- Technical Paper -

LONG-TERM PORE STRUCTURE OF INTERNALLY CURED FLY ASH CEMENT PASTE BY AN INJECTION OF WATER

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ABSTRACT

Long-term pore structure of fly ash cement paste injected with water through a needle from a 1-mL syringe was investigated, to assess the effects of internal curing on long-term mechanical properties and durability. The injection of water at 1 month to simulate internal curing evidently affected pore structure of cement paste having 40% replacement with fly ash, decreasing volume of capillary pores by 6.0–42.9% up to 24 months and increasing that of gel pores by 10.2–99.2% since 6 months in addition to increasing the consumption of $Ca(OH)_2$ by the pozzolanic reaction by 20.1% at 24 months.

Keywords: internal curing, pozzolanic reaction, capillary pores, gel pores, $Ca(OH)_2$ content

1. INTRODUCTION

In recent years, internal curing has been applied to concrete production due to its positive effects. For example, internal curing promotes cement hydration [1, 2, 3] and reduces plastic shrinkage, autogenous shrinkage and cracking [4, 5, 6]. Compared with external curing, internal curing is more effective for paste systems having a low water-to-binder ratio. It has been found that external curing water can penetrate only several millimeters into the surface of system with a low water-binder ratio, whereas internal curing water can be introduced more equally across the cross section and thus produce a cured zone throughout the entire paste system [2]. Some researchers have also begun to study the application of internal curing to high volume fly ash mixtures with a low water-to-binder ratio in order to reduce the autogenous shrinkage and increase the hydration as well as improve the durability of the mortar mixtures [7, 8]. However, these studies have focused solely on the shrinkage, strength, and fluid transport of mixtures consisting of high-calcium fly ash, not low-calcium fly ash.

Porosity is one of the main aspects of the microstructure that affects both the mechanical properties and durability of cement-based materials [9]. According to Mindess and Young, the porosity in hydrated cement paste can be divided into two types: capillary and gel pores. Capillary pores, with a diameter range of 0.01–10 µm, are the remnants of water-filled spaces that exist between partially hydrated cement grains, and affect the strength and permeability of paste. Meanwhile, gel pores, with diameters less than 0.01 µm, can be regarded as part of the calcium silicate hydrate (C-S-H) network, which is the most important hydration product [9]. There are two main methods that are used to assess the pore structures of hydrated cement paste: mercury intrusion porosimetry and physical adsorption of gases. Mercury intrusion porosimetry has been used to measure capillary pores and gel pores, including the small capillary pores of cementitious materials [9, 10, 11].

Based on the above, a long-term study on the pore structure of internally cured cement paste containing low-calcium fly ash was proposed, as a means of assessing the effects of internal curing on the long-term mechanical properties and durability. The present study, therefore, aims at investigating the effects of internal water curing on pore structure over a span of 24 months, by examining cement pastes containing low-calcium fly ash in which water is injected through a 1-mL syringe. This approach was meant to model internal curing, similar to the experimental methods of some previous studies [12, 13, 14]. Because of the difficulty in exactly evaluating the movement of internal water in the fly ash cement system, water was injected 1 month after casting into the fly ash cement system, at which point the system may have had insufficient water for cement hydration and pozzolanic reaction. In addition to the effects of internal water curing on the capillary and gel pores of low-calcium fly ash cement paste, the consumption of $Ca(OH)_2$ by the pozzolanic reaction was also investigated.

2. EXPERIMENTAL INVESTIGATION

2.1 Materials and specimen preparations

A high-early-strength Portland cement conforming to JIS R 5210 (Portland cement) meant to ensure the early strength of the paste, and a low-calcium fly ash conforming to JIS A 6201 (fly ash

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for concrete) were used. The chemical compositions and physical properties of these materials are listed in Table 1.

Paste specimens were prepared with a low water-to-binder ratio of 0.30, which has been shown to be beneficial during the internal curing of pastes and concretes [15]. Low-calcium fly ash was used to replace portions of the high-early-strength Portland cement at mass ratios of 0% (FA0) and 40% (FA40). Superplasticizer and air-entraining agent were used to obtain sufficient consistency for casting. The pastes were mixed in a mechanical mixer and cast in 40-mm-per-side cube-shaped molds. Similar to the experimental method of previous studies [12, 13, 14], a needle with an internal diameter of 0.395 mm was inserted into the center of the sample. During the mixing process, the temperatures of the materials, mixer and mold were all maintained at 20°C. All specimens were demolded 24 h after casting and then sealed with aluminum tape to prevent water loss and carbonation. After that, all specimens were cured at 20°C.

2.2 Model for internal water curing

As noted, deionized water was supplied through a needle from an attached 1-mL syringe from the age of 1 month (see Fig. 1), which was considered to represent a model of internal curing. In addition, control specimens were fabricated with no water injection. The water was allowed to naturally seep into the specimen as a result of the permeability of the paste. That is, no additional pressure was applied to force the liquid into the paste, thus simulating water supplied from internal curing agents. The volume of water that flowed into the sample over time (see Table 2) was tracked for future comparison to the volume of internal water released from internal curing agents throughout the paste over time. In the present study, 1-mL of water added via the syringe was assumed to represent the internal curing water, equal to several percent of the initial mixing water used in the concrete. As shown in Table 2, these volumes are the cumulative water amounts from the initial injection to the designated measuring time, and vary depending on the individual differences in the microstructures of the specimens. As the initial 1-mL of water was consumed by various specimens, more water was added to the syringes inserted into each sample so that the volume of water in all the syringes was constant at 1 mL.

2.3 Mercury intrusion porosimetry

The pore structures in test samples were assessed using mercury intrusion porosimetry (MIP; POREMASTER 60, Quantachrome Instruments, USA). Samples for MIP testing were obtained by crushing the hardened paste cubes within a range of approximately 10 mm from the tip of the syringe needle and selecting material in the size range of 2.5 to 5.0 mm. Samples were subsequently soaked in ethanol for 24 h to avoid any further hydration and dried in a vacuum desiccator for 24 h before MIP measurements. The MIP equipment used in this study operated at a maximum pressure of

Table 1 Chemical compositions and physical properties of cementitious materials

Properties	Cement	Fly ash
SiO_2 (%)	20.30	57.7
Fe_2O_3 (%)	2.71	5.43
Al_2O_3 (%)	4.96	27.54
CaO (%)	65.49	1.26
MgO (%)	1.21	1.06
SO_3 (%)	2.98	0.36
Na_2O (%)	0.22	0.44
K_2O (%)	0.35	0.76
Loss on ignition (%)	1.19	2.8
Density (g/cm^3)	3.14	2.21
Blaine specific surface area (cm^2/g)	4590	3290

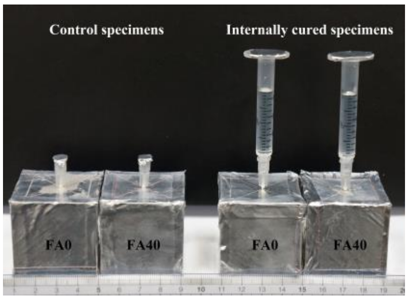

Fig.1 Specimens with no added water or with water added from the tip of a needle at the center of the cube, allowed to naturally flow into the permeable paste as a model of internal curing

Table 2 Volumes (mL) of water transferred into the internally cured specimens over time

Age (months)	FA0	FA40
2	0.10	0.05
4	0.26	0.05
6	0.38	0.35
8	0.69	0.10
10	1.00	0.14
12	0.57	0.20
16	1.16	0.27
20	1.42	1.23
24	0.55	0.10

4.14×10^8 N/m^2. Individual measurements (n = 1) of pore structures in the pastes were performed over a diameter range of 0.003 to 10 μm after aging for 2, 4, 6, 8, 10, 12, 16, 20 and 24 months.

2.4 Thermal gravimetric analysis

The $Ca(OH)_2$ contents in various specimens were measured with a simultaneous differential thermal analysis and thermal gravimetric (DTA-TG) apparatus (DTG-60H, Shimadzu Corporation, Japan) after aging for 1, 2, 4, 6, 8, 10, 12, 16, 20 and 24 months. Individual measurements of the pore structures and $Ca(OH)_2$ contents (n = 1) were conducted on the same specimens. A 4-mm sample was drilled out at the point of the needle and collected as a powder. Each of these samples was soaked in ethanol for 24 h to avoid any further hydration and subsequently dried in a vacuum desiccator for 24 h prior to thermal gravimetric (TG) analysis. During TG trials, the sample temperature was raised at a rate of 20ºC/min and then held at 100ºC for 30 minutes to remove free water, before being raised to 1000°C at 20ºC/min. The $Ca(OH)_2$ content in each specimen was calculated from the mass of the sample after ignition and the mass loss between 400 and 500°C due to dehydration of the $Ca(OH)_2$. This mass loss was determined from the differential thermogravimetry (DTG) plot between the initial and final temperatures of the corresponding DTG peaks [16].

3. RESULTS AND DISCUSSION

3.1 Pore structure

The volumes of total pores in the 0.003–10 µm range, the volumes of capillary pores in the 0.01–10 µm range and the volumes of gel pores in the 0.003–0.01 µm range of both control specimens and internally cured specimens with 0% and 40% replacement with low-calcium fly ash up to the age of 24 months are summarized in Figs. 2 and 3, respectively.

(1) Effect of internal water curing on the total pore volume

According to Fig. 2, the total pore volumes of the control specimens are observed to have decreased over time from 2 to 24 months. This decrease in the total pore volume implies that hydration of the control specimens proceeded throughout aging, generating hydration products that filled the pores. As well, the internal water curing reduced the total pore volumes of the water-injected FA0 specimens by 15.7–40.1% when compared with the control FA0 specimens. It should be noted that the cement hydration promoted by internal water curing was not only found in a previous study [12] but also observed in the present study. Therefore, the additional generation of hydration products in FA0 specimens could have filled the empty spaces and reduced the total pore volume of the cement paste specimens.

As shown in Fig. 3, the total pore volumes of the FA40 specimens decreased with time, regardless of internal curing. Moreover, internal water curing reduced the total pore volumes of the FA40 specimens by 2.9–18.9% when compared with the control FA40 specimens. This result demonstrates that the internal water curing increased the degree of hydration in the low-calcium fly ash cement pastes. However, significant reduction of the total pore volume of the FA40 was not observed at the age of 24 months. It may be due to a small volume of water penetrating into the specimens as shown in Table 2.

(2) Effect of internal water curing on capillary pore volume

The volumes of capillary pores in the 0.01–10 µm range allow us to assess the effects of internal water curing on the material properties such as strength and

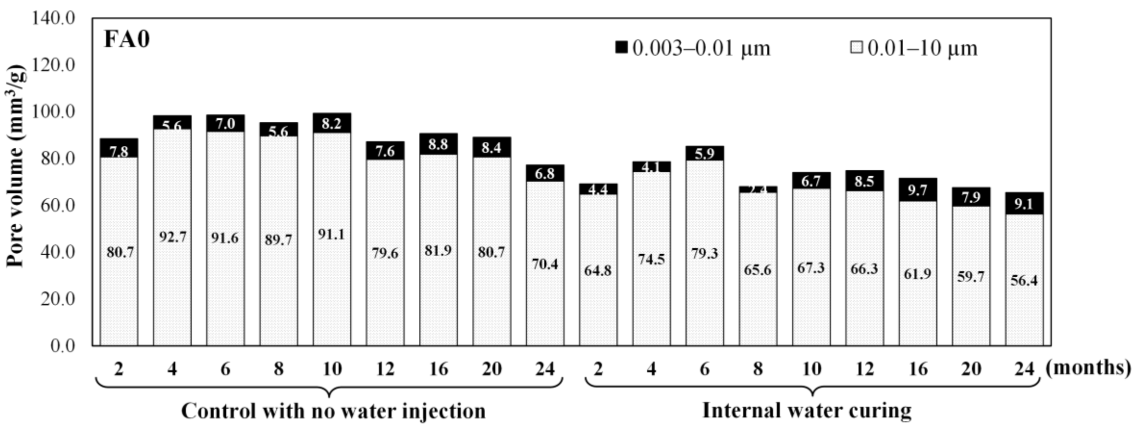

Fig.2 Effects of internal water curing on the pore structure of specimens with no low-calcium fly ash (FA0)

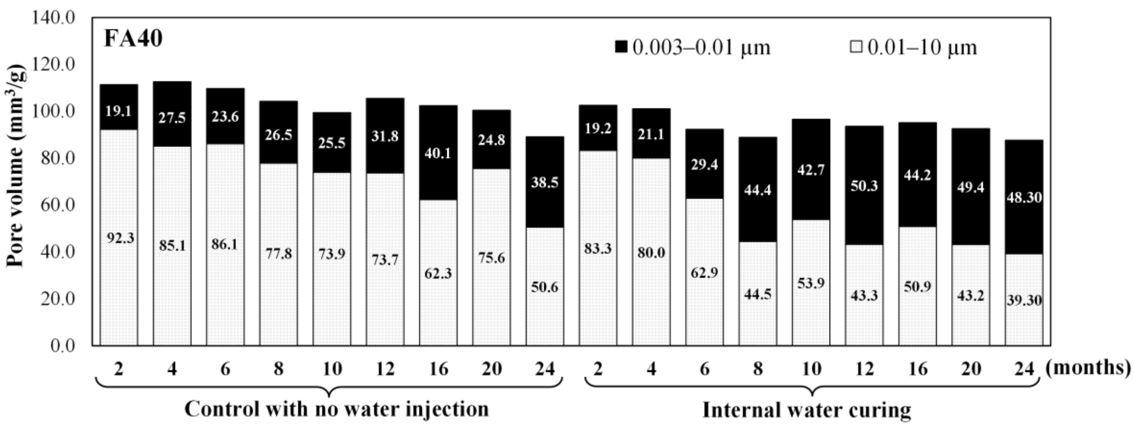

Fig.3 Effects of internal water curing on the pore structure of specimens with 40% replacement with low-calcium fly ash (FA40)

permeability of the paste specimens [9]. According to Figs. 2 and 3, the capillary pore volume decreased with time regardless of fly ash replacement and internal curing, except for the control specimen (FA40) at the age of 20 months. In the case of the FA0 specimens, a slight reduction of the capillary pore volume was observed, while the FA40 specimens showed significant pore volume reduction over the period ranging from 2 to 24 months. This trend could be an indication of the continued pozzolanic reaction of the fly ash in the FA40 over time. Compared with the control specimens, internal water curing reduced the capillary pore volumes of the FA0 and FA40 samples by 13.4–26.9% and 6.0–42.9%, respectively. It is evident that cement paste with sufficient internal water curing may hydrate to a greater extent than paste without internal water curing, resulting in the capillary pores being filled with hydration products. This reduction of the capillary pore volume also explains other effects of internal water curing related to increased strength and decreased permeability of paste/concrete specimens, as reported in some previous studies [1, 2]. Furthermore, the capillary pore volumes of the FA0 specimens were decreased by 19.9% while those of the FA40 specimens were decreased by 22.3% at 24 months by the injection of water. These results demonstrate that internal water curing was effective in decreasing the capillary pore volume of the FA0 and FA40 specimens over time. This implies that internal water curing promoted cement hydration over the short term after injection and then accelerated the pozzolanic reaction of the fly ash in FA40 specimens for a prolonged time span after injection.

(3) Effect of internal water curing on gel pore volume

In the case of the control specimens, the volumes of gel pores in the 0.003–0.01 μm range of the FA0 samples remained constant, whereas those of the FA40 samples increased with time, with the exception of the FA40 specimen at 20 months. Therefore, it appears that the generation of additional hydration products, such as C-S-H, by pozzolanic reactions in the FA40 specimens are related to increases in the gel pore volumes.

Compared with the control specimens, internal water curing did not affect the gel pore volumes of the FA0 specimens. Meanwhile, despite the negligible effect on the gel pore volumes of FA40 specimens at 2 and 4 months, afterwards internal water curing increased the gel pore volumes of FA40 samples by 10.2–99.2%. Additionally, internal water curing increased the gel pore volumes of FA40 samples by 25.5% relative to the pore volumes of control specimen at the age of 24 months, although internal water curing also reduced the total pore volumes of FA40 specimens by a negligible amount (Fig. 3). The increases in the gel pore volumes of FA40 specimens into which water was injected at 1 month implies that internal water curing accelerated the pozzolanic reactions in these materials. In brief, internal water curing promoted the hydration of the cement paste with no low-calcium fly ash (FA0), resulting in the reduction of the total pore volume and the capillary pore volume up to the age of

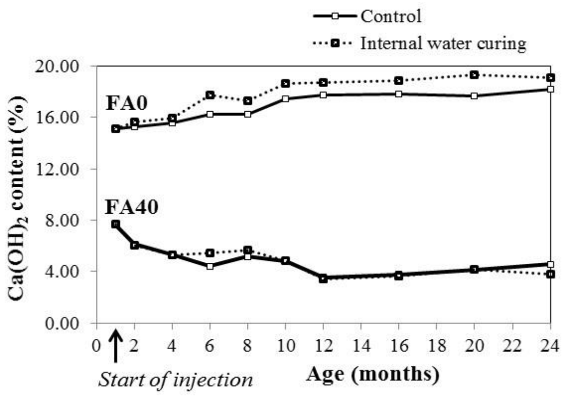

Fig.4 Effects of internal water curing on the $Ca(OH)_2$ contents at the point of the syringe needle of specimens with no low-calcium fly ash (FA0) and 40% replacement with low-calcium fly ash (FA40)

24 months. Meanwhile, it also promoted cement hydration and accelerated the pozzolanic reaction of the cement paste in which there was 40% replacement with low-calcium fly ash (FA40) with the decreases in the total pore volume and the capillary pore volume and an increase in the gel pore volume up to 24 months.

3.2 $Ca(OH)_2$ analysis

(1) $Ca(OH)_2$ content

The $Ca(OH)_2$ content is typically used to evaluate the degree of hydration in a cement paste [17]. Fig. 4 summarizes the $Ca(OH)_2$ content at the point at which the syringe needle was inserted in the control specimens and the internally cured specimens with no low-calcium fly ash (FA0), up to 24 months aging. It can be seen that the $Ca(OH)_2$ content in the FA0 specimens increased with time regardless of internal curing. This is an indication that cement hydration in the case of the FA0 specimens continued to occur over time despite using high-early-strength Portland cement for the FA0 paste in conjunction with a low water-to-binder ratio of 0.30. The increase in the $Ca(OH)_2$ content of the FA0 specimens was also compatible with the reductions in the total pore volumes over time observed in Fig. 2. It is also evident that internal water curing promoted cement hydration in the FA0 specimens, resulting in slightly higher amounts of $Ca(OH)_2$. The acceleration of cement hydration in FA0 specimens was also observed in a study in which water was injected at 3 months [13, 14].

Generally, the pozzolanic activity of fly ash is confirmed by a reduction of the $Ca(OH)_2$ content [18]. Thus Fig. 4 also tracks the $Ca(OH)_2$ contents at the needle point of control specimens and internally cured specimens with 40% replacement with fly ash (FA40) up to 24 months, as a means of evaluating the progress of the pozzolanic reaction. It is evident that the $Ca(OH)_2$ content in the control specimens decreased gradually with time, demonstrating that the pozzolanic reaction of the fly ash in the control specimens was proceeding. The amounts of $Ca(OH)_2$ in the internally cured specimens were almost equal to those of the control specimens at 2, 4, 10, 12, 16, 20 and 24 months, with the only significant differences occurring at 6 and

8 months. The slightly higher $Ca(OH)_2$ contents at 6 and 8 months indicate that cement hydration in the FA40 specimens was promoted by internal water curing. In contrast, the amounts of $Ca(OH)_2$ in the internally cured FA0 specimens were higher than in the control specimens (FA0), while the contents in the internally cured FA40 specimens were slightly lower or nearly the same as those of the control specimens. These data suggest that the additional $Ca(OH)_2$ generated by cement hydration may be subsequently consumed by the pozzolanic reaction, resulting in a slight decrease in the $Ca(OH)_2$ content in the internally cured FA40 specimens. This finding demonstrates that the pozzolanic reaction in the FA40 specimens was accelerated by internal water curing, in agreement with the increased gel pore volumes of FA40 specimens seen in Fig. 3.

(2) Consumption of $Ca(OH)_2$

It is well-known that the hydration of binder consisting of cement and fly ash includes two interrelated process: the cement hydration and the pozzolanic reaction of fly ash which consumes the $Ca(OH)_2$ formed from the cement hydration. The hydration degree of cement in the matrix including cement and fly ash increases more than that in the matrix with only cement [19]. However, it is assumed that the cement hydration in the fly ash cement paste (FA40) is the same as that in the cement paste (FA0). Therefore, the consumptions of $Ca(OH)_2$ by the pozzolanic reaction up to 24 months in the control specimens and the internally cured specimens as shown in Fig. 5 were calculated by the equation (1)

$$CH_{cons.} = CH_{FA0}(c/(c+f)) - CH_{FA40} \qquad (1)$$

where $CH_{cons.}$ is the consumption of $Ca(OH)_2$ by the pozzolanic reaction (%), CH_{FA0} is the $Ca(OH)_2$ content in FA0 specimens (%), CH_{FA40} is the $Ca(OH)_2$ content in FA40 specimens (%), and $(c/(c+f)) = 0.60$ is the mass ratio of cement in the binder (cement + fly ash). Typically, the consumption of $Ca(OH)_2$ by the pozzolanic reaction increases gradually with time regardless of internal curing, demonstrating that the pozzolanic reaction of fly ash proceeds over the course of aging. When compared with the control specimens, internal water curing is seen to have increased the consumption of $Ca(OH)_2$ by the pozzolanic reaction by 20.1% at the age of 24 months. This finding again confirms that internal water curing accelerates the pozzolanic reaction of fly ash up to the age of 24 months.

(3) Relationship between consumption of $Ca(OH)_2$ and pore structure

The consumption of $Ca(OH)_2$ by the pozzolanic reaction showed a negative correlation with the capillary pore volume and a positive correlation with the gel pore volume, as shown in Figs. 6 and 7, respectively. This can be explained by considering that the pozzolanic reaction in the fly ash cement paste proceeded, consuming $Ca(OH)_2$ formed from the

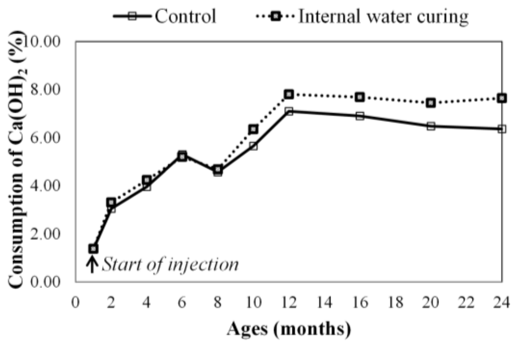

Fig.5 Effects of internal water curing on the consumption of $Ca(OH)_2$ by the pozzolanic reaction

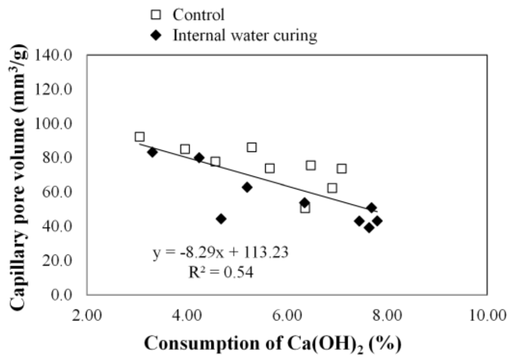

Fig.6 Relationship between the consumption of $Ca(OH)_2$ and the capillary pore volume

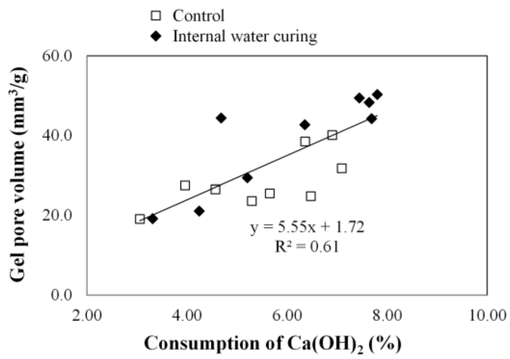

Fig.7 Relationship between the consumption of $Ca(OH)_2$ and the gel pore volume

cement hydration. This process resulted in the increased generation of hydration products filling the capillary pores and forming gel pores.

3. CONCLUSIONS

This work represented a long-term study on the volumes of total pores in the 0.003–10 μm range, the volumes of capillary pores in the 0.01–10 μm range, and the volumes of gel pores in the 0.003–0.01 μm range up to 24 months of aging for internally cured cement pastes. In these specimens, either 0 or 40% of the cement was replaced with low-calcium fly ash in conjunction with a low water-to-binder ratio of 0.30 and the injection of water at the age of one month to simulate internal curing. The following conclusions can be drawn from this study:

(1) Internal water curing reduced the total pore volume by 15.7–40.1% and the capillary pore volume by 13.4–26.9%, but did not affect the gel pore volume of the cement paste with no low-calcium fly ash. The reduction of the capillary pore volume in this study clarified the effects of internal water curing, which has been previously reported to result in increased strength and improved permeability of paste/concrete.

(2) Internal water curing decreased the total pore volume by 2.9–18.9% and the capillary pore volume by 6.0–42.9%, while increasing the gel pore volume by 10.2–99.2% since the age of 6 months for the cement paste in which 40% of the cement was replaced with low-calcium fly ash. This finding demonstrates that internal water curing promotes cement hydration and accelerates the pozzolanic reaction up to 24 months in low-calcium fly ash cement paste.

(3) Internal water curing slightly increased the $Ca(OH)_2$ content in cement paste with no low-calcium fly ash, while it increased the consumption of $Ca(OH)_2$ by the pozzolanic reaction by 20.1% at 24 months in cement paste having 40% replacement with low-calcium fly ash.

Consequently, the injection of water to simulate internal curing increased the consumption of $Ca(OH)_2$ by the pozzolanic reaction, resulting in the increased generation of hydration products filling the capillary pores and forming gel pores not only 1 month but also 23 months after injection. The results of the present study clarified the long-term effects of internal curing on the improvement of mechanical properties and the enhancement of the durability of fly ash concrete.

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