- Technical Paper -

EFFECT OF LOW CURING TEMPERATURE AT EARLY AGES ON EXPANSION BEHAVIOR OF EXPANSIVE MORTAR

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ABSTRACT

This paper investigates the effect of low curing temperatures on the expansion properties of non-expansive and expansive mortar at early ages, considering the different curing conditions and change in curing temperature at later ages. The experiment included visual observation, expansion strain, compressive strength, thermogravimetric differential thermal analysis, quantitative X-ray diffraction, Rietveld analysis, and a scanning electron microscopy test. The crack appeared in the surface of expansive mortar that was first cured at 5℃ for 14 days, then cured at 20℃ and 40℃ until 36 and 18 days, respectively. However, if enough water is supplied, cracks will not appear even after 14 days of curing at low temperatures and then increasing curing temperatures to 20℃ and 40℃. The higher the temperature, the faster cracks appear. The test results also revealed that after expansive mortar was cured in the air at a low curing temperature, there was still an amount of expansive additive in the mortar that had not reacted, which explains the appearance of cracks.

Keywords: expansive additive, ettringite, calcium hydroxide, low curing temperature, cracking.

1. INTRODUCTION

Concrete is one of the most commonly used building materials due to its advantages such as high performance, durability, and low cost. However, drying shrinkage is a significant disadvantage of concrete, which leads to cracks in the concrete. Expansive concrete is widely used to deal with this problem and effectively prevent shrinkage cracks.

However, it is well known that the hydration reaction of expansive additive (EX) is strongly dependent on the curing temperature. This causes a significant effect on the properties of concrete. Several studies were conducted on the effect of curing temperature on the compressive strength and expansion behavior of expansive concrete [1, 2]. These studies reported that the effect of temperature on the expansion properties of expansive concrete is caused by both the curing temperature after casting and the subsequent temperature change. That effect varies depending on the type and amount of expansive material used, as well as the type of cement used. These studies, however, did not take into account the effect of curing conditions such as air curing, sealed curing, and underwater curing on the expansion properties of expansive concrete as the curing temperature changed. Additionally, Van et al. [3] also found that as the curing temperature was reduced, the hydration degree of cement paste with and without EX decreased. This shows that the low curing temperature will delay the hydration reaction of EX. The unhydrated EX will continuously react when temperatures rise. This will affect the properties of expansive concrete. Cracking can occur, especially if there is enough unreacted EX in the concrete to produce an expansion force greater than the concrete's strength. Hence, this phenomenon may be similar to the formation of delayed ettringite in cement concrete. Because the cause of the crack is the formation of ettringite in a later period.

Therefore, the main objectives of this research are to: first, examine the appearance of cracks in expansive mortar by considering the effects of both curing temperatures and curing conditions (air curing and underwater curing); and second, explain the appearance of cracks by investigating the mechanical properties of expansive mortar and the formation of ettringite. This study will also answer two research questions: After expansive mortar is cured at a low curing temperature, which of the curing conditions investigated has cracks? Why does the crack occur in that condition?

2. TEST PROGRAMS

2.1 Materials

Ordinary Portland cement (density and specific surface area were 3.16 g/cm^3 and 3490 cm^2/g, respectively), CSA-type expansive additive (density and specific surface area were 3.05 g/cm^3 and 3260 cm^2/g, respectively) [3], fine aggregate (density and absorption were 2.68 g/cm^3 and 1.78%, respectively) and water were used in this work.

2.2 Experimental Design

Three cases were made with different curing temperatures and curing conditions to investigate the influence of curing conditions on the basic physical properties of expansive mortar.

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Table 1 Experimental design

Cases	Symbol	W/B (%)	B:S	EX (B×wt%)	Curing conditions 1~14 days	Curing conditions 14~91 days	Experiments
A	A-N	0.5	1:3	0	Air curing at 20℃	Air curing at 20℃ and 40℃	Visual Observation (Photography) Expansion Strain Compressive Strength TG-DTA XRD Rietveld SEM
	A-EX5			5			
B	B-N			0	Air curing at 5℃	Air curing at 20℃ and 40℃	
	B-EX5			5			
C	C-N			0	Air curing at 5℃	Water curing at 20℃ and 40℃	
	C-EX5			5			

A compressive strength (at 1, 3, 7, 14, 28, 56, and 91 days) and expansion strain (until 91 days) test were conducted to investigate the mechanical properties of mortar with and without the EX. The thermogravimetric differential thermal analysis (TG-DTA) was used to calculate the Portlandite content (CH) and examine the transformation of ettringite to monosulfoaluminate. The crystal morphology was observed using scanning electron microscopy (SEM) and X-ray diffraction (XRD). Additionally, the ettringite content in expansive mortar was calculated using the Rietveld analysis. Table 1 presents the mix proportions and curing conditions of the experiment. The temperatures of 20 and 40℃ were chosen to simulate the environmental summer temperatures (or heat of hydration in massive concrete).

2.3 Preparing procedure

Before mixing mortar, all of the materials and formwork were placed in a constant temperature and humidity chamber for at least 24 hours to avoid the effect of temperature on the hydration of cement or EX. For example, for Cases A and B, OPC, EX, fine aggregate, water, and formwork were kept in the chamber with temperatures of 20 and 5℃, respectively. After mixing, in order to prevent bleeding, the mixture was also placed in the constant temperature and humidity chamber for 3 hours, during this time, the mixture was stirred by hand every 30 minutes. The mixture was cast into steel prisms measuring $40 \times 40 \times 80$ mm^3 and $\emptyset\, 50 \times 100$ mm^2 cylinders. The specimens were demolded 24 hours after casting and then air-cured at different temperatures for 14 days before being laid in air-curing (Cases A and B) and water-curing conditions (Case C) for another 91 days.

For powder preparation, at the test ages, the mortar was roughly crushed by a hammer. The crushed specimens were immersed in iso-propanol for 1 hours and dried at 40℃ for 24 hours to stop hydration. Afterward, the specimens were crushed to pass through sieve sizes of 0.3, 0.6, 1.7, and 2.5 mm to remove the fine aggregate. Finally, the specimens were finely ground to pass through a 150 m sieve.

2.4 Test Methods

(1) Visual observation

The state of the sample was captured by a camera to check the appearance of cracks on the specimen surface.

(2) Compressive strength test

The compressive strength was conducted in accordance with JIS A 1108 with dimensions of 40×40×80 mm^3.

(3) Expansion strain test

The expansion strain was measured on ∅50×100 mm^2 cylinders. A strain gauge and an embedded strain gauge were used to measure the restrained and free expansion of mortar with and without EX according to JCI S 009-2012 and JIS A 1129, respectively.

(4) TG-DTA test

The TG-DTA test was performed with a heating rate of 20 ℃/min from 30 to 1000℃ in a nitrogen atmosphere. The quantitative amounts of CH and $CaCO_3$ in the test specimen were calculated from weight loss measured from the TG curve around 400−550℃ and 600−800℃, as shown in Eqs. 1 and 2. Moreover, the $CaCO_3$ content in the specimen originates from the carbonation of CH, so the carbonated CH content can be obtained based on the $CaCO_3$ content, as shown in Eq. 3. Therefore, the total CH content of the sample includes the CH content obtained by Eq. 1 and the $CaCO_3$ content obtained by Eq. 2 [4]. The total CH content of the sample was also calculated using Eq. 4.

$$[CH] = m_1 \times 74/18 \quad (1)$$

$$[CaCO_3] = m_2 \times 100/44 \quad (2)$$

$$[CH_C] = [CaCO_3] \times 74/100 \quad (3)$$

$$[CH_T] = [CH] + [CH_C] \quad (4)$$

where,

$[CH]$, $[CaCO_3]$, $[CH_C]$ and $[CH_T]$ are the non-carbonated CH, $CaCO_3$, carbonated CH and total CH content, % respectively. 18, 44, 74 and 100 are the molar masses of H_2O, CO_2, $Ca(OH)_2$, and $CaCO_3$.

(5) XRD test

The XRD test was performed to identify the change of crystalline phase. The following were the XRD conditions: Cu-Kα radiation resource; tube voltage 40 kV; tube current 30 mA; scan range of 5-90°2θ; scan speed 1°/min; step width 0.02°/step. As an internal standard, the specimens were prepared for XRD measurements by adding approximately 15 mass percent alpha-aluminum oxide (α-Al_2O_3) as an internal standard.

(6) Rietveld analysis

For Rietveld analysis, SIROQUANT Ver. 4.0 was used to quantify the ettringite content.

(7) SEM

The ettringite morphology was observed using a NeoScope JCM-7000. The working conditions were as

follows: 15 kV accelerating voltage; 5000 direct magnifications.

3. RESULTS AND DISCUSSION

3.1 Visual Observation

Fig. 1 shows the photography of samples at 28 and 56 days. Cracks on the surface of the samples were clearly visible at 28 and 56 days for Case B (B-EX5), where the samples were post-cured at 40 and 20℃, respectively. In other words, when the curing temperature for the air-curing condition was higher, the crack appeared faster. The crack, however, was not observed in samples that had been cured by underwatering. This can be explained by the curing temperature and the amount of water required for the hydration reactions of both cement and EX, according to the following hypothesis:

The samples were cured at a high temperature (at 20 °C), resulting in a higher amount of hydration products, which increased the strength of the mortar. Meanwhile, with the high curing temperature, the amount of unhydrated EX decreased, and when the curing temperature was raised (during the post-curing period), the expansion force formed by EX hydration was less than the mortar strength. As a result, no cracks were found in Case A.

The samples, however, were cured at a low curing temperature (at 5℃), resulting in less reacted cement and a delay in the development of mortar strength. On the contrary, there will be a significant amount of unreacted expansive additive inside samples after pre-curing (at 5℃ for 14 days). The expansion force generated by EX hydration was greater than the strength of the mortar when the curing temperature was raised, resulting in crack formation in the samples (see Fig. 1). Furthermore, higher curing temperatures speed up the hydration reaction of EX. This is in line with the previous finding [3]. This explained why cracks appeared more quickly in samples cured at 40℃ compared with those cured at 20℃. Unlike Case B, no cracks were observed in the Case C samples, despite the fact that they were also cured for 14 days at the same condition. This could be attributed to the post-curing condition, in which the samples were cured underwater, providing enough water for the hydration reaction of cement. The mortar's strength increased as a result. As a result, the expansion forces generated by unhydrated EX are less than the strength of mortar. Therefore, no cracks were found on the surface of the samples in Case C.

3.2 Expansion Strain

Fig. 2 illustrates the restrained expansion results of non-expansive and expansive mortar with different curing temperatures. As shown in Fig. 2, a significant effect of curing temperature and EX amount was observed on the expansion strain of mortar. The results indicate that the expansion strain increased when the EX-dosage was increased. When compared to the same amount of EX, the expansion strain increased as the curing temperature raised.

The free expansion strain of mortar with and without EX is shown in Fig. 3. It can be seen that with the same EX dosage at the high temperature, the A-EX5 was expanded faster than the B-EX5 and C-EX5 and attained the upper limit of the free expansion strain (approximately 500 μm) at the age of 12 hours, whereas the B-EX5 and C-EX5 reached the upper limit of that (approximately 400 μm) at the age of 2 days. From 14 days on, as the curing temperature increased, the free expansion strains of A-EX5 and B-EX5 increased immediately, however, the free expansion strain also decreased soon, which was due to the effect of the drying shrinkage. For case B, cracks were first visible to the naked eye on the 18th and 36th days after the samples were cured at 40℃ and 20℃, respectively. Meanwhile,

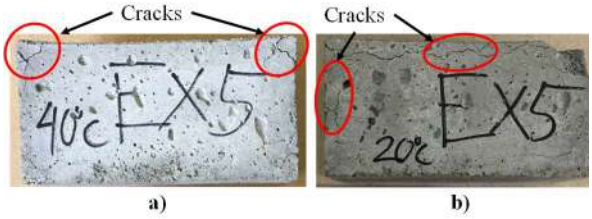

Fig. 1 Photography of expansive mortar: a) at 28 days in temperature of 40℃, b) at 56 days in temperature of 20℃

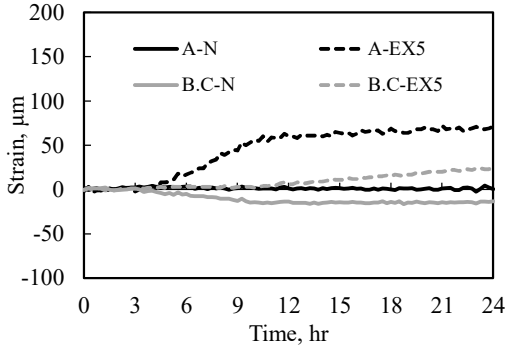

Fig. 2 Restrained expansion of non-expansive and expansive mortar

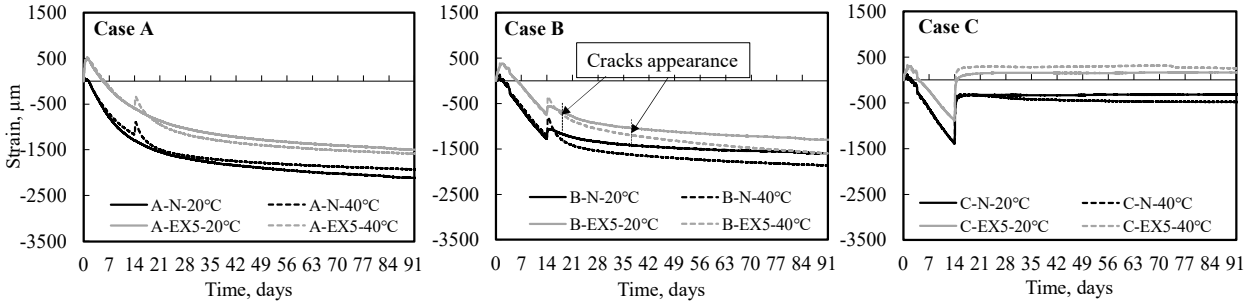

Fig. 3 Free Expansion of non-expansive and expansive mortar until 91 days

when cured in underwater conditions, the samples from Case C showed an increase in free expansion strain when the curing temperature increased immediately and no change until 91 days. The results also showed that the higher the temperature, the greater the free expansion strain.

3.3 Compressive strength

The compressive strength of mortar with and without EX at various temperatures and curing conditions is shown in Fig. 4. The results showed that with a low curing temperature, the samples from Cases B and C had the lowest compressive strength value at 14 days when compared to the sample in Case A. For the samples in Case A, the decrease tendency in compressive strength of mortar was observed when temperature raised to 40℃. This may be due to the expansion of mortar generated by the high temperature, resulting in very small cracks in these samples that are not visible to the naked eye.

However, for the samples in Case B, after 14 days, when the curing temperature was raised, the hydration of EX increased, and more expansion force was generated in the expansive mortar, resulting in the formation of cracks on the sample's surface, as shown in Fig. 1. This explains why the compressive strength of B-EX5 decreased immediately after 14 days at 20℃ and 40℃. The findings also indicated that compressive strength decreased rapidly when the curing temperature was high. In other words, the higher the curing temperature, the greater the decrease in compressive strength. This could be because at higher temperatures, cracks appear faster and strength decreases faster. However, for the samples in Case C, after 14 days, when the curing temperature was increased, the compressive strength increased. Despite the fact that both Cases B and C were cured for 14 days under the same conditions (air curing at 5°C), the compressive strength tendency of Case C differed from that of Case B. This is because the samples in Case C were cured underwater, which supplied enough water for the hydration reaction of cement and led to the high compressive strength of mortar. These results are in line

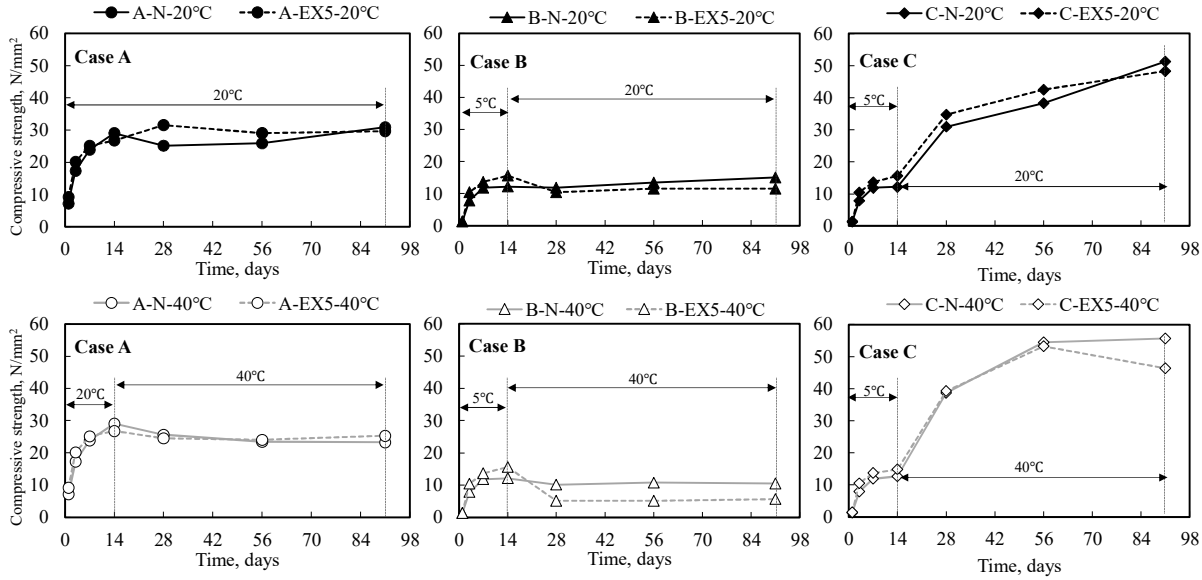

Fig. 4 Compressive strength

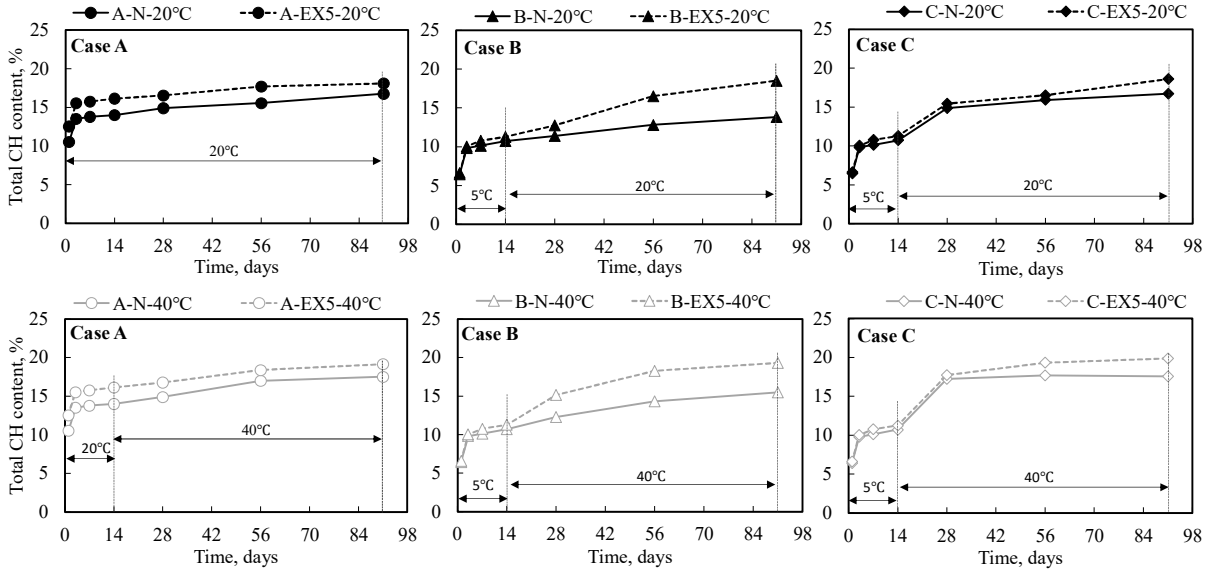

Fig. 5 Total Portlandite content

with the hypotheses discussed in Section 3.1.

3.4 TG-DTA

In this study, the total CH content was calculated by considering the non-carbonated and carbonated CH, which is described in Section 2.4. Fig. 5 presents the total CH content of all samples. In general, the CH content of expansive mortar was higher than that of non-expansive mortar, which was due to the hydration reaction of EX. This is consistent with the findings of Van et al. [3]. At 14 days, Cases B and C had a lower CH content than Case A. This could be explained by the fact that these Cases were cured at a low temperature, resulting in less cement and EX reacting. From 14 days on, the CH content roughly increased when the curing temperature was raised. In other words, many unhydrated EX particles will continue to react, resulting in a greater expansion force. Meanwhile, the strength of mortar was still low at that time. This is a reason why cracks formed on the surface of the B-EX5 sample. Additionally, the results also showed that the speed of increase in the CH content of samples cured at 40℃ was faster than that of samples cured at 20℃ . This is a reason why the cracks that appeared on the surface of the sample (B-EX5) cured at 40℃ much quicker.

Additionally, based on the results of the TG-DTA test, we found that a part of the ettringite tends to transfer to monosulfoaluminate for the samples from Cases A and C, as the DTG results showed a peak corresponding to the decomposition of AFm (around 175℃) [5], as shown in Fig. 6. This tendency was not observed in the sample from Case B. It is well known that the gypsum content influences the transformation of ettringite to monosulfoaluminate. It can be concluded that the high curing temperature and underwater curing, which allow for a smooth hydration process, resulted in more gypsum consumption and the formation of monosulfoaluminate. This transformation may have reduced the expansion force generated by ettringite and CH inside the sample. This also explains why there were no cracks in the samples from Cases A and C.

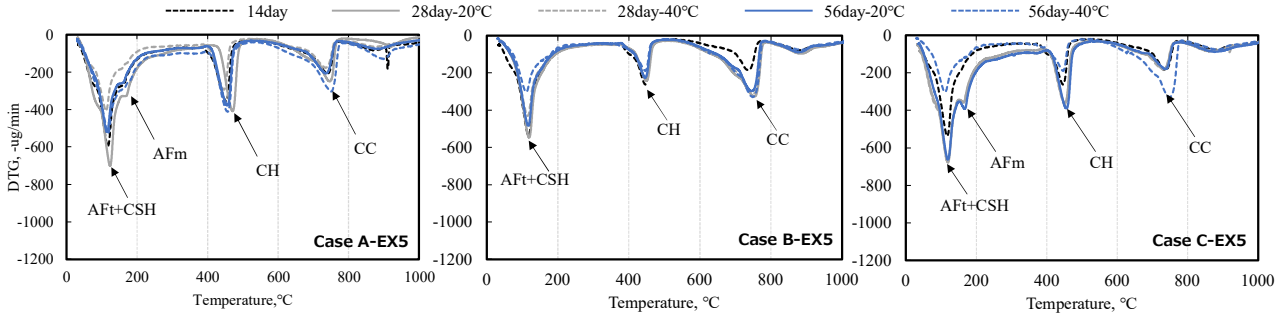

Fig. 6 DTG data recorded by TG-DTA test

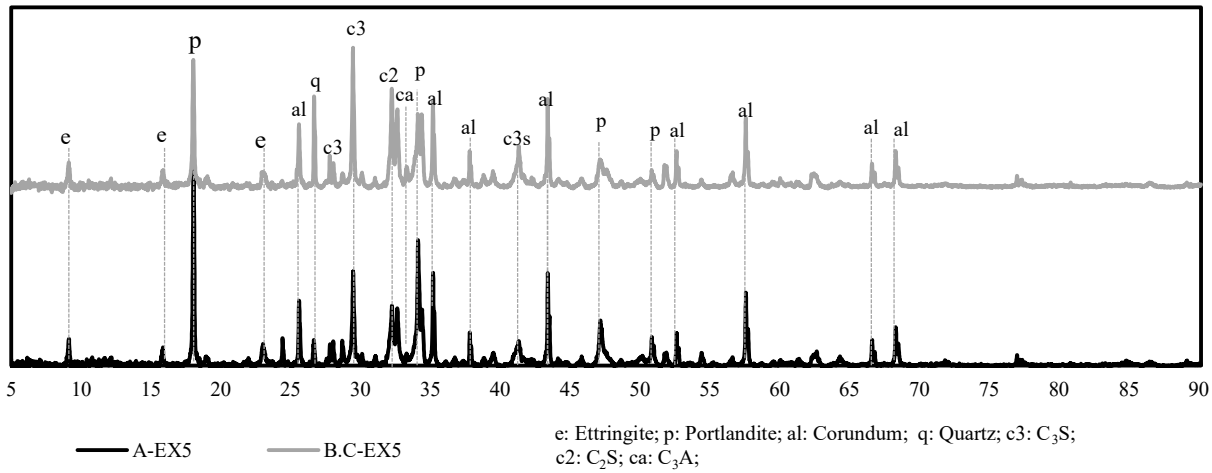

Fig. 7 XRD patterns of expansive mortar at 14 days

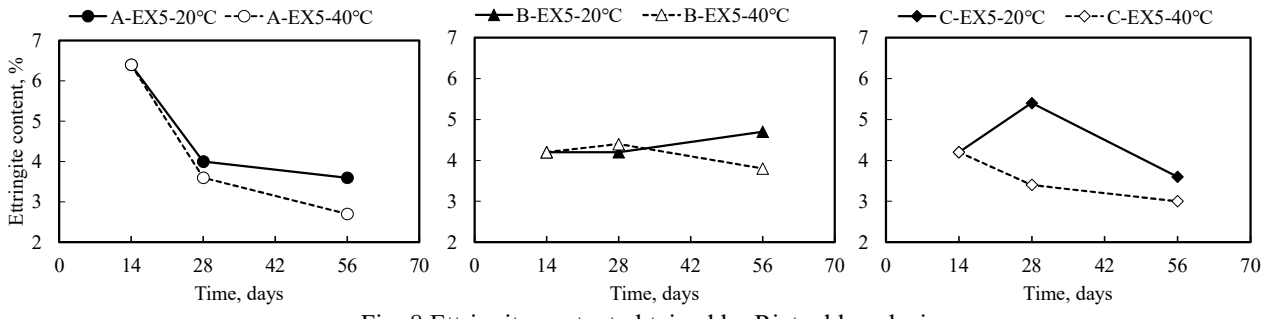

Fig. 8 Ettringite content obtained by Rietveld analysis

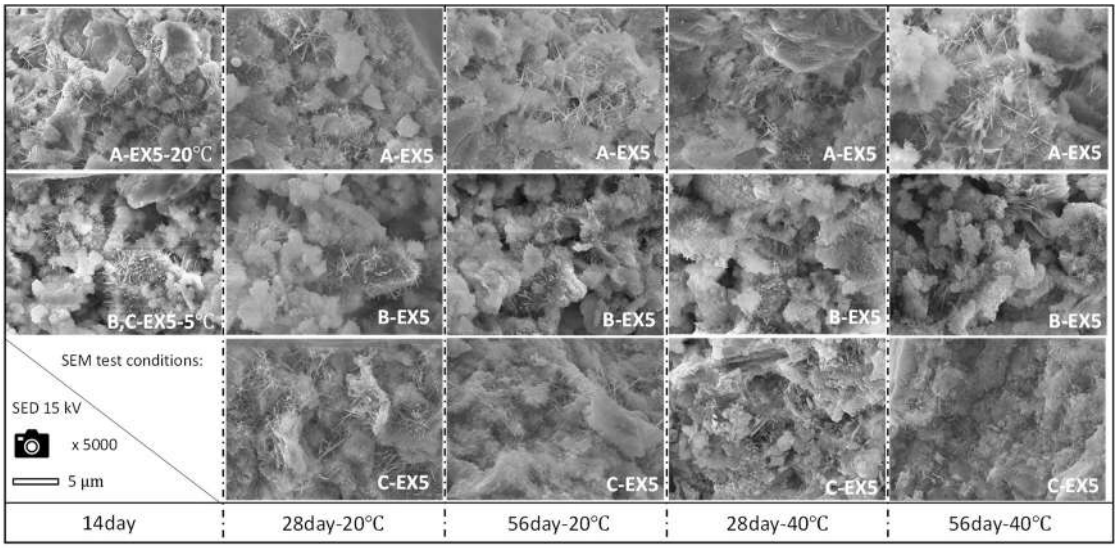

Fig. 9 SEM images of expansive mortar at different curing temperature and curing conditions

3.5 XRD and Rietveld analysis

Fig. 7 depicts the X-ray diffraction patterns of expansive mortar at 14 days. As can be seen in Fig. 7, the intensity of the ettringite and CH peaks in the B-EX5 and C-EX5 samples was lower than in the sample in Case A. The intensity of the cement mineral composition peaks in the B-EX5 and C-EX5 samples at a higher level than that in the sample in Case A. This demonstrates that the hydration of cement and EX was delayed by the low curing temperature. The amount of ettringite was obtained from Rietveld quantitative phase analysis, as presented in Fig. 8. The results show that the ettringite amount for the samples in Cases A and C tended to decrease with the duration. This tendency may be due to the above-mentioned transformation of ettringite to monosulfoaluminate. Meanwhile, after 14 days, the amount of ettringite in the sample from Case B slightly increased with time. This implies that there is still a significant amount of EX that has not fully reacted after curing for 14 days at the low curing temperature. These findings supported the hypothesis discussed above.

3.6 SEM

The ettringite morphology and internal structure of expansive mortar are shown in Fig. 9. As can be seen in Fig. 9, the morphology of ettringite (needle-like shape) was easily observed in all samples. The results also showed that the samples were cured at high temperatures, and that the samples cured underwater (Cases A and C) had a denser structure than the samples cured at low temperatures.

4. CONCLUSIONS

The following major conclusions are drawn based on the results and discussion in this study:

(1) After 14 days of curing at a low temperature, cracks appeared on the surface of the sample cured in the air at 36 and 18 days, when the curing temperature was raised to 20℃ and 40℃, respectively. However, with the same pre-curing conditions as in Case B, no cracks were found in the samples cured underwater (Case C). Therefore, in cold regions, after demoulding, the expansive concrete structure (such as slabs) must be supplied with enough water for the hydration of the cement and EX to avoid cracking due to the delayed hydration of the expansive additives.

(2) Although cracks were observed on the surface of the samples in this study, the exact time when the cracks occurred was not assessed. As a result, more research is necessary in the future to figure out the exact time at which the crack formed.

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REFERENCES

[1] Tsuji, Y.,Yokota, N., Suzuki, Y., Morimoto, H.,"Effect of curing temperature on the expansion behavior and compressive strength of expansive concrete", Proceedings of the 3rd Japanese Concrete Institute Annual Conference,pp. 5-8, 1981.

[2] Togawa, K., Nakamoto, J.,"Effect of temperature on expansion characteristics in expansive concrete", Proceedings of the Japan Society of Civil Engineers, No. 321, pp. 177-187, Apirl 1981.

[3] Van, N.D., Choi, H., Hama, Y., "Modeling early age hydration reaction and predicting compressive strength of cement paste mixed with expansive additives", Construction and Building Materials, Vol. 223, pp. 994-1007, 2019.

[4] Shafiq, N., “Degree of hydration and compressive strength of conditioned samples made of normal and blended cement system, Ksce J. Civil Eng. Vol. 15 (7), pp. 1253-1257, 2011.

[5] Scrivener K., Snellings R., Lothenbach B., A practical guide to microstructural analysis of cementtitious materials, Taylor & Francis Group, 2016, pp. 107-211.