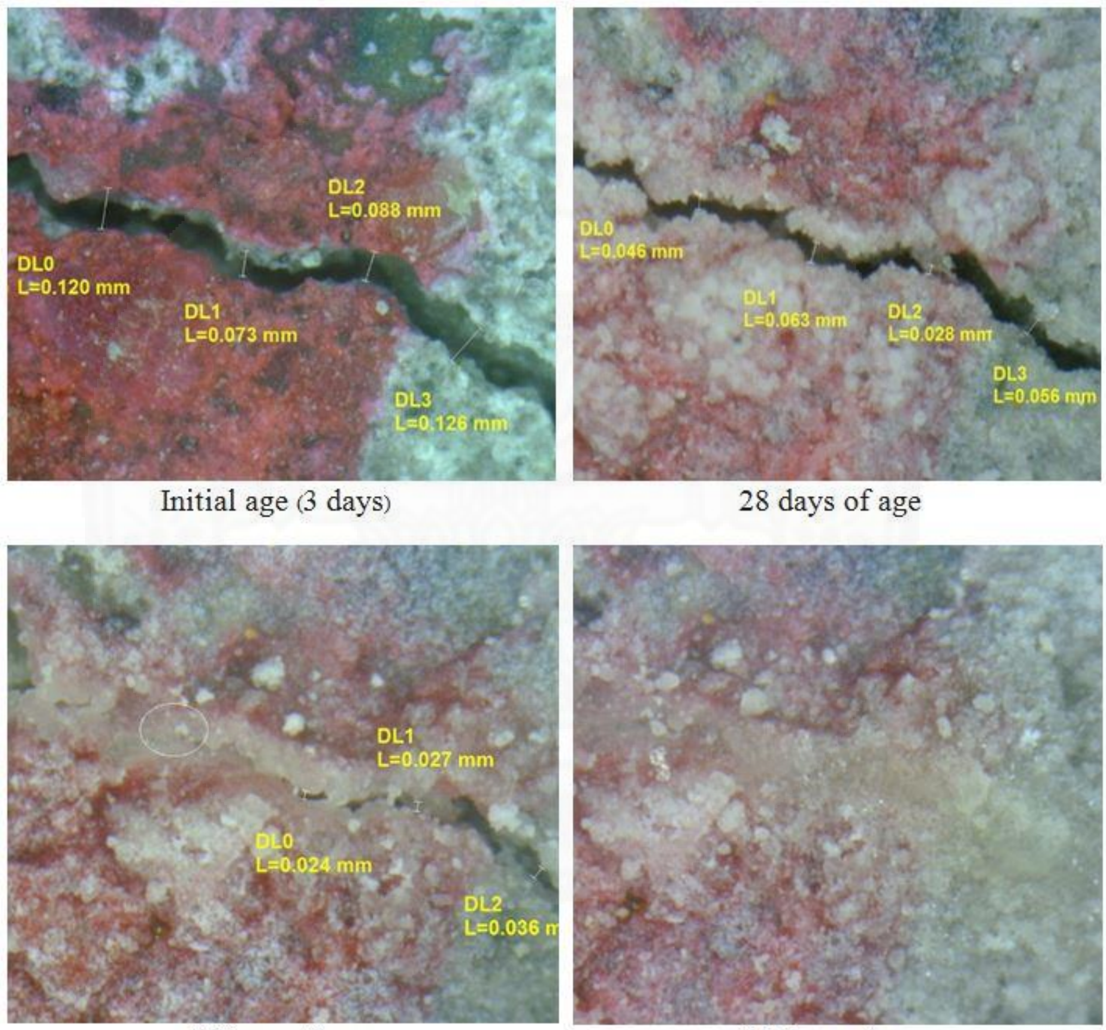


c) Relationship between total chemical compositions in the mixture and results of the crack closing ratio of fly ash mixtures at the age of 28 days.

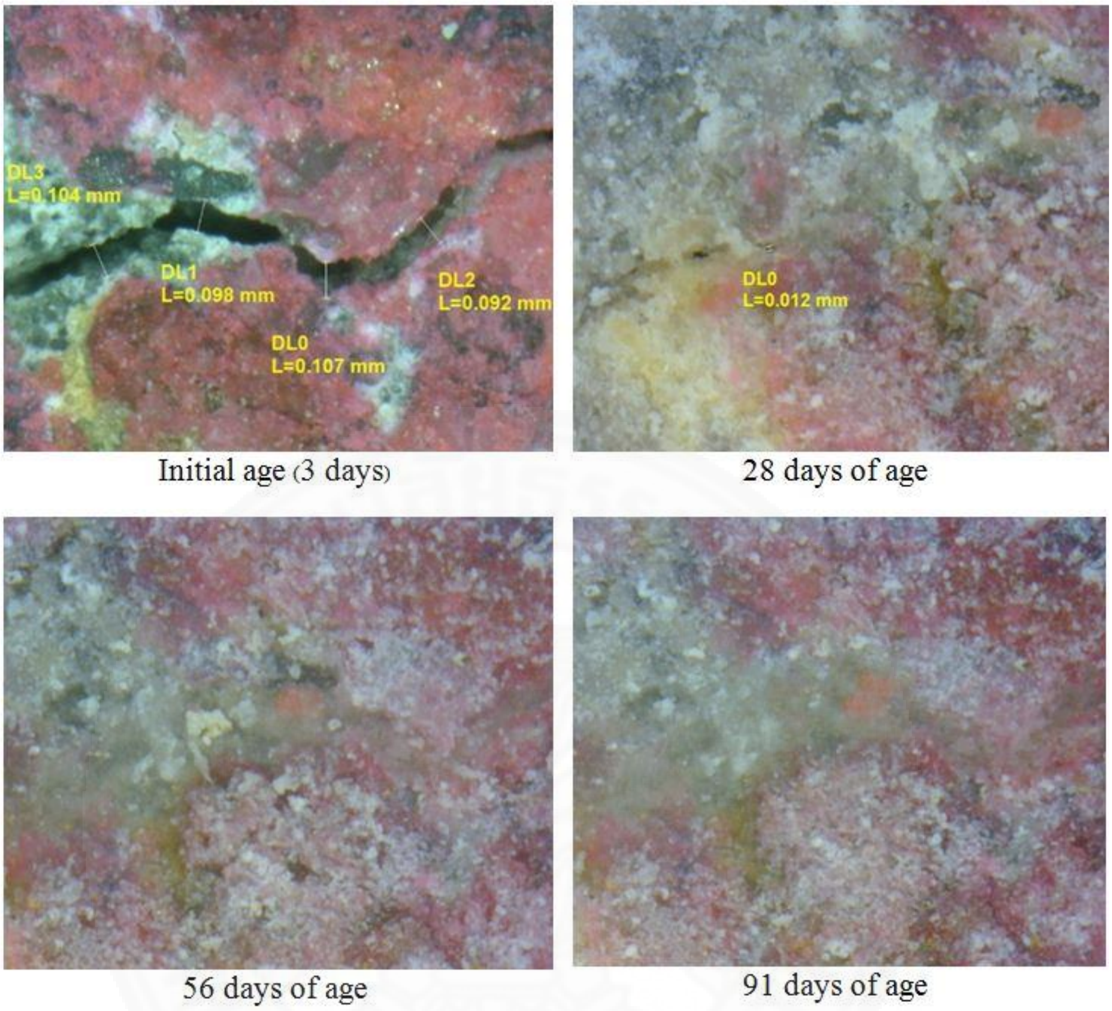
Figure 4.8 Relationship between total chemical compositions in the mixture and results of crack closing ratio of mixtures at the age of 28 days.

Figure 4.9 shows the close-up image of the surface crack closing of tested samples over time. The enhancement of surface crack width over time of mixtures was observed and measured by using the Dino Microscope device. In figure 4.9a, images of cracks of an expansive mortar sample at different ages (3 days of age, as well as a 28, 56, and 91 days of age), 0.1 mm of initial surface crack width, water curing) are illustrated. Figure 4.9b describes the crack images of an expansive mortar with FAB at age of crack introducing (3 days of age), as well as at 28, 56, and 91 days of age (0.1 mm of initial surface crack width, water curing). It is observed that fly ash type B dramatically enhances the self-healing of expansive mortar as previously mentioned.

In summary, the self-healing ability of fly ash mortar samples is improved significantly when using expansive additive and fly ash that contains high amount of calcium oxide, sulfur trioxide, and free lime. The higher amount of CaO, SO_3, and free lime, the better the self-healing ability. These compositions demonstrate major role in the self-healing process. During the hydration reaction, the expansive products which are calcium hydroxide and ettringite are formed. The expansive products have higher volumes when compared to their original products of reactions, leading to the expansion of mortar samples. Furthermore, when ettringite and calcium hydroxide are produced inside the cracks or nearby, they can fill and close the cracks finally.



a) EA6 mortar (in water curing condition).



b) EA6FAB30 mortar (in water curing condition).

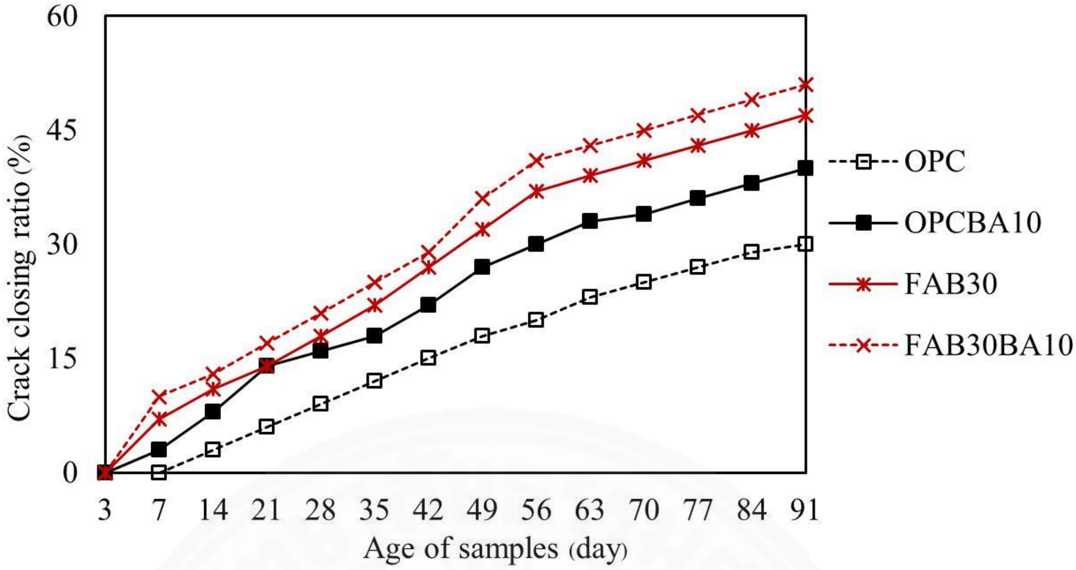
Figure 4.9 Crack closing of mortars at the initial (3 days), as well as at 28, 56, and 91 days of age (initial surface crack width = 0.1 mm) in water curing condition.

4.4 Effect of bottom ash on self-healing ability of mortar

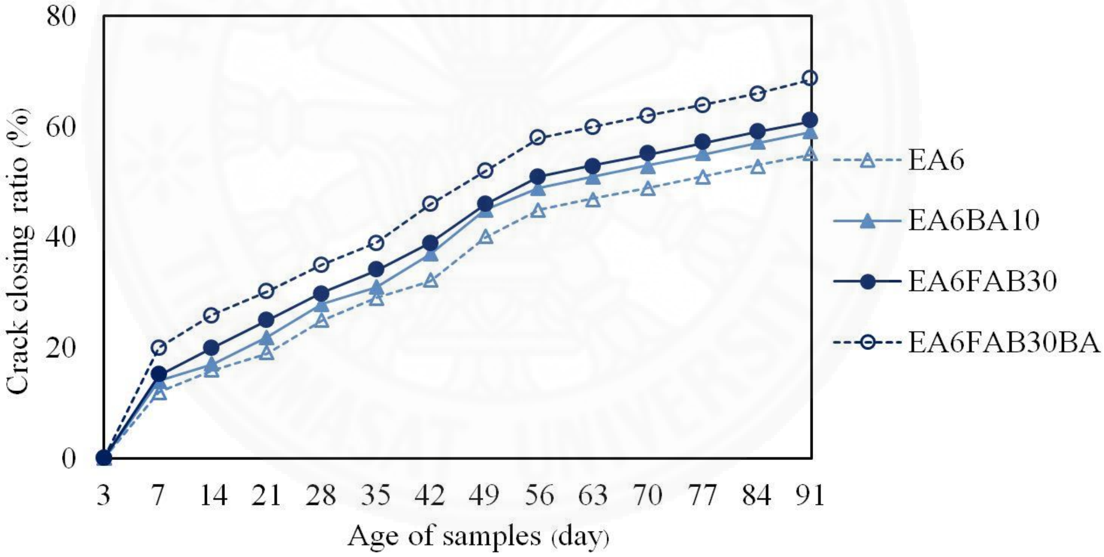
Figure 4.10 demonstrates the SCCR of mixtures with and without bottom ash during 91 days of age. The SCCR of OPC mixtures with and without bottom ash and the SCCR of fly ash mortars with and without bottom ash are demonstrated in Figure 4.10a. It shows that the crack healing of FAB30 sample is faster than that of the OPC sample. With the use of bottom ash, the SCCR of both OPC sample and fly ash sample are remarkably improved. At 91 days of age, the SCCR of OPCBA10 mixture is 10% higher than that of the OPC mixture, and the SCCR of FAB30BA10 mixture is 4% higher than that of the FAB30 mixture as well.

In Figure 4.10b, the SCCR of the expansive OPC mortars with and without BA, and expansive fly ash mortar with and without BA are illustrated. As mentioned in the previous discussion, the effect of FA on OPC mortar and expansive mortar remains a similar tendency though they were cured in an air curing condition. It shows that cracks in expansive mortar with fly ash are healed faster than cracks in expansive mortar without fly ash. Under the effect of internal curing of bottom ash, the SCCR of EA6BA10 mixture is 61% that is higher than 57% of the EA6 mixture. The SCCR of EA6FAB30BA10 at 91 days of age is 68.5% that is 7.5% higher than that of EA6FAB30 mixture.

Figure 4.11 demonstrates the flow rate of mixtures with and without bottom ash during the period of 91 days of age. The effect of BA on the water flow rate of the OPC sample and the fly ash sample is described in Figure 4.11a. There is an improvement in decreasing the water flow rate of OPCBA10 mixture when compared to OPC mixture, due to the use of bottom ash to provide internal curing water for the hydration reaction from inside the sample. The same behavior is recorded when the water flow rate of fly ash sample sharply reduces with the use of bottom ash. It is obviously confirmed that the self-healing of samples is remarkably enhanced with the use bottom ash due to the internal curing effect. Figure 4.11b shows the water flow rate of expansive mortar with and without fly ash under the effect of bottom ash. It indicates the similar effect of bottom ash on the water flow rate of expansive mortar with and without fly ash (mixtures with bottom ash show lower water flow rate).

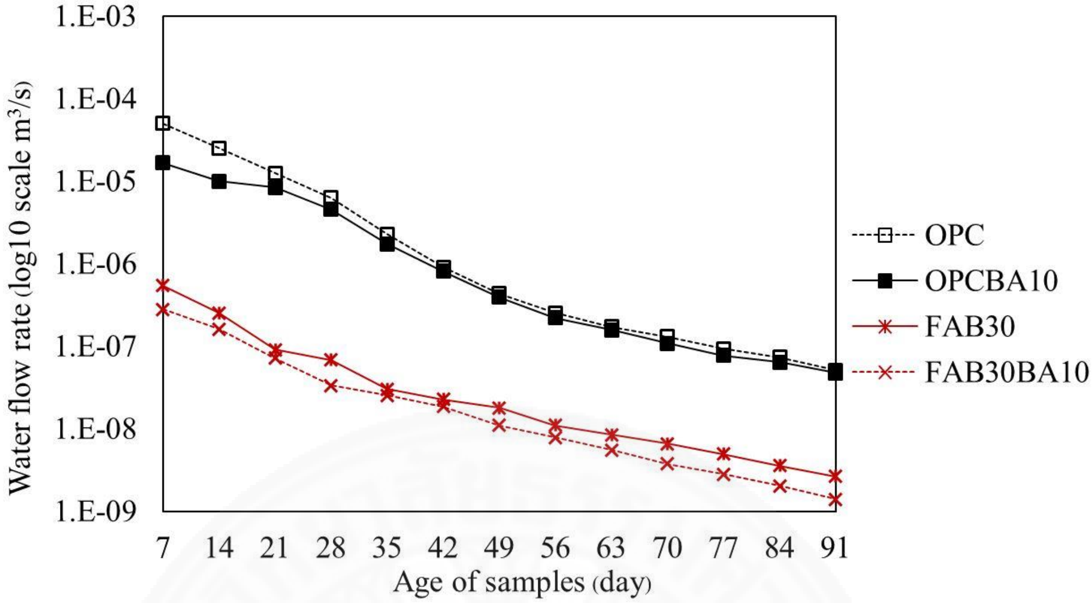


a) OPC mortar and fly ash mortar with and without BA.

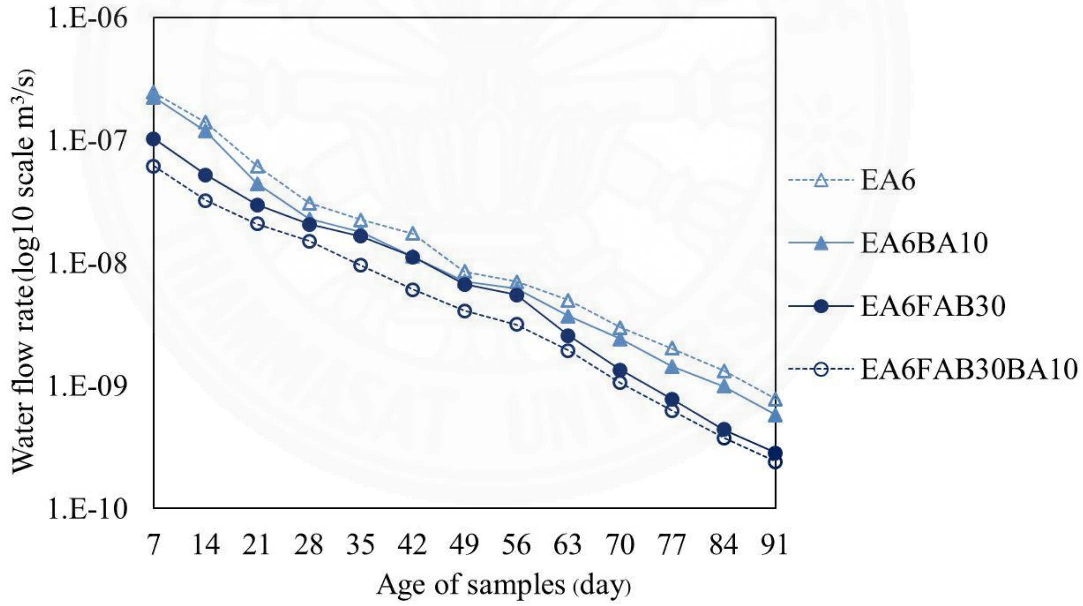


b) Expansive mortar with and without BA.

Figure 4.10 The crack closing ratio of mortars (in air curing condition).



a) OPC mortar and fly ash mortar with and without BA.



b) Expansive mortars with and without BA.

Figure 4.11 Water flow rate of mortars (in air curing condition).

The computer software and the Dino Microscope device were applied to observe the changing of surface crack width. Images of the surface crack of mixtures with and without bottom ash at various ages (3 days of age, as well as at 28, 56, and

91 days of age, 0.1mm of initial surface crack width, air curing) are shown in Figure 4.12, Figure 4.13, Figure 4.14, and Figure 4.15. Figure 4.12 illustrates images of surface crack of expansive mortar without bottom ash at different ages, while Figure 4.13 shows images of surface crack of expansive mortar with bottom ash at different ages. Images of surface crack of expansive fly ash mortar with and without bottom ash at different ages are respectively demonstrated in Figure 4.14 and Figure 4.15. It shows that using bottom ash as the internal curing material is effective in both expansive mortars with and without fly ash.

Due to the outcome of the internal curing effect, the positive role of bottom ash is recorded in all cases of mixture. When using concrete or mortar, pre-soaked bottom ash contains high amount of water, and it will provide water for the continuous reaction of binders in the concrete and mortar in the long term.

Normal water curing method provides curing water from the surface of the structures, which is a very simple and low-cost method. However, it is effective only at the surface of the concrete which is directly in contact with the curing water. For the inner part of mortar or concrete that is far from the surface especially low permeable concrete, normal water curing method is not effective.

On the other hand, internal curing technique is very effective for all locations and all types of structures. Some structures such as underground structures, water-retaining structures, and structures that are very difficult to cure, the internal curing seems to be the most effective curing method that helps structures develop and maintain the properties and durability, as well as the self-healing ability in the long term.

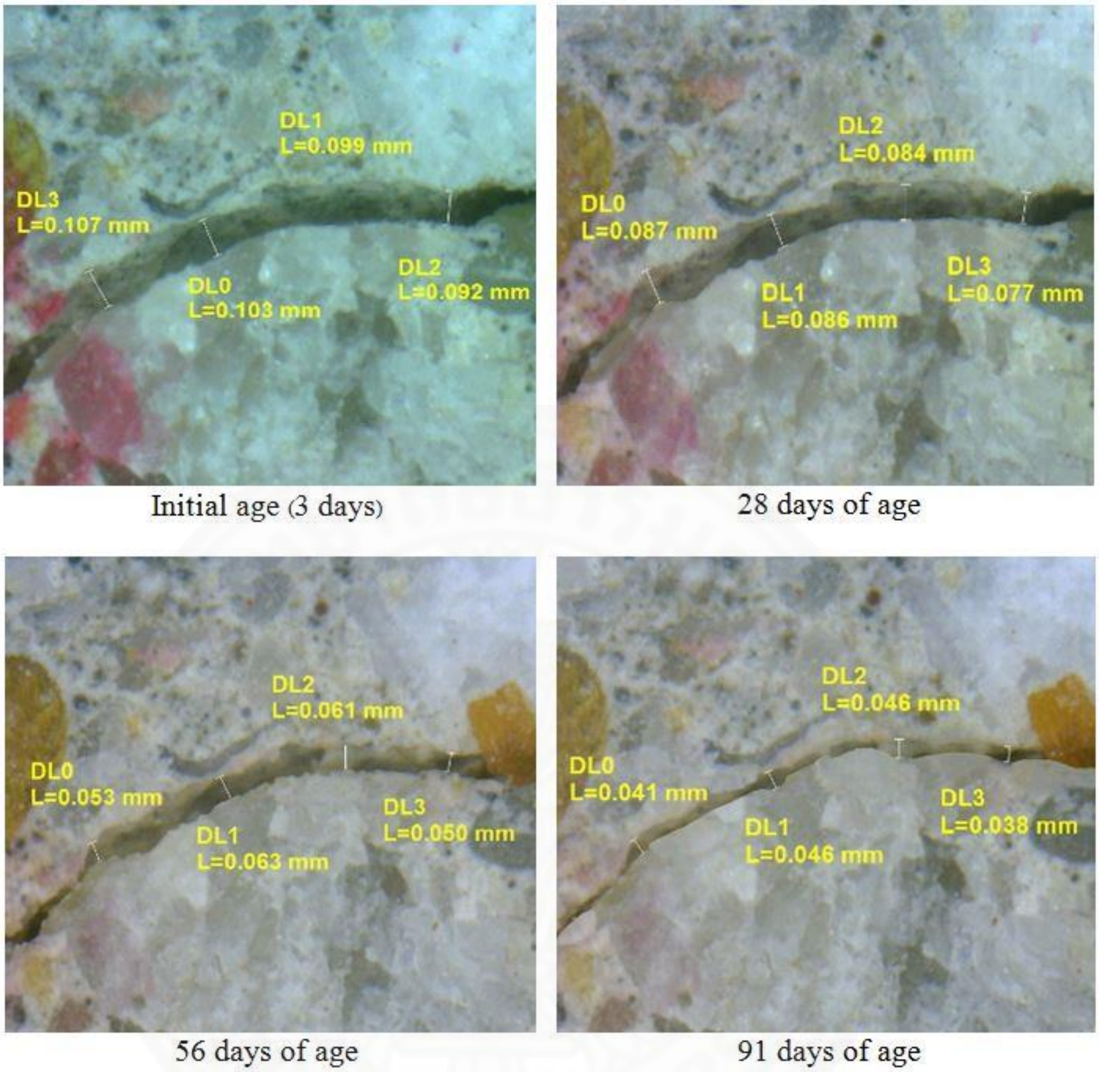
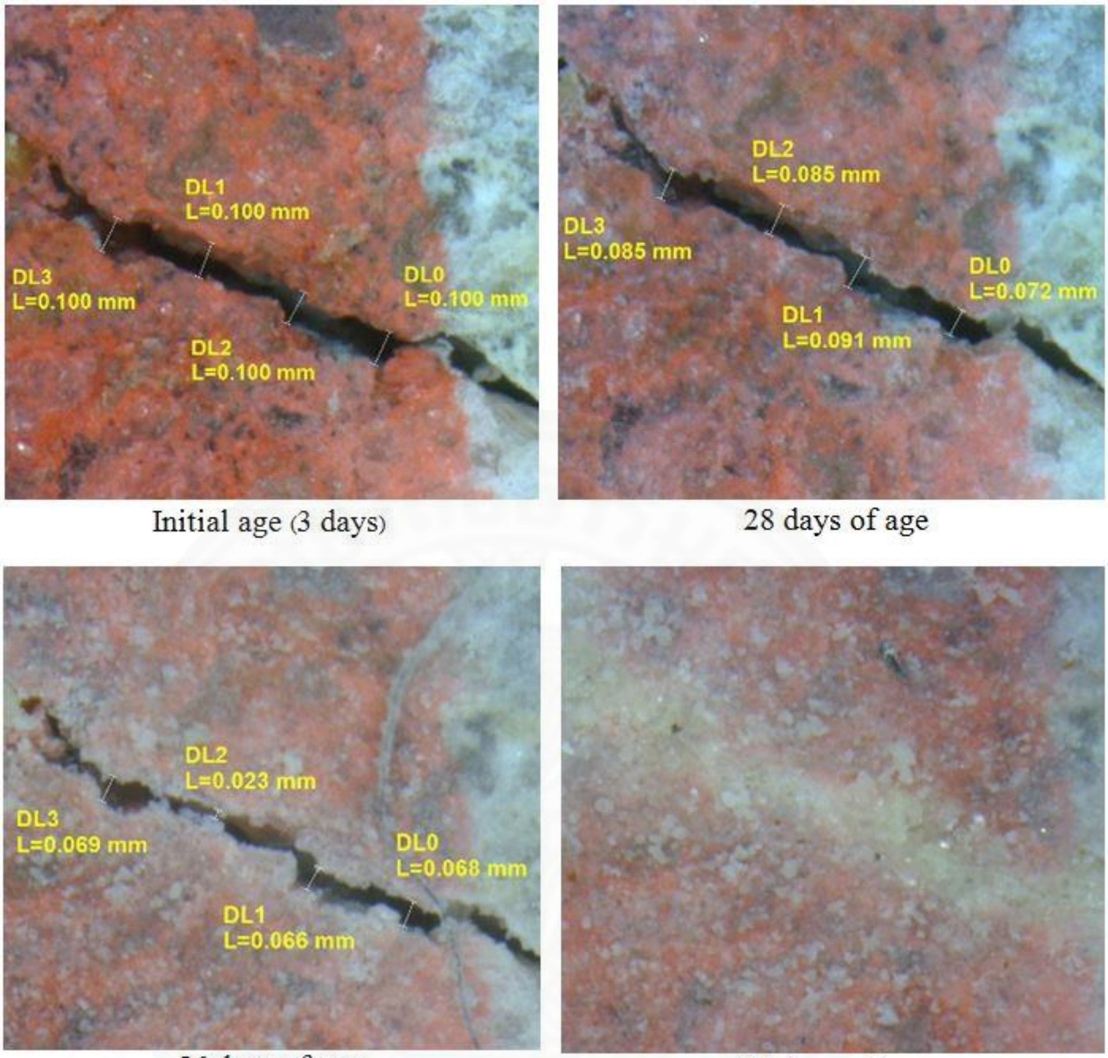


Figure 4.12 Crack closing of EA6 mortar at the crack introducing age (3 days), as well as at 28, 56, and 91 days of age (initial surface crack width = 0.1 mm) in air curing condition.



Initial age (3 days) | 28 days of age

56 days of age | 91 days of age

Figure 4.13 Crack closing of EA6BA10 mortar at the crack introducing age (3 days), as well as at 28, 56, and 91 days of age (initial surface crack width = 0.1 mm) in air curing condition.

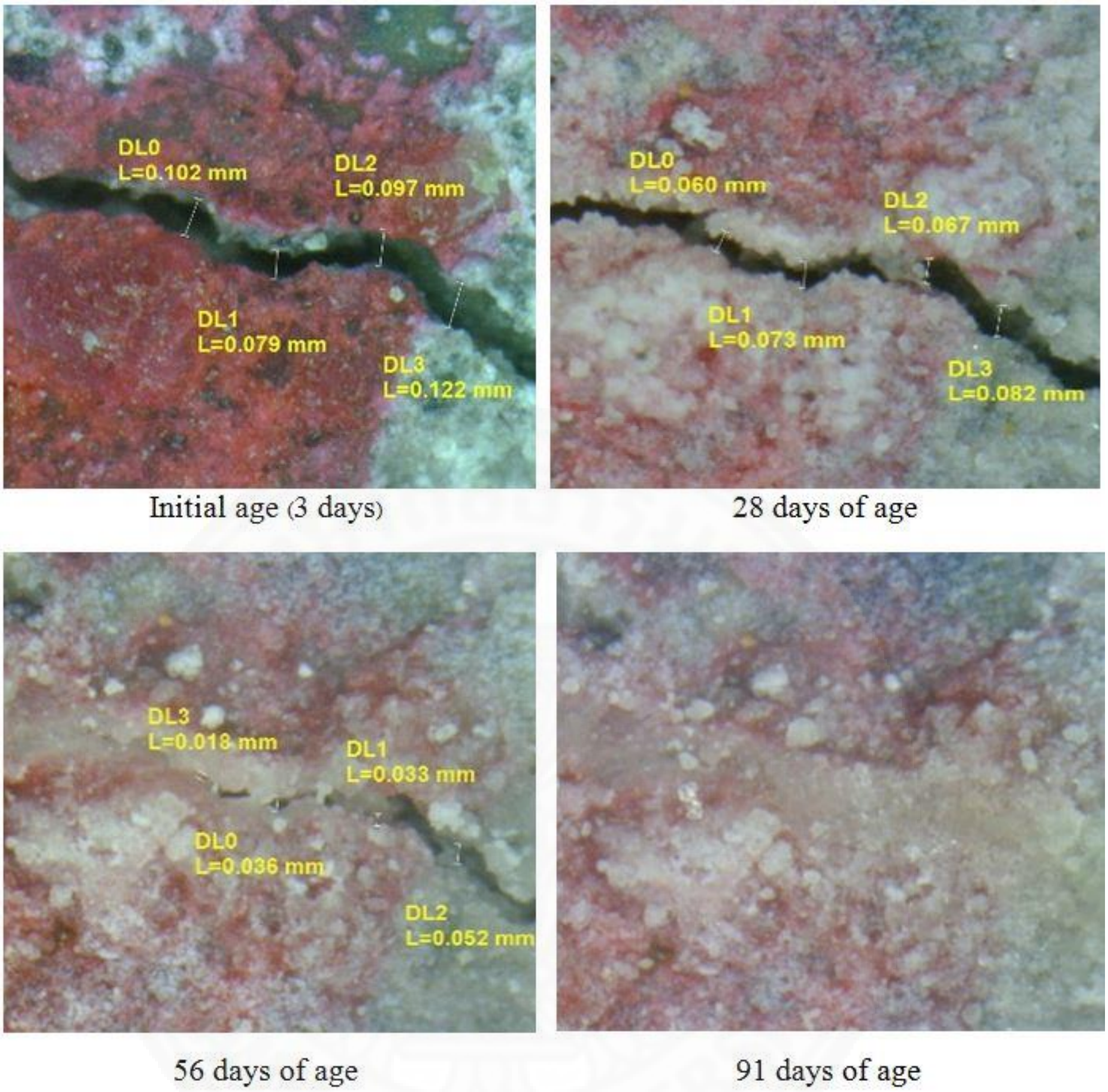


Figure 4.14 Crack closing of EA6FAB30 mortar at the crack introducing age (3 days), as well as at 28, 56, and 91 days of age (initial surface crack width = 0.1 mm) in air curing condition.

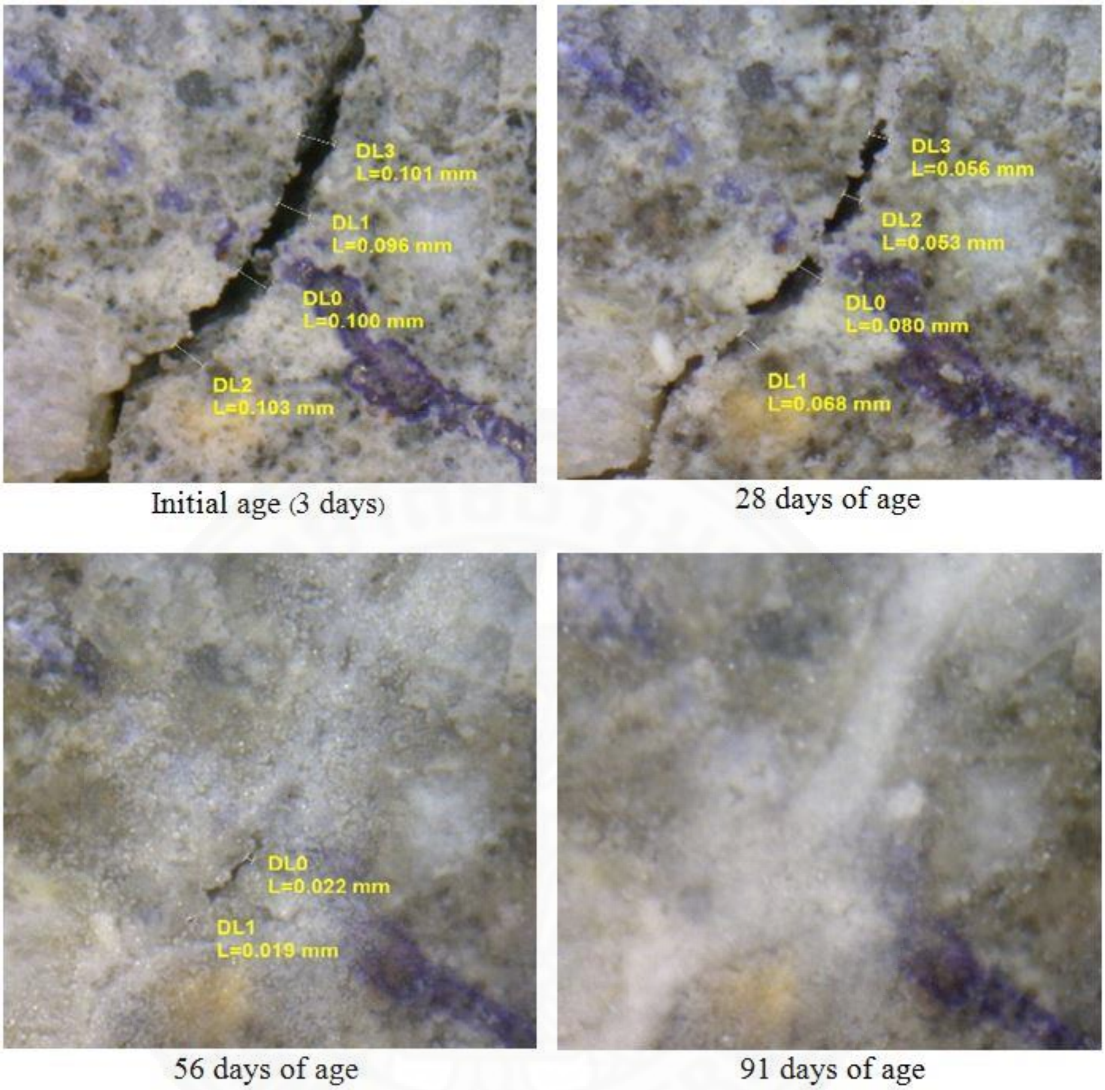


Figure 4.15 Crack closing of EA6FAB30BA10 mortar at the crack introducing age (3 days), as well as at 28, 56, and 91 days of age (initial surface crack width = 0.1 mm) in air curing condition.

CHAPTER 5
CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based on the test results, the following conclusions are obtained:

1) The self-healing behavior of concrete can be quantitatively estimated from the crack-closing ratio and the water flow rate value.
2) The self-healing behavior of mortar cured in water is more effective than mortar cured in the dry-wet cycle and sample cure in air, in order.
3) The S-H ability of mortar was dramatically enhanced with the use of expansive agent:
   - The more amount of expansive additive, the more effective of self-healing ability (Using 6% of expansive additive is more effective than 4% and 0%).
   - The use of expansive additive type A is more effective than expansive additive type T.
4) The S-H ability of mortar samples was improved significantly when using FA. The use of 30% FA resulted in the enhancement of S-H ability of mortar.
5) With the same amount of fly ash used,
   - The effectiveness of FA type on the S-H ability of mortar from low to high is in the following order: low CaO fly ash (FAR), high CaO fly ash (FAA), high CaO-free lime-SO_3 fly ash (FAB).
   - The effectiveness of FA to the S-H ability depends on the amount of total CaO, free lime and SO_3. The higher amount of CaO, free lime and SO_3 in the mixture, the better the S-H ability.
6) Using FA in combination with the EA in mortar samples indicated better self-healing performance than using EA or FA separately.

7) The use of BA with the replacement of 10% of fine aggregate as the water-providing agent indicated the improvement of the S-H ability of mortars in the long term due to the benefit of the IC method.
8) The combination of FA and BA in both OPC mortars and expansive mortars results in the most remarkable improvement in the S-H behavior of mortar.

5.2 Recommendations for future studies

In order to clarify the behavior of self-healing ability of mortars with fly ash and bottom ash, the following future studies should be continued.

1) The effect of crack introducing age on the self-healing ability of mortar with fly ashes and bottom ash should be investigated.
2) Effect of initial crack width on the self-healing ability. Though it was studied in many previous researches, however, the effect of initial crack width on the self-healing ability of concrete/ mortar with the use of bottom ash and different types of fly ash has not been clarified.
3) The curing temperature is an important aspect that may lead to the change of self-healing behavior. Therefore, studying this aspect should be one of the future study items.
4) Products inside crack are very necessary for the explanation of self-healing behavior under the effectiveness of various factors. Then the products inside crack are recommended to be intensively investigated in future.

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