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Effect of water curing conditions on the hydration degree and compressive strengths of fly ash–cement paste

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Abstract

This paper explains the effect of water curing condition on compressive strengths of fly ash–cement paste by quantitative data of hydration degree. Hydration of fly ash–cement paste was estimated by Rietveld analysis and selective dissolution. The result shows that the hydration degree of belite is affected by water curing conditions, more so than that of fly ash and alite. Fly ash still continues to hydrate even without an extra, external supply of water. The strong dependence of fly ash–cement concrete on curing conditions does not come from the hydration degree of fly ash, but rather comes from the hydration degree of cement, especially belite. When the water to binder ratio is low enough, the hydration of cement plus small hydration of fly ash are considered to be enough for adequate compressive strength at the beginning. Then, compressive strength of fly ash–cement paste becomes less sensitive to the water curing period.

Keywords: Curing; Compressive strength; Fly ash; Hydration

1. Introduction

Fly ash–cement concrete has been utilized in buildings and infrastructure systems for many years as it is useful for modifying many properties of concrete such as workability, strength, shrinkage and heat evolution [1–6]. Regarding its properties of strength, numerous studies have been made on the strength characteristics of fly ash–cement concrete [3,7–10].

Generally, it is said that the pozzolanic reaction from fly ash makes the concrete containing fly ash more durable than conventional concrete. As fly ash hydrates, empty space is filled by the hydration product, resulting in a reduction of permeability. However, the main problem is its slow development of strength at early ages, especially when a large amount of fly ash is used.

Fly ash improves the properties of concrete or cement paste due to the pozzolanic reaction and its role as a micro-filler. It is often thought that the first function (pozzolanic reaction) is most important. The hydration reaction depends on curing period so that, in many specifications, it is noted that fly ash–cement concrete needs a longer curing period than conventional concrete. However, in the practical application, longer curing periods are sometimes not properly done or avoided, since longer a curing period usually raises the cost of construction.

Many researchers have studied about the effect of curing condition on the strength of fly ash–cement concrete mortar and paste. Most of them have reported that the influence of curing condition and curing period on fly ash–cement concrete was higher than that of conventional concrete [11–19].

In a recent paper, Ozer and Ozukul [11] reported about the result of the influence of initial water curing on the strength development of ordinary Portland cement and pozzolanic cement concretes. They concluded that poor curing conditions more adversely affects the strength of

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concrete made from pozzolanic cement than that of ordinary Portland cement.

Atis [12] worked on strength properties of high-volume fly ash roller compacted and workable concrete, and the influence of curing condition. He reported that fly ash–cement concrete was more sensitive to dry curing conditions than conventional concrete.

However, above explanation, it should be noted that there is few studies about the relation between individual hydration degree of cement and fly ash and compressive strength under different curing conditions. This is because it is still difficult to directly measure individual hydration degree of fly ash and cement in fly ash–cement system.

For ordinary Portland cement, hydration degree can be estimated by many methods such as thermal analysis (DTA), thermogravimetric (TG) analysis, with a differential scanning calorimeter (DSC), examining non-evaporate water content, and backscattered electron image analysis (BSE). Among these methods, however, there is no method that is able to estimate the composition and hydration degree in binary cementitious systems such as the fly ash–cement system.

It has been reported that the amount of $Ca(OH)_2$ can be quantitatively measured by thermal analysis (DTA) and thermogravimetric (TG) analysis. However, in the reaction processes of fly ash–cement systems, cement produces $Ca(OH)_2$ while fly ash is consuming $Ca(OH)_2$. Thus, the amount of $Ca(OH)_2$ can no longer represent the hydration degree in such binary cementitious systems.

As for BSE image analysis, it was reported that this method is capable of quantitatively measuring the amount of unhydrated Portland Cement [20,21]. Igarashi et al. [22] reported that the brightness of fly ash particles is sufficiently distinct from that of unhydrated cement particles. However, it is quite difficult to measure the area of fly ash distinguished by its brightness by a computer because calcium silicate hydrate gel (C–S–H gel) also has a similar brightness. Some researchers suggest that despite the similarity in the brightness of fly ash and C–S–H gel, the territory of fly ash is clearly visible, and for this reason, a point counting method might be applicable. However, this method requires specialized skills and is tedious and time consuming.

Many researchers discuss the application of Rietveld analysis to a complex system such as cement [23–25]. In the field of hydration of cement, Scrivener et al. [26] has applied Rietveld analysis to obtain quantitative information of Portland cement hydration.

XRD Rietveld analysis is capable of measuring the amount of crystal and estimating the amount of total amorphous material indirectly, however, it cannot distinguish among different types of amorphous material. In a fly ash–cement hydrated sample, there are two main types of amorphous material: that in fly ash and C–S–H gel. In order to determine the amount of each compound, one needs to know the amount of each type of amorphous material. This makes the analysis more complicated.

Although it is difficult to analyze hydration in fly ash–cement systems, fly ash has another advantage; that is, it is very compatible with the use of superplasticizer [6]. In comparison with conventional concrete, the rheology of fly ash–cement concrete is enhanced very much even when a small amount of superplasticizer is added. Because of this, at the same flow ability, fly ash concrete needs a smaller amount of water.

When the water to binder ratio is low, the role of fly ash as micro-filler may be more prominent than the pozzolanic effect. Therefore, hydration degree of cement plus a small hydration degree of fly ash may be enough for sufficient strength development.

This study investigates the effect of initial water curing conditions on the hydration degrees and compressive strengths of fly ash–cement paste prepared with low water to binder ratio at different ages. The hydration degree of cement and fly ash are estimated by the proposed method, as described in a previous paper [27], which is a combination of Rietveld analysis and selective dissolution.

The selective dissolution method is often used to measure the amount of unhydrated fly ash. The procedure for this method is as follows: firstly, an acid solution is used to dissolve all the Ca components in the sample; secondly, an alkaline solution is used to dissolve the gel components (Al, Si, Fe gel). In the final stage, only unhydrated fly ash is left. In this paper an HCl solution is used as the acid solution and Na_2CO_3 is used as the alkaline solution. By using this method, the amount of total unhydrated fly ash can be extracted.

The amount of each crystal phase component (crystal phases of OPC, fly ash and hydrated product) and the total amount of amorphous material can be extracted by Rietveld analysis. On the other hand, selective dissolution can give us the total of unhydrated fly ash. By applying these two methods, the hydration degree of cement and fly ash can be estimated individually.

2. Experiment

2.1. Sample preparation

Ordinary Portland cement and fly ash type II following to JIS R5210 and JIS A6201 are used in this study. The characteristic of cementitious materials are shown in Table 1. Polycarboxylate-based superplasticizer is used to control fluidity of the paste. Replacement ratios of fly ash were 0%, 25% and 50% of total powders. The water to binder ratio is 0.80 and 1.00 by volume. The main experiments were prepared with a water to binder ratio of 1.00. Samples prepared with a water to binder ratio of 0.80 were used to compare the effect of water to binder ratio on hydration degree or the amount of unhydrated of fly ash left.

First, fly ash and the cement particles were mixed together within a 2 l pan mixer for 60 s. After water was added, paste samples were mixed at low speed for 90 s and further mixed at high speed for 90 s. The flow value

Table 1
The chemical and physical properties of fly ash and OPC

	Ignition loss	Chemical composition (mass)									Density (kg/m^3)	Blaine surface area (m^2/kg)	Mineral composition (mass)			
		SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	Na_2O (%)	K_2O (%)	TiO_2 (%)	MnO (%)			C_3S (%)	C_2S (%)	C_3A (%)	C_4AF (%)
OPC	0.77	20.84	5.95	2.62	63.63	1.79	0.18	0.33	0.34	0.10	3150	347	63.09	12.99	11.78	9.23
Fly ash	0.90	59.90	29.60	4.80	1.30	0.60	0.00	0.70	0.00	0.00	2290	376	–	–	–	–

was measured according to JASS15 M-103 [28]. Sample paste was mixed and poured into the cylindrical cone, 50 mm in diameter and 51 mm in height. Then, the cone was slowly pulled up vertically. The flow value was taken as the average value of two crossing diameters of the spreading paste. The flow diameter was controlled to be 200–250 mm. Fly ash–cement paste was poured into 100 by 50 mm cylindrical molds. At 24 h after mixing, the samples were demolded and then cured with one of three different curing conditions. For condition one, the samples were cured in water until the required age. For condition two and three, the samples were initially cured in water for seven and one days, respectively. After that, the samples were kept in 60% moisture storage until the required age. The temperature was controlled to be 20 °C. For the water to binder ratio of 1.00, the samples were prepared with all three curing conditions. For the water to binder ratio of 0.80, the samples were prepared with curing condition in water only. The experiment was continued until a sample age of 182 days. Table 2 indicates the composition and curing conditions for each sample type.

Table 2
The mixed proportion and curing condition

W/B	% FA	Curing condition	Code
0.8	0	In water	0.8-0-W
0.8	25	In water	0.8-25-W
0.8	50	In water	0.8-50-W
1.0	0	In water	1.0-0-W
1.0	25	In water	1.0-25-W
1.0	50	In water	1.0-50-W
1.0	0	In water 7 days	1.0-0-W7
1.0	25	In water 7 days	1.0-25-W7
1.0	50	In water 7 days	1.0-50-W7
1.0	0	In water 3 days	1.0-0-W3
1.0	25	In water 3 days	1.0-25-W3
1.0	50	In water 3 days	1.0-50-W3

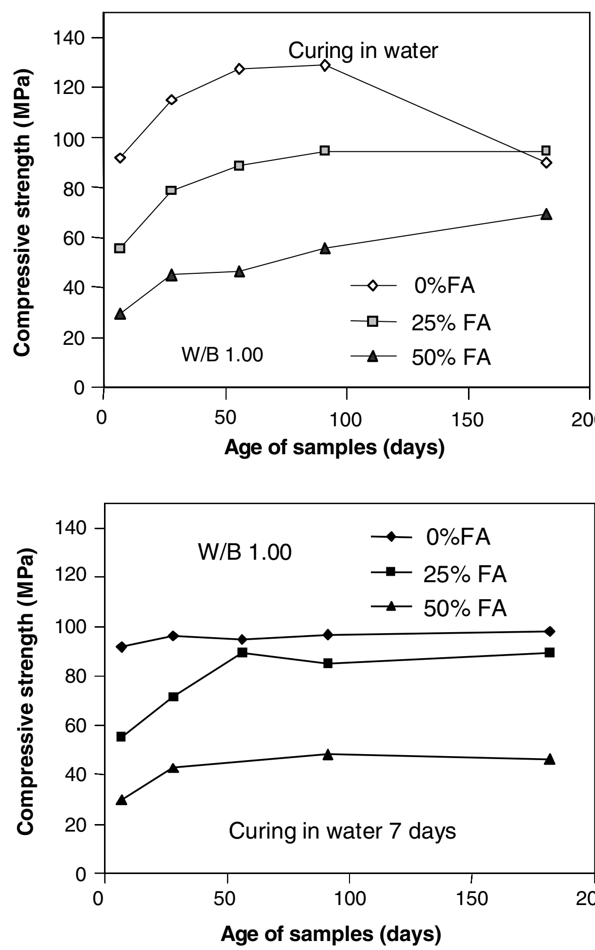

Fig. 1. The effect of replacement ratio of fly ash to the compressive strength of fly ash cement paste.

Table 3
Compression strength of fly ash–cement paste

Age	Compression strength (MPa)											
	0.8-0-W	0.8-25-W	0.8-50-W	1.0-0-W	1.0-25-W	1.0-50-W	1.0-0-W7	1.0-25-W7	1.0-50-W7	1.0-0-W3	1.0-25-W3	1.0-50-W3
7	117.03	80.89	41.45	91.84	55.39	29.93	91.84	55.39	29.93	85.02	65.17	30.78
28	150.91	113.36	65.42	114.71	78.58	44.7	95.89	71.54	42.84	95.47	71.82	40.19
56	145.99	115.06	69.41	127.24	88.72	46.51	94.97	89.37	48.45	94.612	87.68	46.9
91	129.91	116.7	83.72	128.63	94.2	55.69	96.68	84.75	48.29	97.04	77.18	45.04
182	150.11	163.29	104.81	90.19	94.7	69.24	97.94	89.51	46.77	94.01	98.51	48.55

The compressive strength of the cylinder specimens of paste was tested. Three replicate samples were tested. Fractured pieces recovered from the compressive strength tests were further cracked into 2.5–5.0 mm by hammer. Then, the hydration reaction was stopped by acetone. Next, samples were dried at room temperature (20 °C) for 24 h and dried in oven at 40 °C for an additional 3 h. The samples were ground and sieved. The particles passing a 75-μm sieve were used to determine the degree of hydration of fly ash and cement.

The amount of unhydrated fly ash was measured by selective dissolution method. The amount of crystal com-

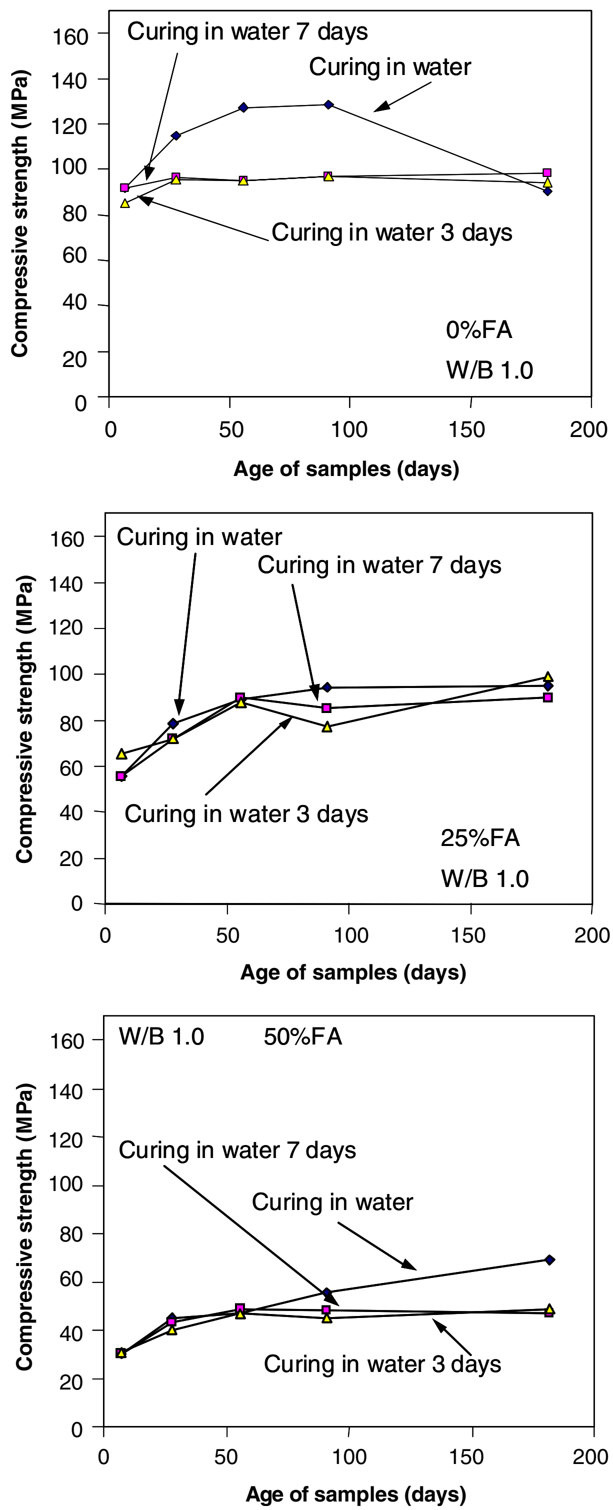

Fig. 2. The effect of curing condition on the compressive strength of fly ash cement paste.

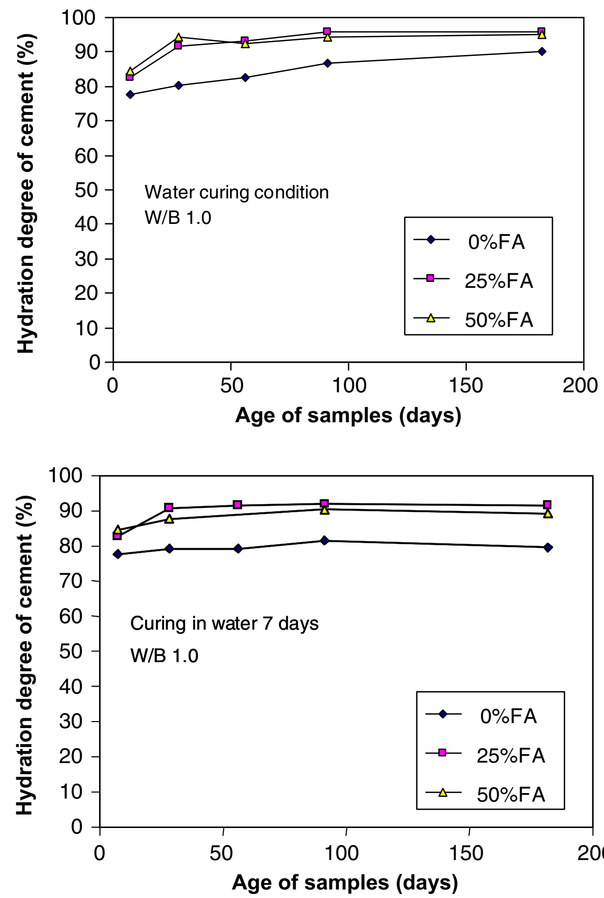

Fig. 3. The relation of hydration degree of cement and replacement ratio of fly ash as a function of time.

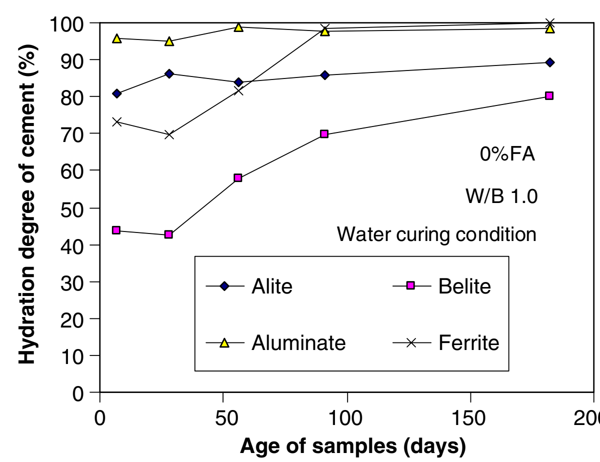

Fig. 4. Hydration degree of each component in cement.

pound and total amorphous material was measured and analyzed by XRD and Rietveld analysis, respectively.

2.2. Selective dissolution method

The determination of hydration degree of fly ash was based on selective dissolution method using HCl and Na_2CO_3 solution [5]. A pre-weighted centrifuge tube was filled with 1 g of sample and 30 cm^3 of HCl solution. Next, the centrifuge tube was placed in a 60 °C hot water bath and stirred for 15 min. Then, the liquid phase was extracted centrifugally from the paste using a centrifugal separator at 4000 rpm for 1 min. The liquid phase was decanted. Only the solid phase would be used in the next step.

After that, the solid phase in the centrifuge tube was filled with hot water, centrifuged again at 4000 rpm for 1 min and decanted. This step was repeated three times. Following this, the centrifuge tube was filled with 30 cm^3 of Na_2CO_3 solution, and placed in an 80 °C hot water bath for 20 min and stirred occasionally. Next, it was again centrifuged at 4000 rpm for 1 min. The supernatant was discarded. The residual was washed with water again three times. Next, the tube with the residue sample was dried at 110 °C and weighed.

The degree of hydration of fly ash can be calculated as follows:

$$\alpha = 1 - \left[\frac{x_s(1 - I_{s'})/(1 - I_s)}{a_1 a_2}\right] \quad (1)$$

$$a_1 = \frac{(1 - I_f) \times R}{(1 - I_f) \times R + (1 - I_o) \times (1 - R)} \quad (2)$$

$$a_2 = \frac{x_f(1 - I_{f'})}{1 - I_f} \quad (3)$$

In the above equations, α the degree of hydration of fly ash; R is the replacement ratio of fly ash by weight; x_s and x_f are the weights after the dissolution process of the hydrated sample and unhydrated fly ash, respectively; I_f, $I_{f'}$, I_o, I_s and $I_{s'}$ refer to the ignition loss of unhydrated fly ash, unhydrated fly ash after the dissolution process, unhydrated OPC, the hydrated sample and the hydrated sample after the dissolution process, respectively; a_1 is the initial fraction of fly ash before the dissolution process. In Eq. (1), $x_s(1 - I_{s'})/(1 - I_s)$ shows the ignition weight of unhydrated fly ash in a hydrated sample after the dissolution process and after calibration from bonding water. $a_1 a_2$ shows the ignition weight of unhydrated fly ash in an unhydrated sample.

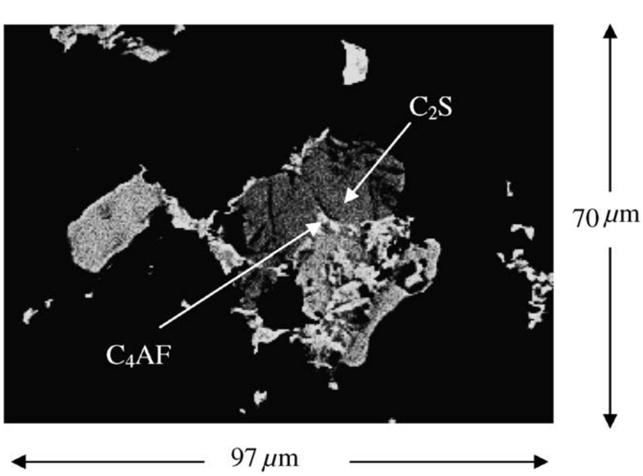

Fig. 5. BSE image analysis.

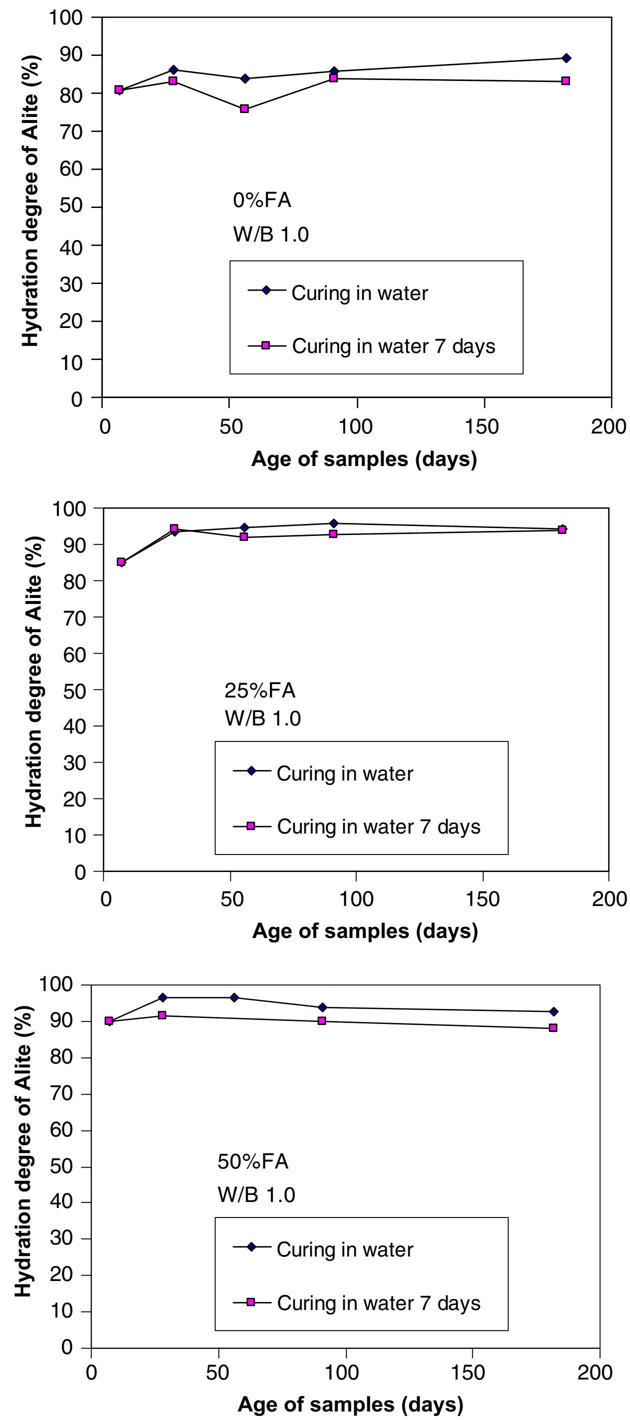

Fig. 6. The effect of curing condition on the hydration degree of alite.

2.3. X-ray diffraction method

The X-ray diffraction apparatus used in this study was Rikaku, CuK_{α}. The determine condition was 5–70° 2θ, 30 kV, 20 mA, 0.02 step scan and 1.00 s step speed. Ten percent of X-ray corundum (SRM 676) was used as internal standard. The XRD data was analyzed by SIROQUANT version 2.5 software.

3. Results

3.1. Compressive strength

The complete set of compressive strengths of the fly ash–cement pastes is shown in Table 3. To help illustrate the trends in strength development, some data in this table are plotted and compared as figures.

3.1.1. Compressive strength and replacement ratio of fly ash

The effects of replacement ratio of fly ash on the compressive strength of samples prepared with water curing condition and 7 days initial water curing condition are shown in Fig. 1(a) and (b), respectively. For the samples prepared with water curing condition, the compressive strength of 0% FA increased until 91 days but suddenly dropped at 182 days. The reason why compressive strength drops at 182 days is still unclear. The compressive strength of 25% FA and 50% FA continually increased until 182 days. At 182 days, the compressive strength of 25% FA was higher than that of the cement paste. For the samples prepared under 7 days initial curing condition, the compressive strength of 0% FA was almost constant. The compressive strength of 50% FA increased until 28 days and after that became nearly constant. In contrast, the compressive strength of 25% FA constantly increased until 56 days. At this age, its compressive strength became close to that of cement paste.

3.1.2. Compressive strength and curing condition

The effect of curing condition on the compressive strength of 0% FA, 25% FA and 50% FA are shown in Fig. 2(a)–(c), respectively. As for 0% FA, the compressive strength of samples cured in water is higher than others except at 182 days. In contrast, there is no significant difference among compressive strength of samples cured in water 7 days and 3 days. As for 25% FA and 50% FA, the effect of water curing condition is small, especially for 25% FA.

3.2. Hydration degree of cement

Fig. 3 shows the relations of the hydration degree of cement and the replacement ratio of fly ash as a function of time. This figure shows that the hydration degree of

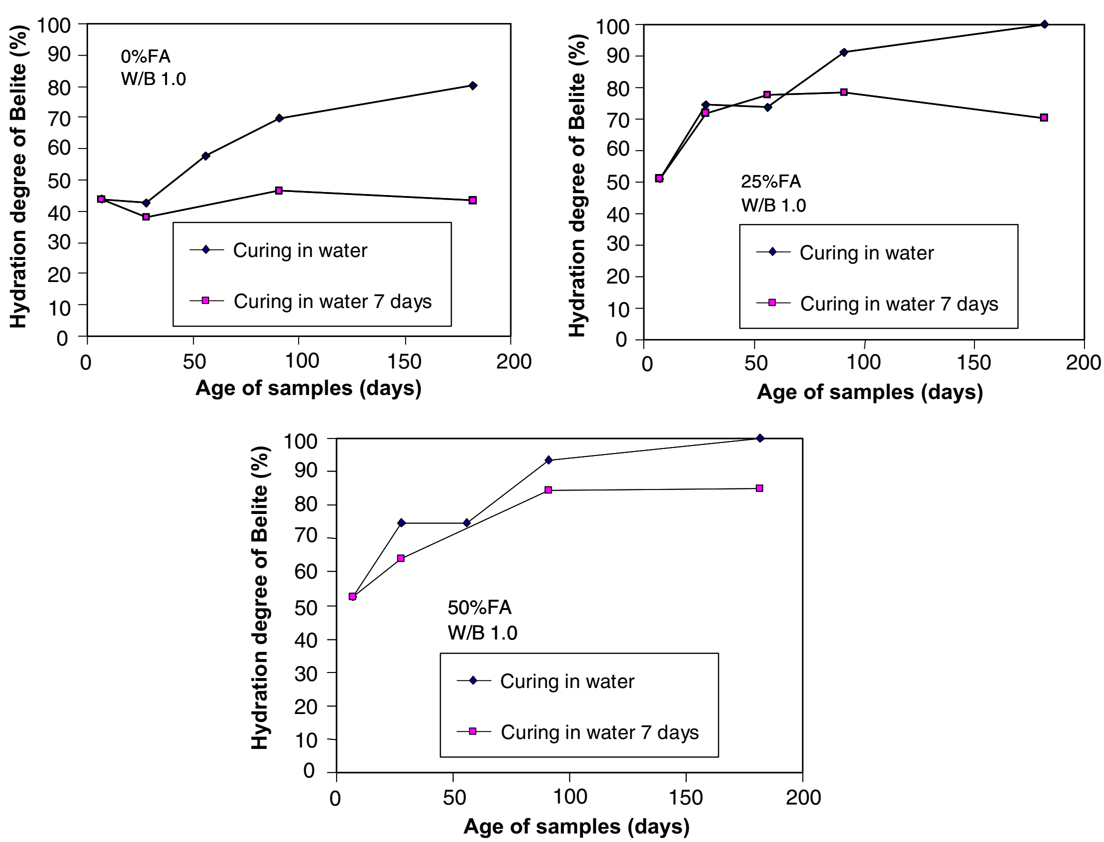

Fig. 7. The effect of curing condition on the hydration degree of belite.

cement in fly ash–cement paste is higher than that of cement paste. The hydration degree of cement is affected by a fly ash particle because of at least two possible reasons. First, although the water to binder ratio is constant, the water to cement ratio increases with the increase in the replacement ratio of fly ash. Another reason is that hydrated product may be accelerated to precipitate on the surface of fly ash, therefore results in the increase of hydration degree of cement.

3.3. Hydration degree of each component in cement

Fig. 4 shows the hydration degree of each component of cement. In Fig. 4, the hydration degree of alite and aluminate are rather constant while the hydration degree of belite gradually increases. The hydration degree of Ferrite is unexpectedly high. However, the BSE image analysis in Fig. 5 shows that even at 182 days, much of the ferrite in the hydrated samples still remained.

In the XRD analysis field, it is well known that a mass absorption of an atom can affect the quantitative analysis of phases using X-ray diffraction peaks. The mass absorption coefficient of Fe for the X-rays of CuK_α is considerably high; $\frac{\mu}{\rho} = 324$ in which μ is the linear absorption coefficient and ρ is density. However, the typical X-ray diffraction machine used in conventional laboratories is a CuK_α X-ray type. In this study, a CuK_α X-ray was also used. It is possible that the amount of Ferrite in this study might contain some error. Therefore, the amount of Ferrite would not be used in the analysis of this study.

It should be known that the hydration of each compound of cement extracted by the proposed method in this study might contain some little error. However, there is no other quantitative method that is capable of interpreting such kinds of data. Nevertheless, the result from the analysis in this study is quite adequate to show the tendency of hydration of each compound in cement, especially alite and belite.

3.4. Hydration degree of alite and belite and curing condition

Fig. 6 shows the effect of curing conditions on the hydration degree of alite. This figure shows that regardless replacement ratio of fly ash, hydration degree of alite does not significantly change when the curing conditions change.

Fig. 7 shows the effect of curing conditions on the hydration degree of belite. Hydration degree of belite is very much affected by curing conditions. Hydration degree of belite of the samples prepared with a water curing condition continues to increase while those prepared with a 7-day initial water curing condition are insignificantly changed. In fact, this phenomenon relates to hydration behavior of alite and belite.

Both alite and belite need water in hydration reaction. Alite reacts faster than belite and consumes a large amount of water at the beginning. It is quite likely that not much water is left for belite. Therefore, belite may need water from outside to react. This explains why hydration of belite is depended on water curing conditions.

In Fig. 7 the effect of curing conditions on hydration of belite becomes smaller as replacement ratio of fly ash increases. The amount of alite decreases as the replacement ratio of fly ash increases. When the amount of alite is not so much, the free water left inside sample may be enough for belite to react. Therefore, the dependent degree of hydration of belite on curing conditions should decrease as replacement ratio of fly ash increases.

3.5. Hydration degree of fly ash

The effect of curing conditions on the hydration degree of fly ash is shown in Fig. 8. Both 25% FA and 50% FA show that the hydration degree of fly ash gradually increases until 91 days. However, from age 91 days to 182 days, it increases rapidly. The hydration degree of sample prepared with the water curing condition is higher than others.

It is interesting to note that fly ash still continues to hydrate even without water supplied from the curing condition. In the pozzolanic reaction, fly ash reacts with

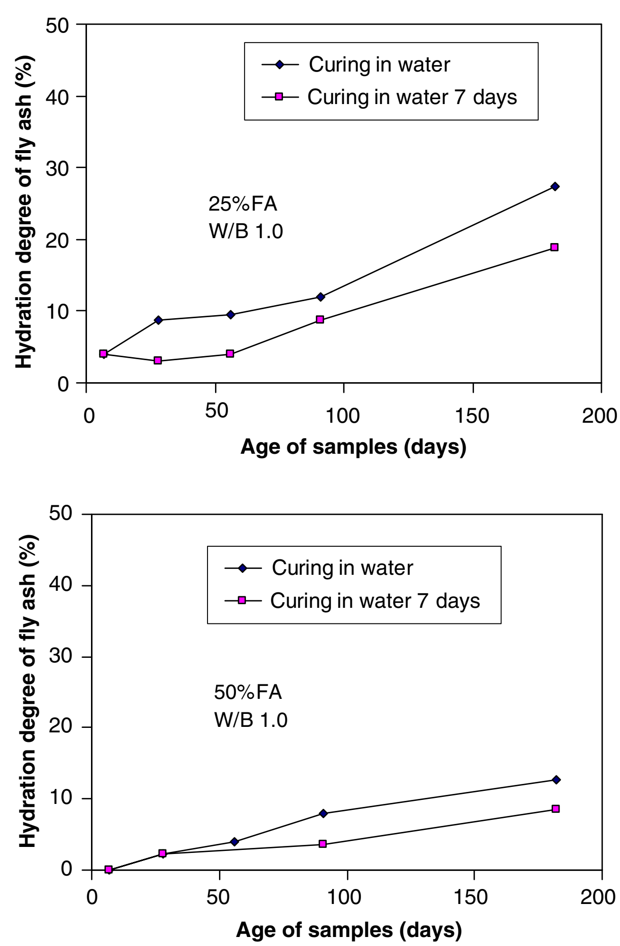

Fig. 8. The effect of curing condition on hydration degree of fly ash.

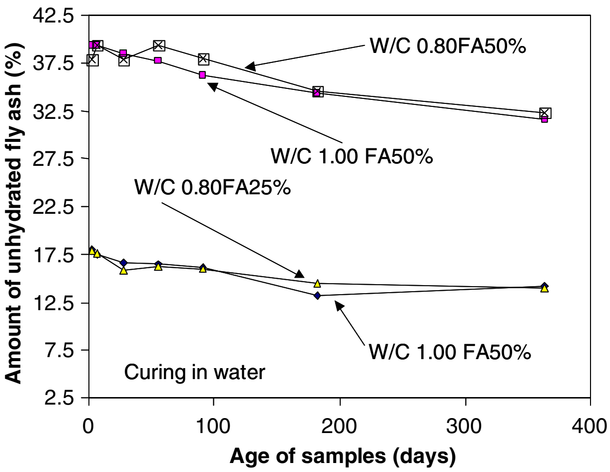

Fig. 9. The effect of water binder ratio on hydration degree of fly ash.

$Ca(OH)_2$ and small amount of water. In general, small amount of water is always left in hardening paste. This water may be enough for pozzolanic reaction. Therefore, the dependent degree of hydration reaction of fly ash on water curing condition should not be major. The corresponding results can be seen in Fig. 2. The compressive strength of fly ash–cement paste does not change so much though the water curing condition is changed.

The effect of water to binder ratio on the hydration degree of fly ash or amount of unhydrated fly ash is shown in Fig. 9. As seen in this figure, the amount of unhydrated fly ash is hardly affected by water to binder ratio.

4. Conclusions

This study investigates the effect of initial water curing conditions on the hydration degree and compressive strengths of fly ash–cement paste prepared with low water to binder ratio. This is the first attempt to explain the compressive strength by quantitatively differentiating the cementitious hydration reactions in binary cementitious systems. From the results, the following conclusions can be made:

1. The strong influence of curing conditions on fly ash–cement concrete is not due to changes in the hydration degree of fly ash, but rather is due to the effect of curing conditions on the hydration degree of cement, especially belite.
2. When water to binder ratio of fly ash–cement paste is 1.00 by volume, the effect of curing conditions on compressive strength is small.
3. Changing water to binder ratio from 1.00 to 0.80 by volume has little effect on hydration degree of fly ash.

These conclusions apply to both the 25% FA and 50% FA contents considered in this study. Further work in this area should consider potential size effects associated with drying of concrete specimens and the corresponding distribution of internal relative humidity.

References

[1] Helmuth RA. Fly ash in cement and concrete. Skokie, Ill.: Portland Cement Association; 1987.
[2] Malhotra VM, Mehta PK. Pozzolanic and cementitious materials (Advances in concrete technology). Taylor & Francis Group; 1996.
[3] Lam L, Wong YL, Poon CS. Degree of hydration and gel/space ratio of high-volume fly ash/cement systems. Cem Concr Res 2000;30(5): 747–56.
[4] Tokyay M. Effects of three Turkish fly ashes on the heat of hydration of PC–FA pastes. Cem Concr Res 1988;18:957–60.
[5] Termkhajornkit P, Nawa T, Nakai M, Saito T. Effect of fly ash on autogenous shrinkage. Cem Concr Res 2005;35(3):473–82.
[6] Termkhajornkit P, Nawa T. The fluidity of fly ash–cement paste containing naphthalene sulfonate superplasticizer. Cem Concr Res 2004;34(6):1017–24.
[7] Poon CS, Lam L, Wong YL. A study on high strength concrete prepared with large volumes of low calcium fly ash. Cem Concr Res 2000;30:447–55.
[8] Bouzoubaa N, Zhang MH, Malhotra VM. Laboratory-produced high-volume fly ash blended cements compressive strength and resistance to the chloride-ion penetration of concrete. Cem Concr Res 2000;30:1037–46.
[9] Jiang LH, Malhotra VM. Reduction in water demand of non-air entrained concrete incorporating large volumes of fly ash. Cem Concr Res 2000;30:1785–9.
[10] Bouzoubaa N, Lachemi M. Self-compacting concrete incorporating high volumes of class F fly ash—preliminary results. Cem Concr Res 2001;31:413–20.
[11] Ozer B, Ozkul MH. The influence of initial water curing on the strength development of ordinary Portland and pozzolanic cement concretes. Cem Concr Res 2004;34(1):13–8.
[12] Atis CD. Strength properties of high-volume fly ash roller compacted and workable concrete, and influence of curing condition. Cem Concr Res 2005;35:1112–21.
[13] Ramezanianpour AA, Malhotra VM. Effect of curing on the compressive strength, resistance to chloride-ion penetration and porosity of concretes incorporating slag, fly ash or silica fume. Cem Concr Compos 1995;17(2):125–33.
[14] Toutanji H, Delatte N, Aggoun S, Duval R, Danson A. Effect of supplementary cementitious materials on the compressive strength and durability of short-term cured concrete. Cem Concr Res 2004; 34(2):311–9.
[15] Cao C, Sun W, Qin H. The analysis on strength and fly ash effect of roller-compacted concrete with high volume fly ash. Cem Concr Res 2000;30(1):71–5.
[16] Freidin C. Hydration and strength development of binder based on high-calcium oil shale fly ash: Part II. Influence of curing conditions on long-term stability. Cem Concr Res 1999;29(11):1713–9.
[17] Khan MS, Ayers ME. Curing requirements of silica fume and fly ash mortars. Cem Concr Res 1993;23(6):1480–90.
[18] Haque MN, Goplan MK, Joshi RC, Ward MA. Strength development of inadequately cured high fly ash content and structural concretes. Cem Concr Res 1986;16(3):363–72.
[19] Zhang MH, Bilodeau A, Malhotra VM, Kim Kwang Soo, Kim Jin-Choon. Concrete incorporating supplementary cementing materials: effect of curing on compressive strength and resistance to chloride-ion penetration. ACI Mater J 1999;96(2):181–9.
[20] Scrivener KL, Pratt PL. Characterisation of Portland cement hydration by electron optical techniques. In: Electron microscopy of materials. Proc Mat Res Soc Symp, vol. 31, 1983. p. 351–6.
[21] Scrivener KL, Pratt PL. Backscattered electron images of polished cement sections in the scanning electron microscope. In: Proceedings

of the sixth international conference on cement microscopy, Albuquerque, 1984. p. 145–55.

[22] Igarashi S, Kawamura M, Watanabe A. Analysis of cement pastes and mortars by a combination of backscatter-based SEM image analysis and calculations based on the Powers model. Cem Concr Compos 2004;26(8):977–85.

[23] Moller H, Polysius AGK. Standardless quantitative phase analysis of Portland cement clinkers. World Cem 1995 (September):75–84.

[24] Taylor JC, Hinczak I, Matulis CE. Rietveld full-profile quantification of Portland cement clinker, The importance of including a full crystallography of the major phase polymorphs. Powder Diffr 2000; 15:7–18.

[25] Taylor JC, Aldridge LP. Full-profile quantitative XRD analysis of Portland cement powder diffraction, testing of possible standard XRD profiles for the major phase, tricalcium silicate. Powder Diffr 1993;8:138–44.

[26] Scrivener KL, Füllmann T, Gallucci E, Walenta G, Bermejo E. Quantitative study of Portland cement hydration by X-ray diffraction/Rietveld analysis and independent methods. Cem Concr Res 2004;34(9):1541–7.

[27] Termkhajornkit P, Nawa T, Kurumisawa K. Quantitative investigation on fly ash and Portland cement hydration. Cem Concr Res, submitted for publication.

[28] Japanese Architectural Standard Specification JASS15 M-103.