

## CONTRIBUTION OF FLY ASH TO MORTAR STRENGTH DEVELOPMENT UNDER STEAM AND INTERNAL CURING

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### ABSTRACT.

The purpose of this study is to quantitatively evaluate the effects of steam curing and internal curing on contribution of fly ash to strength development of mortar by using cementing efficiency factor (k-value) that represents strength development performance as a binder of fly ash. In addition, the pozzolanic reaction of fly ash was evaluated from the viewpoint of calcium hydroxide consumption by using thermogravimetry and differential thermal analysis as well as the degree of fly ash reaction by using selective dissolution method. The result indicated that steam curing improved early compressive strength and internal curing improved compressive strength and k-value at all ages. Also, a linear relationship between the degree of fly ash reaction and the k-value was shown regardless of the age and the replacement ratio of fly ash.

KEYWORDS: Cementing efficiency factor (k-value), fly ash, internal curing, steam curing, strength development of mortar.

## 1. INTRODUCTION

Fly ash, a by-product of coal-fired power generation, has been commonly utilized as a supplementary cementitious material in concrete to obtain advantages such as long-term strength enhancement and densification of the microstructure. Although coal-fired power plant slowly has been shut down in western Europe concerning the environmental impact and the fly ash is decreasing, a certain amount of fly ash production is still produced and desired to be utilized. However, there is a concern that the early strength decreases due to replacing a part of cement with fly ash, and most of fly ash is currently used as a cement raw material in Japan [1].

Steam curing is one of the useful methods for improving the early strength of fly ash concrete, and some properties of steam-cured fly ash concretes have been investigated. Yazici et al. reported that when steam curing was performed, the long-term strength of fly ash concrete was low compared with sealed curing at 20 °C [2]. Meanwhile, pozzolanic reaction of fly ash could continue when the steam-cured fly ash concrete was cured in water after steam curing [3].

On the other hand, there is an internal curing method as a long-term water supply method. Internal curing promotes hydration of cement by supplying

water from the inside by using internal curing materials with high water absorption. Water-absorbing polymer and artificial lightweight aggregate are used as internal curing materials [4, 5]. In addition to these two conventional internal curing materials, roof-tile waste aggregate has been also investigated as an internal curing material. Roof-tile is Japanese traditional roofing material and sintered clay at high temperature. Roof-tile waste aggregate is produced from the roof-tile in its production due to cracking while sintering at high temperature. Sekishu Kawara, one of the Japanese traditional roof tiles, was firstly used as an internal curing material by Suzuki et al. [6], and it was reported that the compressive strength of ultra-high strength concrete was improved and the autogenous shrinkage was reduced by partially replacing the coarse aggregate with the roof-tile waste. It was also reported that the strength of steam cured fly ash concrete was improved by using internal curing material [7]. However, the effects of steam curing as well as internal curing on the reaction of fly ash have not been clearly understood.

The purpose of this study is to evaluate the effects of steam curing and internal curing on contribution of fly ash to strength development of mortar by using cementing efficiency factor (k-value). Furthermore,

	Cement	Fly ash
CaO (%)	65.09	3.59
SiO <sub>2</sub> (%)	20.43	56.74
Al <sub>2</sub> O <sub>3</sub> (%)	4.95	28.06
Fe <sub>2</sub> O <sub>3</sub> (%)	2.52	5.75
MgO (%)	1.62	0.92
Na <sub>2</sub> O (%)	0.25	0.79
SO <sub>3</sub> (%)	2.86	1.07
Loss on ignition (%)	0.76	2.08
Density (g/cm <sup>3</sup> )	3.14	2.23
Blaine fineness (cm <sup>2</sup> /g)	4560	3530

TABLE 1. Chemical compositions and physical properties of cement and fly ash.

	Crushed sand	Roof-tile waste fine aggregate
Density (g/cm <sup>3</sup> )	2.58	2.28
Water absorption (%)	2.05	9.75

TABLE 2. Density and water absorption of crushed sand and roof-tile waste fine aggregate.

	Unit content (kg/m <sup>3</sup> )				
	Water	Cement	Fly ash	Crushed sand	Roof-tile waste fine aggregate
F0S0	278	696	0	1290	0
F20S0	269	537	134	1290	0
F40S0	260	389	260	1290	0
F0S20	278	696	0	1032	228
F20S20	269	537	134	1032	228
F40S20	260	389	260	1032	228

TABLE 3. Mixture proportion.

the pozzolanic reaction of fly ash was assessed, and the relationship between the fly ash reaction and the k-value of fly ash was discussed.

## 2. EXPERIMENTAL METHOD

### 2.1. MATERIALS, MIXTURE PROPORTION AND CURING METHODS

Paste and mortar specimens were prepared in this study. High-early-strength Portland cement and type II fly ash were used as cementitious materials. These cement and fly ash met JIS R 5210 and JIS A 6201, respectively. The chemical compositions and physical properties of cement and fly ash are shown in Table 1. The water to binder ratio was 40% by mass, and fine aggregate to mortar ratio was 50% by volume. Roof-tile used in this study was derived from the Sekishu Kawara, which composed of approximately 70% SiO<sub>2</sub>, and 20% Al<sub>2</sub>O<sub>3</sub>. The density in saturated and surface-dry condition and the water absorption of crushed sand and roof-tile waste fine aggregate are shown in Table 2. The fly ash replacement ratios were 0% (F0), 20% (F20), and 40% (F40) by mass. The roof-tile waste fine aggregate was used for mortar specimens as an internal curing material by replacing crushed sand at 0% (S0) and 20% (S20) by volume.

The mixture proportions are shown in Table 3.

Two curing methods were applied in this study. One was sealed curing at 20 °C at all ages (20 °C sealed curing), while the other was steam curing at a maximum temperature of 50 °C for 6 hours after casting. The steam-cured specimens were demolded at the age of 1 day and stored at 20 °C and 60% relative humidity.

### 2.2. COMPRESSIVE STRENGTH AND K-VALUE

Mortar specimens, which were cast into cylindrical molds of 50-mm diameter and 100-mm height, were used for the compressive strength test. The compressive strength test was conducted according to JIS A 1108 at the ages of 1, 7, and 28 days. The equivalent cement-to-water ratio ( $(C/W)_{eq}$ ) of the fly-ash mortar was calculated from the compressive strength and the relationship between the cement-to-water ratio ( $C/W$ ) and the compressive strength of mortar without fly ash. The k-value was obtained from the  $(C/W)_{eq}$ , the  $C/W$  and the replacement ratio of fly ash ( $r$ ) as shown in Eq. 1 [8].

$$k = \left[ \frac{(C/W)_{eq}}{C/W} - 1 \right] \cdot \frac{1-r}{r} \quad (1)$$

**2.3. CALCIUM HYDROXIDE (CH) CONTENT AND ITS CONSUMPTION PER UNIT FLY ASH CONTENT**

Paste specimens cast into 16-mL polypropylene bottles were used for examining the CH content by thermogravimetry and differential thermal analysis. The CH content in the sample was measured at the ages of 1, 7, and 28 days. Specimens were cut and samples in the size of 2.5 – 5.0 mm range were obtained. The samples were soaked in acetone to stop hydration and dried in a vacuum desiccator for 24 hours. After that the samples were ground into a powder less than 150 μm before the measurements of the degree of fly ash reaction and the CH content.

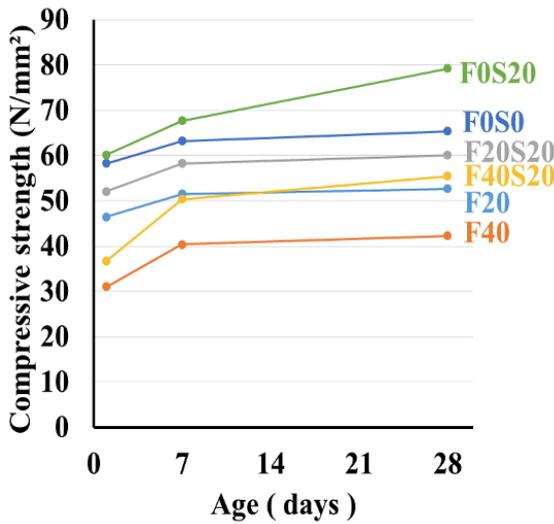


FIGURE 1. Compressive strength of mortar under steam curing.

The CH consumption per unit fly ash content can be calculated using the following Eq. 2.

$$CH_{consumption} = \frac{CH(1 - r) - CH_{FA}}{r} \quad (2)$$

- $CH_{consumption}$ : CH consumption per unit fly ash content (%)
- $CH$ : CH content in paste without fly ash (%)
- $CH_{FA}$ : CH content in paste with fly ash (%)

**2.4. DEGREE OF FLY ASH REACTION**

The sample used for measuring the degree of fly ash reaction was prepared similarly to that for CH content. The degree of fly ash reaction in paste specimens was measured at 1, 7, and 28 days by using selective dissolution method (SDM). To evaluate the reaction ratio of fly ash, Ohsawa et al. proposed a method using 2-M hydrochloric acid (HCl) and 5% sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) aqueous solution [9]. The procedure of SDM is as follows:

A 1-g powder sample was put in a pre-weighed centrifuge tube and weighed precisely, and 30 mL of 2-M HCl solution was added in the tube. Then, the tube

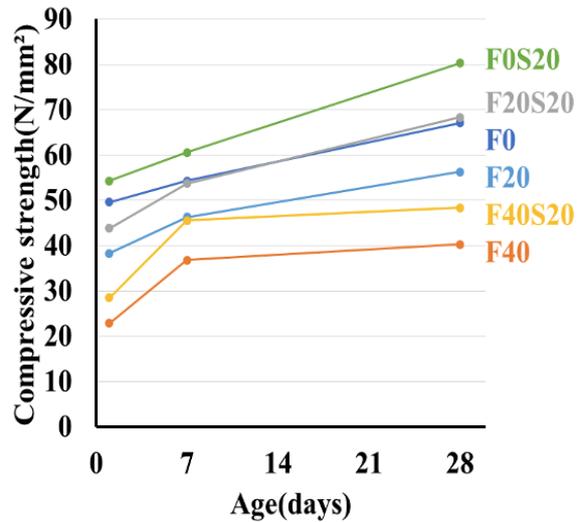


FIGURE 2. Compressive strength of mortar under 20 °C-sealed curing.

was placed in a water bath at 60 °C for 15 min to promote the reaction. The solution was stirred occasionally with a glass rod during this step. The tube was removed from the bath and centrifuged at 4000 rpm (revolutions per minute) for 30 sec to separate solid and liquid phases.

The liquid phase was decanted as much as possible without disturbing the solid residue in the bottom. After that, the tube was filled with hot water, centrifuged again and decanted. This operation was repeated 3 times. The tube was then filled with 30 mL of Na<sub>2</sub>CO<sub>3</sub> solution, placed in a water bath at 80 °C for 20 min and stirred continuously.

The tube was then centrifuged at 4000 rpm for 1 min. The liquid phase was decanted and the residue was washed with hot water and centrifuged. This washing was repeated 3 times. The tube with residue was dried at 105 °C for 24 h and then weighed.

The degree of fly ash reaction is calculated using the following Eq. 3.

$$\alpha = 1 - \frac{X(1 - Ig)}{(1 - Ig)k1k2} \quad (3)$$

- $\alpha$  : degree of fly ash reaction
- $k1$  : original fraction of fly ash in ignited base
- $k2$  : residue extracted of a 1-g fly ash
- $X$  : residue extracted of the hydrated fly ash in a 1-g hydrated sample
- $Ig$  : loss on ignition of the hydrated sample
- $Ig'$  : loss on ignition of the residue

**3. RESULTS AND DISCUSSION**

**3.1. COMPRESSIVE STRENGTH**

The compressive strengths of mortar specimens under steam curing and 20 °C-sealed curing are presented in

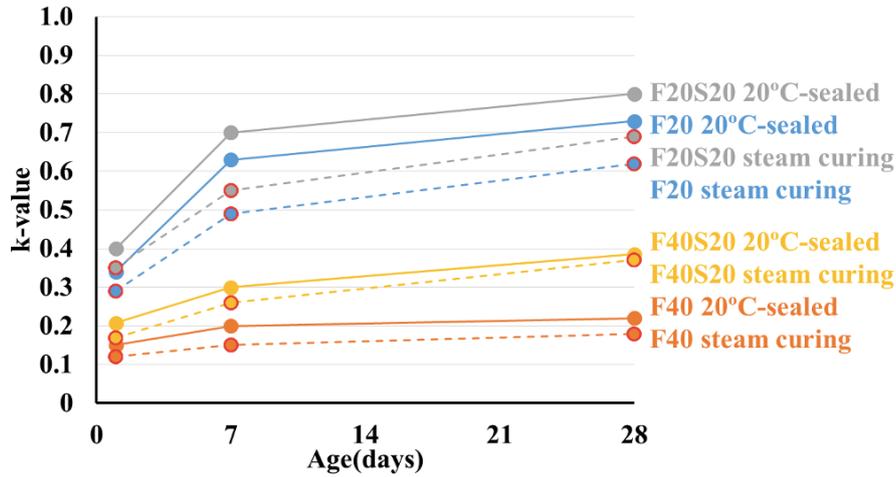


FIGURE 3. k-value of fly ash with age.

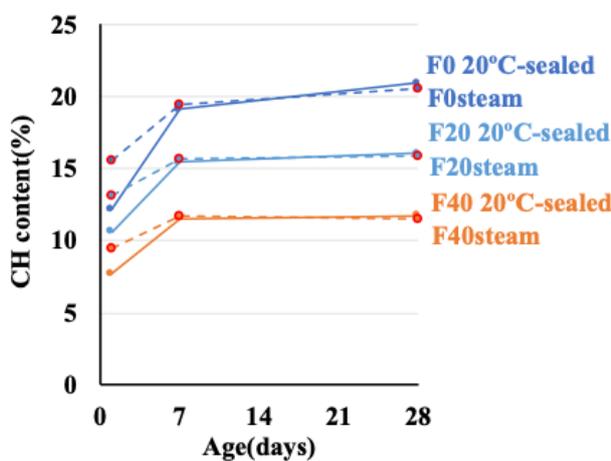


FIGURE 4. CH content with age.

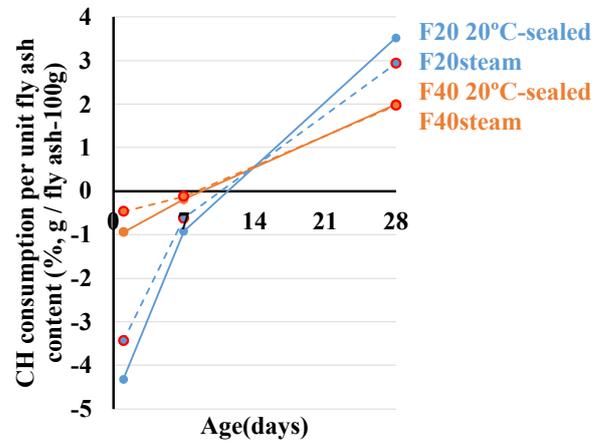


FIGURE 5. CH consumption per unit fly ash content with age.

Figures 1 and 2, respectively. At the age of 1 day, the compressive strength of steam-cured mortar was 10% to 35% higher than that of 20 °C-sealed cured mortar. The roof-tile waste fine aggregate replacement increased the compressive strength of mortar by 15% to 20% at all ages, regardless of curing methods. At the age of 28 days, the mortar specimens with 20% fly ash and 20% roof-tile waste fine aggregate (F20S20) had the same compressive strength as that with 0% fly ash and 0% roof-tile waste fine aggregate (F0) in the case of 20 °C-sealed curing. Meanwhile, the compressive strength of F20S20 was lower than that of F0 in the case of steam curing. This could indicate that steam curing mainly promoted the initial hydration of cement and slightly promoted the pozzolanic reaction of fly ash which can occur at later ages, while internal curing by roof-tile waste fine aggregate could enhance the cement and fly ash reaction at early ages as well as the later ages.

### 3.2. CEMENTING EFFICIENCY FACTOR (K-VALUE)

Figure 3 shows the k-value of fly ash under steam curing and 20 °C-sealed curing with time. The steam curing had a lower k-value than the 20 °C-sealed curing for all mixture proportions. This result also suggests that steam curing mainly promoted the hydration of cement. In addition, the k-value in the case of 20% roof-tile waste replacement (S20) was higher than that in the case of no roof-tile waste. It indicated that the internal curing by using the roof-tile waste fine aggregate promoted the fly ash reaction, and the fly ash contributed to the strength development of mortar. The F40S20 in both curing conditions had a significant increase in the k-value from 7 to 28 days when compared with the F40. This shows that the internal curing continuously promoted the pozzolanic reaction of fly ash for a long term when the replacement ratio of fly ash was high.

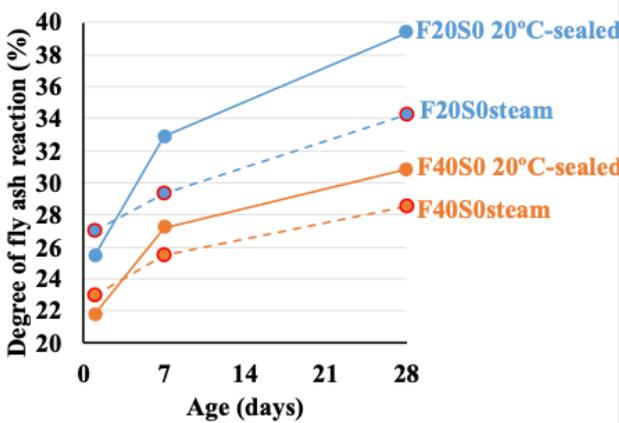


FIGURE 6. Degree of fly ash reaction with age.

### 3.3. CALCIUM HYDROXIDE (CH) CONTENT AND ITS CONSUMPTION PER UNIT FLY ASH CONTENT

Figure 4 shows the CH content under 20 °C-sealed curing and steam curing. At the age of 1 day, the CH content of steam-cured specimens was higher than that of 20 °C-sealed cured specimens. It could imply that steam curing accelerated cement hydration and CH was produced more than that under 20 °C-sealed curing. At the age of 7 days, then, the CH content for each mixture proportion was almost the same, and increased up to the age of 28 days slightly.

Figure 5 shows CH consumption per unit fly ash content by fly ash reaction under 20 °C-sealed curing and steam curing. At the age of 28 days, the CH consumption per unit fly ash content in the case of F20 with 20 °C-sealed curing was greater than that in the case of F20 with steam curing. Besides, the unit CH consumption at the ages of 1 day and 7 days was negative. It may indicate that the cement hydration could be promoted by the incorporation of fly ash and more CH was produced in fly ash cement paste compared to the plain cement paste.

### 3.4. DEGREE OF FLY ASH REACTION

Figure 6 shows the degree of fly ash reaction under steam curing and 20 °C-sealed curing. At all ages, the degree of fly ash reaction in the case of 20 °C-sealed curing was higher than that in the case of steam curing. The longer the material age, the greater the difference between steam curing and 20 °C-sealed curing. It could imply that drying after the end of the steam-supply hindered the fly ash reaction.

Figure 7 shows the relationship between the k-value and the degree of fly ash reaction. The k-value increases as the degree of fly ash reaction increases. The relationship between the degree of fly ash reaction and the k-value can be represented by one straight line regardless of the age and the replacement ratio of fly ash.

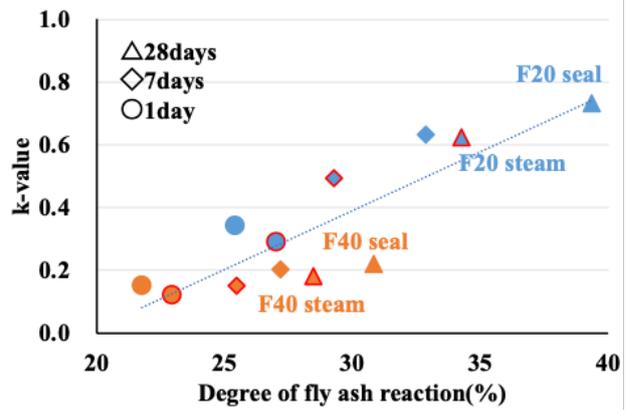


FIGURE 7. Relationship between k-value and degree of fly ash reaction.

## 4. CONCLUSIONS

The present study investigated the k-value of fly ash regarding compressive strength development, CH consumption as well as the degree of fly ash reaction in order to evaluate the effects of steam curing and internal curing on contribution of fly ash to strength development of mortar. The following conclusions can be drawn within the limits of this study.

Steam curing improved early compressive strength of mortar regardless of the fly ash or roof-tile replacement.

The 20% roof-tile waste fine aggregate replacement increased the compressive strength by 15% to 20% for both 20 °C-sealed curing and steam curing. The internal curing could contribute not only to the cement hydration but also to the fly ash reaction.

The k-value in the case of 40% fly ash replacement for both steam and 20 °C-sealed curing conditions increased from 7 to 28 days by using roof-tile waste fine aggregate. It was suggested that when the replacement ratio of fly ash is high, the effect of internal curing on the pozzolanic reaction may continue for a long time.

The k-value in the case of steam curing was lower than that in the case of the 20 °C-sealed curing, and it implies that steam curing significantly promoted the cement hydration whereas it slightly promoted the pozzolanic reaction of fly ash. The relationship between the degree of fly ash reaction and the k-value can be represented by one straight line regardless of the age and the replacement ratio of fly ash.

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