

1 Title:

2 Effects of porous ceramic roof tile waste aggregate on strength development and carbonation
3 resistance of steam-cured fly ash concrete
4

5 Authors:

6 Yuko Ogawa (corresponding author)

7 ogaway@hiroshima-u.ac.jp

8 Department of Civil and Environmental Engineering, Hiroshima University

9 1-4-1, Kagamiyama, Higashi-Hiroshima city, Hiroshima 739-8527, Japan
10

11 Phuong Trinh Bui

12 buiphuongtrinh@hcmut.edu.vn

13 Department of Construction Materials, Ho Chi Minh City University of Technology, VNU-HCM

14 268 Ly Thuong Kiet, Ho Chi Minh city, Vietnam
15

16 Kenji Kawai

17 kkawai@hiroshima-u.ac.jp

18 Department of Civil and Environmental Engineering, Hiroshima University

19 1-4-1, Kagamiyama, Higashi-Hiroshima city, Hiroshima 739-8527, Japan
20

21 Ryoichi Sato

22 sator@hiroshima-u.ac.jp

23 Hiroshima University

24 1-4-1, Kagamiyama, Higashi-Hiroshima city, Hiroshima 739-8527, Japan

Abstract: (95/100 words)

This study aims to investigate the effects of porous ceramic waste coarse aggregate (PCWA), which was used as an internal curing agent, on the compressive strength, shrinkage, and carbonation resistance of steam-cured concrete using fly ash (FA) at the replacement ratios of 0%, 20%, and 40% by mass. The PCWA replacement ratios amounted to 0%, 10%, and 20% of the coarse aggregate volume. The results showed that the PCWA significantly improved compressive strength and carbonation resistance of concrete using 40% FA, whereas it could increase the drying shrinkage of the steam-cured concrete using 40% FA.

Keywords: Roof tile waste, internal curing, fly ash, steam-cured concrete, carbonation resistance

1 Introduction

Fly ash (FA) is a by-product in coal fired power plants, and its utilization is essential to decrease the environmental impact of the cement concrete industry and conserve resources. Therefore, FA has been widely used in construction as a mineral admixture for concrete, a mineral filler for asphalt concrete, a sub-base for pavement layers, and for similar applications. In the construction field, FA is well-known as a useful supplementary cementitious material [1]. The application of FA as a substitute for Portland cement can not only reduce the CO₂ emissions from cement production but also enhance the properties of concrete, such as workability, long-term strength, and chemical resistance. However, concrete including low-calcium FA as a substitute for Portland cement has lower early-age strength and slower strength development than that without FA owing to the slow FA reaction. This lower early-age strength can prevent the wider application of FA as a cementitious material. To increase its applications, the utilization of FA in pretensioned prestressed concrete has been investigated [2–5]. The manufacture process of pretensioned prestressed concrete

generally involves steam curing to obtain the required early-age strength for prestressing, and the steam-cured concrete members are demolded and exposed to the air at an earlier age than those cured at the normal temperature. Lothenbach et al. reported that concrete cured at the higher temperature has lower long-term strength because of its higher porosity due to the denser inner C-S-H and the decrease in the ettringite content at higher temperature [6]. It was also reported that, compared with the standard curing (i.e., curing at 20 or 27 °C), steam curing causes concrete to become more porous, resulting in lower durability and higher sorptivity [4, 7, 8]. Some researchers showed that the supply of water/moisture to the demolded concrete after steam curing could effectively improve concrete strength [9]. Meanwhile, Zou et al. reported that the performance of steam-cured concrete, including permeability and compressive strength, decreased due to steam curing followed by water curing [10]. It is debatable whether supplying water to concrete after steam curing is effective at enhancing the qualities of concrete.

Internal curing was devised as a method for reducing the autogenous shrinkage in high-performance concrete. According to Lura [11], the internal curing effect obtained using saturated porous aggregate was first presented in 1967 by Aroni and Polivka, and the method has been developed as a new technique that supplies water from the internal curing agent after mixing. Later, the ACI Committee 308 defined “internal curing” as the process by which the hydration of cement occurs because of the availability of additional internal water that is not part of the mixing water [12]. Lightweight aggregates and superabsorbent polymers are well-known as internal curing agents that reduce early-age shrinkage. Philleo was the first to propose the use of lightweight fine aggregate as an internal curing agent [13]. The internal curing effect of lightweight aggregate has been investigated [14–17], and Nie et al. indicated recently that internal curing with lightweight aggregate improved the performance of heat-cured concrete [9]. Jensen and Hansen were the first to propose a superabsorbent polymer as an internal curing agent [18], and later, many researchers

investigated the use of such polymers as internal curing agents in concrete [19–21]. Besides these two materials, one natural and the other artificial, some by-products or recycled materials such as bottom ash and recycled aggregate have been investigated as internal curing agents [22–24]. Moreover, a waste aggregate derived from roof tiles was reported as an effective internal curing material [25]. In the north part of Chugoku district in Japan, it is estimated that approximately 10,000 tons of roof tiles, called *Sekisyu Kawara*, were discarded as waste in 2015 alone owing to thermal cracking during the cooling process [26]. To obtain a roof tile with the high freezing and thawing resistance required in the cold weather region, *Sekisyu Kawara* is produced by sintering clay at high temperatures exceeding 1200 °C, higher than the temperature applied in the production of other roof tiles that are produced for use in warm regions in Japan. Suzuki et al. studied the effects of *Sekisyu Kawara*, which has relatively high water absorption and relatively low crushing values, as an internal curing agent, and found that the roof tile waste can develop the compressive strength and reduce the autogenous shrinkage of high-performance concrete with a very low water-to-binder ratio of 0.15 [25, 27]. Sato et al. used roof tile waste as the internal curing agent in slag concrete, and reported that this slag concrete had higher compressive strength and denser structure than that without the roof tile waste as the internal curing agent [28]. It has also been reported that internal curing using roof tile waste can effectively increase the compressive strength of high-volume FA concrete cured at 20 °C [29]. Moreover, the properties of steam-cured FA concrete including porous ceramic roof tile waste were investigated in some earlier works [30–32]. It was reported that addition of roof tile waste at a replacement ratio of 10% increased the compressive strength and carbonation resistance of steam-cured FA concrete [30, 32], whereas that of 20% significantly decreased the autogenous shrinkage in steam-cured FA concrete [31]. However, these results have not been discussed sufficiently yet.

The purpose of this study is to investigate the effect of roof tile waste as an internal curing agent on

the quality of steam-cured FA concrete by providing additional data and furthering the discussions of the previous literature [30-32]. In this study, pre-saturated roof tile waste replaced 10% or 20% of the coarse aggregate volume in steam-cured high strength FA concrete, and its effects on the compressive strength, shrinkage, and carbonation resistance of the concrete were investigated.

2 Experimental procedures

2.1 Materials

High-early-strength Portland cement meeting the standard values of the Japanese Industrial Standards JIS R 5210 (Portland cement) [33] was used in this study. Low-calcium FA meeting the standard values of Type II as per JIS A 6201 (Fly ash for use in concrete) [34] was also used as cementitious material. The chemical composition and physical properties of these cementitious materials are listed in Table 1.

Table 1 Chemical composition and physical properties of cementitious materials used in this work

	High-early-strength Portland cement	Low-calcium FA
Notation	C	F
SiO ₂ (%)	20.15	59.20
Al ₂ O ₃ (%)	4.80	23.87
Fe ₂ O ₃ (%)	2.71	5.43
CaO (%)	65.32	2.38
MgO (%)	1.15	1.01
SO ₃ (%)	3.08	0.27
Na ₂ O (%)	0.16	0.30

K ₂ O (%)	0.40	0.87
Loss on ignition (%)	1.37	2.7
Density (g/cm ³)	3.14	2.33
Blaine fineness (cm ² /g)	4490	3200

Crushed quartz-porphry was used as the conventional fine and coarse aggregates in the concrete. These fine and coarse aggregates met the standard values for manufactured sand and crushed stone as per JIS A 5005 (Crushed stone and manufactured sand for concrete) [35]. Porous ceramic waste aggregate (PCWA) derived from crushed roof tile waste was also used as an internal curing agent, partially replacing the coarse aggregate. PCWA was used in a saturated surface-dry condition after 7 d of water immersion. Table 2 lists the physical properties of these aggregates. The crushing values of the coarse aggregates based on BS 812-110 (Testing aggregates. Methods for determination of aggregate crushing value) [36] are also listed in Table 2. The crushing value of the PCWA is approximately twice as high as that of the conventional coarse aggregate.

Table 2 Physical properties of aggregates

	Crushed quartz-porphry		Porous ceramic aggregate
	for fine aggregate	for coarse aggregate	for coarse aggregate
Notation	S	G	PCWA
Density (g/cm ³)	2.60	2.62	2.26
Water absorption (%)	1.16	0.62	8.70
Crushing value (%)	Not measured	11.7	20.6

2.2 Mixture proportions

Eight mixtures were prepared in this study, as presented in Table 3. The water-to-cementitious materials ratio (W/C+F) and the water content were fixed at 0.30 and 165 kg/m³, respectively. The replacement ratios of cement with FA were 0%, 20%, and 40% by mass, and those of coarse aggregate with PCWA were 0% (G0) and 10% (G10) by volume. For the concretes including 20 mass% and 40 mass% of FA in the cementitious materials, 20 vol.% (G20) of the coarse aggregate was also replaced with PCWA. The grading of the PCWA itself as well as the mixed coarse aggregates are presented in Fig. 1. The range limits of the 5–20 mm coarse aggregate defined as “Crushed stone 2005 for concrete” as per JIS A 5005 [35] are also shown in Fig. 1. Although the PCWA was finer than the reference coarse aggregate (G0), the differences in grading among G0, G10, and G20 were negligible within the low replacement ratios. A superplasticizer and an air-entraining agent were also used to obtain the same slump of 20.0 ± 2.0 cm and air content of 4.5 ± 1.0%. The slump, air content, and temperature of the concrete, measured just after mixing, are also listed in Table 3.

Table 3 Mixture proportions of concrete and measured properties of fresh concrete

	Unit content (kg/m ³)						Properties of fresh concrete		
	W	C	F	S	G	PCWA	Slump (cm)	Air content (%)	Temperature (°C)
HF0-G0	165	550	0	751	854	0	19.0	3.9	21.0
HF0-G10	165	550	0	751	780	74	20.0	3.9	19.0
HF20-G0	165	440	110	714	854	0	18.5	3.5	19.0
HF20-G10	165	440	110	714	780	74	20.0	3.8	20.0
HF20-G20	165	440	110	714	684	147	21.3	4.0	20.5

HF40-G0	165	330	220	677	854	0	20.0	3.8	19.0
HF40-G10	165	330	220	677	780	74	21.5	3.5	18.5
HF40-G20	165	330	220	677	684	147	21.3	4.2	20.1

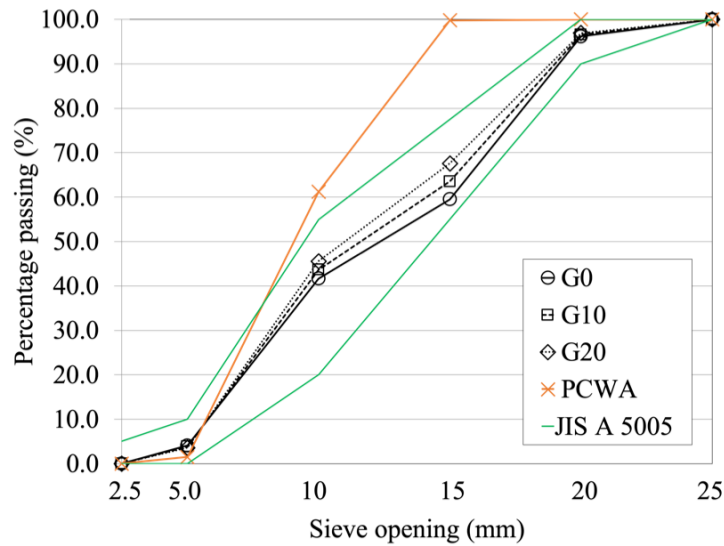


Figure 1 Gradings of coarse aggregates

2.3 Curing conditions

All the concrete specimens were cured under a steam curing condition after casting. One of the general steam curing conditions, as described in Fig. 2, was applied. The temperature was raised to 50 °C at a rate of 15 °C/h after initial curing for 3 h at 20 °C and 60% relative humidity (RH). Thereafter, it was maintained at the maximum temperature of 50 °C for 6 h. The chamber was cooled down to 20 °C at a rate of 5 °C/h and then maintained at 20 °C for 7 h. Then, the specimens were demolded 24 h after casting and stored at 20 ± 2 °C and 60 ± 2% RH. The specimens were steam-cured from the age of 3 to 11 h during the time when the temperature was raised and maintained at the maximum.

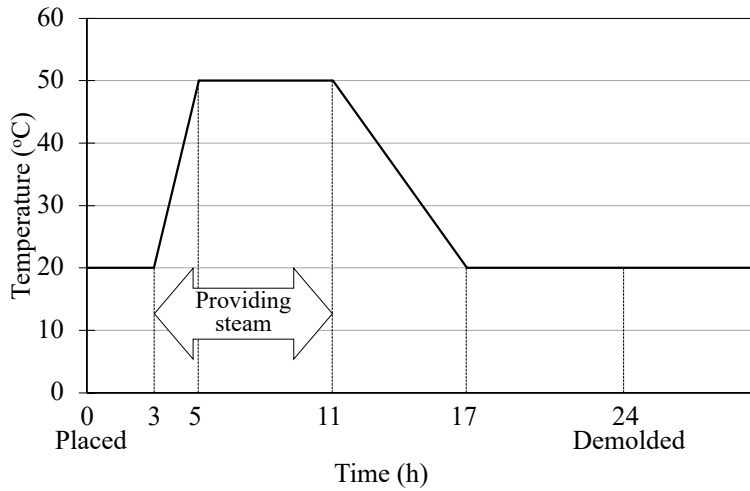


Figure 2 Temperature history of steam curing

2.4 Specimens and testing procedure

2.4.1 Compressive strength

Compressive strength tests were conducted at the ages of 1, 7, 28, 56, 91, 182, and 364 d in accordance with JIS A 1108 (Method of test for compressive strength of concrete) [37]. Three cylindrical specimens, each measuring 100 mm in diameter and 200 mm in height, were used for each condition.

2.4.2 Length change

A prism specimen, 100 mm in height, 100 mm in width, and 400 mm in length, was prepared to measure the variation of length in the concrete. The change in length was measured with an embedded gauge (KM-100BT, Tokyo Measuring Instruments Lab., Japan) located at the center of the prismatic specimens immediately after placing. To evaluate the length change in the concrete due to autogenous shrinkage and drying shrinkage, the temperature in the specimen was also measured with a thermocouple included in the gauge to obtain the thermal strain from the measured

free strain. Similar to the measurements of the compressive strength of the specimens, these specimens were also steam-cured and demolded at the age of 1 d. Then, they were stored at 20 °C and 60% RH.

2.4.3 Accelerated carbonation

Three prism specimens, each measuring 100 mm in height, 100 mm in width, and 400 mm in length, were prepared for the accelerated carbonation test for each mixture. These specimens were cured under the aforementioned conditions until the age of 91 d, and were then stored at 20 ± 2 °C, $60 \pm 5\%$ RH, and $5.0 \pm 0.2\%$ CO₂. The age of 91 d was selected because FA concrete would have been sufficiently cured within this time. All the surfaces, except for one of the 100 × 400 mm sides, which was not the top or bottom surface at the casting stage, were coated with epoxy resin. Only the uncoated 100 × 400 mm side was exposed to the accelerated carbonation condition. The carbonation depths at six positions for each specimen were measured using the phenolphthalein method at the accelerated carbonation periods of 1, 4, 8, 13, and 26 weeks.

2.4.4 Pore size distribution

The pore size distributions of six mixtures, namely HF0-G0, HF0-G10, HF20-G0, HF20-G10, HF40-G0, and HF40-G10, were measured at the age of 364 d using a mercury intrusion porosimeter (POREMASTER 60, Quantachrome Instruments, USA). The samples for the test, each ranging from 2.5 to 5.0 mm in size, were obtained after the compressive strength test was completed. These samples were soaked in acetone to stop further hydration, and then dried in a vacuum desiccator before the test. The pore size distribution was measured from 3 nm to 300 μm.

3 Results and discussion

3.1 Compressive strength

The compressive strengths of all the mixtures are shown in Fig. 3 (a), and the standard errors ranged from 0.2 to 4.6 N/mm². In addition, the compressive strengths normalized by the strength of each mixture without PCWA are shown in Fig. 3 (b). Although the compressive strength decreased with the increase in FA fraction, the values for HF20-G0, HF20-G10, and HF20-G20 at the age of 364 d were almost the same as that of HF0-G0 (see Fig. 3 (a)). It implies that when FA was used with the low replacement ratio of 20%, it may have reacted well and contributed to the development of the same compressive strength as that of the concrete using only high-early-strength Portland cement in the long term. It can be found that the compressive strengths of concrete using PCWA were equal to or higher than those without PCWA in all the mixtures and for all the ages except for HF20-G10 at the age of 1 d. This indicates that the internal curing using PCWA is likely to have developed the compressive strength of concrete. The PCWA with higher crushing value than conventional aggregates has the risk of inducing strength reduction of concrete, while the reduction rate should depend on the replacement ratio. According to a previous study [27], however, the compressive strength of ultra-high-strength concrete with the replacement of 40% coarse aggregate by PCWA was 160 N/mm², which was 30% higher than the ultra-high-strength concrete without PCWA. That is, there were no particular negative effects of PCWA on the strength development of concrete. Moreover, judging from the present test results, the strength reduction is not significant so long as the replacement ratio is below 20% and the enhancement effect of the internal curing is high beyond this negative effect. According to Fig. 3(b), using PCWA can improve the compressive strength of concrete by up to 25%. This means that the internal water of PCWA can be supplied to the mortar matrix naturally, and the PCWA can cure the concrete internally. In the case of concrete using only high-early-strength Portland cement, the gain in compressive strength using 10% PCWA was approximately 5% at the age of 1 d and increased to 10% at the ages of 182 and 364 d (see Fig. 3

(b)). This means that the effect of PCWA increased slightly over time. However, when the FA replacement ratio was 20%, the PCWA increased the compressive strength by 10% and 5% at the ages of 7 and 28 d, respectively, and the compressive strength of concrete using PCWA was almost the same as that without PCWA at the other ages regardless of the PCWA replacement ratio. It is assumed that the internal water of PCWA could participate in the development of compressive strength at a relatively early age. In the case of the 40% FA replacement, the gain in compressive strength due to PCWA was more than 10% for the early and later ages. The normalized compressive strength of HF40-G20 was 1.25 at the ages of 1 and 364 d, which indicated that the PCWA increased the compressive strength by 25%. Notably, this was the highest value for all the ages. A previous study reported that the application of 40% PCWA to high-strength FA concrete using 60% ordinary Portland cement and 40% FA with the water-to-cementitious materials ratio of 0.30 improved the compressive strength by 8.4–16.5% when it was cured under sealed conditions at 20 °C [29]. When compared to the result from the previous study [29], the addition of 20% PCWA improved the compressive strength to quite an extent both at the early and later ages, whereas while the addition of 10% PCWA provided a notable improvement in the compressive strength at the early age, this gain gradually decreased. This implies that the use of PCWA at the replacement ratio of 20% in the case of the 40% FA fraction in steam-cured concrete could effectively enhance cement hydration at an early age and the FA reaction in the long term. Furthermore, when compared with the previous study [29], the development of the compressive strength of the FA concrete in this study was not significant even in the long term, and the compressive strengths of HF40-G10 and HF40-G20 decreased slightly from 182 to 364 d. This result may be attributed to the negative impact of the steam curing, that is, the acceleration of cement hydration and FA reaction at the early age.

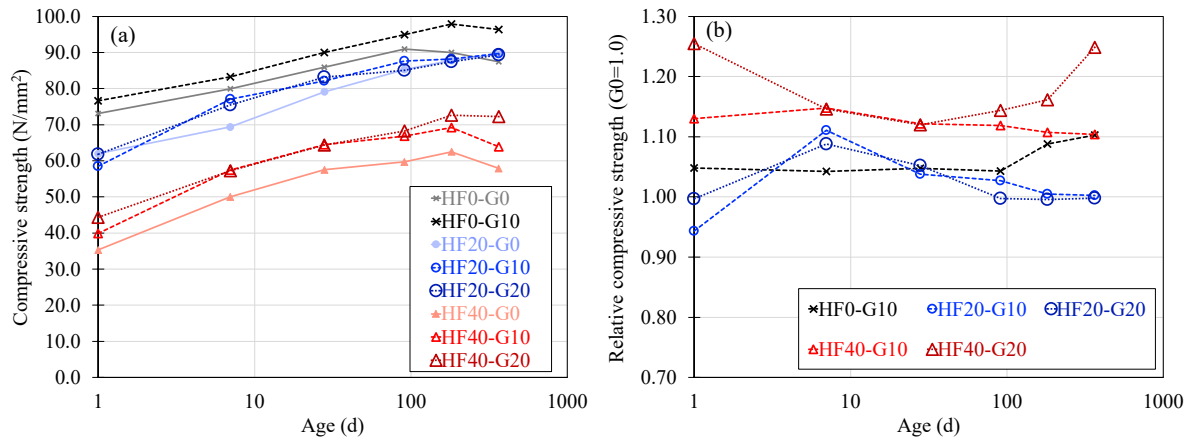


Figure 3 Effect of PCWA on compressive strength: (a) compressive strength of concrete with age, (b) compressive strength normalized by that of concrete without PCWA

3.2 Length change

The length change until the age of 1 d, as shown in Fig. 4, was obtained by subtracting the thermal strain from the measured free strain, where the thermal expansion coefficient is assumed to be a constant of $10 \times 10^{-6}/^{\circ}\text{C}$. It should be noted that the first peak of the measured value of length change in concrete corresponded to zero shrinkage in concrete up to the age of 1 d, as seen in Fig. 4. For the FA replacements, except for HF20-G10, the shrinkage in concrete using PCWA was smaller than that without PCWA. This result implies that PCWA can effectively reduce the autogenous shrinkage by supplying the internal water to the mortar matrix self-desiccated due to hydration. For the FA concrete, a PCWA replacement ratio of 20% could reduce the autogenous shrinkage to a greater extent than the replacement ratio of 10%. The shrinkage in Fig. 4, however, could be overestimated with the assumption that the thermal expansion coefficient is $10 \times 10^{-6}/^{\circ}\text{C}$ as this value can change at early ages [38]. Thus, further investigation into the autogenous shrinkage while considering the time dependency of the thermal expansion coefficient is required to ascertain its absolute value.

The length changes immediately after demolding and at the age of 200 d after demolding are also

shown in Fig. 5 (a) and (b), respectively. The value of zero length change was set to the strain at the age of 1 d (i.e., at the time of the demolding). Although there was no significant difference in the length change until the age of 3 d, the addition of PCWA increased the length change in concrete after its exposure to air until the age of 200 d, especially for the concrete with 40% FA (see Fig. 5 (b)). It appears that the internal water supplied during steam curing may not have been consumed completely for cement hydration and evaporated at a later age, resulting in the higher drying shrinkage in the concrete with PCWA at a higher replacement ratio, especially when the water-to-cement ratio was relatively high (i.e., the FA replacement ratio was high). Although the internal water of PCWA could play a role in internal curing, the water supplied from PCWA also could evaporate and cause drying shrinkage. This idea is in line with the results of a previous study [30]; though the length change in concrete without PCWA was almost the same or lower than that in concrete with PCWA for the same mass loss in concrete after demolding, the mass loss in concrete with PCWA increased to a greater extent than that in the concrete without PCWA, resulting in the larger shrinkage in the former.

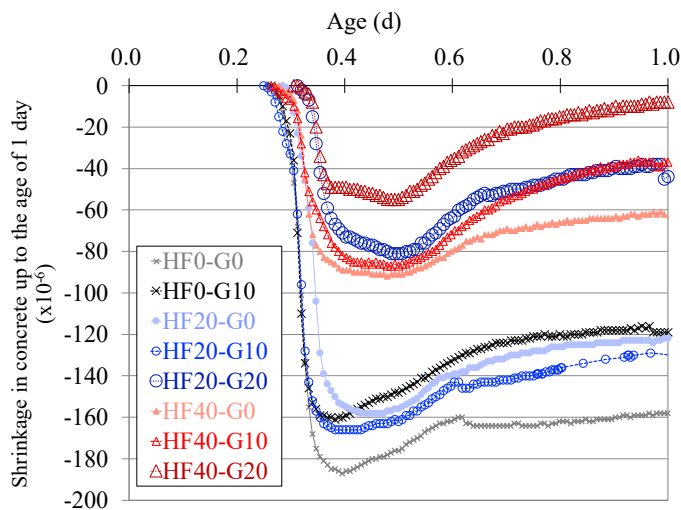


Figure 4 Shrinkage in concrete up to the age of 1 d before demolding

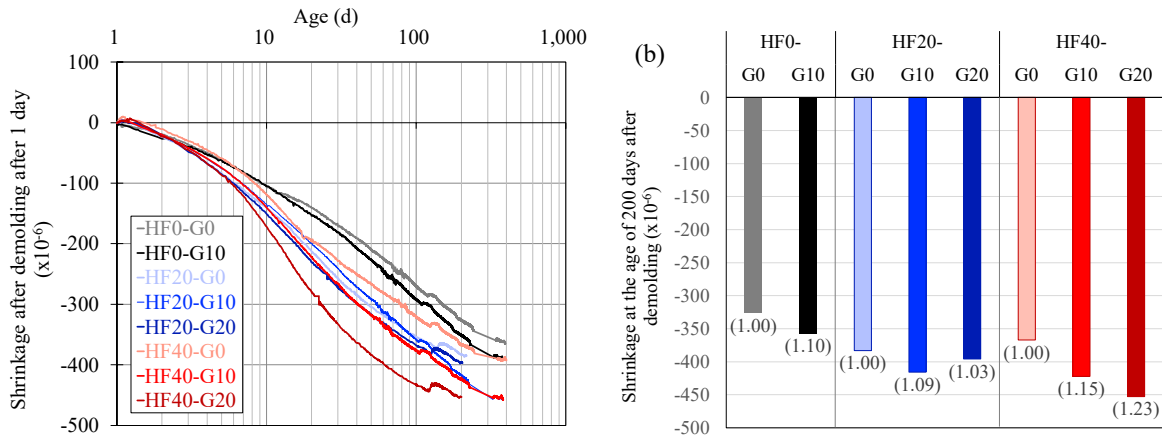


Figure 5 Shrinkage in concrete after demolding at 1 d: (a) with age, (b) at the age of 200 d

3.3 Accelerated carbonation

The carbonation depths of FA concrete are shown in Fig. 6. It can be observed that the carbonation depth of HF40-G0 was higher than that of any other mixture regardless of the carbonation period. Thus, the internal curing by PCWA was effective in lowering the carbonation depth for the concrete using 40% FA, whereas no significant effect of PCWA was observed for the concrete using 20% FA. The tendency was the same as that of compressive strength (see Fig. 3 (a)). Moreover, the carbonation rate after 4 or 8 weeks reduced compared to that before 4 or 8 weeks, except for HF40-G0. It implies that the steam curing and storage at 60% RH made the surface layer of the concrete porous, resulting in fast carbonation, whereas long-term storage had relatively less influence within the concrete, resulting in slow carbonation. Further investigation into properties such as pore structure is needed to confirm the aforementioned idea of inhomogeneity.

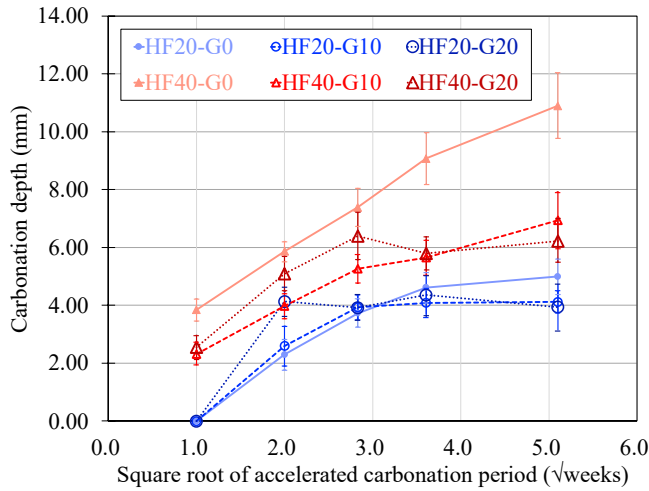


Figure 6 Carbonation depth of FA concrete under accelerated carbonation conditions

The carbonation coefficients were calculated using regression analysis with an empirical relationship based on the square root law, as expressed by Eq. (1). The carbonation coefficients were obtained using all the plotted data in Fig. 6.

$$y = \alpha\sqrt{t} \quad (1)$$

where y is the carbonation depth (mm), t is the period of accelerated carbonation (week), and α is the accelerated carbonation coefficient ($\text{mm}/\text{week}^{0.5}$). As shown in Table 4, the accelerated carbonation coefficients of HF40-G10 and HF40-G20 were approximately 65% of that of HF40-G0. It appears that internal curing by the PCWA could have effectively enhanced the carbonation resistance in concrete using 40% FA by approximately 35%. This means that the internal water of PCWA can be supplied to the mortar matrix as curing water to promote cement hydration and FA reaction, resulting in denser microstructure and enhancement of carbonation resistance. The relationships between the accelerated carbonation coefficient and compressive strength at each age are shown in Fig. 7. The compressive strength increased with the decrease in the accelerated carbonation coefficient regardless of the inclusion of PCWA. The effects of the internal curing on

compressive strength as well as carbonation resistance could be attributed to the improvement in pore structure. That is, using PCWA can make the concrete microstructure denser, resulting in better quality concrete, as reiterated in the next section. Furthermore, the relationship between the accelerated carbonation coefficient and compressive strength suggests that the CO₂ gas might have penetrated only the mortar matrix, and that the porous structure of PCWA did not adversely affect the carbonation resistance of concrete since the relationship can be approximated by a line regardless of the PCWA replacement. Further, the carbonation rate can be estimated using the compressive strength even when adding PCWA.

Table 4 Accelerated carbonation coefficient (mm/week^{0.5})

HF20-G0	HF20-G10	HF20-G20	HF40-G0	HF40-G10	HF40-G20
1.10	1.00	1.06	2.40	1.56	1.60

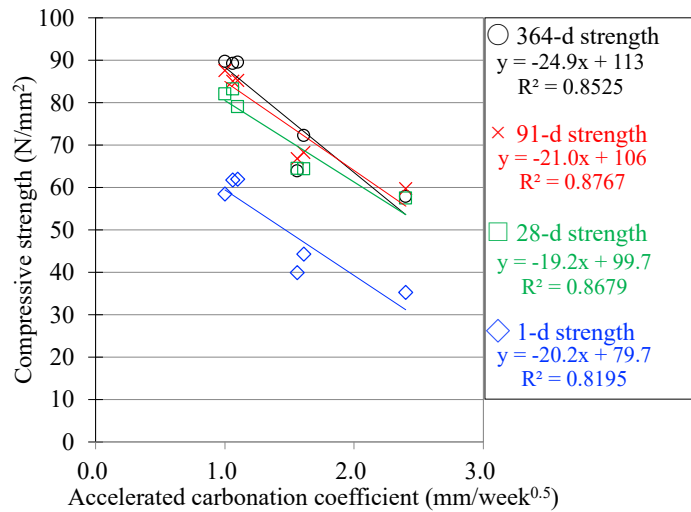


Figure 7 Relationship between compressive strength and accelerated carbonation coefficient

3.4 Pore size distribution

Figure 8 shows the cumulative intrusion curves for six samples at the age of 364 d [32]. Although using 10% PCWA in the concrete with 40% FA replacement slightly reduced the cumulative pore volume, this effect was not observed clearly for the concrete with 0% and 20% FA replacements. The trend was almost the same as that of the effects on compressive strength and carbonation resistance. Figure 9 shows the pore size distributions of these samples. According to Yamamoto and Kanazu [39], the pore volume for the pore size ranges of 0.003 μm to 0.02 μm increased, whereas that for the size ranges of 0.02 μm to 0.33 μm decreased as the pozzolanic reaction of FA progressed. Figure 9 also shows that the volumes of pores smaller than 0.02 μm increased significantly and those of pores ranging from 0.02 μm to 0.33 μm in size decreased for HF40 concrete with PCWA. This result indicates that the internal curing using PCWA could effectively enhance the pozzolanic reaction of FA in HF40 concrete, resulting in higher compressive strength and a lower accelerated carbonation coefficient. Moreover, the pore size distribution of HF20-G10 was almost the same as that of HF20-G0, which reflected the slight effect of PCWA on the compressive strength and carbonation resistance of steam-cured concrete using 20% FA replacement. The same pore size distribution tendency as that of HF40 concrete can be observed for the HF0 concrete (i.e., in the concrete without FA). This result may also indicate the promotion of cement hydration by PCWA, which is in agreement with the findings of a previous study [29]. These results imply that the internal water of PCWA can enhance the cement hydration and FA reaction by being supplied as the curing water. Moreover, a denser pore structure can be formed.

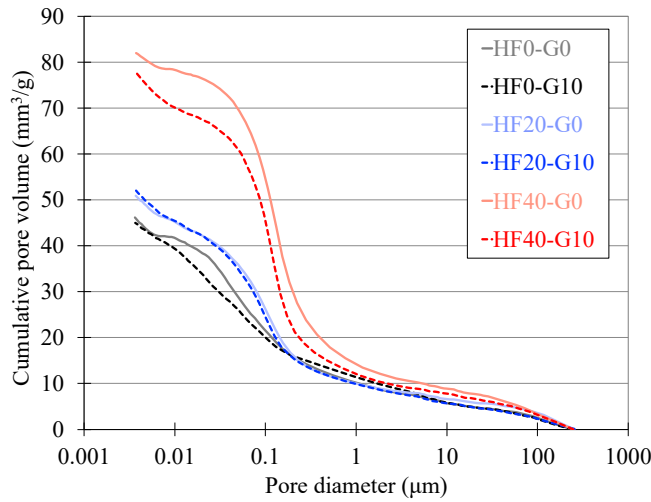


Figure 8 Cumulative intrusion curves of the concrete specimens at the age of 364 d [32]

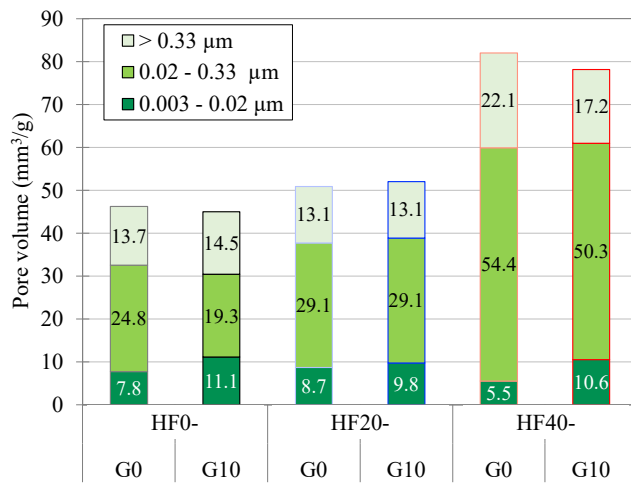


Figure 9 Pore size distributions of the concrete specimens at the age of 364 d

In general, macropores (pores larger than $0.05 \mu\text{m}$) play a dominant role in the mechanical properties and mass transfer resistance of concrete [40]. Figure 10 shows the relationship between the macropore volume and the 364-d compressive strength. The relationships between the macropore volume and the accelerated carbonation coefficient are also shown in Fig. 10, wherein the accelerated carbonation coefficient of HF0 is plotted as 0. As the macropore volume reduces, the compressive strength increases and the accelerated carbonation coefficient reduces regardless of

the mixture proportion. It can also be observed that for HF40 concrete alone, the use of PCWA decreased the macropore volume, resulting in higher compressive strength and lower accelerated carbonation coefficient. Consequently, internal curing using PCWA can assist in generating a denser microstructure and improving the compressive strength and carbonation resistance.

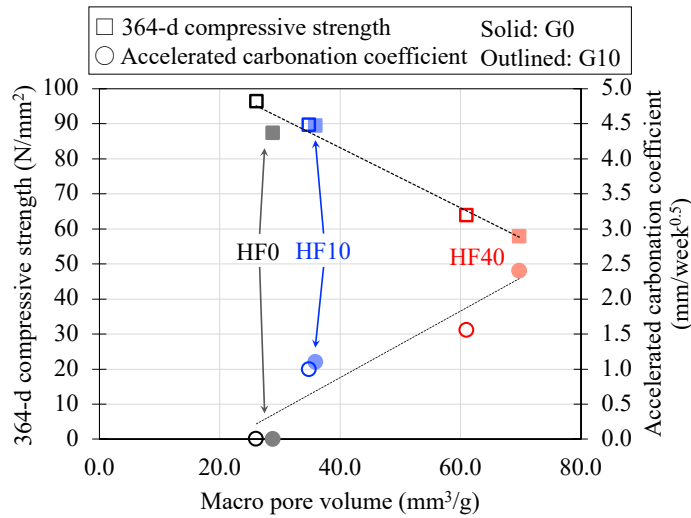


Figure 10 Relationship between macropore volume and other properties

4. Conclusions

The purpose of this study was to investigate the effects of PCWA as an internal curing agent on the compressive strength, shrinkage, and carbonation of steam-cured FA concrete, where the replacement ratios of FA were 0%, 20%, and 40% by mass, and those of PCWA were 0%, 10%, and 20% by volume.

The following conclusions can be drawn:

- (1) The internal curing using PCWA effectively enhanced the compressive strength of steam-cured FA concrete, especially when the replacement ratio of FA was 40%. PCWA increased the compressive strength of the concrete using 40% FA by 10–25%. This effect was obtained at the early age as well as in the long term. No adverse effect owing to the porous structure of PCWA

was observed for up to 20% replacement by PCWA up to the age of 364 d.

(2) PCWA could effectively reduce autogenous shrinkage and increase drying shrinkage in the steam-cured FA concrete.

(3) The accelerated carbonation resistance in the steam-cured concrete with 40% FA improved significantly with the addition of PCWA, and the effects of PCWA at the replacement ratios of 10% and 20% on the carbonation resistance were almost the same. PCWA replacements of 10% and 20% reduced the accelerated carbonation coefficient of 40% FA concrete by approximately 35%.

(4) The inverse linear relationship between the compressive strength and the accelerated carbonation coefficient was observed regardless of PCWA replacement. This result clarified that the CO₂ mainly penetrates the mortar matrix, not the PCWA, and that the porous structure of PCWA does not affect the carbonation resistance of concrete adversely.

(5) Considering the pore size distribution, the internal curing using PCWA could effectively enhance the FA reaction and cement hydration. PCWA decreased the macropore volume in mortar matrix, resulting in enhanced compressive strength and improved carbonation resistance.

Consequently, although PCWA is derived from waste, it is beneficial as an internal curing agent to improve the quality of not only steam-cured cement concrete but also steam-cured FA concrete. The quality improvement from using PCWA can greatly contribute to promoting the utilization of the waste of PCWA as well as the by-products of FA in the construction field. Further investigations on microstructure and/or internal relative humidity change of the concrete with time are needed to explain the mechanism of the internal curing effect of PCWA. This would also be useful for the design of the PCWA replacement ratio.

Conflicts of interests

The authors declare no conflicts of interest at this time.

Acknowledgment

The authors would like to thank Mr. Naoki Doi and Mr. Yusuke Muragishi, Master's students at Hiroshima University, for their help in conducting the experiments. This research was partially supported by a JSPS KAKENHI Grant-in-Aid for Young Scientists (B) (No. JP25820191).

References

- [1] J. Bijen, Benefits of slag and fly ash, *Constr. Build. Mater.* 10 (1996) 309–314.
[https://doi.org/10.1016/0950-0618\(95\)00014-3](https://doi.org/10.1016/0950-0618(95)00014-3).
- [2] T.R. Naik, B.W. Ramme, High early strength fly ash concrete for precast/prestressed products, *PCI J.* 35 (1990) 72–78. <https://doi.org/10.15554/pcij.11011990.72.78>.
- [3] J. Zachar, Sustainable and economical precast and prestressed concrete using fly ash as a cement replacement, *J. Mater. Civ. Eng.* 23 (2011) 789–792.
[https://doi.org/10.1061/\(asce\)mt.1943-5533.0000243](https://doi.org/10.1061/(asce)mt.1943-5533.0000243).
- [4] Z. He, J. Liu, K. Zhu, Influence of mineral admixtures on the short and long-term performance of steam-cured concrete, *Energy Procedia* 16 (2011) 836–841.
<https://doi.org/10.1016/j.egypro.2012.01.134>.
- [5] B. Liu, Y. Xie, J. Li, Influence of steam curing on the compressive strength of concrete containing supplementary cementing materials, *Cem. Concr. Res.* 35 (2005) 994–998.
<https://doi.org/10.1016/j.cemconres.2004.05.044>.
- [6] B. Lothenbach, F. Winnefeld, C. Alder, E. Wieland, P. Lunk, Effect of temperature on the pore solution, microstructure and hydration products of Portland cement pastes, *Cem. Concr. Res.* 37 (2007) 483–491. <https://doi.org/10.1016/j.cemconres.2006.11.016>.

- 416 [7] D.W.S. Ho, C.W. Chua, C.T. Tam, Steam-cured concrete incorporating mineral admixtures,
417 Cem. Concr. Res. 33 (2003) 595–601. [https://doi.org/10.1016/S0008-8846\(02\)01028-1](https://doi.org/10.1016/S0008-8846(02)01028-1).
- 418 [8] G. Long, Z. He, A. Omran, Heat damage of steam curing on the surface layer of concrete,
419 Mag. Concr. Res. 64 (2012) 995–1004. <https://doi.org/10.1680/mac.11.00164>.
- 420 [9] S. Nie, S. Hu, F. Wang, P. Yuan, Y. Zhu, J. Ye, Y. Liu, Internal curing – A suitable method
421 for improving the performance of heat-cured concrete, Constr. Build. Mater. 122 (2016)
422 294–301. <https://doi.org/10.1016/j.conbuildmat.2016.05.159>.
- 423 [10] C. Zou, G. Long, C. Ma, Y. Xie, Effect of subsequent curing on surface permeability and
424 compressive strength of steam-cured concrete, Constr. Build. Mater. 188 (2018) 424–432.
425 <https://doi.org/10.1016/j.conbuildmat.2018.08.076>.
- 426 [11] P. Lura, Autogenous Deformation and Internal Curing of Concrete, Delft University Press,
427 Delft, 2003.
- 428 [12] CT-18: ACI Concrete Terminology, American Concrete Institute, Farmington Hills, Mich.,
429 (2018) 36.
- 430 [13] R. Philleo, Concrete science and reality, in: J.P. Skalny, S. Mindess (Eds.), Materials
431 Science of Concrete II, American Ceramic Society, Westerville, Ohio, 1991, pp. 1–8.
- 432 [14] T.A. Hammer, High strength LWA concrete with silica fume effect of water content in the
433 LWA on mechanical properties, in: Suppl. Pap. Fourth CANMET/ACI Int. Conf. Fly Ash,
434 Silica Fume, Slag, Nat. Pozzolans Concr., Istanbul, Turkey, 1992: pp. 314–330.
- 435 [15] D.P. Bentz, P. Lura, J.W. Roberts, Mixture proportioning for internal curing, Concr. Int. 27
436 (2005) 35–40.
- 437 [16] D.P. Bentz, Internal curing of high-performance blended cement mortars, ACI Mater. J. 104
438 (2007) 408–414.
- 439 [17] D. Cusson, T. Hoogeveen, Internal curing of high-performance concrete with pre-soaked

- fine lightweight aggregate for prevention of autogenous shrinkage cracking, *Cem. Concr. Res.* 38 (2008) 757–765. <https://doi.org/10.1016/j.cemconres.2008.02.001>.
- [18] O.M. Jensen, P.F. Hansen, Water-entrained cement-based materials, *Cem. Concr. Res.* 31 (2001) 647–654. [https://doi.org/10.1016/s0008-8846\(01\)00463-x](https://doi.org/10.1016/s0008-8846(01)00463-x).
- [19] B. Craeye, M. Geirnaert, G. De Schutter, Super absorbing polymers as an internal curing agent for mitigation of early-age cracking of high-performance concrete bridge decks, *Constr. Build. Mater.* 25 (2011) 1–13. <https://doi.org/10.1016/j.conbuildmat.2010.06.063>.
- [20] J. Justs, M. Wyrzykowski, D. Bajare, P. Lura, Internal curing by superabsorbent polymers in ultra-high performance concrete, *Cem. Concr. Res.* 76 (2015) 82–90. <https://doi.org/10.1016/j.cemconres.2015.05.005>.
- [21] O.M. Jensen, P. Lura, Techniques and materials for internal water curing of concrete, *Mater. Struct. Constr.* 39 (2006) 817–825. <https://doi.org/10.1617/s11527-006-9136-6>.
- [22] S.T. Yildirim, C. Meyer, S. Herfellner, Effects of internal curing on the strength, drying shrinkage and freeze-thaw resistance of concrete containing recycled concrete aggregates, *Constr. Build. Mater.* 91 (2015) 288–296. <https://doi.org/10.1016/j.conbuildmat.2015.05.045>.
- [23] F. Liu, J. Wang, X. Qian, J. Hollingsworth, Internal curing of high performance concrete using cenospheres, *Cem. Concr. Res.* 95 (2017) 39–46. <https://doi.org/10.1016/j.cemconres.2017.02.023>.
- [24] M. Wyrzykowski, S. Ghourchian, S. Sinthupinyo, N. Chitvoranund, T. Chintana, P. Lura, Internal curing of high performance mortars with bottom ash, *Cem. Concr. Compos.* 71 (2016) 1–9. <https://doi.org/10.1016/j.cemconcomp.2016.04.009>.
- [25] M. Suzuki, I. Maruyama, T. Kawabata, R. Sato, A study on deformation of ultra high strength concrete containing crushed roof tile aggregate (in Japanese), *Proc. Japan Concr.*

Inst. 29 (2007) 651–656.

[26] S. Suzuki, Survey research on manufacture and supply of structural concrete using crushed roof tile waste as aggregate (in Japanese), *Japan Test. Cent. Constr. Mater. J.* 51 (2015) 14–19.

[27] M. Suzuki, M. Seddik Meddah, R. Sato, Use of porous ceramic waste aggregates for internal curing of high-performance concrete, *Cem. Concr. Res.* 39 (2009) 373–381. <https://doi.org/10.1016/j.cemconres.2009.01.007>.

[28] R. Sato, A. Shigematsu, T. Nukushina, M. Kimura, Improvement of properties of Portland blast furnace cement type B concrete by internal curing using ceramic roof material Waste, *J. Mater. Civ. Eng.* 23 (2010) 777–782. [https://doi.org/10.1061/\(asce\)mt.1943-5533.0000232](https://doi.org/10.1061/(asce)mt.1943-5533.0000232).

[29] P.T. Bui, Y. Ogawa, K. Nakarai, K. Kawai, R. Sato, Internal curing of Class-F fly-ash concrete using high-volume roof-tile waste aggregate, *Mater. Struct.* 50 (2017) 203. <https://doi.org/10.1617/s11527-017-1073-z>.

[30] Y. Muragishi, Y. Ogawa, K. Kawai, R. Sato, Effect of porous ceramic waste aggregate on durability of steam cured fly ash concrete (in Japanese), *Cem. Sci. Concr. Technol.* 68 (2014) 337–344. <https://doi.org/10.14250/cement.68.337>.

[31] P.T. Bui, Y. Ogawa, N. Doi, K. Kawai, R. Sato, Properties of steam-cured fly ash concrete using porous ceramic waste aggregate, in: *Proc. 13th Int. Conf. Recent Adv. Concr. Technol. Sustainability Issues*, Ottawa, Canada, ACI SP303, 2015: pp. 323–336.

[32] P.T. Bui, Y. Muragishi, Y. Ogawa, K. Kawai, R. Sato, Effects of porous ceramic waste aggregate as an internal curing agent on steam-cured high strength fly ash concrete, in: *Proc. Int. Conf. Sustainable Struct. Concr.*, La Plata, Argentina, 2015: pp. 66–76.

[33] Japanese Standards Association, JIS R 5210:2009. Portland cement, Japanese Standards

Association, Tokyo, Japan, 2009.

[34] Japanese Standards Association, JIS R 6201:2015. Fly ash for use in concrete, Japanese Standards Association, Tokyo, Japan, 2015.

[35] Japanese Standards Association, JIS A 5005:2009. Crushed stone and manufactured sand for concrete, Japanese Standards Association, Tokyo, Japan, 2009.

[36] British Standards Institution, BS 812-110:1990. Testing aggregates. Methods for determination of aggregate crushing value (ACV), British Standards Institution, London, United Kingdom, 1990.

[37] Japanese Standards Association, JIS A 1108:2018. Method of test for compressive strength of concrete, Japanese Standards Association, Tokyo, Japan, 2018.

[38] I. Maruyama, A. Teramoto, Impact of time-dependant thermal expansion coefficient on the early-age changes in cement paste, *Cem. Concr. Res.* 41 (2011) 380–391.
<https://doi.org/10.1016/j.cemconres.2011.01.003>.

[39] T. Yamamoto, T. Kanazu, Experimental explanation of compacting effect on hydration phases and strength development mechanism derived from pozzolanic reaction of fly ash (in Japanese), *J. Japan Soc. Civ. Eng. Ser. E.* 63 (2007) 52–65.

[40] P.K. Mehta, P.J. Monteiro, *Concrete. Microstructure, properties, and materials*, third ed., McGraw-Hill, New York, 2006.