USE OF FLY ASH TO ENHANCE PERFORMANCE OF EXPANSIVE CONCRETE

BY

RACHOT CHATCHAWAN

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE (ENGINEERING AND TECHNOLOGY)
SIRINDHORN INTERNATIONAL INSTITUTE OF TECHNOLOGY
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A Thesis Presented

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Submitted to

Sirindhorn International Institute of Technology

Thammasat University

In partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE (ENGINEERING AND TECHNOLOGY)

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FEBRUARY 2018

Acknowledgements

The author wishes to express his profound gratitude and appreciation to his advisor, Asst. Prof. Dr. Warangkana Saengsoy, for her continuous encouragement, guidance and supports which enormously assisted in working towards the completion of his research work.

The author wishes to extend his gratitude and appreciation to Prof. Dr. Somnuk Tangtermsirikul and Dr. Parnthep Julnipitawong, for interesting advices, comments and devoting valuable time to serve as committee members. His gratitude and appreciation also appreciatively expressed to Asst. Prof. Dr. Taweechai Sumranwanich, external committee member.

The author also wishes to extend his gratitude towards Sirindhorn International Institute of Technology (SIIT), Thammasat University which enabled him to learn from international professionals and provided scholarship during his study period. Special appreciation is forwarded to all researchers in Construction and Maintenance Technology Research Center (CONTEC) for kind encouragement and suggestions during his study.

The authors would like to acknowledge the partial research fund and consulting received from Siam Research and Innovation Co., Ltd. for this project.

Sincere appreciations are given to faculty members of School of Civil Engineering and Technology who taught the author and also its staff who facilitated the author during study.

Finally, sincere gratitude and utmost admiration is dedicated to his parents and family members who supported him all through his life

Abstract

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by

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The use of expansive additive, a kind of mineral admixture, is an effective method for reducing the shrinkage cracking due to the induced expansion at the early age by the production of $Ca(OH)_2$ and ettringite which compensate the effect of subsequent shrinkage. Although the use of expansive additive is effective to reduce the shrinkage cracking, the high price of expansive additive is a factor resulting in the high cost of concrete. At present, some substandard fly ashes consisting high amounts of free lime and SO_3 have been occasionally produced and have eventually become a main reason of unused fly ash in Thailand due to the problem of undesirable expansion when used in concrete. In order to find the way for relieving the problem of construction due to expansive additive usage and the problem of unused high free lime and SO_3 fly ash in Thailand, the possibility for a combined use of the substandard fly ash and expansive additive in concrete was challenged.

This research aims to study performances and clarify performances of expansive concretes containing fly ashes. Both effects of expansive additive types and fly ash types were focused. Setting time, slump, free expansion, autogenous shrinkage, total shrinkage, and unconfined and confined compressive strength were preliminary investigated and microstructural or mineralogical investigation were investigated for clarifying the results. The standard fly ashes which are a low CaO fly ash (FAR) and a high CaO fly ash (FAA), and substandard fly ashes which are high free lime and SO_3

fly ashes (FAB and FAC) were used in this study. All types of fly ash were used as a cement replacing material at 0 and 30% by weight of total binders. Four expansive additives referred to as EAD, EAT, EAA and EAB were also used in this study. All types of expansive additive were used for replacing binder at 0 and 5% by weight of total binders for paste and mortar mixtures, and 0 and 20 kg/m^3 for concrete mixtures, respectively.

The test results on the concrete containing different types of expansive additive revealed that when compared with Non-EA mixtures, the EAT, EAA and EAB mixtures showed better performances in all properties. The EAT, EAA and EAB mixtures showed the comparable autogenous and total shrinkages, and in order, showed slightly lower slump, faster setting, higher free expansion, and higher unconfined and confined compressive strength. When the EAD mixture was cooperatively considered (only compared in case of Non-FA mixture), the EAD mixture showed best performances in results of free expansion, autogenous shrinkage and total shrinkage when compared with the Non-EA and expansive additive mixtures, but the compressive strength of EAD mixture was the lowest in case of unconfined condition, but could turn into the same level with EAT mixture when was made under confined condition.

The test results for the effect of concrete containing different types of fly ash revealed that the low CaO fly ash mixtures showed slower setting than that of the Non-FA mixture, but the Non-FA and low CaO fly ash mixtures showed comparable performances in the results of slump, free expansion, and autogenous and total shrinkages. All above mentioned results continuously showed better performances in the high CaO fly ash, and high free lime and SO_3 fly ashes mixtures, respectively. The results of unconfined and confined compressive strength showed the same tendency. The early age compressive strength showed the largest in the Non-FA mixtures and respectively followed by the high free lime and SO_3 fly ashes, high CaO fly ash and low CaO fly ash mixtures. At long term, the compressive strength of low CaO fly ash mixtures showed the largest or the same with the Non-FA and high CaO fly ash mixtures, and the high free lime and SO_3 fly ashes showed the lowest.

The results of free expansion and shrinkage also additionally showed that the use of 10% high free lime and SO_3 fly ash could enhance the performance of expansion in a similar level to the use of 30% high CaO fly ash. In addition, the use of 30% high free lime and SO_3 fly ash, 30% high CaO fly ash, and 10% high free lime and SO_3 fly ash in EAT and EAA concretes could reduce the expansive additive usage approximately to 22, 12 and 8 kg /m^3, respectively. Moreover, when the expansion at the early age was not considered, the use of expansive additives in concrete was not beneficial to the shrinkage reduction at long term, but it became effective when the expansive additive was applied with fly ash.

The different expansion in each mixture could be proved by the results of produced expansive products which were analyzed by X-ray diffraction. The cause for expansion in each type of expansive concrete mainly occurred from different types of produced expansive products which were results of the amounts of different constituents for producing expansive products in each type of expansive additive. The expansion enhancement of expansive concrete containing different types of fly ash was not due to the increased amount of $Ca(OH)_2$, but well correlated with the increased amount of ettringite. The highest ettringite formation was observed in the high free lime and SO_3 fly ash mixtures, followed by the high CaO fly ash and low CaO fly ash mixtures, respectively.

For the clarifications in the results of unconfined and confined compressive strength, in case of unconfined compressive strength, the different unconfined compressive strength results in each type of expansive concrete was mainly caused by the different porosity values. The mixtures with lower porosity, indicating to a denser paste structure, resulted in higher compressive strength. Comparing between the specimens made under unconfined and confined conditions, the results of compressive strength were higher when the specimens were prepared in confined condition. The confinement of the specimens led to the reduction of porosity of concrete, indicating a denser paste structure, therefore resulted in an enhancement of compressive strength.

Keywords: Expansive Concrete, Fly Ash, Free Expansion, Shrinkage, Compressive strength

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Chapter 1

Introduction

1.1 General

Shrinkage is a phenomenon of concrete which occurs from the volume reduction mainly due to combined effects of drying shrinkage which is caused by loss of moisture to the environment, and autogenous shrinkage which is caused by the loss of moisture in the pores due to water consumption in hydration and pozzolanic reactions [1-3]. In the practical situation, a concrete structure cannot shrink freely because it is usually restrained by steel bars or other nearby structures. Therefore, the concrete structure under restrained condition will crack if the induced stress is higher than the strength capacity of the concrete. When cracks occur, the risk of deteriorations increases which can reduce the service life of the structure.

There are many applicable methods to reduce and control the problem of shrinkage cracking in concrete. Generally, sufficient curing, joint provision and use of additional reinforcement are some practical methods for reducing and controlling the shrinkage cracking. However, the problem of shrinkage cracking can be considered since the designing state by, for examples, reducing amount of paste or increasing amount of aggregate in the proportioning of concrete or use of mineral admixtures as a replacing material. At present, the use of expansive additive, a kind of mineral admixture, is an effective method for reducing the shrinkage cracking due to the induced expansion at the early age by the production of $Ca(OH)_2$ and ettringite which compensate the effect of subsequent shrinkage [4].

1.2 Statement of problems

Fly ash is not an industrial product, but a by-product of coal combustion. Therefore, its properties vary based on many factors. Many standards have been established to specify the quality of fly ash for being used in cement and concrete, such as ASTM C618, EN 450 and TIS 2135 [5-7]. The use of standard quality fly ash in concrete improves many basic and durability properties of concrete [8-15]. However, some substandard fly ashes consisting high amounts of free lime and SO_3 have been occasionally produced and have eventually become a main reason of unused fly ash in Thailand [16]. Some sustainable ways to solve this problem have been processed. Recently, few studies investigated about the use of this substandard fly ash in concrete and reported that there was a problem about the expansion property if the free lime and SO_3 contents were excessive. Kaewmanee et al. [17] found that the presence of free lime content up to 4.51% in fly ash slightly affected concrete properties such as faster setting, higher compressive strength at early age, and higher autoclave expansion. Atis et al. [18] reported that expansion was induced at the 40% replacement of cement by the substandard fly ashes which contained high CaO, free lime and SO_3 contents. Nawaz et al. [19] concluded that the free lime content in fly ash should be reduced when SO_3 content was higher for controlling the excessive expansion.

Several studies reported that the use of standard fly ash, Class 2b fly ash according to the TIS 2135 standard, could improve the expansion of expansive concrete. This is due to the presence of some amounts of free lime and SO_3 in this type of fly ash which might participate in enhancing expansive products of the expansive concrete [20, 21]. Therefore, it is possible that this substandard fly ash, showing the problem of expansion when used in normal concrete due to the presence of high amounts of free lime and SO_3 contents, will be more effective to improve expansion of expansive concrete than the standard fly ash. If the use of this fly ash, with a proper content, in expansive concrete is accomplished, it is economical for construction because it can reduce the expansive additive dosage which is considered as the expensive material. Most importantly, it will be another effective way to relieve the problem of unused fly ash in Thailand due to the increased amounts of free lime and SO_3.

1.3 Objectives of study

From the above discussion, the objectives are focused as follows:

- To study the effect of the use of expansive additives with fly ashes in concrete
- To clarify and explain the combined effect of expansive additives and fly ashes on the properties of concrete

1.4 Scope of study

For the scope of this study, various parameters are studied to achieve the objectives as follows:

a. Materials

- Type of cement : Ordinary Portland cement type 1
- Type of expansive additive : 4 types of commercial expansive additive
- Type of fly ash : Low CaO fly ash, high CaO fly ash and high free lime and SO_3 fly ash

b. Paste mixtures

- Water to binder ratio : 0.30 and normal consistency
- Amount of expansive additive : 0 and 5% by weight of total binders
- Amount of fly ash : 0 and 30% by weight of total binders
- Curing condition : Water

c. Mortar mixtures

- Water to binder ratio : Water content at constant flow value of 110±5 %
- Amount of expansive additive : 0 and 5% by weight of total binders
- Amount of fly ash : 0 and 30% by weight of total binders
- Curing condition : Water
- Experimental condition : Free and restrained

d. Concrete mixtures

- Water to binder ratio : 0.45
- Amount of expansive additive : 0, 20 and 30 kg/m^3 of concrete
- Amount of fly ash : 0, 10 and 30% by weight of total binders
- Curing condition : Water, sealed and air
- Experimental condition : Free and restrained

e. Experimental programs

- Setting time of paste
- Water requirement of mortar
- Slump
- Free expansion
- Autogenous shrinkage
- Total shrinkage
- Unconfined compressive strength
- Confined compressive strength
- Porosity of concrete
- XRD analysis

Chapter 2

Literature Review

2.1 Effects of free lime and SO_3 on properties of concrete

Kaewmanee et al. [17] clarified the characteristics of free lime in fly ash and investigated the properties of cement pastes and mortars containing fly ash with different free lime contents. To study the effect of free lime content in fly ash, the original fly ash was added by free lime at varying total free lime contents. The results showed that free lime particles were mostly found outside the fly ash particles, with only negligible encapsulated amount of free lime inside the fly ash particles. This means that the originally high free lime and added free lime have similar effects on the properties of fly ash mixtures. The test results of properties of cement pastes and mortars showed that the free lime did not affect much the physical properties. The free lime content up to 4.51% caused faster setting, higher compressive strength especially at the early age and higher autoclave expansion (still within standard limit). Whereas, the higher free lime content in the mixtures tended to cause higher expansion due to alkali-aggregate reaction (still lower than cement-only mixture). Carbonation depth, shrinkage, chloride resistance and sulfate resistance were only slightly affected by the added free lime.

Atis et al. [18] studied the compressive and flexural strengths, and drying shrinkage of mortar containing non-standard fly ash which contained high amount of CaO (51.29%) and SO_3 (12.06%). The fly ash was replaced at 0, 10, 20, 30 and 40% by weight of total binders. The water to cement ratio was kept constant at 0.4. The relative humidity and temperature of 65% and 20±2 ºC were used for curing their specimens. The test results of compressive and flexural strengths of mortars containing non-standard fly ash were pleasantly developed. The strength at 28 days of mixtures with 10% and 20% of fly ash replacement were comparable with the cement-only mixture. The result of shrinkage showed that the shrinkage was lower when higher amount of fly ash was used, and the expansion occurred at 40% of fly ash replacement.

From the results, it indicated that this fly ash might be used for compensating drying shrinkage of cement-based materials.

Nawaz et al. [19] investigated the limitation of free lime content in fly ash. The free lime was added into four different types of fly ash until over all free lime contents equal to 5, 7 and 10%. The basic properties and durability properties were tested by using the replacement percentages at 20 and 40%. The results revealed that the increase of free lime content in fly ash lead to the increase of water requirement, faster setting, improvement of compressive strength and higher expansion when tested for autoclave expansion, alkali-aggregate reaction and sulfate expansion. The amount of free lime up to 10% in the mixture with 20% fly ash and amount of free lime up to 7.72% in the mixture with 40% of fly ash did not excessively expand over the specified limit of ASTM C618 in the tests of autoclave expansion. The expansion which occurred due to the alkali-aggregate reaction was lower than cement-only when the free lime content was 7.95% in mixture with 20% of fly ash, and the free lime content was 10% in mixture with 40% of fly ash. For the result of sulfate expansion, the fly ash mixtures tended to show higher expansion than the cement-only mixtures, especially for the case of fly ash which contained high amount of SO_3 (> 5%). For the mixture with 20% fly ash which contained SO_3 content lower than 5%, the free lime could be up to 10% without compromising the basic and durability properties. For the mixture with 40% fly ash which contained SO_3 content lower than 5%, the free lime could be up to 4.23%. In case of the fly ash which contain high SO_3 content, the limit of free lime content of fly ash which obtain desirable durability properties is reduced to 5.31% and 3.73% for 20% and 40% fly ash replacements, respectively.

Sung et al. [22] studied properties of mortars which contained ground granulated blast-furnace slag and circulating fluidized bed boiler co-firing fly ash. The circulating fluidized bed boiler co-firing fly ash contained the total amount of Fe_2O_3, SiO_2, and Al_2O_3 less than 70%, but contained 37.8% of CaO and 12.9% of SO_3. The replacing ratios by weight of circulating fluidized bed boiler co-firing fly ash to granulated blast-furnace slag were 1:9, 2:8, 3:7, 4:6, 5:5, 6:4 and 7:3. The binders to aggregate ratio was 1:2.75 and water to binder ratio was 0.50. The test results showed that the compressive strength of 3:7 of circulating fluidized bed boiler co-firing fly ash to granulated blast-furnace slag was the highest when compared with others (except only-cement mixture). The compressive strength of mixtures with higher than 40% of circulating fluidized bed boiler co-firing fly ash were unstable. The increasing of circulating fluidized bed boiler co-firing fly ash led to the volumetric expansion. The C-A-S-H and $CaSO_4{\cdot}(H_2O)_8$ was observed from SEM and XRD analysis which might be the cause of volume expansion due to the excessive SO_3 content in the mixtures.

Tudjono et al. [23] investigated the mortar containing nano fly ash and nano lime. The class F fly ash and lime were prepared by a milling machine. The Portland cement to nano fly ash ratio was 1:12.24 and the mixture of nano fly ash and lime in the ratio of 1.11:1 was replaced into 0, 2.5, 5, 7.5, 10, 12.5 and 15% of total binders. The binders to aggregate ratio was 1 to 1.70 and 0.49 of water to binder ratio was used. The specimens with size 50×50×50 mm were tested for the compressive strength at the age of 7 days. The results illustrated that the compressive strength of mixture with only-nano fly ash was lower than that of control specimen (only-cement mixture). The compressive strength of mixtures with nano fly ash and nano lime at 2.5, 5 and 7.5% of total binders were higher than that of only-nano fly ash mixture. The 2.5% of nano fly ash and nano lime mixture showed the highest strength enhancement.

Elaty and Ghazy [24] investigated the properties of cement paste, mortar and concrete with silica fume and mixed with saturated lime water. The mixing solution types and the percentage of silica fume were main parameters for this study. The results showed that the consistency was enhanced by using lime water when compared with the control mixture. The initial and final setting times were delayed when compared with the mixture mixed using traditional water due to the principles of common ion effect. The pozzolanic reaction was enhanced in mixture with lime water and silica fume which was identified by the development of strength at early and later ages. The results of XRD analysis showed that the CH crystals for further reaction at later ages was found in mixture with higher silica fume percentages which was up to 30%. In addition, the high alkaline media around steel bars from the moment of ingredients mixing as well as at later ages despite pozzolanic reaction that was identified from results of chloride attack were ensured in mixtures with silica fume and lime water.

Barbhuiya et al. [25] investigated the properties of fly ash concrete containing hydrated lime and silica fume. In the investigation, the workability, compressive

strength and permeability were tested in the mixtures with water to binder ratios of 0.30 and 0.35. Portland cement was constantly replaced by fly ash at 30% and 50% for mixtures with water to binder ratios of 0.30 and 0.35, respectively. The 5% hydrated lime was added by weight of total binders and the 5% silica fume was added by weight of Portland cement. The cement paste mixtures were made with the same mix proportions for investigating the characteristics of pore structure by mercury intrusion porosimeter and determining the hydrated phases by thermogravimetric methods. The test results revealed that the the early age compressive strength of fly ash concrete was improved by added lime and silica fume. The compressive strength at the age of 28 days was significantly increased when silica fume was used. The air permeability of concrete with lime and silica fume decreased or remained almost the same when compared to the OPC concrete. The sorptivity of concrete was also improved by adding lime and silica fume. The results of the differential scanning calorimetry and thermogravimetric analysis showed that the $Ca(OH)_2$ content in the cement paste was increased by adding hydrated lime, whereas was decreased by the adding silica fume. The results of mercury intrusion porosimetry confirmed that the use of hydrated lime and silica fume were beneficial to the decreasing of total pore volume of fly ash cement paste.

2.2 Standard and classification of expansive concrete

ACI 223R-10 [4] defined the expansive concrete as the concrete which is made from expansive cement or cement with expansive additive. The expansion will be generally generated after setting and the compressive stress would be induced under the restrained condition. These expansion or compressive stress that are generated will be compensated with the subsequent shrinkage. The expansive cement and expansive additives were classified based on their principal constituents which were available for ettringite and calcium hydroxide formation (Table 2.1).

Table 2.1 Classification of expansive cement and expansive additive following to ACI 223R-10

Types	Expansive cement	Expansive additive
K	- Portland cement - Anhydrous tetracalcium trialuminate sulfate - Calcium sulfate - Lime	- Calcium sulfoaluminate - Calcium sulfate
M	- Portland cement, - Calcium aluminate cement - Calcium sulfate	- Calcium aluminate cement - Calcium sulfate
S	Portland cement containing high amount of - Tricalcium aluminate - Calcium sulfate	- Tricalcium aluminate cement - Calcium sulfate
G	-	- Calcium dioxide - Aluminum dioxide

The Japan Society of Civil Engineers (JSCE) [26] classified the expansive concrete based on size of expansive force as shrinkage compensating and chemical prestressing concrete. It is classified into shrinkage compensated or chemically prestressed concrete based on function. The qualification of maximum uniaxial expansion rate within 7 days according to the test method A of JIS A 6202 (0.95% of restraining steel ratio) were set following the point of expansive force as follows: [27]

- 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 prestressing concrete was 200×10^{-6} to 700×10^{-6}.
- The standard range of uniaxial expansion rate for chemical prestressing concrete used for factory products was 200×10^{-6} to 1000×10^{-6}.

2.3 Expansive cement and expansive additive

Wczelik et al. [28] studied the expansive additive which was produced by grinding a high alumina cement with a special sulphate–lime sinter. The synthesis of the sulphate–lime sinter and the expansion or compensation of shrinkage during the hydration process of the mixing between the Portland cement, calcium aluminate cement and sulphate–lime components were focused. To find the optimum blended cementitious material proportions, the volume changes, compressive strength, hydration rate, phase composition and hydration products microstructure were carried out. The results revealed that the mixtures with alumina calcium aluminate cement at 40%, 50% and 70% and the lime–anhydrite sinter produced the ettringite during hydration, showing the expansive agent role. The shrinkage compensating of expansive cement mixtures could be produced by replacing by weight of cement by 8–16% of the blending of 51% calcium aluminate cement or 40% alumina and lime–sulfate components, but the mixtures with high calcium aluminate cement should not be used due to the falling down strength. The formed ettringite was almost the same after 2 days and 7 days of hydration. Calcium silicate hydrates and calcium hydroxide or even gypsum were formed. Homogenous, porous structure was formed by the ettringite, calcium silicate hydrates and calcium hydroxide products.

Konik et al. [29] investigated the sulfate–calcium component, prepared as the expansive additive by flue gas desulphurization gypsum and calcareous raw material mixture burning, and aluminate clinker, containing 60% Al_2O_3. The ground sulfate component and ground aluminate clinker were mixed with expansive additive. The expansive material showed the main phases of anhydrite, monocalcium aluminate and calcium oxide. The expansive additive at 7% and 12% by weight of binder were mixed with Portland cement for studying the initial and final setting time, expansion, length changes, compressive strength and flexural strength. The results showed that the 7% expansive additive mixture was a low-shrinkage material (reduce shrinkage), while the mixture with 12% expansive additive was an expansive binder. The cement pastes which were examined by XRD revealed the peaks of ettringite growing at 3 and 7 days of hydration.

Odler and Subauste [30] studied the ettringite formation and the associated expansion of cement pastes made from gypsum, Al-bearing compound, and tricalcium silicate. The results indicated that there were significant differences between the ettringite formation rate and the capacity of the formed ettringite for generating the expansion. The formed ettringite in some systems was not related to the expansion. The consuming water from the environment seems to be inessential for producing the expansion, but intensifies its extent.

Bizzozero et al. [31] studied the expansion at long-term of samples with calcium aluminate cement and calcium sulfoaluminate cement which were added higher amount of gypsum. The results showed that there was a critical gypsum amount which leads to unstable expansion and the failure of samples. The microstructures of systems with added gypsum which is below and above the initial were similar. Pore solution analysis showed that supersaturation with respect to ettringite increased with the calcium sulfate content, which lead to an increase of the crystallization pressure. The supersaturation determined the minimum pore size in which crystals could grow. Therefore, at a higher supersaturation value, a larger pore volume was accessible for ettringite crystals to grow, thereby exerting the pressure in the porous skeleton.

2.4 Properties of expansive concrete

Nagataki and Gomi [32] explained that the expansion of the expansive concrete became larger when the amount of expansive additive was increased. When the expansive additive content did not exceed 30 kg/m^3 of concrete, unconfined compressive strength, tensile strength, bond strength, bending strength, Young's Modulus and creep of the expansive concrete were similar to those of the normal concrete. For the results of shrinkage, the expansion of expansive concrete which was cured long time in water did not change when it reached to a some settled value. The specimen which was kept in the dry condition after water curing showed the shrinkage, but was smaller than the normal concrete. For the results of sulfate resistance and wear resistance, they were the same as the normal concrete.

Shuguang and Yue [33] investigated the microstructure and hydration rate of high-performance expansive fly ash concrete (HPEC) by XRD and SEM methods. The high-performance concrete and HPEC which cured under confined and unconfined conditions were used for comparing the different mechanical properties. The results showed that the inner expansive cement paste and concrete microstructure which loose, and cracks were observed in the interface paste and paste-aggregate for specimens under the unconfined condition. For confined condition, the expansive concrete microstructure was improved by the denser paste-aggregate interface.

Carballosa et al. [34] studied the structural elements of self-stressing self-compacting concretes. Two types of cements and type K and G expansive additives were used. The effect of different parameters on their performance was investigated. The results showed that, with the use of expansive additives, the compressive strength reduction mainly depended on the reached expansion. The magnitude of expansion depended on the reaction of alumina and sulfates of cement when using type K expansive additive and, without watering, type G expansive additive produced expansion by amorphous calcium hydrated agglomerates formation. Inversely, an unmethodical formation of ettringite was found when type K additive was used.

Ozawa et al. [35] studied the effectiveness of expansive strain in concrete with curing at different temperatures. The study was conducted to clarify the expansive energy and hydration products of expansive mortar at 20, 30 and 50°C under restrained testing conditions. The results showed that the maximum values of an expansive energy were 0.75×10^{-4} Nmm/mm^3 for the curing at 20°C, 1.1×10^{-4} Nmm/mm^3 for the curing at 30°C and 0.9×10^{-4} Nmm/mm^3 for the curing at 50°C. The rates of expansive energy of specimen curing at 30 and 50°C were larger than that at 20°C. Differential scanning calorimetry results and scanning electron microscopy observation showed that the ettringite (AFt) needle crystals were observed at 7 days for 20°C and at 1 day for 30 and 50°C. It was also found that AFt changed to monosulfate (AFm) at 50°C. It could be inferred that AFt formation was dependent on temperature history.

Sun-Gyu [36] studied the effectiveness of expansive additive for minimizing the restrained shrinkage cracking of high-strength concrete at early age. Free shrinkage test and simulated-completely restrained test were conducted. Creep and shrinkage of high-strength concrete with and without expansive additive under restrained conditions were characterized by experiments that provided data on free shrinkage and restrained shrinkage. The test results showed that the autogenous shrinkage and tensile stress were effectively reduced by adding expansive additive. In addition, the shrinkage stress was relaxed by 70% in ordinary high-strength concrete and 80% in expansive high-strength concrete.

Yan and Qin [37] investigated the possibility of delayed ettringite formation of massive expansive concrete. For the first 7 days, the temperature match condition system was developed to simulate the temperature development in the core of fresh massive concrete. The restrained mortar specimens were cured from ambient temperature to 70°C in 48 hours and was kept over 70°C for 72 hours. The relative humidity was higher than 95%. After that, the specimens were left cooling down to 60°C in 48 hours. Another kind of curing was curing at ambient temperature. After 7 days of curing, the specimens were cured at ambient temperature but different relative humidity. The result showed that the use of expansive additive in massive concrete could not provide significant effect due to its shrinkage-compensating effect, which results from the decomposition of ettringite when the curing temperature was higher than 70°C in early age. Harmful of delayed expansion occurred due to delayed ettringite formation at a later age in shrinkage compensating massive concrete. It appeared most severe when specimens were cured under water and mild temperatures in humid air.

But specimens which were cured in dry air showed the result of shrinkage. Those possessed potential danger for the durability of concrete structures in service.

Sisomphon et al. [39] studied the potential for self-healing of cement-based materials incorporating calcium sulfoaluminate based expansive additive and crystalline additive. The surface crack width between 100 to 400 µm were generated in mortar specimens having age of 28 days and the self-healing process was initiated by submerging the cracked specimens in the water after that. It was observed from the experimental results that the mixtures with calcium sulfoaluminate based expansive additive and crystalline additive showed satisfactory surface crack closing ability. The optimal mix design was found to be a ternary blend of Portland cement with 10% and 15% by weight of calcium sulfoaluminate based expansive additive and crystalline additive of which the surface crack width up to about 400 µm was completely closed, and the rate of water passing was dropped to zero within 28 days. It was hypothesized that the leaching amount of Ca^{2+} from the matrix played an important role on the precipitation of calcium carbonate which was the major healing product. The analysis showed that the amount of Ca^{2+} was more released in those specimens with additions of calcium sulfoaluminate based expansive additive to crystalline additive than that of the control specimen. Moreover, those specimens with those additives had higher pH value which might promote calcium carbonate precipitation.

2.5 Properties of expansive concrete with fly ash, blast furnace slag and bottom ash

Lam et al. [20] studied the expansion and compressive strength of concrete containing expansive additive. The effect of dosage of expansive additive, type of binder, water to binder ratio, curing temperature, curing condition and restraining ratio by steel were investigated. The results showed that the free expansion and restrained expansion of fly ash concrete containing expansive additive was higher when compared to cement-only expansive concrete. The expansion of expansive concrete developed within 3 days, after that it was constant or slightly turned into shrinkage. The curing condition was important for expansive concrete. Water curing resulted in higher expansion than the other curing conditions. The free expansion of concrete containing expansive additive increased when water to binder ratio increased, especially in the fly ash concrete mixtures. The loss of strength was a problem in case of fly ash concrete containing high amount of expansive additive under free condition, but it relieved when the concrete was restrained.

Lam et al. [39] studied the durability properties of expansive concrete. The effect of expansive additive content, fly ash content and water to binder ratio on durability such as carbonation resistance of concrete, sulfate resistance of mortar, chloride penetration and chloride binding capacity of paste were studied. The results indicated that when the mixtures with expansive additive content lower than 30 kg/m^3 were used, carbonation resistance of both cement-only expansive concrete and fly ash expansive concrete were better than that of concrete without expansive additive. But when the mixtures with expansive additive content higher than 30 kg/m^3 were used, carbonation resistance of fly ash expansive concrete becomes worse. The use of expansive additive showed the negative effect on sulfate resistance and chloride binding capacity of concrete but tended to reduce chloride permeability.

Zhang and Li [40] studied the effects of ground granulated blast furnace slag (GGBS) and fly ash (FA) on the expansion performance of expansive concrete (sulphoaluminate base). The specimens were cured at simulated temperature as the internal temperature of mass concrete. In the study, gypsum was added to eliminate the negative effects of GGBS and FA usage in expansive concrete. The hydration process, hydration products and the morphology of products were analyzed by XRD, TG-DTG and SEM. The results revealed that, in case of mixtures without the addition of gypsum at simulated curing temperature, the use of GGBS and FA decreased the expansion and compressive strength when compared with non-GGBS/FA mixtures. In case of mixtures with the addition of gypsum, GGBS mixtures increased expansion and compressive strength when compared with non-GGBS mixture, because the gypsum promoted the ettringite formation and stimulated the hydration of GGBS. For mixtures with FA, the addition of gypsum also promoted the ettringite formation, but the expansion was decreased when compared with non-FA mixture. This is because the FA mixtures prolonged setting time.

Yan et al. [41] investigated the characteristics of hydration reaction of expansive concrete containing mineral admixtures, which are fly ash and ground granulated blast furnace slag (GGBS), by calorimetry. The results indicated that the total and final heat of hydration of mixtures decreased when water to binder ratios was decreased. The ternary binder system, mixtures with Portland cement, mineral admixture and expansive agent, showed low hydration heat and rate of heat evolution, but their total heat of hydration continuously increased and higher than that of binary binder system in later period at low w/b ratio.

Sutthiwaree et al. [42] investigated the early age length change under free and restrained conditions and total shrinkage of expansive concrete. The effect of bottom ash, identified as an internal curing material, was used for partially replacing fine aggregate. The results revealed that the expansion under free and restrained conditions could be enhanced by using bottom ash. On the other hand, the use of bottom ash increased subsequent shrinkage.

Chapter 3

Experimental Program

3.1 General

This research was designed to investigate effect of free lime and SO_3 in fly ash which may affect properties of expansive concrete. It also covers the clarification of their effects. The variables and mix proportions of cement paste, mortar and concrete are shown in Tables 3.1 to 3.3, respectively. Primarily, the properties of expansive concrete, i.e., setting time, slump, compressive strength and especially expansion/shrinkage were focused. After that, microstructural and mineralogical investigations were conducted for clarifying the test results. It can be seen from outline of study in Figure 3.1.

Table 3.1 Variables and conditions of mix proportion of cement paste

W/B ratio	Fly ash (%)	Expansive additive (%)	Curing condition
0.30 and NC*	0 and 30	0 and 10	Water cured

* Normal consistency

Table 3.2 Variables and conditions of mix proportion of mortar

W/B ratio	Fly ash (%)	Expansive additive (%)	Restrained condition
WR*	0 and 30	0 and 5	• Free • Restrained

*Water requirement (water content at a constant flow of 110±5 %)

Table 3.3 Variables and conditions of mix proportion of concrete

W/B ratio	Fly ash (%)	Expansive additive (kg/m^3)	Curing condition	Restrained condition
0.45	0, 10 and 30	0, 20 and 30	• Water cured • Sealed cured • Air cured	• Free • Restrained

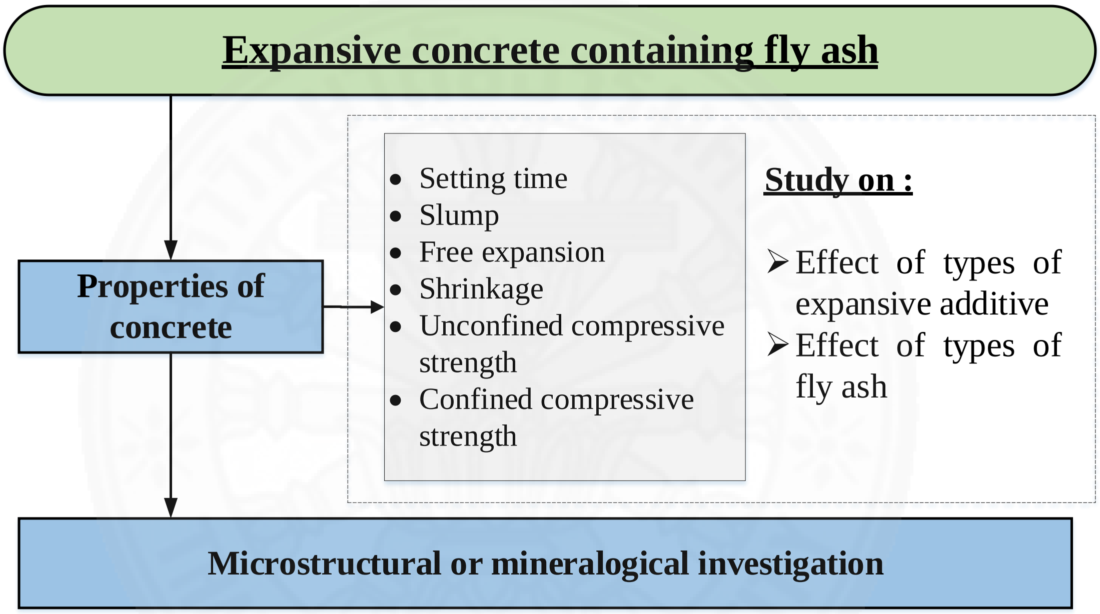


Figure 3.1 Outline of the study

3.2 Materials

3.2.1 Cement

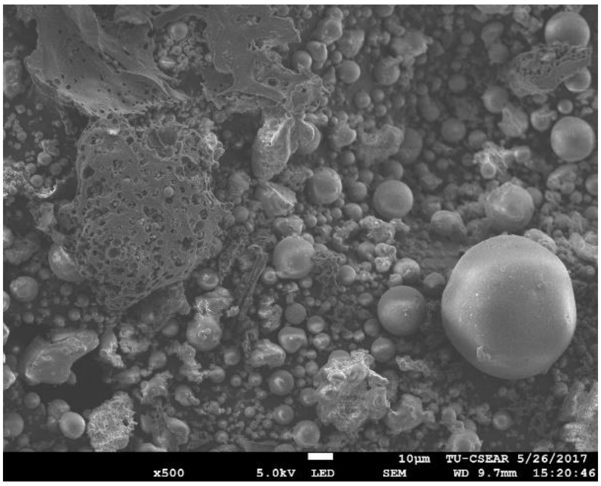
Ordinary Portland cement type 1 (OPC) according to TIS 15 [43] was used as a binder for all mixtures. The chemical compositions and physical properties of the ordinary Portland cement type 1 are shown in Table 3.4.

3.2.2 Expansive additives

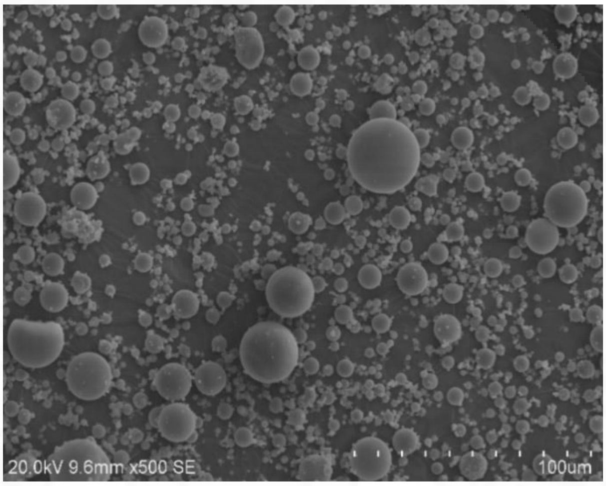
Two types of commercial expansive additive denoted by EAD and EAT, and two types of domestically manufactured expansive additive denoted by EAA and EAB were used as cement replacing materials. The chemical compositions and physical properties of expansive additives are shown in Table 3.5, and the phase analysis of the expansive additives by XRD is shown in Table 3.6.

3.2.3 Fly ashes

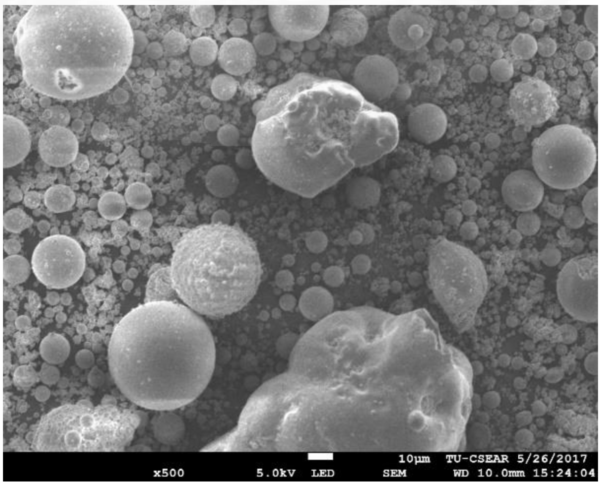
Three types of fly ash were used for replacing cement in percentage by weight of binder. FAR was fly ash from the BLCP power plant, while FAA, FAB and FAC were fly ashes from Mae Moh power plant. FAR fly ash contained lowest CaO, free lime and SO_3 contents. FAA fly ash contained higher CaO, free lime and SO_3 contents when compared with the FAR fly ash. FAB and FAC fly ashes contained the highest amount of CaO, free lime and SO_3 when compared with other types of fly ash. According to TIS 2135 [7], FAR and FAA could be classified as class 2a and 2b, respectively, but FAB and FAC contained excessive amount of SO_3 according to this standard. Moreover, FAB and FAC also showed the presence of high amount of free lime which was not limited in the standard. However, BS EN 450 [6] recommends that the amount of free lime should not be over 2.5%, so FAB and FAC had the over amount of free lime according to this standard. Therefore, FAB and FAC fly ashes were classified as the non-standard fly ash. The particle shapes of the fly ashes, and chemical compositions and physical properties of the fly ashes are shown in Figure 3.2 and Table 3.4, respectively.



a) FAR (BLCP power plant)



b) FAA (Mae Moh power plant)



c) FAB fly ash (Mae Moh power plant)

Figure 3.2 Particle shapes of fly ashes

Table 3.4 Chemical compositions and physical properties of ordinary Portland cement type 1 and fly ashes

Chemical compositions (% by weight)/ physical properties	OPC	FAR	FAA	FAB	FAC
SiO_2	19.70	61.09	35.71	26.61	25.22
Al_2O_3	5.19	20.35	20.44	13.60	13.88
Fe_2O_3	3.34	5.20	15.54	18.34	17.39
CaO	64.80	2.32	16.52	24.97	26.25
MgO	1.20	1.35	2.00	2.33	2.38
Na_2O	0.16	0.79	1.15	1.75	1.40
K_2O	0.44	1.36	2.41	1.77	1.92
SO_3	2.54	0.28	4.26	8.53	9.44
Free lime	-	0.03	1.71	3.93	3.06
LOI	2.10	5.68	0.49	0.53	0.56
Specific gravity	3.13	2.11	2.21	2.57	2.57
Blaine fineness (cm^2/g)	3,660	3,400	2,867	2,820	2,722

Table 3.5 Chemical compositions and physical properties of expansive additives

Chemical compositions (% by weight)/ physical properties	EAD	EAT	EAA	EAB
SiO_2	1.93	9.04	2.12	1.54
Al_2O_3	5.03	2.53	4.75	13.70
Fe_2O_3	1.22	1.47	0.14	0.10
CaO	69.47	68.60	61.19	50.29
MgO	1.04	0.78	0.73	0.40
SO_3	17.67	12.19	26.46	29.30
Na_2O	< 0.01	0.23	-	-
K_2O	0.05	0.13	-	-
TiO_2	0.05	0.14	-	-
P_2O_5	0.09	0.10	-	-
MnO	0.03	0.03	-	-
SrO	0.02	0.03	0.11	0.08
ZnO	0.01	0.02	-	-
CuO	0.02	0.02	-	-
Free lime	46.98	27.98	28.94	11.77
LOI	3.30	4.65	4.48	4.51
Specific gravity	2.90	3.02	2.94	2.79

Table 3.6 Phases analysis of expansive additives by XRD

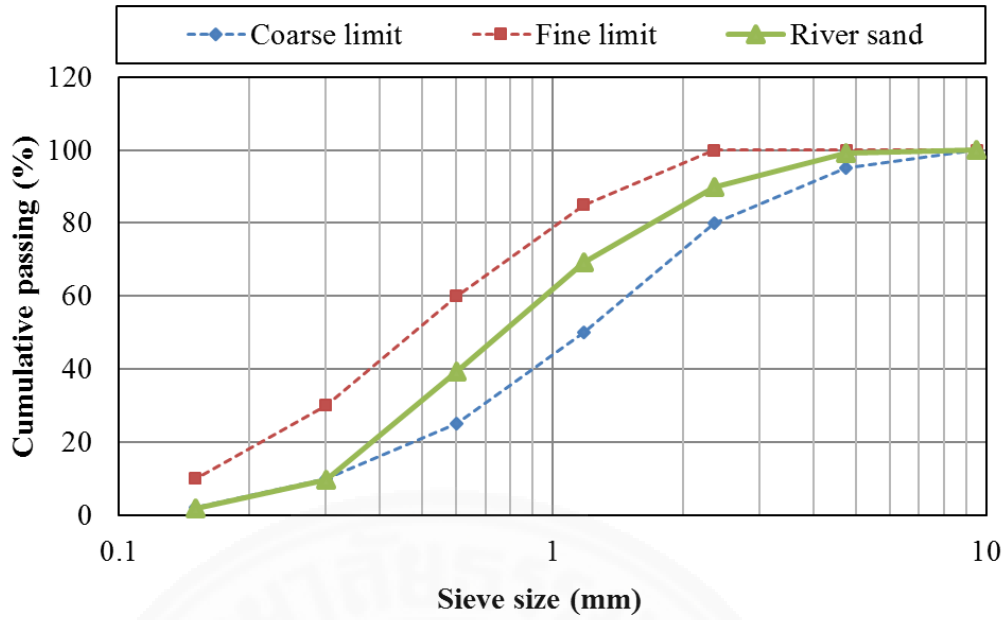
Phases analysis (%)	EAD	EAT	EAA	EAB
Yeelimite (C_4A_3S)	9.77	-	13.03	37.23
Anhydrite ($CaSO_4$)	31.80	19.45	47.72	42.46
Lime	46.98	27.98	28.94	11.77
Portlandite	3.97	2.15	2.65	1.61
Calcite	1.51	2.15	1.80	1.20
Gypsum	-	-	5.09	5.24
Quartz	-	-	0.78	0.49
Bassanite	-	1.04	-	-
Fluorellestadite	1.37	-	-	-
Periclase	0.86	-	-	-
C_2S	1.85	11.23	-	-
C_3S	-	27.18	-	-
C_3A	-	5.00	-	-
C_4AF	1.88	3.82	-	-

3.2.4 Aggregates

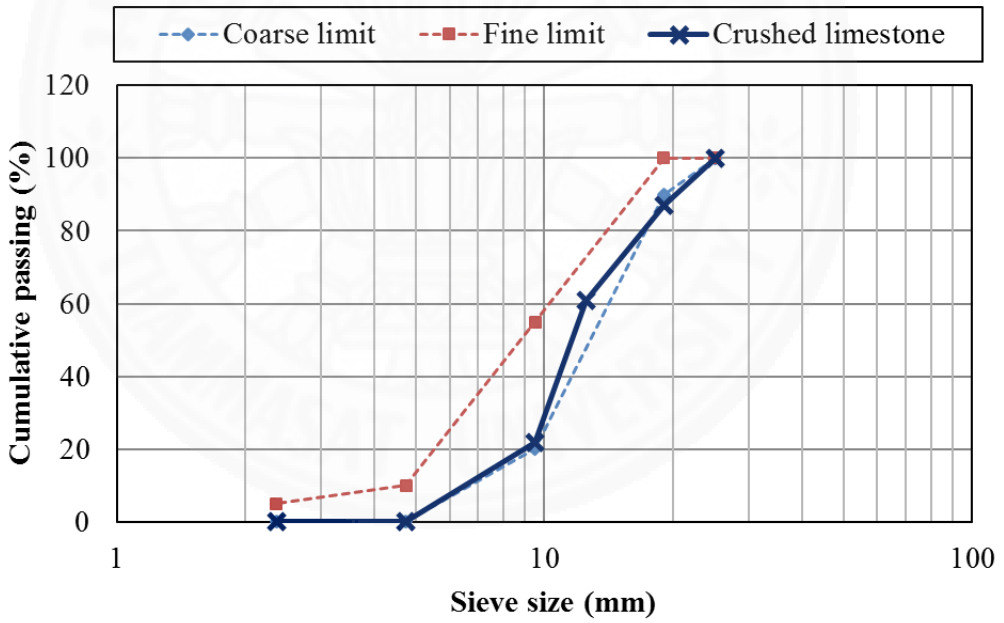
Natural river sand was used as the fine aggregate and crushed limestone was used as the coarse aggregate. Gradations of the fine and coarse aggregates were in accordance with ASTM C 33 [44]. The nominal size of the coarse aggregate was between 19 to 4.75 mm. The results of sieve analysis and physical properties of the fine and coarse aggregates can be seen in Figure 3.3 and Table 3.7, respectively.

Table 3.7 Physical properties of fine and coarse aggregates

Properties	Fine aggregate	Coarse aggregate
Absorption (%)	1.08	0.34
Specific gravity (SSD)	2.60	2.83
S/A at minimum void (by volume)	0.44	
Minimum void (%)	23.52	



a) Fine aggregate



b) Coarse aggregate

Figure 3.3 Sieve analysis results of aggregates

3.3 Mix proportions

In order to design the mix proportions, Ordinary Portland cement type 1 was used as the main binder while expansive additive and fly ash were used as cement replacing materials. The expansive additive (0 and 5%) and fly ash (0 and 30%) were replaced in percentage by weight of total binders. The water content of the first group of cement paste mixtures was controlled at the normal consistency for testing setting time. A constant water to binder ratio of 0.30 was used for the remaining groups of cement paste mixtures for mineralogical investigation. They are listed in Table 3.8. The mortar mixtures were designed for examining different water requirements in each mixture. The expansive additive (0 and 5%) and fly ash (0 and 30%) were also replaced in percentage by weight of total binders for mortar mixtures which were similar to the cement paste design concept. The sand to binder ratio was kept at 2.75 for all mixtures. The mix proportions of tested mortars are given in Table 3.9. For concrete mixtures, the expansive additives were used to partially replace cement by absolute weight in kg/m^3 of concrete (0, 20 and 30 kg/m^3), but fly ashes were used to partially replace cement in percentage by weight of total binders (0, 10 and 30%). The water to binder ratio and paste volume to void volume of aggregate phase were kept at 0.45 and 1.4, respectively, for all mixtures. The designed concrete mixtures which were used for studying the properties of concrete could be categorized into 4 groups, i.e., cement-only concrete, expansive concrete, fly ash concrete and expansive concrete containing fly ash. The mix proportions of the above mentioned groups are given in Table 3.10

Table 3.8 Mix proportions of pastes

No.	Mix ID	Portland cement type 1, ratio by weight	Expansive additives, ratio by weight				Fly ashes, ratio by weight				W/B
			EAD	EAT	EAA	EAB	FAR	FAA	FAB	FAC	
1	OPC	1.00	-	-	-	-	-	-	-	-	Normal consistency
2	EAT5	0.95	-	0.05	-	-	-	-	-	-	
3	EAA5	0.95	-	-	0.05	-	-	-	-	-	
4	EAB5	0.95	-	-	-	0.05	-	-	-	-	
5	FAR30	0.70	-	-	-	-	0.30	-	-	-	
6	FAA30	0.70	-	-	-	-	-	0.30	-	-	
7	FAB30	0.70	-	-	-	-	-	-	0.30	-	
8	FAC30	0.70	-	-	-	-	-	-	-	0.30	
9	EAT5FAR30	0.65	-	0.05	-	-	0.30	-	-	-	
10	EAT5FAA30	0.65	-	0.05	-	-	-	0.30	-	-	
11	EAT5FAB30	0.65	-	0.05	-	-	-	-	0.30	-	
12	EAT5FAC30	0.65	-	0.05	-	-	-	-	-	0.30	
13	EAA5FAB30	0.65	-	-	0.05	-	-	-	0.30	-	
14	EAB5FAB30	0.65	-	-	-	0.05	-	-	0.30	-	
15	W30	1.00	-	-	-	-	-	-	-	-	0.30
16	W30EAD5	1.00	0.05	-	-	-	-	-	-	-	
17	W30EAT5	0.95	-	0.05	-	-	-	-	-	-	
18	W30EAA5	0.95	-	-	0.05	-	-	-	-	-	
19	W30EAB5	0.95	-	-	-	0.05	-	-	-	-	
20	W30EAT5FAR30	0.65	-	0.05	-	-	0.30	-	-	-	
21	W30EAT5FAA30	0.65	-	0.05	-	-	-	0.30	-	-	
22	W30EAT5FAB30	0.65	-	0.05	-	-	-	-	0.30	-	
23	W30EAA5FAR30	0.65	-	-	0.05	-	0.30	-	-	-	
24	W30EAA5FAA30	0.65	-	-	0.05	-	-	0.30	-	-	
25	W30EAA5FAB30	0.65	-	-	0.05	-	-	-	0.30	-	

Table 3.9 Mix proportions of mortars

No.	Mix ID	S/B	Portland cement type 1, ratio by weight	Expansive additives, ratio by weight			W/B
				EAT	EAA	EAB	
1	OPC	2.75	1.00	-	-	-	Water requirement*
2	EAT5	2.75	0.95	0.05	-	-	
3	EAA5	2.75	0.95	-	0.05	-	
4	EAB5	2.75	0.95	-	-	0.05	

*Water content at constant flow of 110±5 %

Table 3.10 Mix proportions of concrete

No.	Mix ID	γ	Proportions of concrete per 1 m^3, kg							
			Portland cement type 1	Expansive additives		Fly ashes		Sand	Lime stone	water
1	W45	1.4	416.02	-		-		767.30	1,062.96	187.21
2	W45EAD20	1.4	395.31	EAD	20.00	-		767.30	1,062.96	186.89
3	W45EAT20	1.4	395.67	EAT	20.00	-		767.30	1,062.96	187.05
4	W45EAA20	1.4	395.43	EAA	20.00	-		767.30	1,062.96	186.94
5	W45EAB20	1.4	394.95	EAB	20.00	-		767.30	1,062.96	186.73
6	W45FAR30	1.4	274.43	-		FAR	117.61	767.30	1,062.96	176.42
7	W45FAA30	1.4	276.61	-		FAA	118.55	767.30	1,062.96	177.82
8	W45FAB10	1.4	370.96	-		FAB	41.22	767.30	1,062.96	185.48
9	W45FAB30	1.4	283.28	-		FAB	121.41	767.30	1,062.96	182.11
10	W45FAC30	1.4	283.28	-		FAC	121.41	767.30	1,062.96	182.11
11	W45EAT20FAR30	1.4	254.19	EAT	20.00	FAR	117.51	767.30	1,062.96	176.27
12	W45EAT20FAA30	1.4	256.38	EAT	20.00	FAA	118.45	767.30	1,062.96	177.67
13	W45EAT20FAB10	1.4	350.64	EAT	20.00	FAB	41.18	767.30	1,062.96	185.32
14	W45EAT20FAB30	1.4	263.04	EAT	20.00	FAB	121.30	767.30	1,062.96	181.95
15	W45EAT20FAC30	1.4	263.04	EAT	20.00	FAC	121.30	767.30	1,062.96	181.95
16	W45EAT30FAA30	1.4	246.26	EAT	30.00	FAA	118.40	767.30	1,062.96	177.60
17	W45EAT30FAB10	1.4	340.48	EAT	30.00	FAB	41.16	767.30	1,062.96	185.24
18	W45EAT30FAB30	1.4	252.92	EAT	30.00	FAB	121.25	767.30	1,062.96	181.88
19	W45EAA20FAR30	1.4	254.04	EAA	20.00	FAR	117.45	767.30	1,062.96	176.17
20	W45EAA20FAA30	1.4	256.22	EAA	20.00	FAA	118.38	767.30	1,062.96	177.57
21	W45EAA20FAB10	1.4	350.43	EAA	20.00	FAB	41.16	767.30	1,062.96	185.21
22	W45EAA20FAB30	1.4	262.88	EAA	20.00	FAB	121.23	767.30	1,062.96	181.85
23	W45EAA20FAC30	1.4	262.88	EAA	20.00	FAC	121.23	767.30	1,062.96	181.85
24	W45EAA30FAA30	1.4	246.03	EAA	30.00	FAA	118.30	767.30	1,062.96	177.44
25	W45EAA30FAB10	1.4	340.17	EAA	30.00	FAB	41.13	767.30	1,062.96	185.08
26	W45EAA30FAB30	1.4	252.68	EAA	30.00	FAB	121.15	767.30	1,062.96	181.72
27	W45EAB20FAR30	1.4	253.73	EAB	20.00	FAR	117.31	767.30	1,062.96	175.97
28	W45EAB20FAA30	1.4	255.90	EAB	20.00	FAA	118.24	767.30	1,062.96	177.37
29	W45EAB20FAB30	1.4	262.55	EAB	20.00	FAB	121.09	767.30	1,062.96	181.64
30	W45EAB20FAC30	1.4	262.55	EAB	20.00	FAC	121.09	767.30	1,062.96	181.64

3.4 Method of testing

3.4.1 Setting time of paste

Setting time of paste was determined according to ASTM C 191 [45]. The apparatus for testing setting time of paste is shown in Figure 3.4.

Figure 3.4 Apparatus for testing setting time of paste

3.4.2 Water requirement of mortar

Water requirement of mortar was tested following the procedure in ASTM C 1437 [46]. Water requirements of different proportions of mortar mixtures were determined for a constant flow value at 110±5 %. The apparatus for testing water requirement of mortar is shown in Figure 3.5.

Figure 3.5 Apparatus for testing water requirement

3.4.3 Slump

Slump of fresh concrete was tested according to ASTM C 143 [47]. After mixing concrete, the fresh concrete was immediately filled into a damped slump cone with three layers where each layer of the filled concrete was one third of the volume of the slump cone. Each layer of the filled concrete was tamped 25 times by using a tamping rod. The slump cone was vertically lifted after smoothening the surface and removing spilled concrete. The distance between the top of slump cone to the surface of sample was measured. The apparatus for slump test is shown in Figure 3.6.

Figure 3.6 Apparatus for slump test

3.4.4 Free expansion and shrinkage

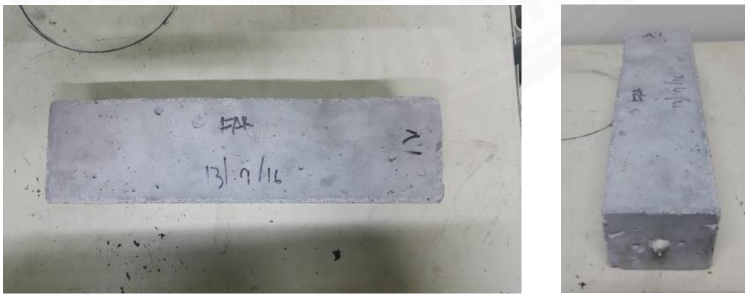
Free expansion and shrinkage tests were conducted according to ASTM C 157/C 157M [48]. Concrete specimens with a size of 75×75×285 mm were used for the tests. For an autogenous shrinkage, the specimens were sealed with paraffin, plastic sheet and aluminum foil to prevent the loss of moisture after demolding. Total shrinkage test specimens were cured in the water for the first 7 days and subsequently exposed to drying environment (28°C and 75% RH). The initial length of the specimens was recorded at the age of 8 hours after mixing. After that, the length change of each specimen was measured every day for the first 7 days, every 2 days for the second week and every week until the shrinkage gradually reduced with time. The length changes of specimens for total shrinkage test at the first 7 days (cured in water) were used as the results of free expansion. The apparatus and specimens for free expansion and shrinkage tests are shown in Figures 3.7 and 3.8.

a) Length comparator

b) Mold for free expansion and shrinkage tests

Figure 3.7 Apparatus for free expansion and shrinkage tests

a) Autogenous shrinkage



b) Free expansion and total shrinkage

Figure 3.8 Specimens for free expansion and shrinkage tests

3.4.5 Compressive strength

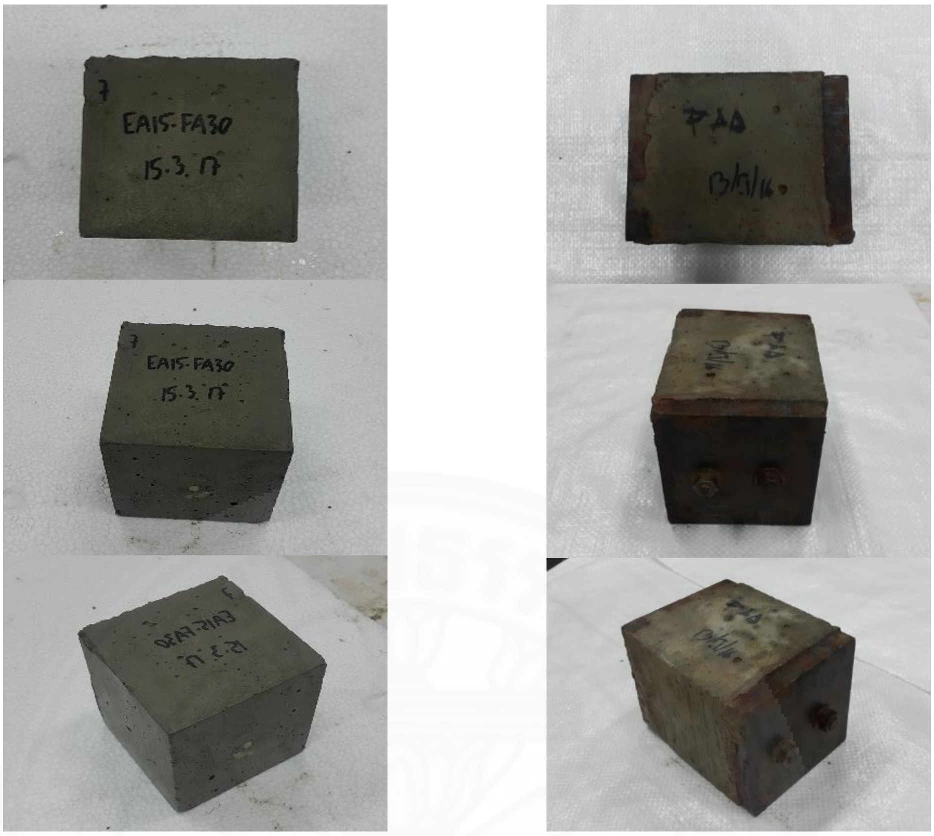
Compressive strength was tested in two conditions, unconfined and confined conditions. The unconfined compressive strength was conducted according to TIS 409 [49]. The concrete cube specimens with a size of 100×100×100 mm were demolded at 24 hours after casting, and cured in water for 3, 7, 28, and 91 days. After curing, the compressive strength of the specimens was measured. The confined compressive strength simulates the concrete in general structures which are always in confined condition. The specific confinement was designed. Two deformed bars with a diameter of 12 mm (DB 12) were fixed at the ends by steel plates with a size of 100x100 mm. To prohibit adhesion between concrete and deformed bars, the deformed bars were sheathed by plastic tubes. The specimens were demolded at 24 hours after casting, and cured in water for 3,7, 28 and 91 days. The steel plates, deformed bars and plastic tubes were cured together with the concrete specimens and were removed before testing. The apparatus and specimens for unconfined and confined compressive strength tests are shown in Figures 3.9 and 3.10.

a) Machine for compressive strength test

b) Mold for unconfined compressive strength test

c) Mold for confined compressive strength

Figure 3.9 Apparatus for compressive strength test



a) Unconfined compressive strength b) Confined compressive strength

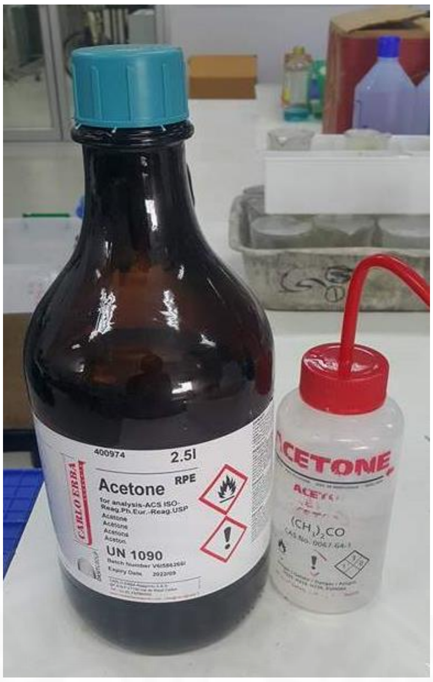
Figure 3.10 Specimens for compressive strength test

3.4.6 Porosity of concrete

Concrete specimens with the same size as the unconfined and confined compressive strength tests were used for testing porosity of concrete. The test was conducted following ASTM C 642 [50]. After curing the specimens in water for 7 days, the specimens were dried in an oven at 100-110 °C for 24 hours and the weights of the specimens were recorded. The specimens were again dried in the oven for more than 24 hours until the weight decrease was not over 0.5%. The final weights of the oven-dried specimens were recorded. After that, the specimens were immersed in the water for 48 hours and the weights of saturated surface-dried specimens were recorded. The specimens were again immersed in water for more than 24 hours until the weight increase was not over 0.5%. The final immersion weights of the surface-dried specimens were recorded. After that, the specimens were boiled for 5 hours, let them naturally cool down for not less than 14 hours to 20-25 °C and the weights of the saturated surface-dried specimens were recorded. After that, the boiled specimens were weighed in water and recorded. After all processes, the density, absorption and permeable voids were determined.

3.4.7 XRD analysis

X-ray Diffraction was used for mineralogical investigation. Paste specimens were cast and cured in water for 7 days. After that, the specimens were cursorily ground to a size between 2.36-4.75 mm (passing sieve No.4 but retained on sieve No.8), and immersed in the acetone for 24 hours. Then the samples were dried in the oven at 50°C for 24 hours to stop the hydration of cement. The dried specimens were ground to the particle size range of 45-75 μm by a planetary ball mill and mixed with corundum (aluminium oxide), an internal standard, by a ground powder to corundum ratio of 9:1. After that, the tests of the prepared specimens were conducted on Bruker D4 Endeavor. Eva program was used for determining the composition of the compound and Rietveld analysis implemented in TOPAS software was used for quantifying the amounts of compounds. The measurement conditions were defined as 0.02 degree for the step angle, 0.2 second for the count time and 5 to 70 degrees for the range of 2θ. The tube voltage and current were 35 kV and 45 mA, respectively. Chemicals for specimen preparation, equipment for specimen preparation and investigation, and specimen for XRD investigation are shown in Figures 11, 12 and 13, respectively.



a) Acetone

b) Corundum

Figure 3.11 Chemicals for specimen preparation of XRD investigation

a) Sieve No.4 and No.8

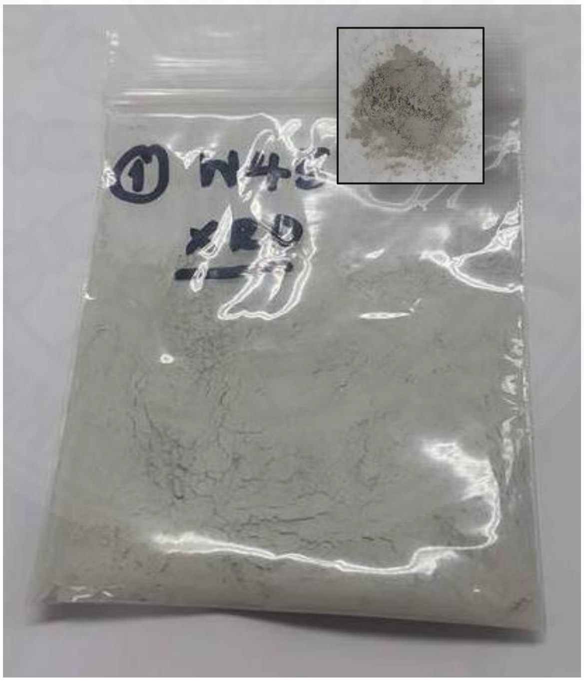
b) 4 decimal place analytical balance

c) Planetary ball mill

d) X-ray Diffraction (XRD) Bruker D4 Endeavor (SRI, Saraburi, Thailand)

Figure 3.12 Equipment for specimen preparation and XRD investigation

a) Sieved specimen with size between 2.36-4.75 mm



b) Specimen for X-ray Diffraction

Figure 3.13 Specimen for XRD investigation

Chapter 4

Results of Setting Time and Slump

4.1 Experimental results on setting time of paste

4.1.1 Effect of expansive additive types

Figure 4.1 shows the results of initial and final setting times of pastes containing different types of expansive additive. The mixtures containing expansive additive showed faster setting than that of the Non-EA (expansive additive) mixture (OPC mixture). The EAT, EAA and EAB mixtures showed faster setting than the Non-EA mixture. When different types of expansive additive were considered, the EAB mixture showed the fastest setting, followed by the EAA and EAT mixtures, respectively. It is possibly because of amount of ettringite which is differently produced in each mixture. The EAT, EAA and EAB expansive additives which respectively contain higher constituents for ettringite formation (Table 3.6). The setting time is accelerated due to the produced ettringite which holds high amount of water in its structure, reducing free water in the mixture [51].

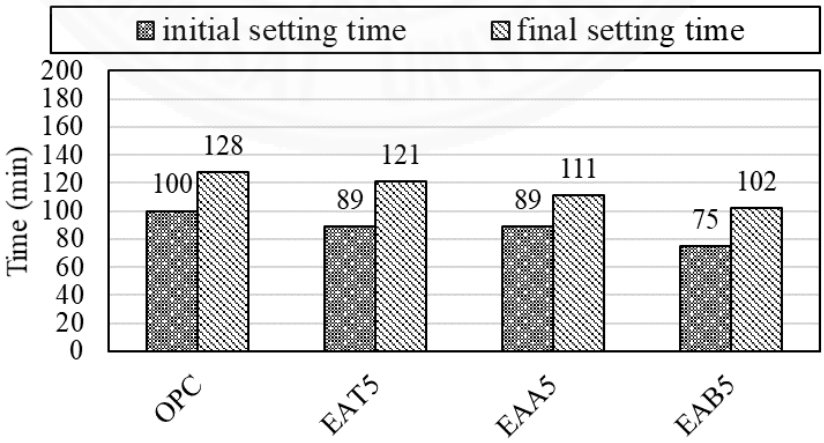
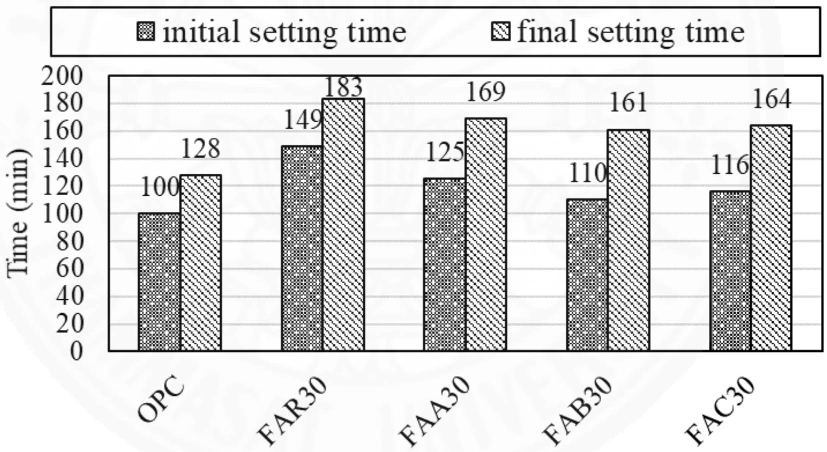


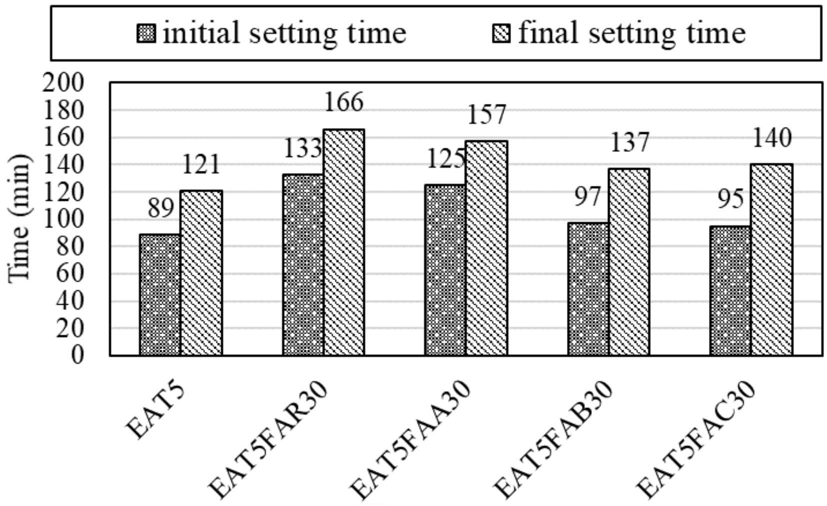
Figure 4.1 Results of initial and final setting times of pastes containing different types of expansive additive

4.1.2 Effect of fly ash types

Figure 4.2 shows the results of setting times of pastes containing different types of fly ash. The use of fly ashes in Non-EA pastes and paste with expansive additive showed similar tendency. It can be seen that the mixtures containing all types of fly ash showed slower setting than that of Non-FA (fly ash) mixtures (both OPC and EAT mixtures). When different types of fly ash were considered, the low CaO fly ash (FAR) mixtures showed the slowest setting, followed by the high CaO fly ash (FAA), and high free lime and SO_3 fly ashes (FAB and FAC) mixtures, respectively (Figure 4.2 a-b). This is because the setting time is accelerated by formation of $Ca(OH)_2$ and ettringite which are enhanced by the free lime and SO_3 in the high free lime and SO_3 fly ashes (FAB and FAC). Moreover, high free lime and SO_3 fly ashes (FAB and FAC) also contain higher amount of CaO which possess some cementitious properties, resulting in faster paste setting when compared with the high CaO fly ash (FAA) (Table 3.4).



a) Non-EA pastes



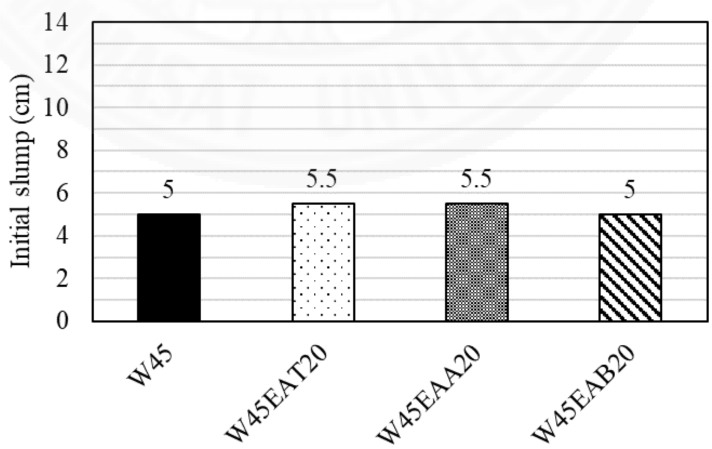
b) Pastes with EAT expansive additive

Figure 4.2 Results of initial and final setting times of pastes containing different types of fly ash

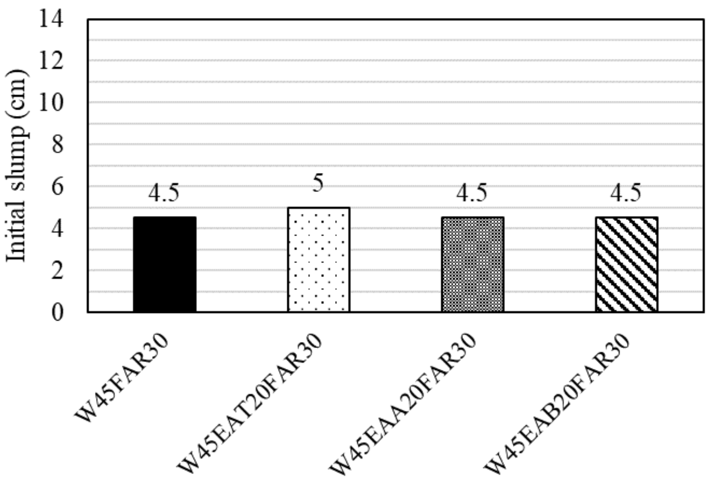
4.2 Experimental results on slump of concrete

4.2.1 Effect of expansive additive types

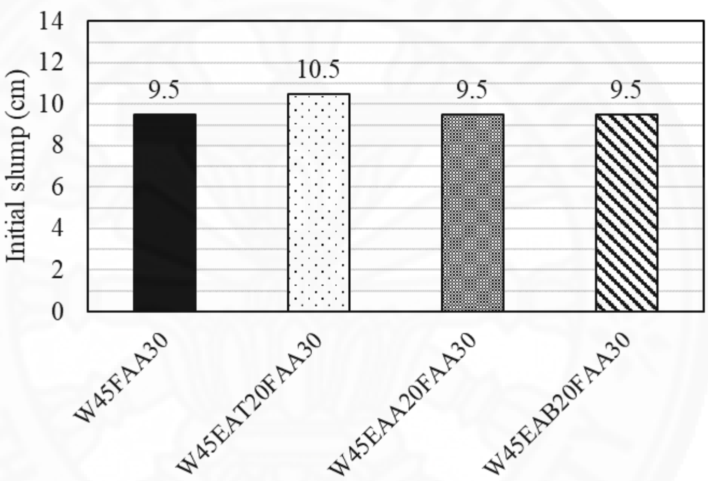
Figure 4.3 shows the results of initial slump of concrete containing different types of expansive additive. Considering the effect of expansive additive types, the slump values showed similar tendency for Non-FA concrete (Figure 4.3a) and fly ash concretes (Figure 4.3b-e). The results of slump showed the same range in each mixture, but the tendency also could be seen. The results of slump were slightly different in mixtures with different types of expansive additive. The initial slump tended to be slightly higher in EAT mixtures, comparable in EAA mixtures and slightly lower in EAB mixtures when comparing with their respective Non-EA mixtures. When different types of expansive concrete were considered, the EAB mixtures showed the lowest slump, and showed slightly higher values in EAA and EAT mixtures, respectively. The tendency of initial slump could be explained by the different water requirement of mortars. It can be seen in Table 4.1, showing the result of water requirement of mortars, that the EAB mixture showed the highest water requirement, and showed slightly lower for the EAA, EAT mixtures, respectively. It corresponds to the results of slump of concrete because the mixture with high amount of water requirement reduces free water in the mixture with controlled water content, resulting in a reduction of slump.



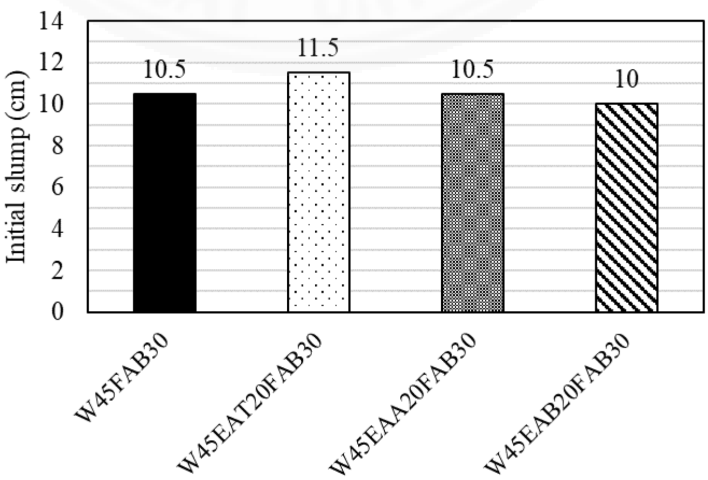
a) Non-FA concrete



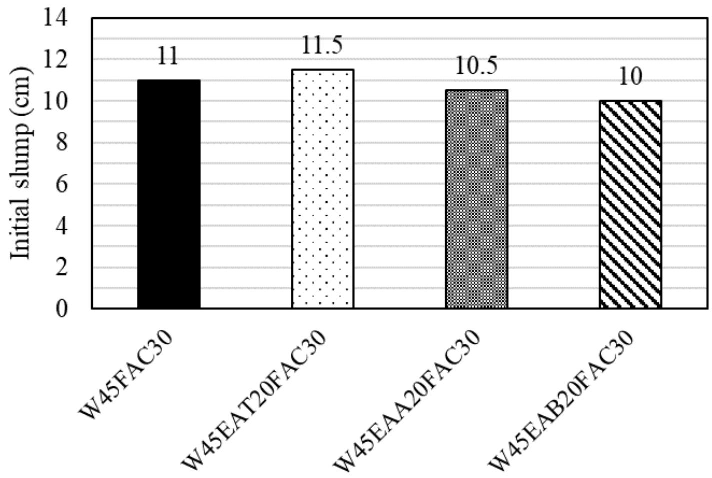
b) FAR fly ash concrete



c) FAA fly ash concrete



d) FAB fly ash concrete



e) FAC fly ash concrete

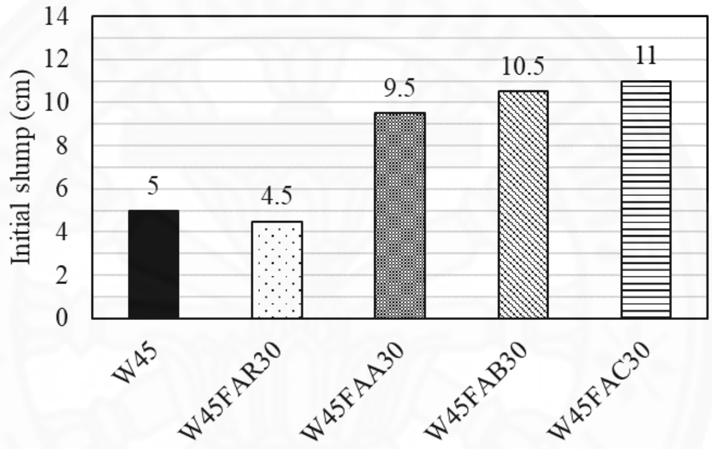
Figure 4.3 Results of initial slump of concrete containing different types of expansive additive

Table 4.1 Result of water requirement of mortars

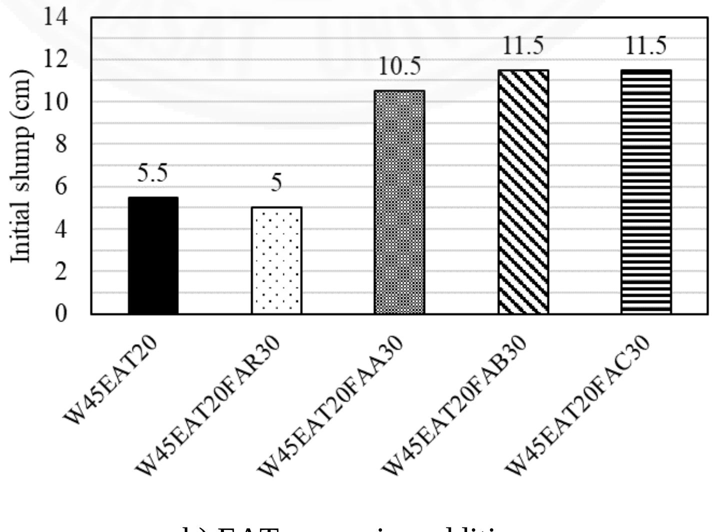
Mix ID	Water requirement (%)
OPC	100.00
EAT5	98.18
EAA5	99.79
EAB5	103.59

4.2.2 Effect of fly ash types

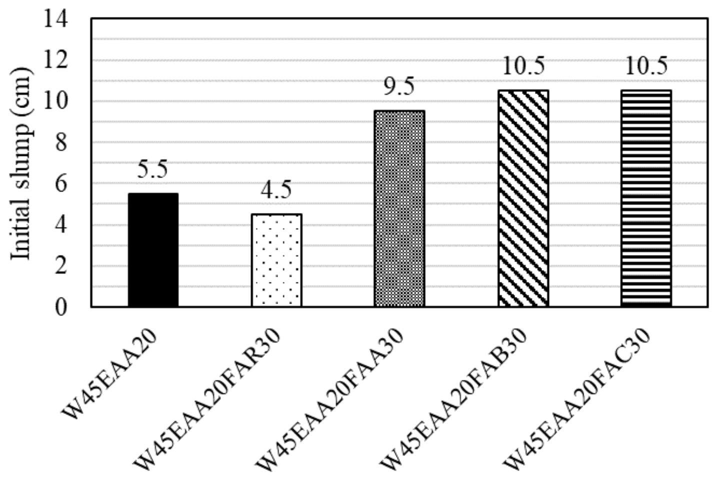
Figure 4.4 shows the results of initial slump of concrete containing different types of fly ash. The slump of concrete containing different types of fly ash showed similar tendency for Non-EA concrete (Figure 4.4a) and expansive concretes (Figure 4.4b-d). The low CaO fly ash (FAR) mixtures gave comparable slump with the Non-FA mixtures, and gave the lowest slump when compared with other types of fly ashes mixtures. The mixtures with high CaO fly ash (FAA) and high free lime and SO_3 fly ashes (FAB and FAC) mixtures gave higher slump than that of the Non-FA and low CaO fly ash mixtures. The above results corresponded to the results of water requirement of mortars containing different types of fly ash which was revealed by Nawaz et al. that the water requirement of mortars was the lowest in the high free lime and SO_3 fly ashes and the high CaO fly ash mixtures which is followed by the low CaO fly ash mixtures [19]. This is because the low CaO fly ash (fly ash from BLCP power plant) is finer, consists higher amount of LOI than the other fly ashes, and contains much larger amount of non-spherical particles (Table 3.4 and Figure 3.2a), resulting in higher water requirement. Therefore, the slump is lower when compared with the other fly ashes mixtures. On the other hand, high CaO fly ash and high free lime and SO_3 fly ashes (fly ashes from Mae Moh power plant) are coarser, consist of lower amount of LOI than the FAR fly ash, and contain much larger amount of spherical particles, resulting in lower water requirement. Therefore, the slump is higher when compared with the other fly ash mixtures [52, 53].



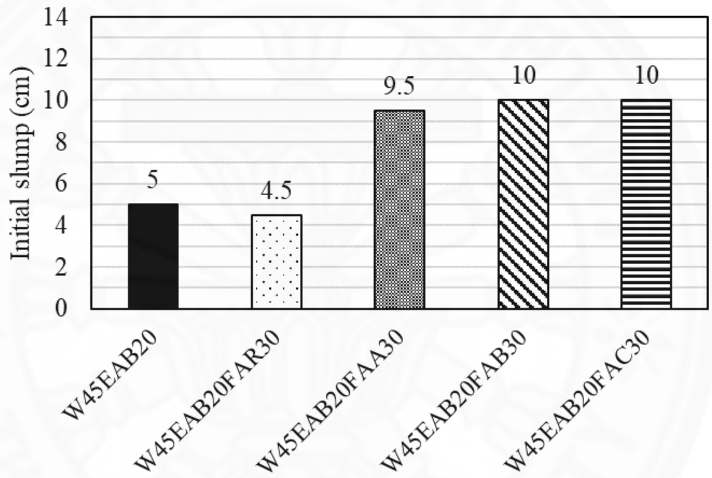
a) Non-EA concrete



b) EAT expansive additive



c) EAA expansive concrete



d) EAB expansive concrete

Figure 4.4 Results of initial slump of concrete containing different types of fly ash

Chapter 5

Results and Clarifications of Free Expansion and Shrinkage

5.1 Experimental results on free expansion

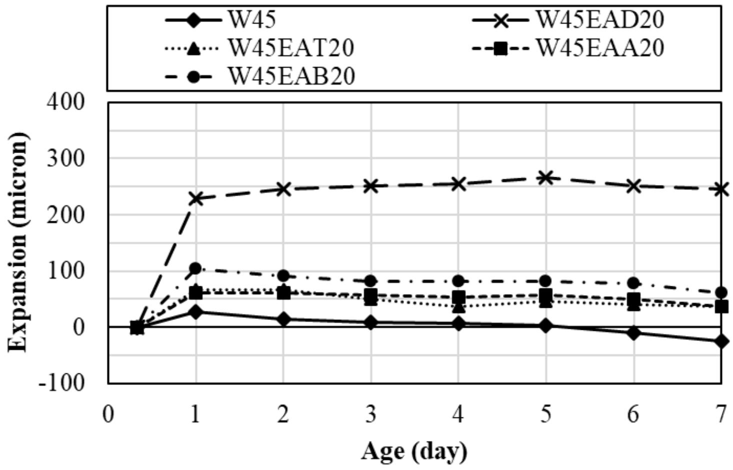
5.1.1 Effect of expansive additive types

Figure 5.1 shows the results of free expansion of concrete containing different types of expansive additive. In case of Non-FA concrete (Figure 5.1a), all types of expansive concrete showed larger expansion than that of Non-EA concretes. The EAT and EAA expansive concretes had similar expansion, and the EAB expansive concrete had larger expansion than that of EAT and EAA expansive concretes. However, the expansion capacity of EAT, EAA and EAB expansive concretes congregated at same range, while the EAD expansive concrete had the highest expansion and showed obvious difference when compared with the other types of expansive concrete. In case of fly ash concretes (Figure 5.1b-e), the tendency of the expansion also showed the same as that of the Non-FA concrete, but the performance of expansion enhancement in each types of expansive concrete were different. The EAT and EAA mixtures also had similar expansion capacity while the EAB mixture had larger expansion than those of EAT and EAA mixtures. The different expansion between EAB expansive concrete, EAA and EAT expansive concretes could be more and more obviously seen in low CaO fly ash, high CaO fly ash and high free lime and SO_3 fly ashes mixtures, respectively.

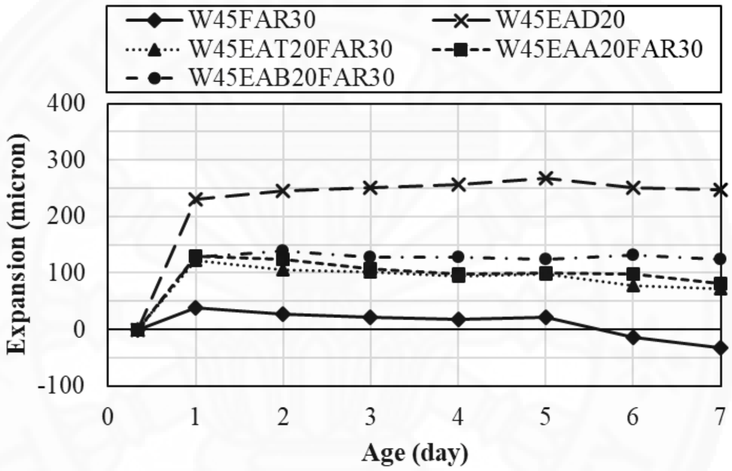
Although the EAD was the most effective expansive additive for generating expansion when compared with others, its cost must be considered. It is because the EAD expansive additive is the imported material from aboard. However, the domestic materials usage that have comparative performances can solve this problem because it is cheaper than the imported material. Unfortunately, the use of EAA and EAB expansive additives which are the domestically manufactured expansive additives showed lower expansion performance than the use of EAD expansive additive (Figure 5.1a). Therefore, fly ashes which are the domestically waste materials were used for enhancing the expansion performance of EAA and EAB expansive concretes. It can be noted that, although, the EAT expansive additive is not the domestic material, the expansion performance was similar with those of EAA and EAB expansive additive when used in concrete. Therefore, the expansion performance of concrete with EAT expansive additive was tested in the same manner with the case of EAA and EAB expansive additives.

When all types of expansive concrete (EAT, EAA and EAB expansive additives) with fly ashes were compared with the EAD (Non-FA) expansive concrete (Figure 5.1b-e), the expansion were lower in case of low CaO fly ash and high CaO fly ash mixtures, but were at least comparable or tended to be higher in case of high free lime and SO_3 fly ashes mixtures. This means that the use of EAT, EAA and EAB expansive additives which showed the low expansion in case of Non-FA concrete, but by incorporating the high free lime and SO_3 fly ashes (FAB and FAC) in concrete, could enhance the expansion to be comparable to the EAD (Non-FA) expansive additive which was the most effective mixture giving the highest expansion in case of Non-FA concrete (Figure 5.1a).

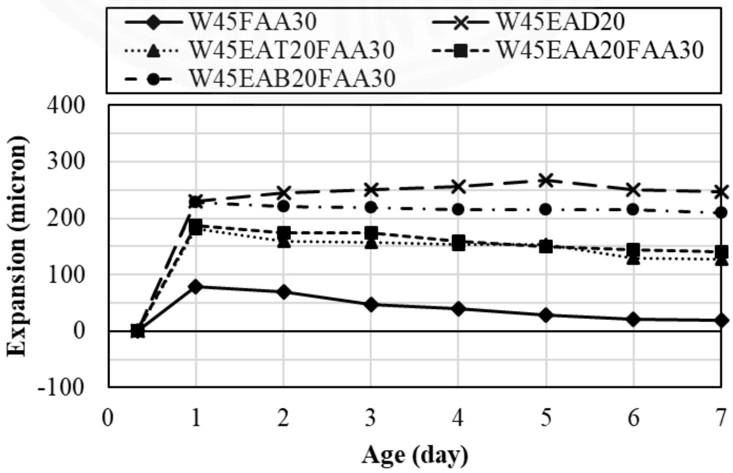
The effectiveness of expansion in different types of expansive concrete may occur due to many factors. Firstly, from the chemical aspects, the difference of ingredients for expansive products formation in each type of the expansive additive may be the main factor [54]. The ettringite may be more produced in EAB, EAA and EAT expansive concretes, in order, because of the Yeelimite (C_4A_3S), which is the main constituent for ettringite formation, mostly contains in the EAB expansive additive, followed by the EAA and EAT additives, respectively (Table 5.1 and Table 3.6). Inversely, mixtures with EAT and EAA expansive additives may produce the highest amount of $Ca(OH)_2$ due to the higher free lime content than the mixture with EAB expansive additive. However, the period of expansive products formation may be another factor resulting in the effectiveness of the generated expansion. The expansion is only effective when expansive products are generated after setting, but it is not productive if the expansive products are produced before setting time. Moreover, the different paste stiffness in each types of expansive concrete may result in the different effectiveness of generated expansion. Since these factors are combined, it is difficult to verify each effect separately.



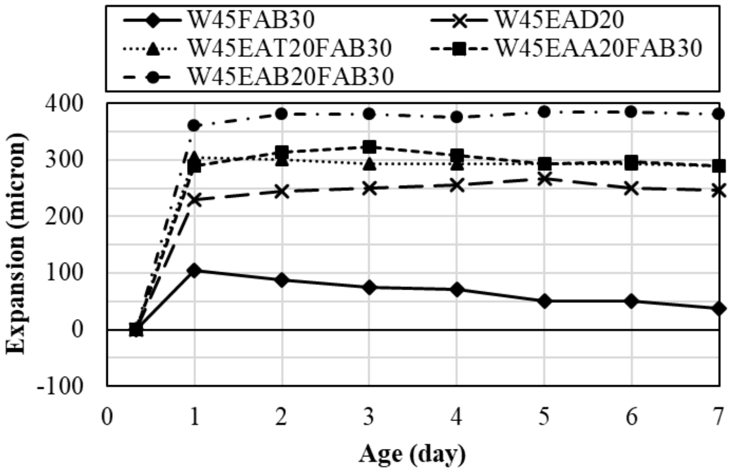
a) Non-FA concrete



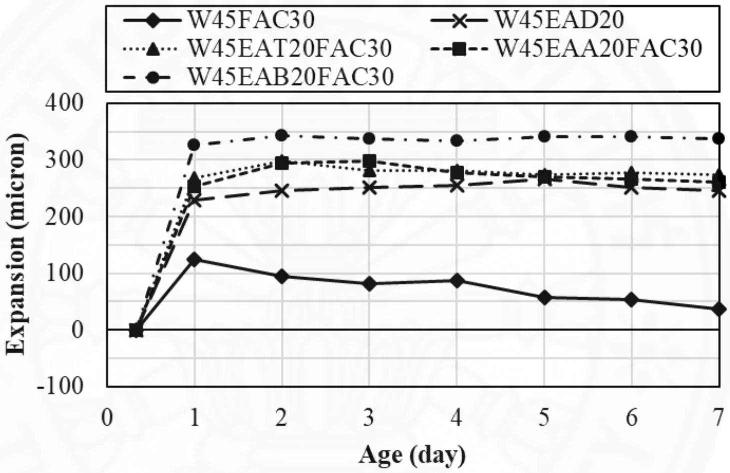
b) FAR fly ash concrete



c) FAA fly ash concrete



d) FAB fly ash concrete



e) FAC fly ash concrete

Figure 5.1 Results of free expansion of concrete containing different types of expansive additive

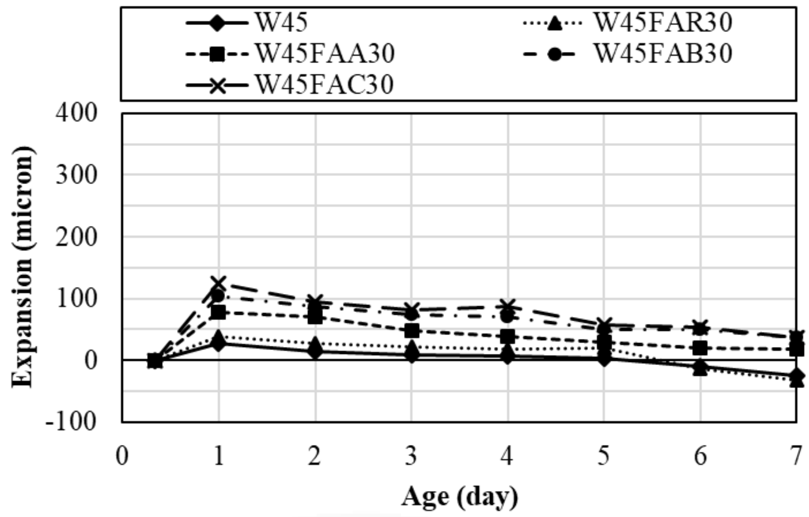
Table 5.1 Chemical compounds for ettringite formation of different types of expansive additive [55]

Type of expansive additive	Chemical formula	
K	$C_4A_3S + 8CS + 6CH + 90H$	$\rightarrow 3C_6AS_3H_{32}$
M	$CA + 3CS + 2CH + 30H$	$\rightarrow C_6AS_3H_{32}$
S	$C_3A + 3CS + 32H$	$\rightarrow C_6AS_3H_{32}$

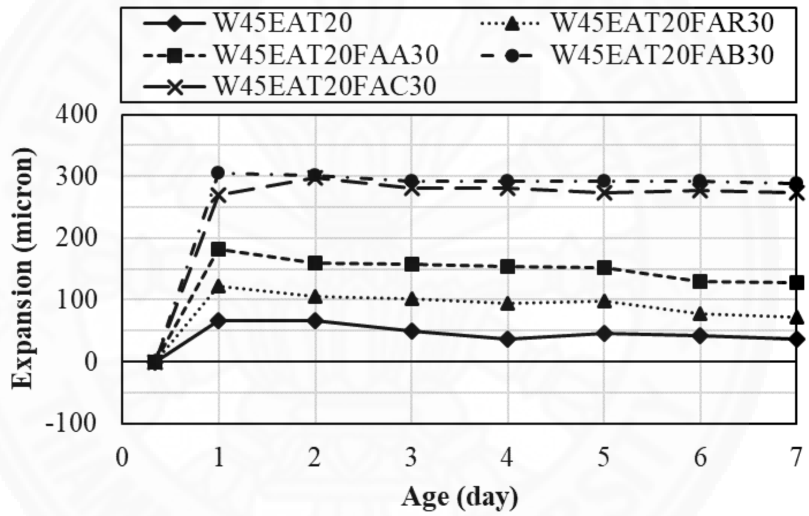
C_4A_3S : Calcium sulfoaluminate
CS : Calcium sulfate
CH : Calcium hydroxide
H : Water
CA : Calcium aluminate
C_3A : Tricalcium aluminate
$C_6AS_3H_{32}$: Ettringite

5.1.2 Effect of fly ash types

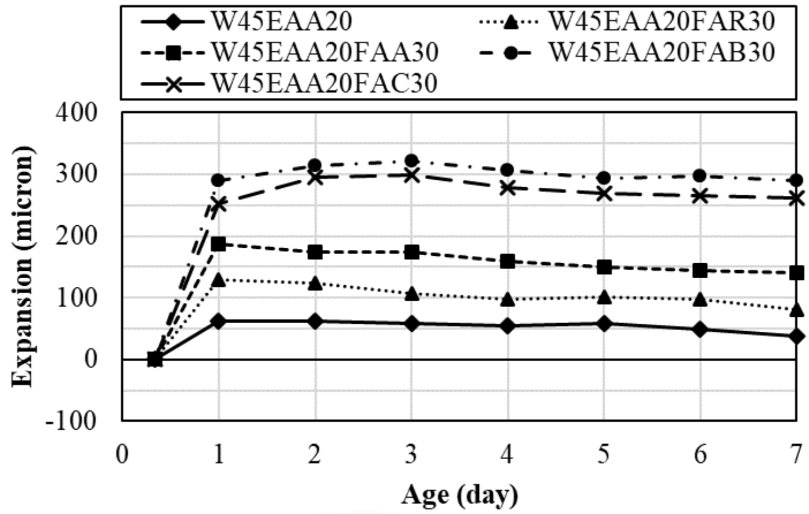
Figure 5.2 shows the results of free expansion of concrete containing different types of fly ash. In case of Non-EA concrete (Figure 5.2a), the Non-FA and low CaO fly ash (FAR) concretes showed almost the same expansion. The expansion at the age of 7 days of concrete showed the highest in high free lime and SO_3 fly ashes concrete, followed by the high CaO fly ash concrete, and Non-FA and low CaO fly ash concretes, respectively. It is possibly because free lime and SO_3 contents in fly ash generate expansion due to formation of $Ca(OH)_2$ and ettringite. Therefore, the expansion of concrete showed the highest in mixture with fly ash containing the highest amounts of free lime and SO_3. In case of expansive concretes (Figure 5.2b-d), the expansion also showed similar tendency with the case of Non-EA concrete, but the expansion was further enhanced when low CaO fly ash, high CaO fly ash and high free lime and SO_3 fly ashes were used, respectively. The cause of the different expansion enhancement in each types of fly ash mixture also occur from the early age paste stiffness of fly ash mixture which is lower than that of mixture without fly ash, therefore concrete expand easily. In addition, the additional expansive products are produced from the presence of free lime and SO_3 in fly ashes [20]. Moreover, it is also possible that fly ash delays the expansive additive reaction causing less expansion to occur before the mixtures get hardened. It is noted that the expansion reaction occurring during fresh and plastic states is considered ineffective since it does not contribute to the expansion of the hardened mixtures.



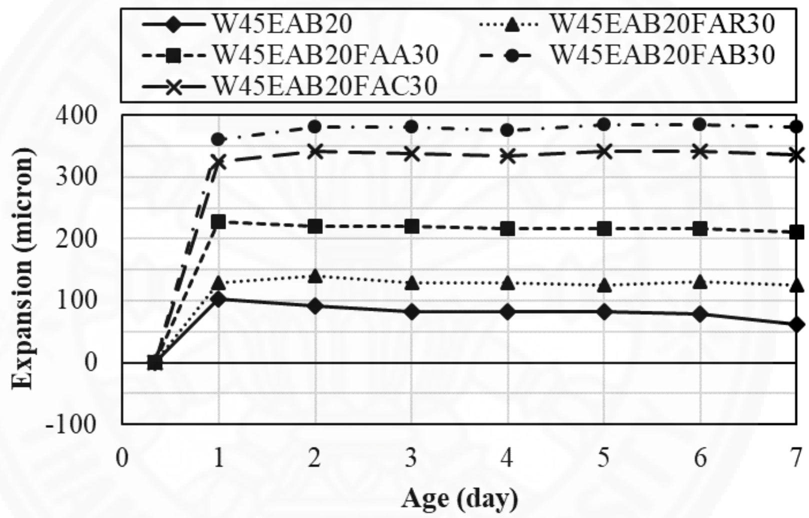
a) Non-EA concrete



b) EAT expansive concrete



c) EAA expansive concrete

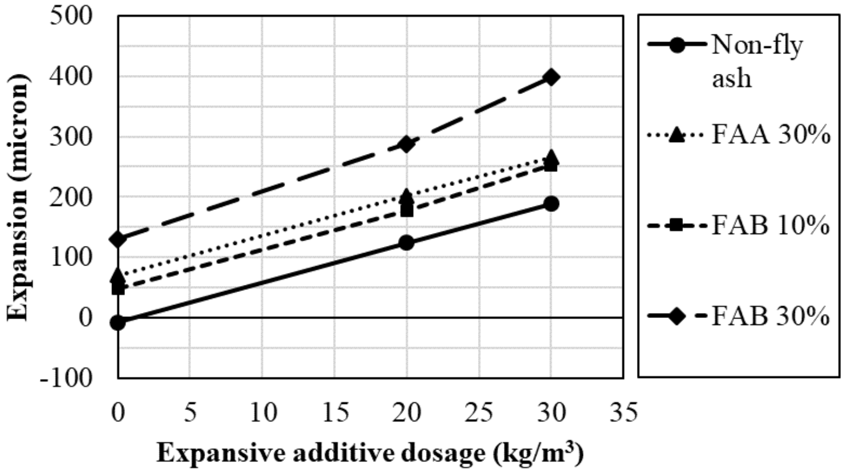


d) EAB expansive concrete

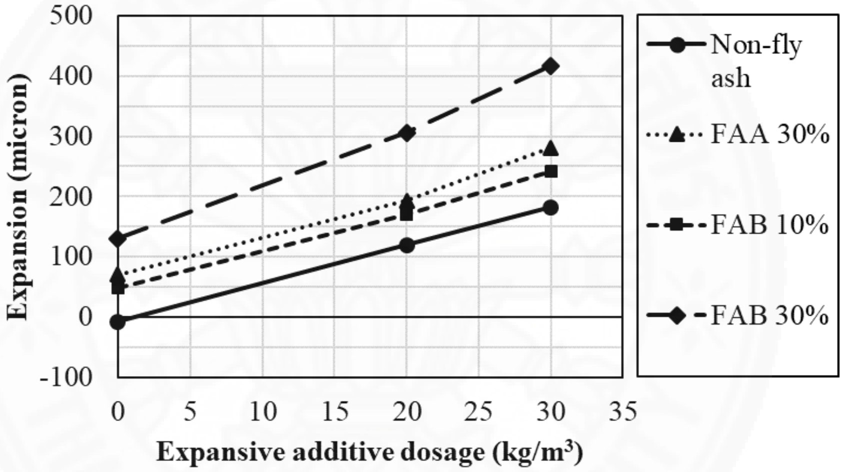
Figure 5.2 Results of free expansion of concrete containing different types of fly ash

5.1.3 Effectiveness of fly ashes for reducing expansive additives usage

Figure 5.3 shows the results of free expansion of Non-FA and fly ash concretes containing different dosages of expansive additives at the age of 7 days. it is observed that the use of fly ash especially in the case of high free lime and SO_3 fly ash was effective to enhance expansion of expansive concrete. It can be seen from the expansion of EAT and EAA expansive concretes (Figure 5.3a-b) that the mixtures replacing at 30% high free lime and SO_3 fly ash (represented by FAB fly ash) was the most effective mixture to enhance expansion at all dosages of expansive additive, followed by the mixtures replacing 30% high CaO fly ash (FAA) and 10% high free lime and SO_3 fly ash, respectively. However, the 10% high free lime and SO_3 fly ash mixtures also showed almost similar expansion values to the 30% high CaO fly ash mixtures. In the same way, the use of fly ash could reduce the use of expansive additive dosage. For example, when considering fly ash concrete having similar expansion values with the Non-FA concrete containing 30 kg/m^3 of EAT and EAA expansive additive, the use of 30% high free lime and SO_3 fly ash, 30% high CaO fly ash and 10% high free lime and SO_3 fly ash could produce the expansion at same range of Non-FA mixtures at the expansive additive usage approximately of 8, 18 and 22 kg /m^3, respectively. This means that the use of fly ashes could reduce the expansive additive usage approximately to 22, 12 and 8 kg/m^3 for mixtures with 30% high free lime and SO_3 fly ash, 30% high CaO fly ash and 10% high free lime and SO_3 fly ash, respectively.



a) EAT expansive concrete



b) EAA expansive concrete

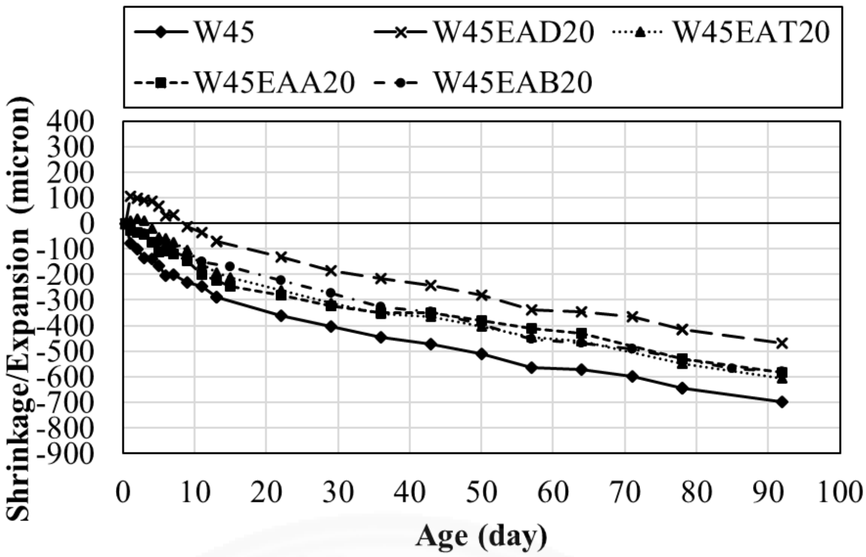
Figure 5.3 Results of free expansion of Non-FA and fly ash concretes containing different dosages of expansive additive at the age of 7 days

5.2 Experimental results on shrinkage

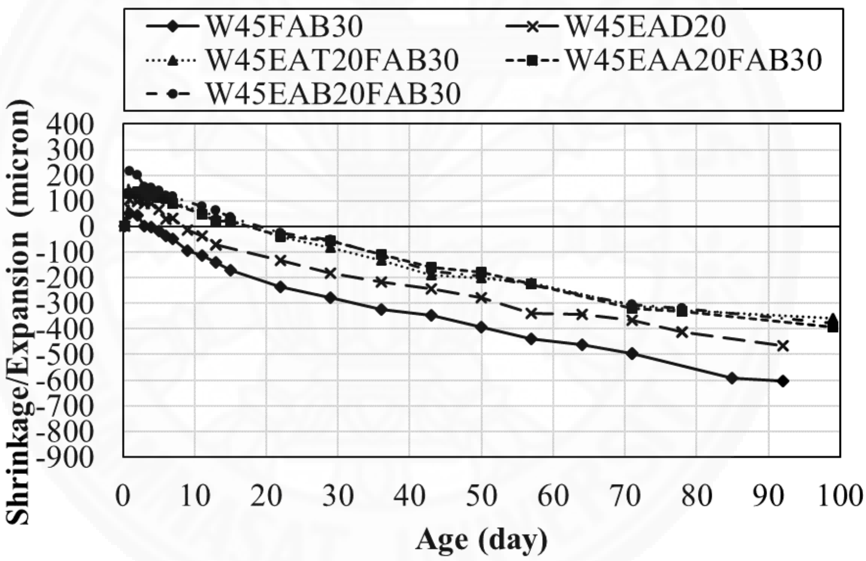
5.2.1 Autogenous shrinkage

5.2.1.1 Effect of expansive additive types

The use of expansive additives could reduce the autogenous shrinkage of concrete. It can be seen in Figure 5.4, showing the results of autogenous shrinkage of concrete containing different types of expansive additive, that all types of expansive concrete resulted in lower autogenous shrinkage than that of Non-EA concrete. In case of Non-FA concrete (Figure 5.4a), the EAD expansive concrete had the smallest autogenous shrinkage when comparing with other types of expansive concrete. The EAT, EAA and EAB expansive concretes showed similar autogenous shrinkage values which were higher than that of the EAD expansive concrete. However, for fly ash concretes (Figure 5.4b), the EAT, EAA and EAB mixtures showed similar autogenous shrinkage values, and were lower than that of the EAD (Non-FA) expansive concrete which was the most effective mixture giving the highest expansion at the early age in case of Non-FA concrete (Figure 5.1).



a) Non-FA concrete

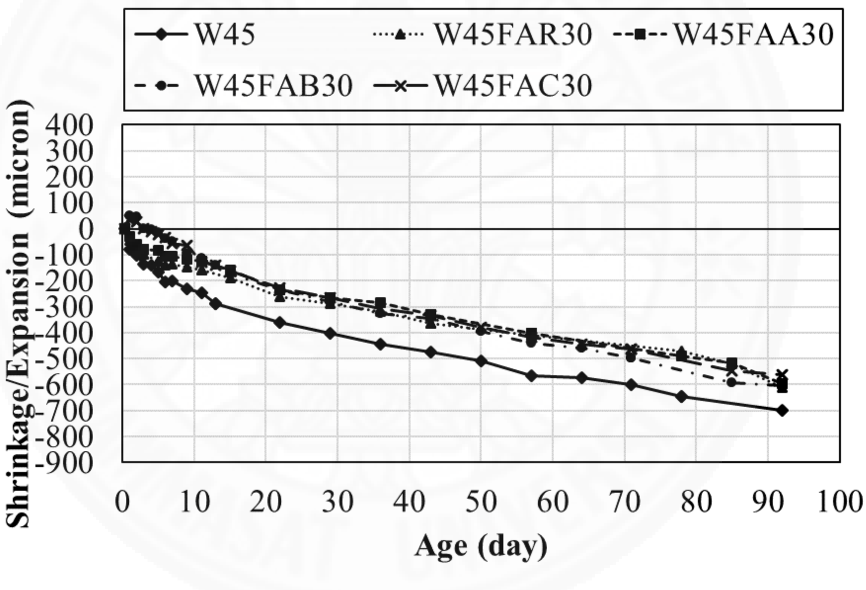


b) FAB fly ash concrete

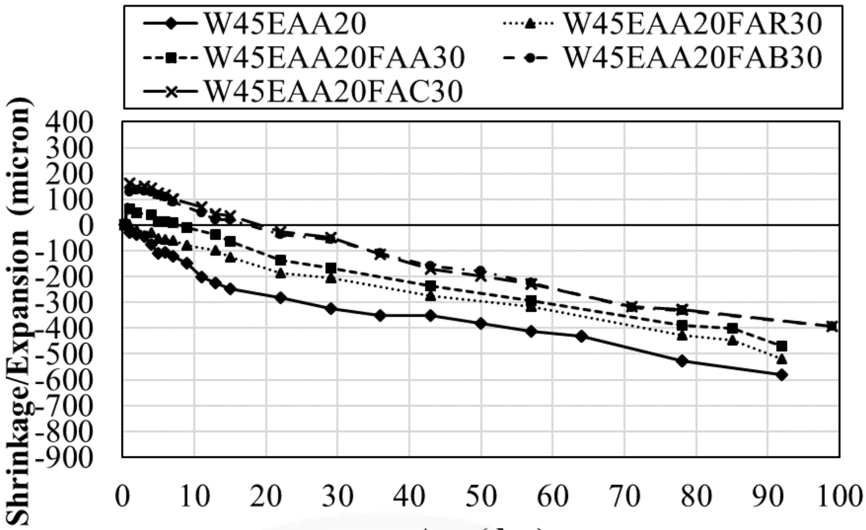
Figure 5.4 Results of autogenous shrinkage of concrete containing different types of expansive additive

5.2.1.2 Effect of fly ash types

The use of all types of fly ash in Non-EA concrete and expansive concrete could reduce the autogenous shrinkage. It can be seen from Figure 5.5, showing the results of autogenous shrinkage of concrete containing different types of fly ash, that all types of fly ash mixtures had smaller autogenous shrinkage than that of Non-FA mixtures. In case of Non-EA concrete (Figure 5.5a), the low CaO fly ash (FAR), high CaO fly ash (FAA) and high free lime and SO_3 fly ashes (FAB and FAC) mixtures had similar autogenous shrinkage values. Inversely, in case of expansive concrete (Figure 5.5b), the autogenous shrinkage values evidently showed the smallest in high free lime and SO_3 fly ashes mixtures, followed by high CaO fly ash and low CaO fly ash mixtures, respectively.



a) Non-EA concrete



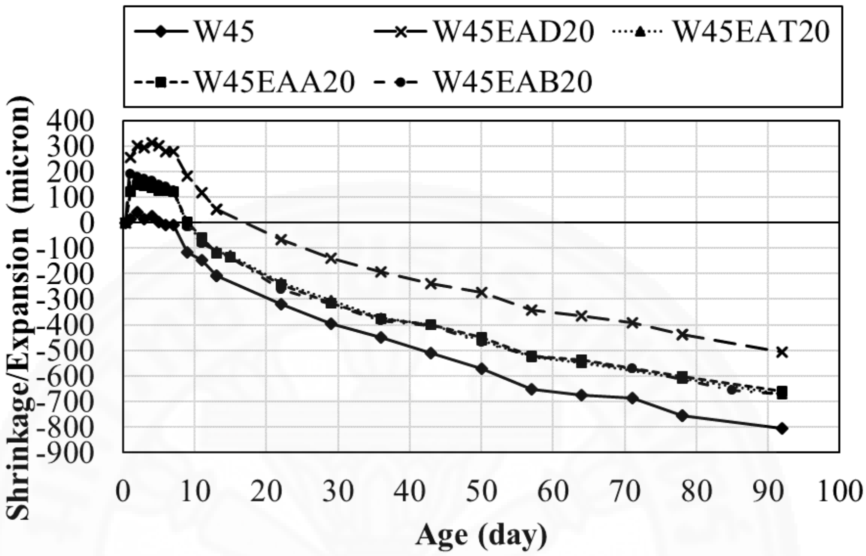
b) EAA expansive concrete

Figure 5.5 Results of autogenous shrinkage of concrete containing different types of fly ash

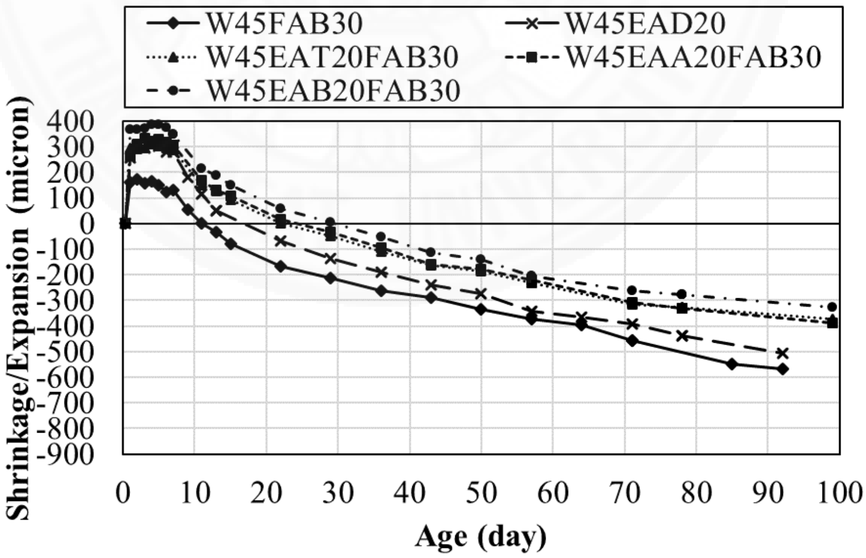
5.2.2 Total shrinkage

5.2.2.1 Effect of expansive additive types

Figure 5.6 shows the results of total shrinkage of concrete containing different types of expansive additive. It can be seen that the results of the effect of expansive additive types on total shrinkage showed similar tendency to the results of autogenous shrinkage. The use of all types of expansive concrete could reduce the total shrinkage when compared to that of the Non-EA concrete. In case of Non-FA concrete (Figure 5.6a), the EAD expansive concrete had the smallest total shrinkage when compared with other types of expansive concrete. Similar to the results of autogenous shrinkage, the EAT, EAA and EAB expansive concretes showed similar total shrinkage values, and had higher total shrinkage values than that of the EAD expansive concrete. In case of fly ash concrete (Figure 5.6b), the total shrinkage of EAT, EAA and EAB mixtures were lower than that of the EAD (Non-FA) expansive concrete which was the most effective mixture giving the highest expansion at the early age in case of Non-FA concrete (Figure 5.1). When different types of expansive additive were used in fly ash concrete, the results of total shrinkage had a slightly different tendency to the results of autogenous shrinkage. The EAT and EAA mixtures had similar total shrinkage values, whereas the EAB expansive concrete with fly ash showed slightly lower values than that of the EAT and EAA mixtures. It can be noted that, if the sufficient curing at the early age is provided, the expansion could be effectively enhanced, especially in fly ash mixtures. By this reason, it directly resulted in the reduction of shrinkage at long term.



a) Normal concrete

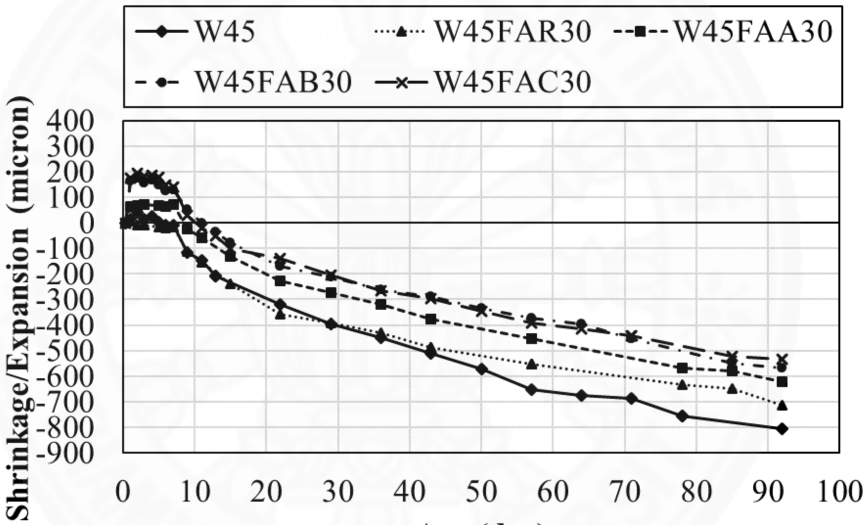


b) FAB fly ash concrete

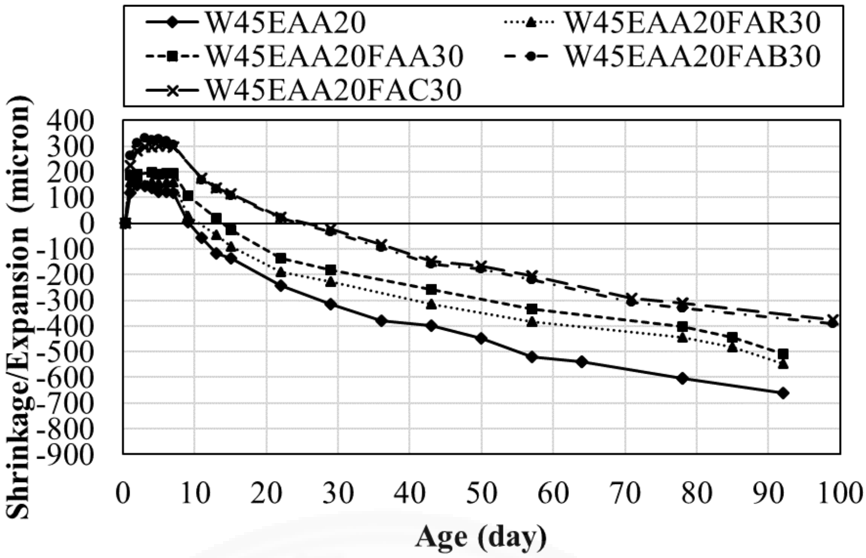
Figure 5.6 Results of total shrinkage of concrete containing different types of expansive additive

5.2.2.2 Effect of fly ash types

Figure 5.7 shows the results of total shrinkage of concrete containing different types of fly ash. The results of total shrinkage showed similar tendency for both Non-EA concrete (Figure 5.7a) and expansive concrete (Figure 5.7b). All types of fly ash mixtures had lower total shrinkage values than that of Non-FA mixtures. When different types of fly ash were considered, the total shrinkage values showed the lowest in high free lime and SO_3 fly ashes mixtures (FAB and FAC), followed by high CaO fly ash (FAA) and low CaO fly ash (FAR) mixtures, respectively. It can be noted that the reduction of total shrinkage was mainly caused by the difference of the significant expansion which was generated at the early age in each mixture.



a) Non-EA concrete



b) EAA expansive concrete

Figure 5.7 Results of total shrinkage of concrete containing different types of fly ash

5.2.3 Shrinkage of concrete in dry curing period

To clearly see the effectiveness of shrinkage reduction at long term state, the results of total shrinkage which excluded the generated expansion during water-cured period at the early age was simply calculated by Equation (1) and the results are shown in Figures 5.8 and 5.9.

$$\mathcal{E}S_{dry}(t) = \mathcal{E}S_{total}(t) - \mathcal{E}S_{total}(7) \qquad (1)$$

Where $\mathcal{E}S_{dry}(t)$ = Shrinkage values in dry curing period after 7 days of water curing (micron)

$\mathcal{E}S_{total}(t)$ = Total shrinkage values at the age of *t* days (micron)

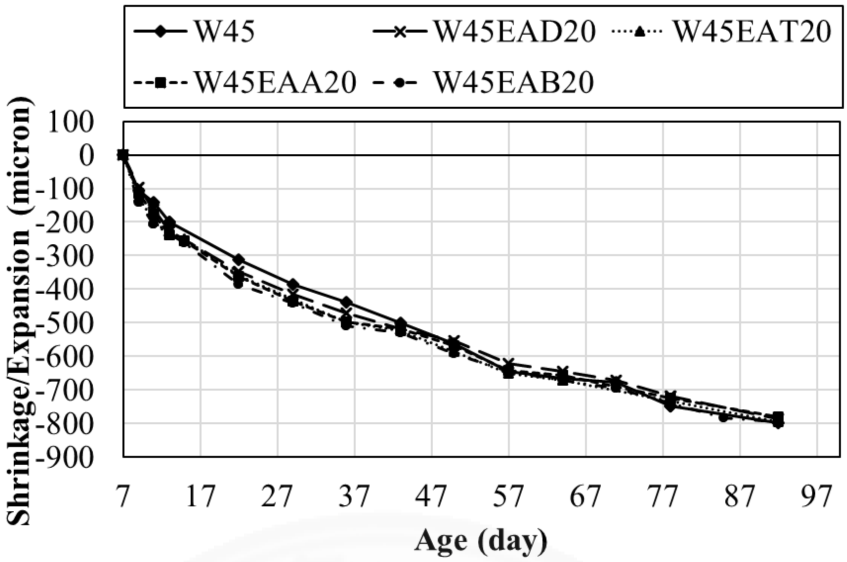
$\mathcal{E}S_{total}(7)$ = Total shrinkage values at the age of 7 days (micron)

t = Age of total shrinkage specimen (days)

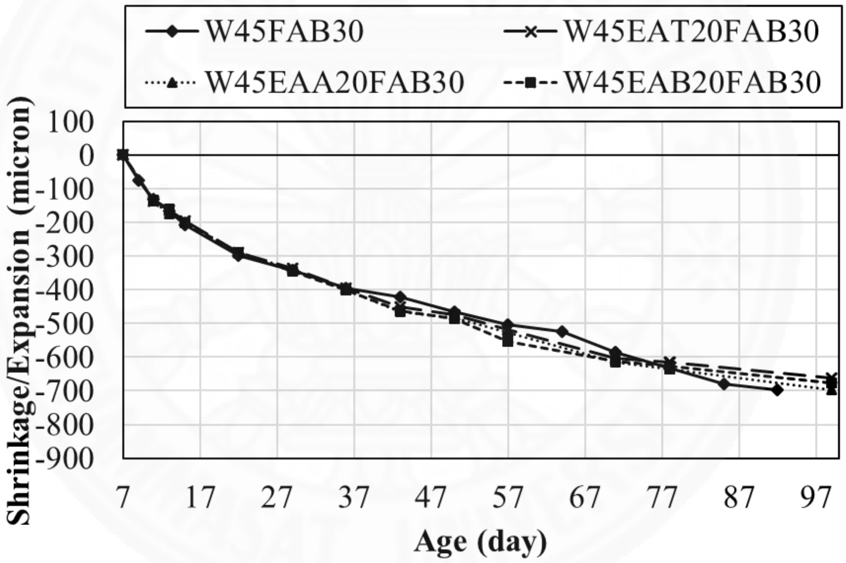
In case of the effect of expansive additive types, it can be seen in Figure 5.8, showing the results of total shrinkage in dry curing period of concrete containing different types of expansive additive, that all types of expansive concrete showed similar shrinkage values in dry curing period to the Non-EA concrete. Moreover, the shrinkage values in dry curing period also did not show significant differences between each type of expansive concrete. These tendencies could be seen in both of Non-FA concrete (Figure 5.8a), and fly ash concrete (Figure 5.8b).

In case of the effect of fly ash types, it can be seen in Figure 5.9, showing the results of total shrinkage in dry curing period of concrete containing different types of fly ash, that all types of fly ash concrete showed lower shrinkage values in dry curing period than that of the Non-FA concrete (Figure 5.9a). However, the shrinkage values in dry curing period did not show significant differences between each type of fly ash concrete. These tendencies could be seen in both of Non-EA concrete (Figure 5.9a), and expansive concrete (Figure 5.b).

All of the above results for free expansion and shrinkage revealed that the reduction of shrinkage values in expansive concretes mainly depended on the magnitude of generated expansion at the early age, but did not participate in shrinkage reduction at long term. When fly ash was cooperatively used, especially in expansive concrete, the shrinkage could be further reduced because of expansion enhancement at the early age by the hypothesis of the paste stiffness reduction, the additional expansive products from free lime and SO_3 in fly ashes, and the delayed expansive additive reaction by fly ash. Moreover, the use of fly ash also reduced shrinkage at long term. It is partly because of the reduction of autogenous shrinkage at early age due to the lower water retainability of fly ash, thereby providing more free water for hydration process, as well as the retardation of hydration. Drying shrinkage at long term is also reduced due to the improvement of pore structure by pore refinery effect of fly ash which reduces the evaporation of free water to the environment [56]. In addition, it may be because of the pore refinery effect which occurs from the additional produced expansive products in expansive concrete with fly ashes which reduces permeability of the concrete. Therefore, it is beneficial to use fly ash in expansive concrete in the aspect of shrinkage reduction due to combined effects of the significant enhancement of expansion at the early age and properties of fly ash which could reduce shrinkage at long term.

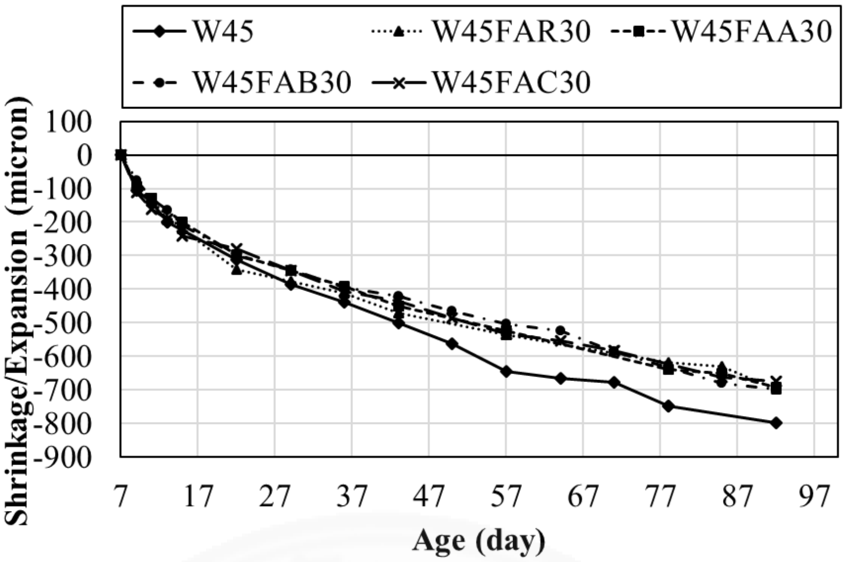


a) Non-FA concrete

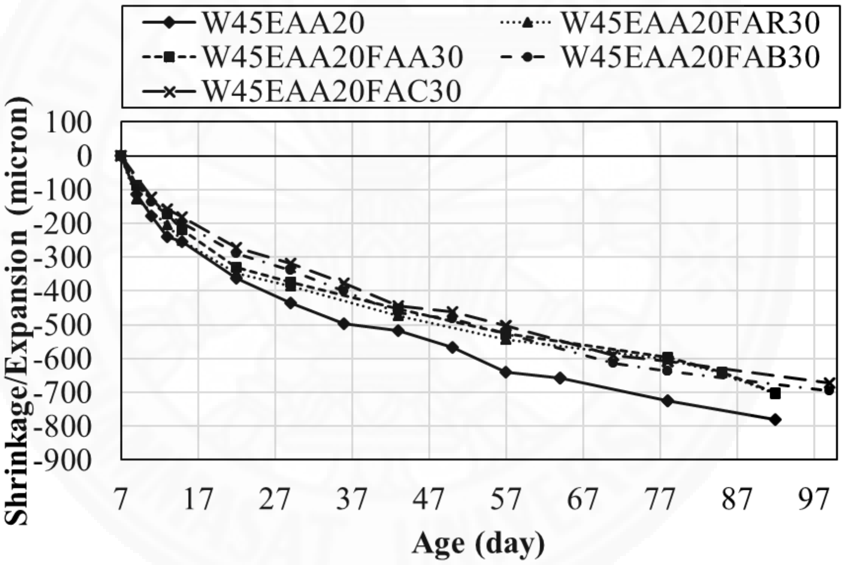


b) FAB fly ash concrete

Figure 5.8 Results of total shrinkage in dry curing period of concrete containing different types of expansive additive



a) Non-EA concrete



b) EAA expansive concrete

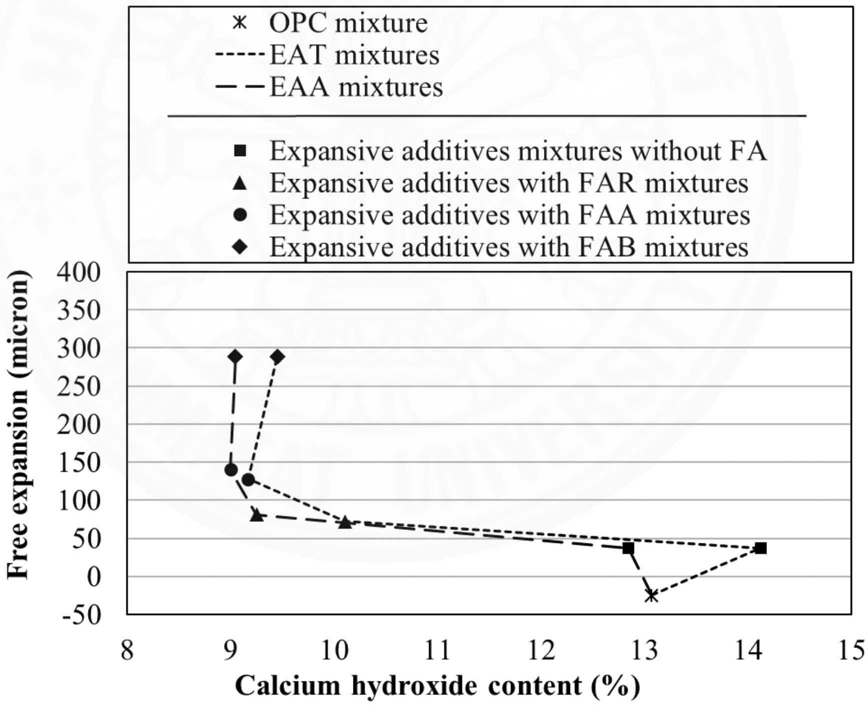
Figure 5.9 Results of total shrinkage in dry curing period of concrete containing different types of fly ash

5.3 Mechanism clarifications for expansion enhancement

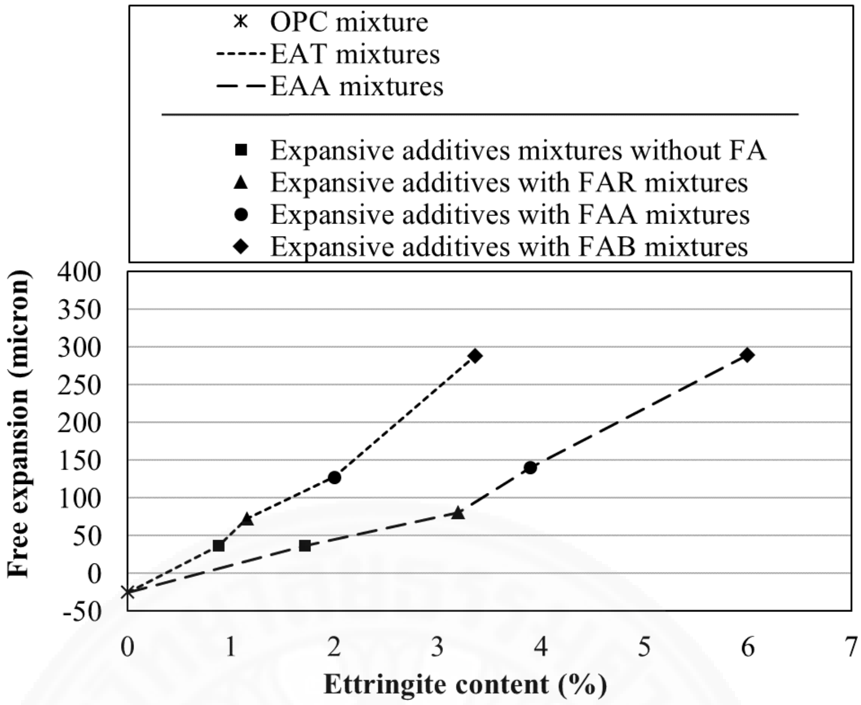
According to the hypothesis on the effect of expansive additive types which were described earlier, the produced expansive products are forecasted to be caused by the different proportions of constituents which are available for expansive products formation in different types of expansive additive. For the effect of fly ash types, the additional amounts of free lime and SO_3 in fly ashes may join the formation of expansive products, resulting in expansion enhancement. In order to confirm these hypothesis, the XRD analysis was used to examine the amounts of expansive products ($Ca(OH)_2$ and ettringite) in each mixture. The results of expansive products derived from XRD analysis in each mixture are illustrated using a relationship between the products and the free expansion of concrete at the age of 7 days. This relationship which is categorized by the effect of expansive additive types and the effect of fly ash types is shown in Figures 5.10 and 5.11, respectively.

In case of different types of expansive additive (Figure 5.10), the results of XRD analysis showed similar tendency as the described hypothesis. Despite the similar expansion shown for the EAT and EAA mixtures, the produced amounts of expansive products were different. At the same level of free expansion, the EAT mixtures tended to have larger amount of $Ca(OH)_2$ left in the mixtures (Figure 5.10a), but EAA mixtures produced much larger amount of ettringite (Figure 5.10b). It is corresponded to the chemical constituents of each expansive additive type. It can be seen in Table 3.6 that the EAT expansive additive mainly contains free lime, $CaSO_4$, and basic oxide compounds of cement (C_2S, C_3S, C_3A and C_4AF), but the EAA expansive additive mainly contains yeelimite, anhydrite and free lime. When considering the mechanism of ettringite formation in Table 5.1, the ettringite formation of EAA expansive additive is caused by the reaction between C_4A_3S, $CaSO_4$, $Ca(OH)_2$ and water. Therefore, some produced $Ca(OH)_2$ in the EAA mixtures are consumed for ettringite formation. This is the reason that the EAA mixtures had lower $Ca(OH)_2$ left in the mixtures than that of the EAT mixtures although both consist of similar amount of free lime. In addition, C_3S and C_2S in EAT expansive additive also produce $Ca(OH)_2$ from their hydration. However, the different effectiveness of expansive products for generating expansion in each type of expansive can also be caused by the different setting time and stiffness of the mixtures. These factors must be studied further.

In case of mixtures containing different types of fly ash (Figure 5.11), the amounts of produced expansive products were assumed as a result of expansion enhancement from fly ash containing higher amounts free lime and SO_3. However, the results did not always correlate with the hypothesis. It is found that the expansion enhancement was not due to the increased amount of $Ca(OH)_2$, but well correlated with the increased amount of ettringite. It can be seen in the Figure 5.11a that the free expansion was enhanced at the lower levels of $Ca(OH)_2$ content in the mixture. The cause for $Ca(OH)_2$ reduction is because it is consumed by pozzolanic reaction and ettringite formation [23,25,55]. In contrast, it can be seen in Figure 5.11b that the free expansion linearly increased when the produced ettringite content was increased. The high free lime and SO_3 fly ash (FAB) mixtures which were the mixtures with the highest ettringite formation showed the highest free expansion, followed by the high CaO fly ash (FAA) and low CaO fly ash (FAR) mixtures, respectively.

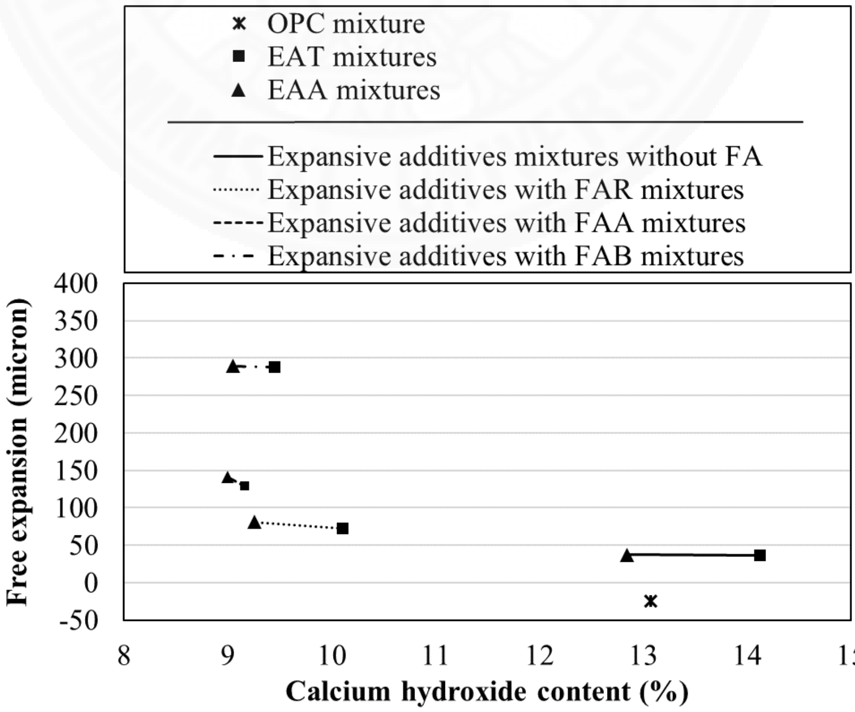


a) Calcium hydroxide content

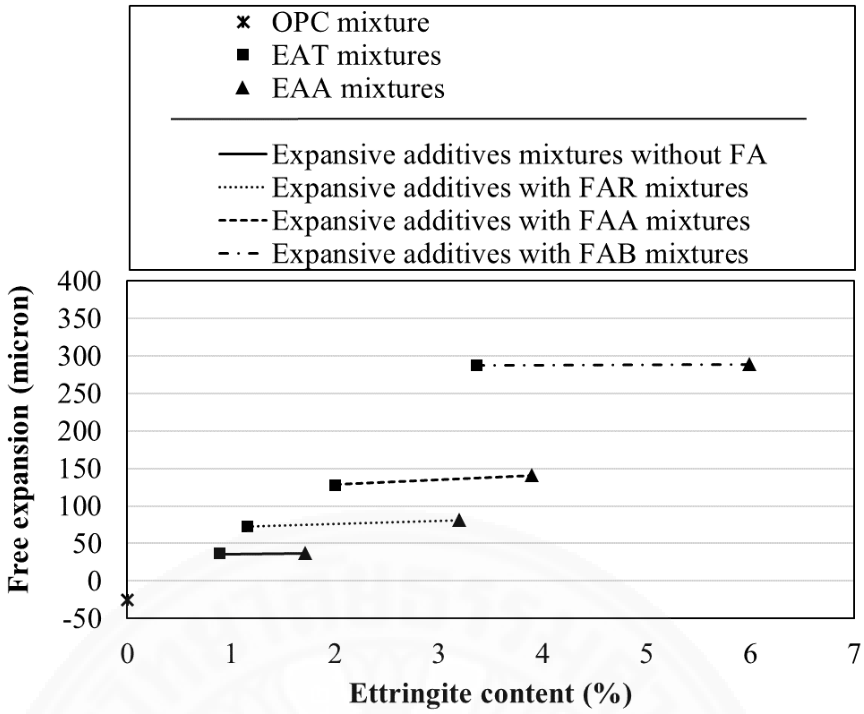


b) Ettringite content

Figure 5.10 Relationship between results of expansive products derived from XRD analysis and free expansion of concrete containing different types of expansive additive at the age of 7 days



a) Calcium hydroxide content



b) Ettringite content

Figure 5.11 Relationship between results of expansive products derived from XRD analysis and free expansion of concrete containing different types of fly ash at the age of 7 days

Chapter 6

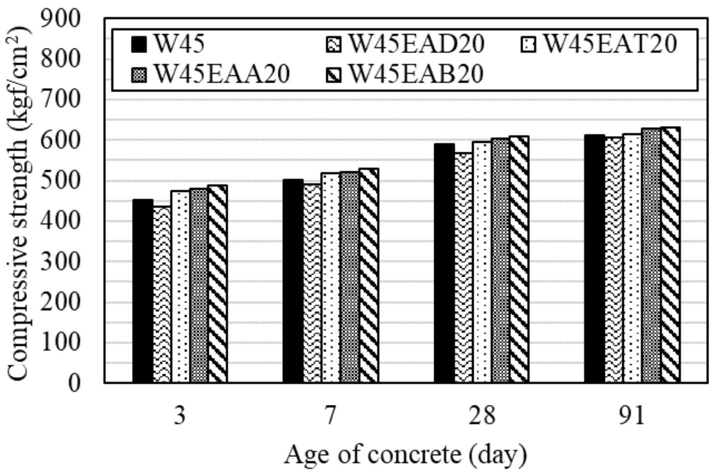
Results and Clarifications of Compressive Strength

6.1 Experimental results on compressive strength

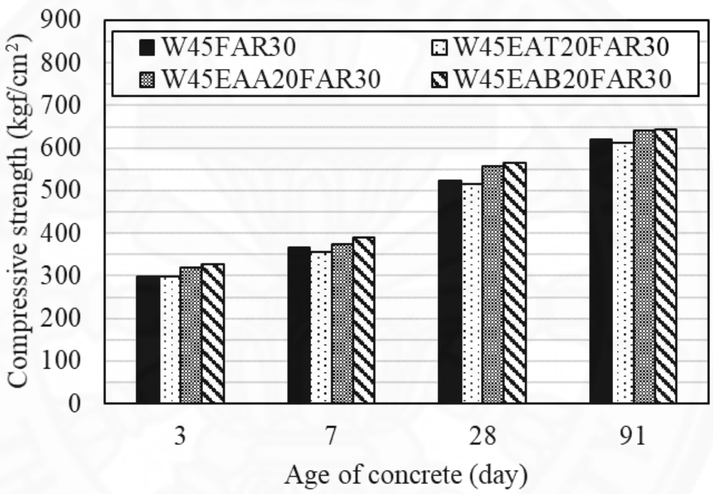
6.1.1 Unconfined compressive strength

6.1.1.1 Effect of expansive additive types

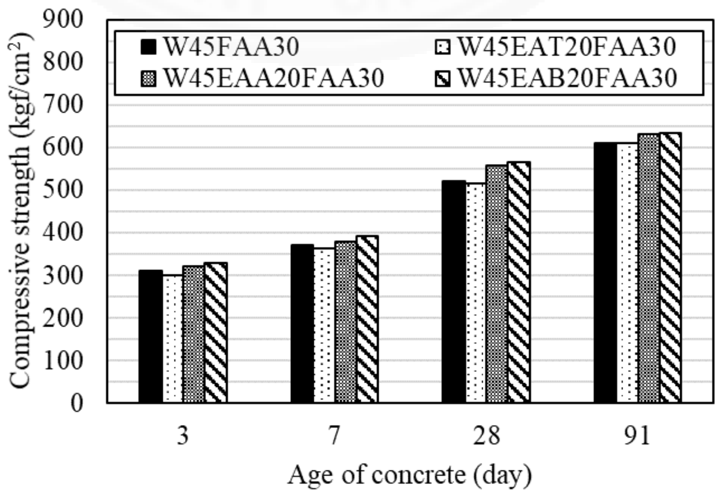
Figure 6.1 shows the results of unconfined compressive strength of concrete containing different types of expansive additive. In case of Non-FA concrete (Figure 6.1a), when the compressive strength was compared between the Non-EA concrete and expansive concrete, the EAD expansive concrete had lower compressive strength than that of Non-EA concrete. The EAT expansive concrete had similar compressive strength to the Non-EA concrete. The EAA and EAB expansive concretes had higher compressive strength than that of the Non-EA concrete. When the compressive strength was compared in each type of expansive concrete, the EAB expansive concrete had the highest compressive strength, followed by the EAA, EAT and EAD expansive concretes, respectively. The low compressive strength of EAD expansive concrete may occur from produced micro crack which is generated due to the large expansion. In case of fly ash concretes (Figure 6.1b-e), the compressive strength results also showed similar tendency to the case of Non-FA concrete (Figure 6.1a). When the compressive strength was compared between Non-EA concrete and expansive fly ash concrete, the EAT mixtures had similar compressive strength to the Non-EA mixtures, and the EAA and EAB mixtures had higher compressive strength than that of Non-EA mixtures. When the compressive strength was compared in each type of expansive fly ash concrete, the EAB mixtures had the highest compressive strength, followed by the EAA and EAT mixtures, respectively.



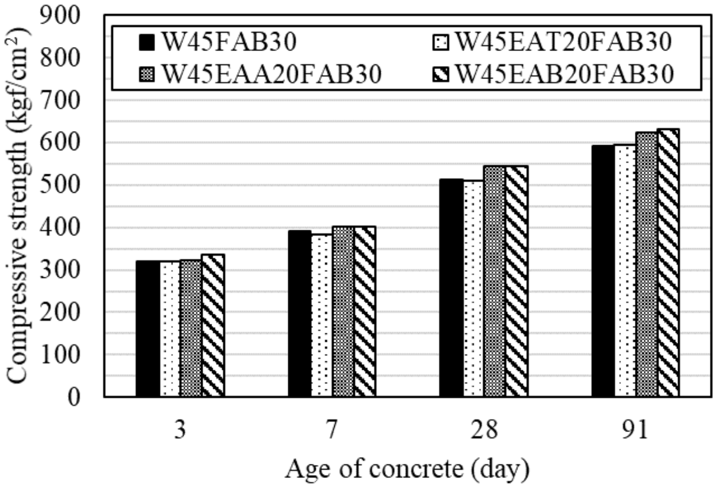
a) Non-FA concrete



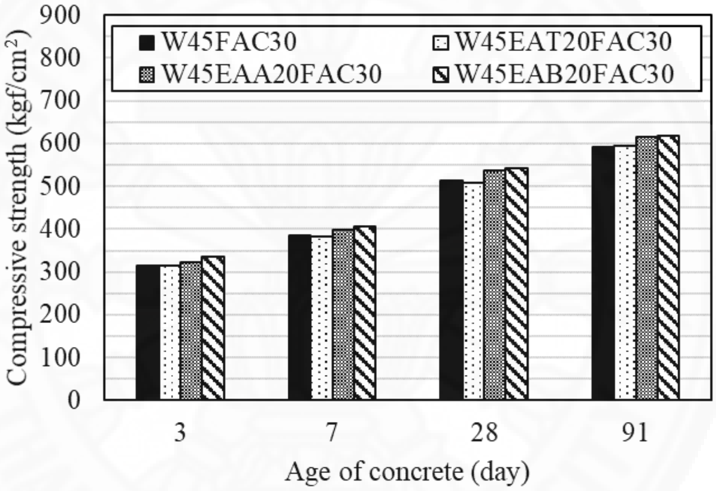
b) FAR fly ash concrete



c) FAA fly ash concrete



d) FAB fly ash concrete

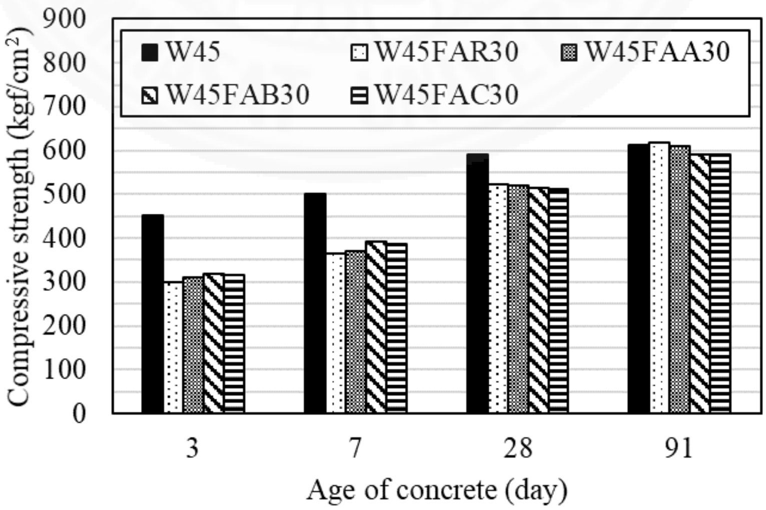


e) FAC fly ash concrete

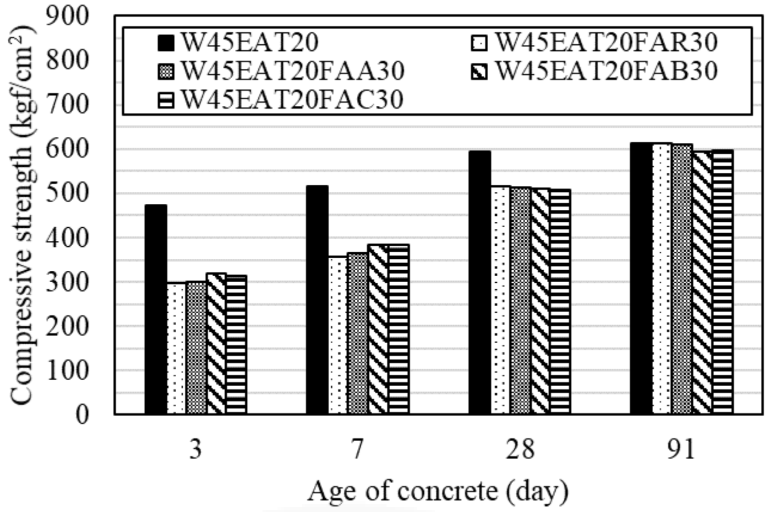
Figure 6.1 Results of unconfined compressive strength of concrete containing different types of expansive additive

6.1.1.2 Effect of fly ah types

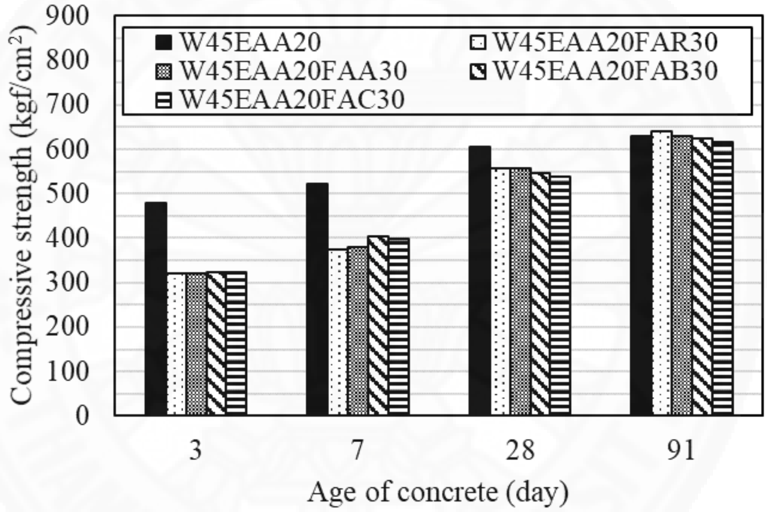
Figure 6.2 shows the results of unconfined compressive strength of concrete containing different types of fly ash. The use of different types of fly ash showed similar trend of compressive strength results in Non-EA concrete (Figure 6.2a). and expansive concretes (Figure 6.2b-d). When comparing between Non-FA mixtures and fly ash mixtures, all types of fly ash mixtures showed significantly lower compressive strength than that of the Non-FA mixtures at the early age. However, the compressive strength of fly ash mixtures was enhanced at the later age due to the pozzolanic reaction. When different types of fly ash were considered, the compressive strength of fly ash mixtures at the early age was mainly affected by the CaO and free lime contents in the fly ashes. The low CaO fly ash (FAR) mixtures showed the lowest compressive strength, and it was higher in high CaO fly ash (FAA) and high free lime and SO_3 fly ashes (FAB and FAC) mixtures, respectively. It is because of the activation of pozzolanic reaction with the produced $Ca(OH)_2$ at the early age from CaO and free lime contents in fly ash [23,25]. At the later age, the compressive strength of fly ash mixtures was mainly affected by the availability of the SiO_2 content for pozzolanic reaction. The low CaO fly ash mixtures, having the highest SiO_2 content, turned into the fly ash mixtures which resulted in the highest compressive strength, followed by the high CaO fly ash and high free lime and SO_3 fly ashes mixtures, respectively.



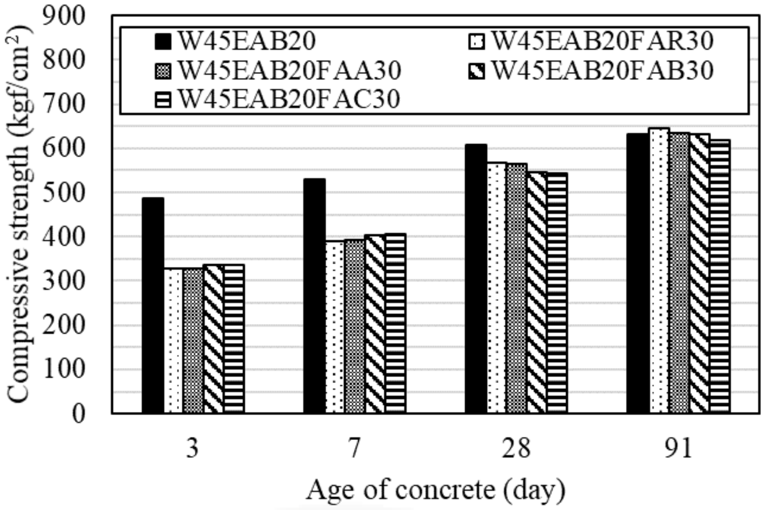
a) Non-EA concrete



b) EAT expansive concrete



c) EAA expansive concrete



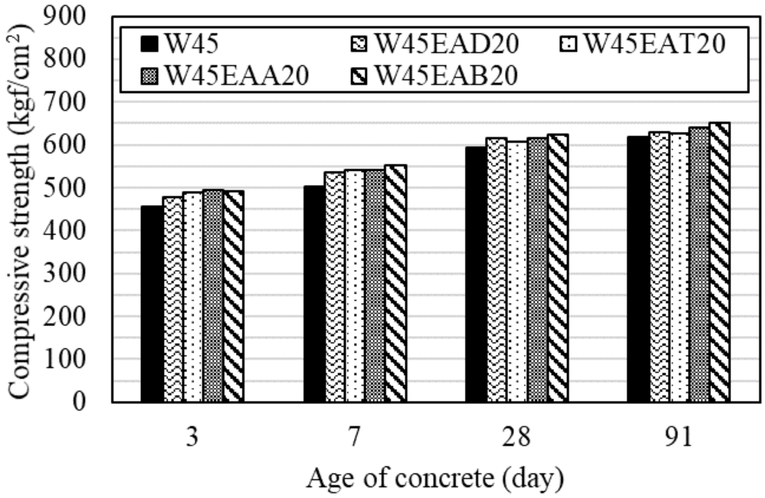
d) EAB expansive concrete

Figure 6.2 Results of unconfined compressive strength of concrete containing different types of fly ash

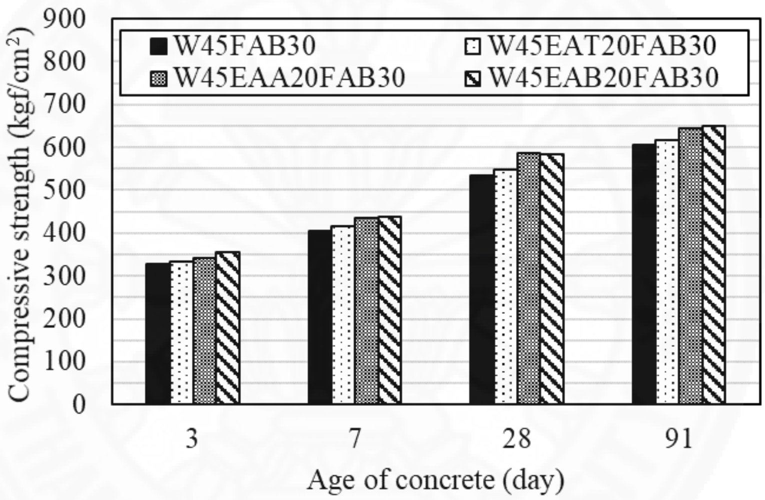
6.1.2 Confined compressive strength

6.1.2.1 Effect of expansive additive types

Figure 6.3 shows the results of confined compressive strength of concrete containing different types of expansive additive. In case of Non-FA concrete (Figure 6.3a), all types of expansive concrete had higher compressive strength than that of the Non-EA concrete. When different types of expansive concrete were compared, the EAD and EAT concrete showed similar compressive strength while the EAA and EAB concretes had slightly higher confined compressive strength. In case of fly ash concrete (Figure 6.3b), the compressive strength also showed similar tendency to the case of Non-FA concrete (Figure 6.3a). All types of expansive concrete had higher compressive strength than that of the Non-EA concrete. The EAT concrete had the lowest compressive strength, followed by the EAA and EAB concretes, respectively. It should be noted that the EAD concrete was not used for testing unconfined compressive strength of fly ash concrete.



a) Non-FA concrete

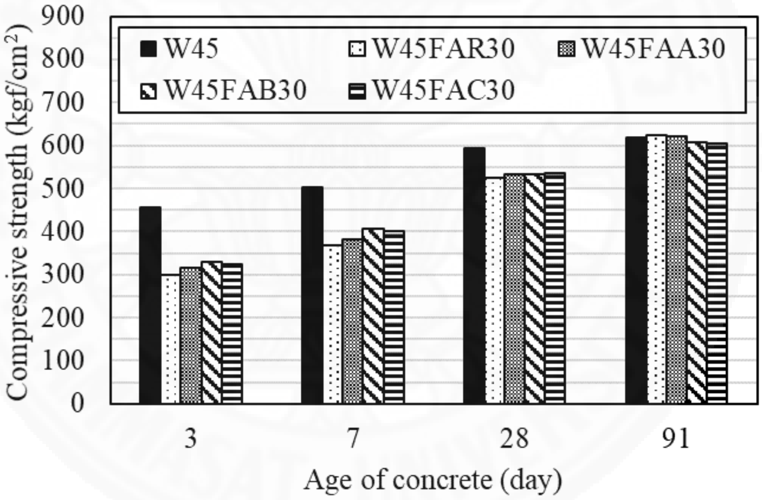


b) FAB fly ash concrete

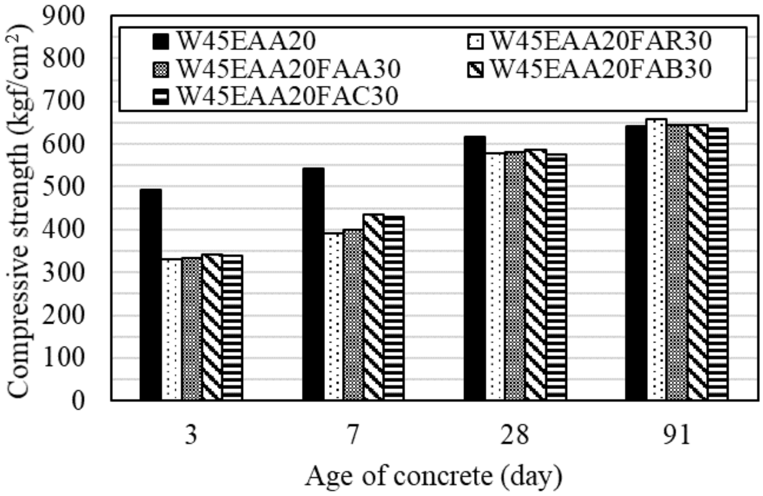
Figure 6.3 Results of confined compressive strength of concrete containing different types of expansive additive

6.1.2.2 Effect of fly ash types

The effect of fly ash types on confined compressive strength showed similar tendency with the unconfined compressive strength in both cases of Non-EA concrete (Figure 6.4a) and expansive concrete (Figure 6.4b). It can be seen that all types of fly ash mixtures showed lower compressive strength than the Non-FA mixtures at the early age. However, the compressive strength of fly ash mixtures was enhanced at the later age. When different types of fly ash mixtures were compared, at the early age, the low CaO fly ash (FAR) mixtures showed the lowest compressive strength, followed by the high CaO fly ash (FAA) and high free lime and SO_3 fly ashes (FAB and FAC) mixtures, respectively. At the later age, the highest compressive strength was observed in low CaO fly ash mixtures. This is because of the effect of chemical compositions (CaO, free lime and SiO_2) which are different in each type of fly ash as described earlier.



a) Non-EA concrete

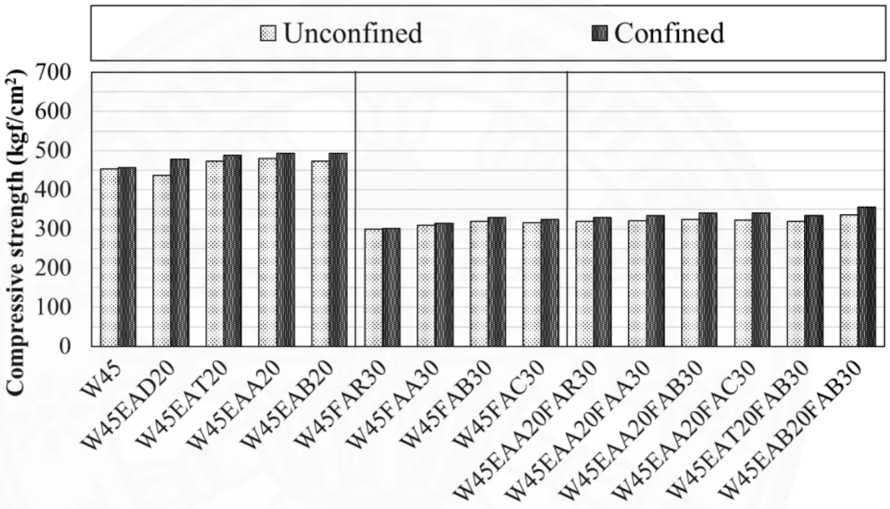


b) EAA expansive concrete

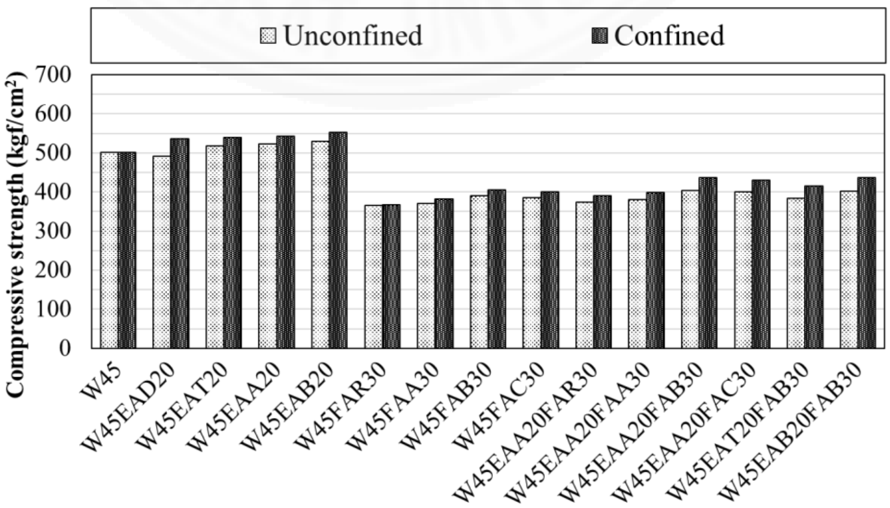
Figure 6.4 Results of confined compressive strength of concrete containing different types of fly ash

6.1.3 Comparison between unconfined and confined compressive strength results

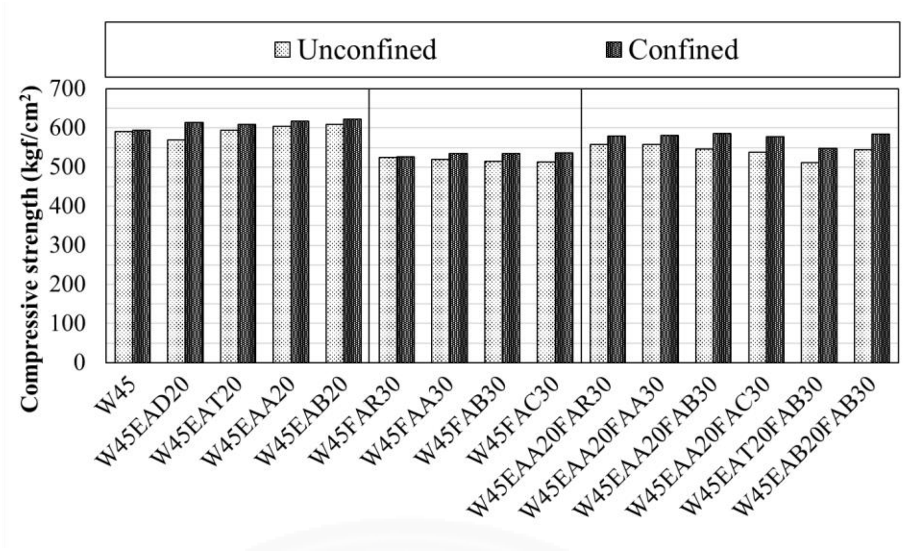
The results of unconfined and confined compressive strengths of concrete are compared and shown in Figure 6.5. It can be seen that the concrete mixtures which were made under confined condition had higher compressive strength than those of unconfined condition. This tendency could be seen in all ages of concrete (Figure 6.5a-d). The strength improvement of concrete made under confined condition occurs from the restricted expansion, improving microstructure and so making denser paste structure [33].



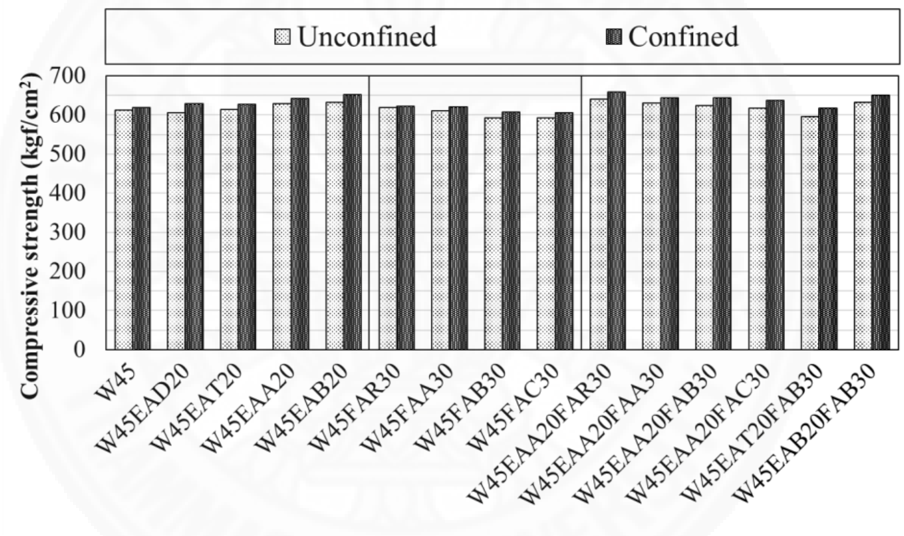
a) Age of concrete at 3 days



b) Age of concrete at 7 days



c) Age of concrete at 28 days



d) Age of concrete at 91days

Figure 6.5 Results of unconfined and confined compressive strengths of concrete

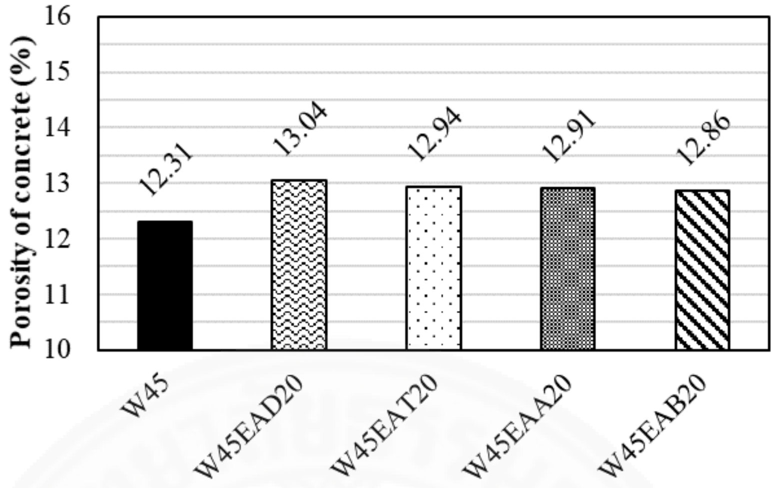
6.2 Mechanism clarifications for strength enhancement

6.2.1 Unconfined compressive strength

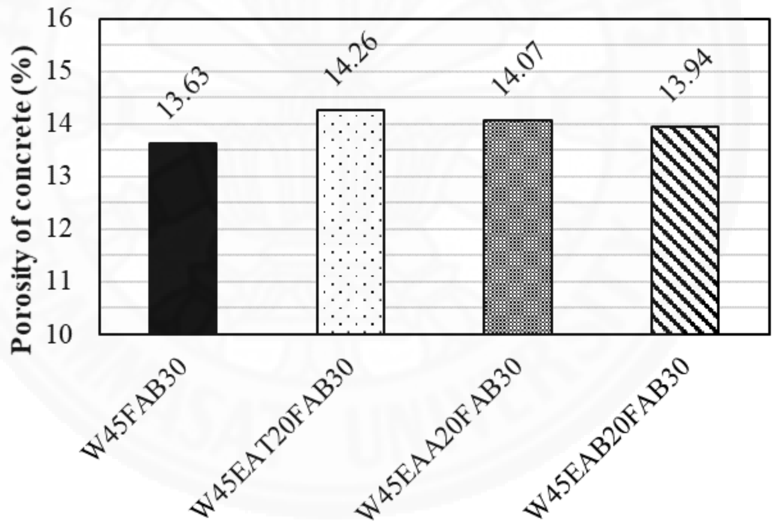
Figure 6.6 shows the results of porosity of concrete made under unconfined condition at the age of 7 days. It can be seen that the porosity of concrete in each types of expansive concrete was different. In case of Non-FA concrete (Figure 6.6a), the EAB expansive concrete had the lowest porosity, followed by EAA, EAT and EAD expansive concrete, respectively. In case of fly ash concrete (Figure 6.6b), the EAB expansive concrete had the lowest porosity, followed by EAA and EAT expansive concrete, respectively. It should also be noted that the EAD concrete was not tested for fly ash concrete.

The results of porosity of concrete made under unconfined condition were plotted against the results of unconfined compressive strength as expressed in Figure 6.7. The highest unconfined compressive strength was shown in mixture with EAB, followed by the mixtures with EAA, EAA and EAD for Non-FA concrete, respectively. For fly ash concrete, the highest unconfined compressive strength was shown in mixture with EAB, followed by the mixtures with EAA and EAT, respectively. This tendency corresponded to the results of porosity which showed the smaller values in the order of EAB, EAA, EAT and EAD expansive concrete for the case of Non-FA concrete and in the order of EAB, EAA and EAT expansive concrete for the case of fly ash concrete. Therefore, the different unconfined compressive strength results in different types of expansive concrete were supposed to be mainly caused by the different porosity values. The mixtures with lower porosity, indicating a denser paste structure, resulted in higher compressive strength.

However, when comparing between Non-EA and expansive concretes, the expansive concrete had larger porosity than that of the Non-EA concrete (Figure 6.6), whereas the results of unconfined compressive strength showed comparable strength between the EAT expansive concrete and the Non-EA concrete and showed higher strength in EAA, EAB expansive concretes than the Non-EA concrete (Figure 6.1). It is possible that there may be other factors than the total porosity that affect the compressive strength behavior. The pore structures may be an important factor which must be investigated further.



a) Non-FA concrete



b) FAB fly ash concrete

Figure 6.6 Results of porosity of concrete made under unconfined condition at the age of 7 days

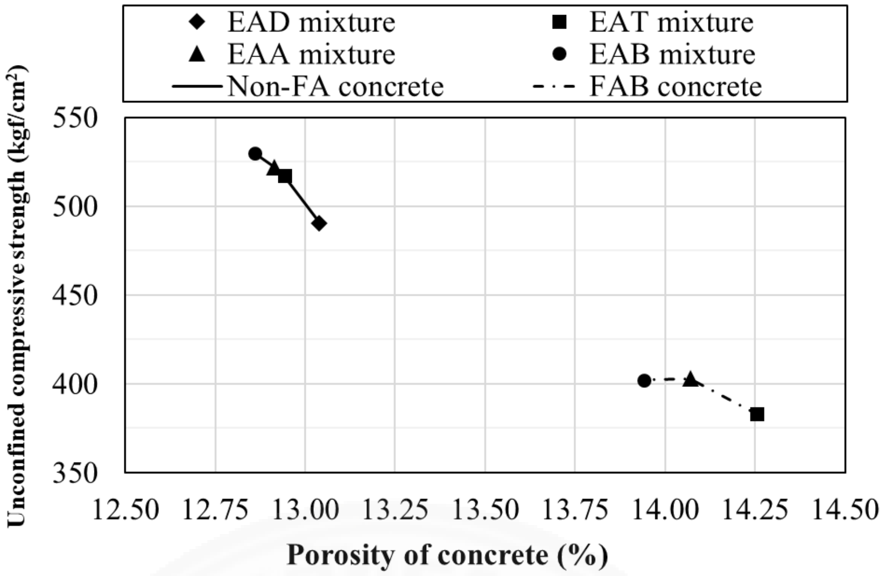


Figure 6.7 Relationship between results of porosity and compressive strength of concrete made under unconfined condition at the age of 7 days

6.2.2 Confined compressive strength

Figure 6.8 shows the results of porosity of the concrete made under unconfined and confined conditions at the age of 7 days. The porosity of concrete of all confined mixtures showed lower values when they were compared with the unconfined mixtures. This is caused by the denser paste structure which is produced under confined condition.

The results of porosity of concrete made under unconfined and confined expansion conditions were plotted against the results of unconfined and confined compressive strength as shown in Figure 6.9. It can be seen that the porosity in each type of mixture was reduced when they were made under confined expansion condition. Moreover, the results of compressive strength of concrete also showed higher values in specimens made under confined condition. This means that the confinement of the specimens led to the reduction of porosity of concrete, indicating a denser paste structure, therefore resulted in an enhancement of compressive strength.

In addition, when comparing at the same values of total porosity of Non-EA and EA mixtures (both confined and unconfined specimens), the EA mixtures had higher compressive strength than that of the Non-EA mixtures (Figure 6.10). It is possible that the use of expansive additive reduces size of pores, resulting in the higher strength. For confirming this hypothesis, this effect must be studied further.

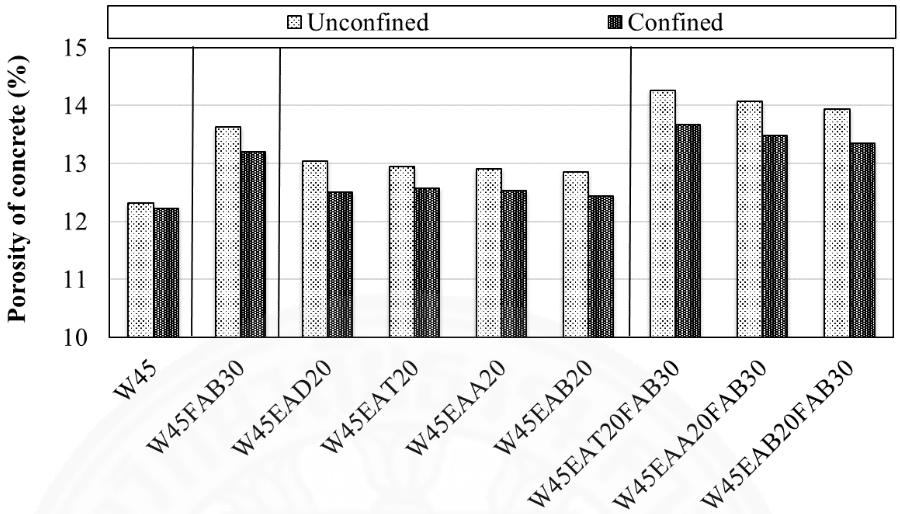


Figure 6.8 Results of porosity of concrete made under unconfined and confined conditions at the age of 7 days

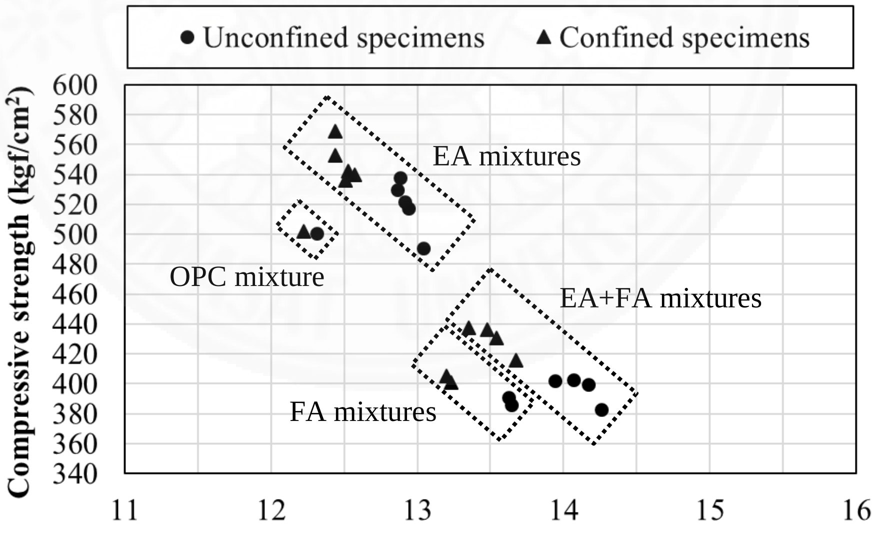


Figure 6.9 Relationship between results of porosity and compressive strength of concrete made under unconfined and confined conditions at the age of 7 days

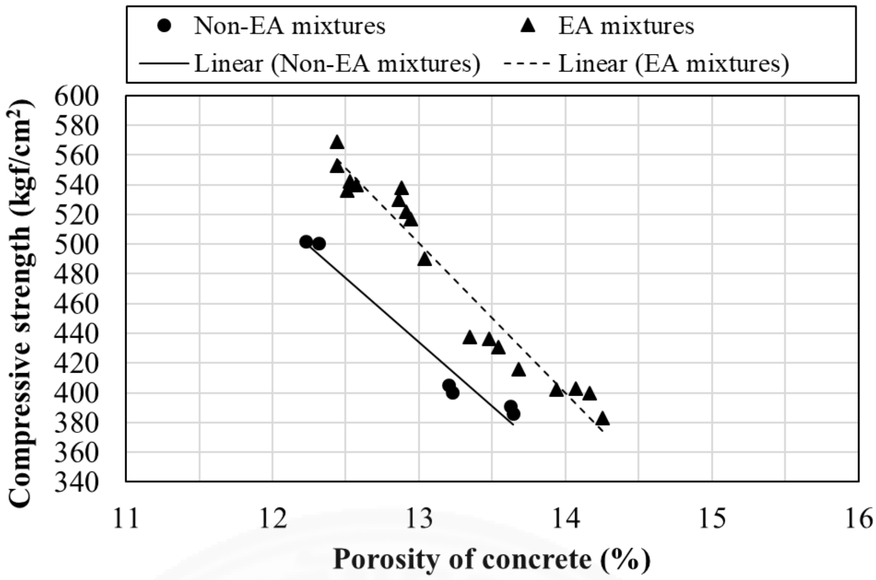


Figure 6.10 Relationship between results of porosity and compressive strength of concrete with and without expansive additive at the age of 7 days

Chapter 7

Conclusions and Recommendations

7.1 Conclusions

According to all results of the study, the performances of concrete which were considered for the effect of expansive additive types and fly ash types were summarized and shown in Tables 7.1 and 7.2 respectively.

Table 7.1 Performances of concrete with and without fly ash by considering the effect of expansive additive types

Properties	Performances				
	Non-expansive additive mixtures	**EAD mixture**	**EAT mixtures**	**EAA mixtures**	**EAB mixtures**
Setting time	●	-	●●	●●●	●●●●
Slump	●●	-	●●●	●●	●
Free expansion	●	●●●●	●●	●●	●●●
Autogenous shrinkage	●	●●●	●●	●●	●●
Total shrinkage	●	●●●	●●	●●	●●
Unconfined compressive strength	●●	●	●●	●●●	●●●●
Confined compressive strength	●	●●	●●	●●●	●●●●

Remark: The performance level is represented by the numbers of symbol (●).

Table 7.2 Performances of concrete with and without expansive additive by considering the effect of fly ash types

Properties	Performances			
	Non-fly ash mixtures	**Low CaO fly ash mixtures**	**High CaO fly ash mixtures**	**High free lime and SO_3 fly ashes mixtures**
Setting time	●●●●	●	●●	●●●
Slump	●	●	●●	●●●
Free expansion	●	●●	●●●	●●●●
Autogenous shrinkage	●	●●	●●●	●●●●
Total shrinkage	●	●●	●●●	●●●●
Unconfined compressive strength				
- Early age	●●●●	●	●●	●●●
- Long term	●●	●●●/●●	●●	●
Confined compressive strength				
- Early age	●●●●	●	●●	●●●
- Long term	●●	●●●/●●	●●	●

Remark: The performance level is represented by the numbers of symbol (●).

Apart from the performances of concrete which are summarized and shown earlier, the results in the other aspects are also found. From the results of free expansion and shrinkage, it can be additionally concluded that;

- The use of 10% high free lime and SO_3 fly ash could enhance the performance of expansion in a similar level to the use of 30% high CaO fly ash.
- The use of 30% high free lime and SO_3 fly ash, 30% high CaO fly ash, and 10% high free lime and SO_3 fly ash in EAT and EAA concretes could reduce the expansive additive usage approximately to 22, 12 and 8 kg /m^3, respectively.
- When the expansion at the early age was not considered, the use of expansive additives in concrete was not beneficial to the shrinkage reduction at long term, but it became effective when the expansive additive was applied with fly ash.

In addition, the results clarifications for the free expansion and compressive strength revealed that;

- The cause for expansion in each type of expansive concrete mainly occurred from different types of produced expansive products which were results of the amounts of different constituents for producing expansive products in each type of expansive additive.
- The expansion enhancement of expansive concrete containing different types of fly ash was not due to the increased amount of $Ca(OH)_2$, but well correlated with the increased amount of ettringite. The highest ettringite formation was observed in the high free lime and SO_3 fly ash mixtures, followed by the high CaO fly ash and low CaO fly ash mixtures, respectively.
- The different unconfined compressive strength of each type of expansive concrete was mainly caused by the different porosity values. The mixtures with lower porosity, indicating a denser paste structure, resulted in higher compressive strength.
- Comparing with the unconfined expansion, the results of compressive strength were higher when the specimens were prepared in confined condition. The confinement of the specimens led to the reduction of porosity of concrete, indicating a denser paste structure, therefore resulted in an enhancement of compressive strength.

7.2 Recommendations for future studies

In addition to the mentioned clarifications in this study, further investigations are recommended for future studies as follows:

- The reduction of early age paste stiffness and the delaying expansive additive reaction by fly ash incorporation are the reasons for expansion enhancement in expansive concrete.
- Apart from the effect of shrinkage reduction at long term by fly ash, the pore refinery effect which occurs from the additional produced expansive products in expansive concrete with fly ashes may be a participated factor.
- Apart from the results of total porosity of concrete, the size of pores may be an important factor which affect the compressive strength, especially in case of EA mixtures which show higher compressive strength when compared to Non-EA mixtures at the same total porosity.
- The effect of degree of restrained expansion under confinement conditions which results in the enhancement of compressive strength should also be investigated.

In practice, concrete structures are always at risk of the deteriorations and under restrained condition. Overall concrete properties are necessary to be known for evaluating the proper expansive concrete usage. Because this study is only emphasized in basic properties and some aspects of durability properties, the other properties must be additionally investigated such as slump loss, chloride penetration resistance, sulfate resistance, alkali-aggregate reaction, carbonation, tensile strain capacity, expansion and shrinkage under restrained condition, self-healing ability, etc.

Apart from the fly ash, other waste materials such as blast furnace slag and bottom ash may also be used for enhancing properties and reducing cost of expansive concrete. In case of bottom ash, the use of the bottom ash as a material for partially replacing fine aggregate is beneficial because the internal curing property of the bottom ash provides additional water for expansive additive reaction. Moreover, the properties of bottom ash to improve concrete performance is normally similar to that of fly ash when it is pulverized. Therefore, the pulverized bottom ash can possibly be used for enhancing expansive concrete properties in the same manner with the use of fly ash. In order to reduce cost of pulverization, the bottom ash may be interground with other materials during manufacturing process of expansive additive. In addition, blast furnace slag is a pozzolanic material which is similar to fly ash. Therefore, the properties of expansive concrete may be enhanced by using blast furnace slag.

Apart from finding way to promote fly ashes usage, another main target of this study is finding a way to reduce cost of expansive concrete. It is revealed from this study that the use of fly ashes can enhance expansive concrete properties and reduce expansive additive dosages. These lead to a cost reduction of the expansive concrete. Therefore, the effectiveness of the cost reduction of the expansive concrete by fly ashes usage should be evaluated.

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