- Technical Paper -

AN INVESTIGATION ON CORRELATION BETWEEN FLY ASH REACTION AND CEMENTING EFFICIENCY FACTOR FOR STRENGTH DEVELOPMENT OF CONCRETE

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ABSTRACT

This paper aims at investigating the contribution of fly ash to strength development of concrete by evaluating the relationship between cementing efficiency factor *k* (*k*-value) and degree of fly ash reaction. The compressive strength of concretes was examined and the degree of fly ash reaction in paste specimens was measured by selective dissolution method at the ages of 7, 28 and 91 days. The results showed that the *k*-value of fly ash increased proportionally with an increase in the degree of fly ash reaction for each mixture proportion.

Keywords: *k*-value of fly ash, degree of fly ash reaction, strength development of fly-ash concrete

1. INTRODUCTION

Although there are many studies [1−6] on the efficiency of fly ash as a supplementary cementitious material, it is rather difficult to determine *k*-value of fly ash in concrete in a most accurate way. The *k*-value of fly ash was introduced firstly by I.A. Smith [1]. In his work, a constant *k*-value of 0.25 was drawn from 7-day and 28-day compressive strengths of fly-ash concrete. It means only 25% of fly ash, in this case, can be considered as cement in contribution to strength development of concrete up to 28 days. Nevertheless, it was found that the *k*-value of fly ash tends to decrease with an increase in water-to-cement ratio [2] but increases with age [4]. Most of the proposed methods for determining the *k*-value are based on compressive strength obtained experimentally. However, it is necessary to clarify the contribution of fly ash to strength development of concrete under various conditions at the same time such as water-to-binder ratio, type and replacement ratio of fly ash.

On the other hand, there are a lot of studies [7−12] which investigated degree of fly ash reaction in fly-ash cement system when fly ash was used to replace a part of cement in concrete industry. Nevertheless, the contribution of fly ash to the strength development due to the fly ash reaction have not been evaluated sufficiently and/or quantitatively. Therefore, there were two purposes of this study:

(1) To evaluate the effects of fineness of fly ash and water-to-binder ratio on the contribution of fly ash to strength development of concrete.

(2) To clarify the correlation between the degree of fly ash reaction and the *k*-value of fly ash for strength development of concrete.

2. TEST PROGRAMS

2.1 Materials

Ordinary Portland cement (OPC) and two low-calcium type II fly ashes were used as cementitious materials. These cement and fly ashes met JIS R 5210 and JIS A 6201, respectively. The chemical compositions and physical properties of cementitious materials are shown in Table 1. It should be noted that the fly ash named FA-H has higher Blaine fineness of 4790 cm^2/g than FA-M (3503 cm^2/g). Besides, crushed quartz porphyry was used as fine and coarse aggregates. The densities of these aggregates in saturated surface-dry conditions were 2.61 and 2.62 g/cm^3, respectively. In addition, admixtures including water reducing/superplasticizer and air-entraining agent were used to adjust the slump and air content of fresh concrete.

Table 1 Chemical compositions and physical properties of cement and fly ashes

	Fly ash		Cement
Notation	FA-H	FA-M	OPC
CaO (%)	4.17	3.12	65.05
SiO_2 (%)	53.32	53.53	20.29
Al_2O_3 (%)	28.27	28.11	4.91
Fe_2O_3 (%)	4.62	5.17	2.96
MgO (%)	2.05	1.21	1.20
K_2O (%)	0.84	0.9	0.39
Na_2O (%)	0.54	0.79	0.26
SO_3 (%)	0.21	0.63	1.93
Loss on ignition (%)	2.64	2.08	2.44
Density (g/cm^3)	2.31	2.37	3.16
Blaine fineness (cm^2/g)	4790	3503	3290

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Table 2 Mixture proportions and properties of fresh concrete

Group	Mixture	W/(C+F) (wt. %)	F/(C+F) (wt. %)	Unit content (kg/m^3)					Properties of fresh concrete		
				W	C	F	S	G	Temp. (ºC)	Slump (cm)	Air (%)
Control	C30	30	0	170	567	0	653	932	18.6	11.0	5.2
	C40	40	0	170	425	0	770	932	14.3	7.0	5.2
	C50	50	0	170	340	0	840	932	17.5	11.5	5.3
	C60	60	0	170	283	0	887	932	13.7	8.5	5.2
FA-H	50H20	50	20	170	272	68	819	932	19.3	10.5	4.7
W/B	50H30	50	30	170	238	102	809	932	26.8	18.5	5.0
0.50	50H40	50	40	170	204	136	798	932	27.1	11.0	5.1
FA-M	50M20	50	20	170	272	68	821	932	21.6	11.0	4.7
W/B	50M30	50	30	170	238	102	812	932	16.9	12.5	5.4
0.50	50M40	50	40	170	204	136	802	932	16.9	13.0	4.8
FA-M	30M20	30	20	170	453	113	621	932	23.6	9.5	5.1
W/B	30M30	30	30	170	397	170	606	932	23.7	13.0	4.7
0.30	30M40	30	40	170	340	227	590	932	23.8	12.0	4.8

W/(C+F) = W/B is water-to-binder (cement + fly ash) ratio by mass, F/(C+F) is replacement ratio of fly ash by mass, W is water, C is cement, F is fly ash, S is fine aggregate (crushed sand) and G is coarse aggregate (crushed stone)

2.2 Mixture proportions

Two water-to-binder ratios (W/B) of 0.50 and 0.30 (only for FA-M) and four replacement ratios of fly ash; 0, 20, 30 and 40% by mass were adopted to make concrete as shown in Table 2. Moreover, control mixtures with water-to-cement ratios of 0.40 and 0.60 were also prepared to obtain a relationship between cement-to-water ratio (C/W) and compressive strength of concrete without fly ash for evaluation of the equivalent cement-to-water ratio ($(C/W)_{eq.}$) and the cementing efficiency factor experimentally.

It is noticed that the masses of water and gravel were kept constant at 170 and 932 (kg/m^3) respectively in all mix proportions of concrete. Regarding properties of fresh concrete, slump and air content were measured in accordance with JIS A 1116 and JIS A 1128, respectively. The targets of slump and air content in this present work were set at 10.0 ± 2.0 (cm) and 5.0 ± 0.5 (%), respectively. In addition, temperature of fresh concrete was also recorded immediately after mixing as shown in Table 2.

To examine the degree of fly ash reaction, paste specimen was exactly prepared with the same W/B ratio, kind and replacement ratio of fly ash as used in the concrete test but no admixtures.

2.3 Specimen preparation

Preparations of materials were carried out at 20 ºC. Concretes were mixed in laboratory by using a mechanical mixer. Concrete specimens used for the compressive strength test were cast into twenty-four cylindrical molds of 100-mm diameter and 200-mm height, while paste specimens used for examining the degree of fly ash reaction in paste were cast into polypropylene bottles of 16 mL. All specimens including concrete and paste, were cured under a sealed condition and stored at 20 ºC until the designated test ages of 7, 28 and 91 days.

Moreover, samples used for examining the degree of fly ash reaction were obtained by cutting the hardened pastes and selecting material in the size of 2.5–5.0 mm range. To stop further hydration, the cutting samples were soaked in acetone and placed in a vacuum under a pressure of 0.1 MPa for 30 min. This operation was repeated 3 times with changing acetone. Then, the samples were dried in a vacuum desiccator for 3 days. After that the samples were ground into a powder of less than 150 µm before measuring the degree of fly ash reaction in paste specimen using selective dissolution method (SDM).

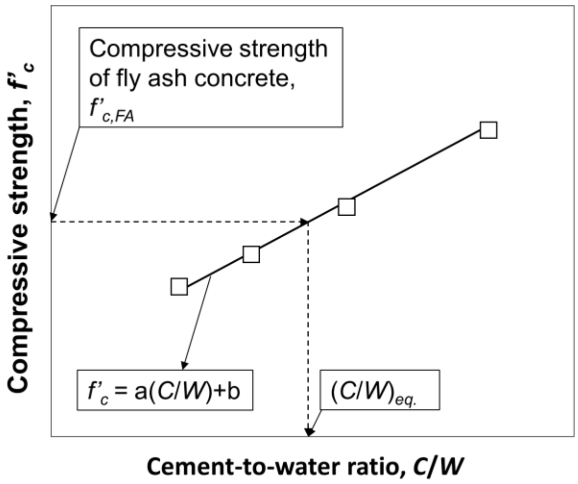

Fig.1 Principle of calculating the equivalent cement-to-water ratio $(C/W)_{eq.}$

2.4 Compressive strength of concrete and evaluation of k-value of fly ash

The test for compressive strength was conducted at the ages of 7, 28 and 91 days according to JIS A 1108 (Method of test for compressive strength of concrete). Three specimens of each mixture were tested, and the mean value of these measurements was reported.

As illustrated in Fig. 1, the equivalent cement-to-water ratio, $(C/W)_{eq.}$, [1] was determined by substituting the value of compressive strength of fly-ash concrete into the relationship between the compressive strength of control concrete and cement-to-water ratio at each age.

The k-value of fly ash can be calculated using the following Eq. (2) which is derived from Eq. (1).

$$(C/W)_{eq.} = (C + kF)/W \quad (1)$$

$$k = \left\{ \frac{(C/W)_{eq.}}{(C/W)} - 1 \right\} \times \left(\frac{1-r}{r} \right) \quad (2)$$

where,
$(C/W)_{eq.}$ is the equivalent cement-to-water ratio,
C, F, and W are unit contents of cement, fly ash, and water (kg/m^3),
k is the k-value of fly ash,
r is replacement ratio of fly ash (i.e. $F/(C+F)$).

2.5 Degree of fly ash reaction in paste specimens

Ohsawa et al. [8] proposed a method using 2 M hydrochloric acid and 5% sodium carbonate aqueous solution to investigate the degree of fly ash reaction which is calculated using the following Eq. (3):

$$\alpha = 1 - \frac{X(1 - Ig')/k_2}{(1 - Ig)k_1} \quad (3)$$

where,
α is degree of fly ash reaction (fraction);
k_1 is original fraction of fly ash in ignited base (fraction);
k_2 is residue extracted of a 1-g fly ash (fraction);
X is residue extracted of the hydrated fly ash in a 1-g hydrated sample (fraction);
Ig is loss on ignition of the hydrated sample (fraction);
Ig' is loss on ignition of the residue extracted (fraction).

It is noted that the amounts of Ig and Ig' were calculated from the mass losses between 105 and 1000 ºC by TG-DTA. More details about this testing could be also found in the study by Termkhajornkit et al. [9].

3. RESULTS AND DISCUSSION

3.1 Compressive strength of concrete and evaluation of k-value of fly ash

The compressive strength of concrete was measured at the designated test ages as summarized in Table 3. As the same tendency as general, the compressive strength of concrete increased with time as well as the decrease in the W/B ratio. Addition of fly ash to replace a part of cement resulted in reduction of compressive strength of fly-ash concrete at the early ages as compared to the control mixture. However, the compressive strength of fly-ash concrete increased considerably at the age of 91 days and even became higher than that of the control i.e. C50 for two mixtures of 50H20 and 50M20. To evaluate the k-value of fly ash as mentioned above, relationship between the compressive strength of control concrete and cement-to-water ratio was obtained as shown in Fig. 2.

The k-values for each fly ash replacement ratio were also summarized in Table 3 as well as illustrated obviously in Fig. 3. As shown in Fig. 3(a), although k-value of 20% fly ash in FA-H concrete with W/B ratio of 0.50 i.e. 50H20 inhibited a lowest value of 0.26 at 7 days, it showed the highest value at the ages of 28 and 91 days as compared to the others. In detail, it reached approximately 1.2 at 91 days. With the same W/B ratio of 0.50, the k-value of 20% fly ash in FA-M concrete i.e. 50M20 increased significantly after 7 days and was also larger than 1 at 91 days. In this case, it can be said that the contribution of FA-H or FA-M to strength development of concrete with the high W/B ratio of 0.50 was more significant than that of cement at the later age. On the other hand, it can be seen the k-value of 20% fly ash in FA-M concrete with the low W/B ratio of 0.30 i.e. 30M20 was the highest value of 0.55 at 7 days but became the lowest one of 0.74 at 91 days. This might be explained that the contribution of fly ash to strength development of concrete varied with time due to both effects which are physical effect of fly ash at the early ages and a pozzolanic reaction at the later ages for concrete with low W/B ratio of 0.30.

Table 3 Compressive strengths of concrete and cementing efficiency factors of fly ash

Group	Mixture	F/(C+F) (wt. %)	Compressive strength (N/mm^2) at			k-value of fly ash at		
			7 days	28 days	91 days	7 days	28 days	91 days
Control	C30	0	61.6 ± 1.3	71.9 ± 2.7	79.6 ± 2.9	–	–	–
	C40	0	43.5 ± 0.4	51.9 ± 0.7	55.9 ± 0.6	–	–	–
	C50	0	33.8 ± 0.5	40.7 ± 0.4	44.5 ± 1.0	–	–	–
	C60	0	28.1 ± 1.4	33.7 ± 0.7	37.8 ± 0.2	–	–	–
FA-H W/B 0.50	50H20	20	28.2 ± 0.7	39.3 ± 1.0	47.1 ± 1.5	0.26	0.86	1.23
	50H30	30	24.7 ± 0.4	35.2 ± 1.1	43.8 ± 0.7	0.22	0.61	0.94
	50H40	40	17.9 ± 0.5	27.6 ± 0.5	35.7 ±1.1	-0.01	0.29	0.55
FA-M W/B 0.50	50M20	20	29.2 ± 0.3	36.8 ± 0.1	45.5 ± 1.5	0.38	0.55	1.05
	50M30	30	22.2 ± 0.4	31.5 ± 0.8	39.1 ± 1.6	0.01	0.32	0.61
	50M40	40	18.8 ± 0.3	28.5 ± 0.7	36.4 ± 0.6	0.05	0.32	0.58
FA-M W/B 0.30	30M20	20	55.1 ± 1.3	66.1 ± 1.7	74.2 ± 0.7	0.55	0.64	0.74
	30M30	30	49.0 ± 0.5	63.5 ± 0.6	72.3 ± 0.8	0.40	0.65	0.75
	30M40	40	41.0 ± 1.9	55.8 ± 0.7	65.5 ± 0.3	0.25	0.49	0.61

For 30% fly ash replacement ratio, the effect of W/B ratio on the contribution of fly ash to strength development of concrete can be seen obviously through comparing both 50M30 and 30M30 as shown in Fig. 3(b). The lower the W/B ratio, the higher the *k*-value of fly ash, especially at the early ages. Besides, the *k*-value of fly ash in FA-H concrete (50H30) was always higher than that in FA-M one (50M30) at the same W/B ratio of 0.50. It can be said that the *k*-value increased with an increase in the Blaine fineness of fly ash in the case of fly ash replacement ratio of 30%. Furthermore, the *k*-value of 50H30 showed the highest value of 0.94 at the age of 91 days, meanwhile that of 30M30 became the maximum one at the early ages i.e. 7 and 28 days. From the results as shown in Table 3, it can be also said that the fly ash used to replace a part of cement with replacement ratio of 30% contributed most significantly to strength development of FA-M concrete with the low W/B ratio of 0.30, especially at the early age due to the physical effect of fly ash.

As illustrated in Fig. 3(c), all *k*-values of fly ash replacement ratio of 40% were almost the same at 91 days. They were 0.55, 0.58 and 0.61 for mixtures of 50H40, 50M40 and 30M40, respectively. Effect of W/B ratio on the *k*-value was slightly significant only at the early ages whereas the fineness effect of fly ash became less in this case. It can be said that the effect of high Blaine fineness of fly ash on the *k*-value varied with the replacement ratio of cement with fly ash. In other words, the *k*-value of fly ash in the case of 40% fly ash replacement ratio might be independent on the Blaine fineness of fly ash. Nevertheless, the *k*-value of 30M40 always showed the highest value. This means that the addition of fly ash in concrete with the low W/B ratio of 0.30 resulted in better contribution of fly ash to strength development of concrete especially at the early ages due to mainly both effects of space filling and hydration enhancement.

As mentioned before, Smith [1] suggested a constant *k*-value of 0.25 for fly ash whereas Bijen and Van Selst [2] found that the *k*-value tends to decrease as water-to-cement ratio increases. Moreover, it was found by Babu and Rao [4] that the *k*-value of fly ash increases with age. Thus, the *k*-value of fly ash cannot be a constant value, and it varied with time as well as fly ash replacement levels as demonstrated in this study. These results are consistent with the results in some studies [2-4]. In addition, the effects of fineness of fly ash and W/B ratio on the contribution of fly ash to strength development of concrete were obviously significant and varied with replacement ratio of fly ash. Especially, at the early ages the contribution of fly ash to strength development of concrete at W/B ratio of 0.30 was more considerable than that at W/B ratio of 0.50 due to physical effect of fly ash including space filling and hydration enhancement effects, but it became less significant at 91 days. It can be explained in more detail in the next section because the effect of fly ash reaction contributed considerably to strength development of concrete at the later ages and increased with an increase in the W/B ratio.

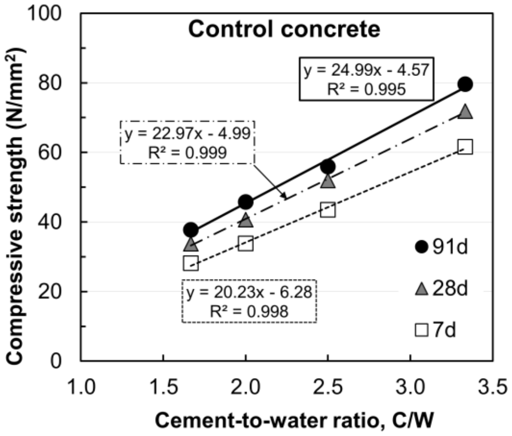

Fig.2 Compressive strength of control concrete

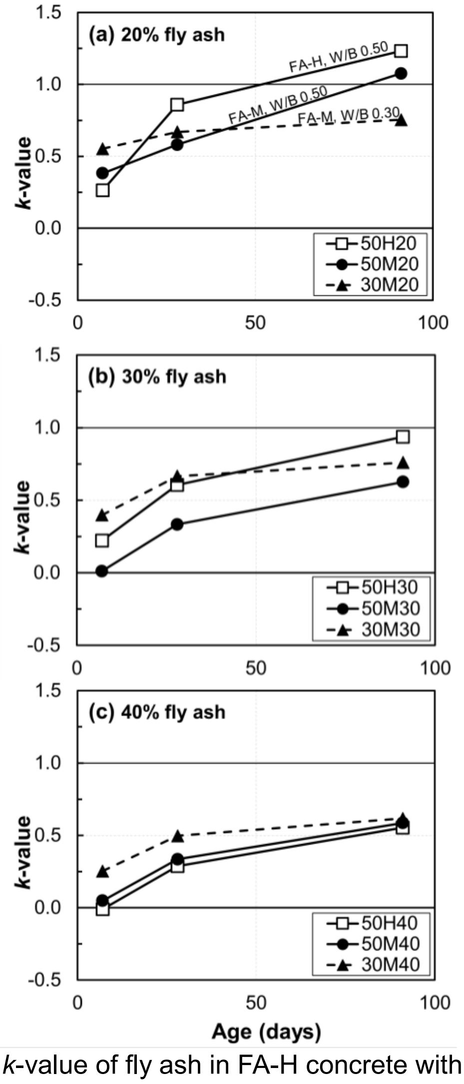

Fig.3 *k*-value of fly ash in FA-H concrete with W/B ratio of 0.50 (square) and in FA-M concrete with W/B ratios of 0.50 (circle) and 0.30 (triangle) for (a) 20%, (b) 30% and (c) 40% replacement ratios of fly ash

3.2 Degree of fly ash reaction in paste specimens

Fig. 4 shows the experimental results of the degree of fly ash reaction in paste specimens at the ages of 7, 28 and 91 days. Each value presented in Fig. 4 is a mean experimental value of two measurements (n=2).

As the same tendency as general, the degree of fly ash reaction increased with time and became high significantly at 28 days regardless of fineness of fly ash as well as W/B ratio. However, the higher the Blaine fineness of fly ash, the more the fly ash reacted especially at the early ages. Furthermore, the lower the replacement ratio of fly ash, the higher the degree of fly ash reaction at all ages. This might be due to the higher concentration of Ca^{2+} ions for the fly ash reaction in the pore solutions of the pastes with the lower replacement ratio of fly ash [7]. In addition, the degree of fly ash reaction increased considerably with an increase in the W/B ratio especially after 28 days. In detail, the degrees of fly ash reaction in the FA-M paste with the W/B ratio of 0.50 were small and almost the same as those of 0.30 at 7 and 28 days. However, at 91 days, the degree of fly ash reaction in FA-M paste with W/B ratio of 0.50 increased significantly and was higher than that of 0.30 as compared with the results in Figs. 4(b) and 4(c). In the case of the low W/B ratio of 0.30 and a high fly ash replacement ratio of 40%, the degree of fly ash reaction increased significantly at 28 days then increased slightly at 91 days. These results are consistent with the results in some studies [9−11].

The effect of the fineness of fly ash on the degree of fly ash reaction can be compared in Figs. 4(a) and 4(b). It can be seen that the degree of fly ash reaction in the FA-H paste was higher than that in the FA-M one, especially at the early ages. This might be explained by the fact that the fly ash with a higher Blaine fineness reacted more effectively and contributed significantly to strength development of concrete due to pozzolanic reaction. However, it should be noted that the degree of fly ash reaction in FA-H paste was equal to or slightly higher than that in FA-M paste at 91 days.

Briefly, at the early ages, the degree of fly ash reaction showed a small value and it was involved mainly in chemical reaction forming ettringite as demonstrated in the study by Berry et al. [12]. Nevertheless, it increased significantly from 7 to 91 days independently of fineness of fly ash as well as W/B ratio. Moreover, it can be concluded that the higher the ratio of W/B, the more the degree of fly ash reaction, especially at the later ages whereas the higher the Blaine fineness of fly ash, the higher the degree of fly ash reaction at the early ages. These results seem not to be agreed well with the results of *k*-value which showed the highest contribution of fly ash for 30% fly ash replacement ratio to strength development of concrete with a lower W/B ratio of 0.30. Furthermore, the *k*-value of 50H40 was smaller than that of 50M40 although the degree of fly ash reaction in 50H40 paste was higher than that in 50M40 one. It means the *k*-value of fly ash might depend not only on the degree of fly ash reaction but also on the physical effect of fly ash including space filling and hydration enhancement effects.

3.3 Relationship between the *k*-value and the degree of fly ash reaction

In this study, the degree of fly ash reaction was used to evaluate the contribution of fly ash to strength development of concrete in a chemical approach as shown in Fig. 5. On the whole, the *k*-value increases with the increase of the degree of fly ash reaction. It means that the fly ash reaction contributed to the strength development with time.

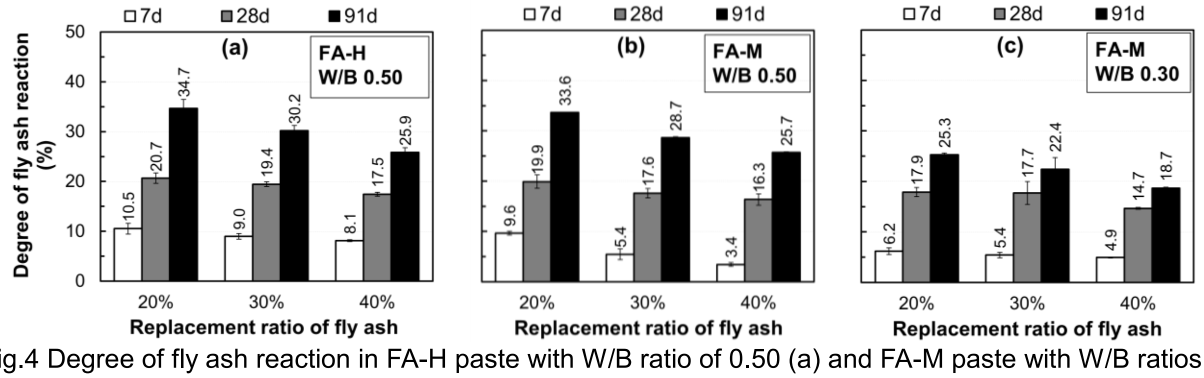

Fig.4 Degree of fly ash reaction in FA-H paste with W/B ratio of 0.50 (a) and FA-M paste with W/B ratios of 0.50 (b) and 0.30 (c)

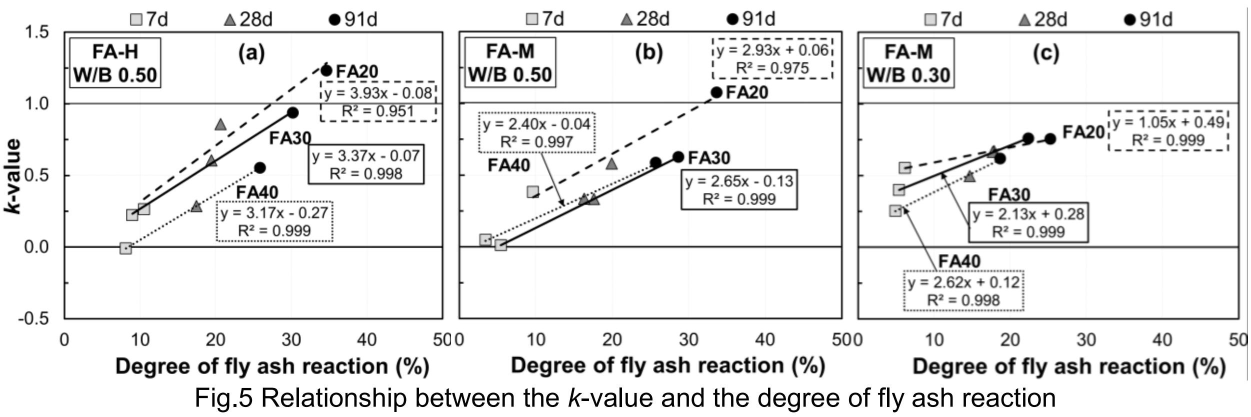

Fig.5 Relationship between the *k*-value and the degree of fly ash reaction

For each replacement ratio of fly ash, the relationship between the *k*-value and the degree of fly ash reaction in FA-H paste with W/B ratio of 0.50 (a) and in FA-M paste with W/B ratios of 0.50 (b) and 0.30 (c) can be obtained with a very high correlation coefficient R^2. From these linear correlations, the effect of W/B ratio on the *k*-value of fly ash was also observed due to the different slope of these relationships. The lower the W/B ratio, the smaller the slope of these relationships especially obviously for the case of fly ash replacement ratio of 20% i.e. FA20 (dash line). This can be implied that the *k*-value of fly ash might depend not only on the degree of fly ash reaction but also on the physical effect of fly ash including space filling and hydration enhancement especially at the early ages.

In the case of FA-H concrete with a W/B ratio of 0.50 as shown in Fig. 5(a), the higher the degree of fly ash reaction, the higher the *k*-value, especially for two lower fly ash replacement ratios of 20% and 30%. Meanwhile, in the case of FA-M concrete with the same W/B ratio of 0.50 as shown in Fig. 5(b), only the *k*-value of 20% fly ash replacement ratio became high significantly as compared to the others. Combining Figs. 5(a) and 5(b), it can be said that the fly ash replacement ratio of 40% seems to have no obvious effect of the fineness on the *k*-values. It implies that the fly ash having a higher Blaine fineness might have higher contribution to strength development of concrete only for two lower fly ash replacement ratios of 20% and 30% within the limits of the present work.

As illustrated in Fig. 5(c), the *k*-value of fly ash in FA-M concrete at a lower W/B ratio of 0.30 was high significantly at the early ages despite of a low degree of fly ash reaction as compared with that at a higher W/B ratio of 0.50. It is presumed that the physical effect of fly ash including space filling and hydration enhancement contributed significantly to strength development of concrete with a lower W/B ratio at the early ages. Moreover, the efficiency of space filling due to fly ash reaction product is also remarkable in paste at W/B ratio of 0.30 having a low initial capillary porosity. Briefly, the addition of fly ash in high-strength concrete resulted in the better contribution of fly ash to strength development of concrete due to mainly both effects of space filling and hydration enhancement at the early ages.

4. CONCLUSIONS

The following conclusions can be drawn within the limits of the present experiments:

(1) With a very high correlation coefficient, a relationship between the *k*-value of fly ash and the degree of fly ash reaction for each fly ash replacement ratio can be obtained to evaluate the contribution of fly ash to strength development of concrete in a chemical approach in each mixture proportion.
(2) However, the *k*-value of fly ash might depend not only on the degree of fly ash reaction but also on the physical effect of fly ash.
(3) According to the relationships between *k*-value and degree of fly ash reaction, the effects of space filling and hydration enhancement of fly ash on the *k*-value might increase with a decrease in the W/B ratio, especially at the early ages but not increase with time. Meanwhile, the degree of fly ash reaction increased not only with time but also with an increase in the W/B ratio.

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