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Research Article

Late-Age Properties of Concrete with Different Binders Cured under 45°C at Early Ages

Hu Jin

Sichuan College of Architectural Technology, Deyang 618000, China

Correspondence should be addressed to Hu Jin; hujinthu@163.com

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It is commonly accepted that high curing temperature (near 60°C or above) results in reduced mechanical properties and durability of concrete compared to normal curing temperature. The internal temperature of concrete structures at early ages is not so high as 60°C in many circumstances. In this paper, concretes were cured at 45°C at early ages and their late-age properties were studied. The concrete cured at 20°C was employed as the reference sample. Four different concretes were used: plain cement concrete, concrete containing fly ash, concrete containing ground granulate blast furnace slag (GGBS), and concrete containing silica fume. The results show that, for each concrete, high-temperature curing after precuring does not have any adverse effect on the nonevaporable water content, compressive strength, permeability to chloride ions, and the connected porosity of concrete at late ages compared with standard curing. Additionally, high-temperature curing improves the late-age properties of concrete containing fly ash and GGBS.

1. Introduction

Cement concrete is one of the most widely used construction materials today. It is well known that the hydration of cement releases heat. In common engineering practices, the internal temperature of concrete is higher than 20°C at early ages for the following reasons. First, the amount of cementitious materials is occasionally large to meet the strength grade requirements. Second, heat dissipation is always low in large-volume concrete members (e.g., foundation plates) [1]. Third, the initial casting temperature of concrete is usually high [2]. Thus, the actual temperature of concrete at early ages is higher than the standard curing temperature in the laboratory, which makes the concrete used in practice behave differently from the concrete used in the laboratory.

High temperature accelerates the early hydration of cement, thereby increasing the early strength and elastic modulus of concrete. However, a high early age curing temperature may result in a lower ultimate strength and elastic modulus of concrete [3–7]. It has also been found [8, 9] that an increased curing temperature leads to the increased diffusion and penetration of the hydrate. Shen et al. [10] found that the autogenous shrinkage of high performance concrete (HPC) increased with increasing curing temperature and that HPC specimens cured isothermally at 20°C showed better cracking resistance than those cured at higher temperatures. These phenomena can be explained by exploring the microstructure of the cured HPC specimens. Some researchers [4, 11] have found that concrete cured at elevated temperatures shows accelerated hydration and nonuniform distribution of hydration products, which results in greater porosity and an increase in compressive strength at an early age, followed by a decrease at later ages [9, 12]. Kjellsen et al. [13] found that at a higher temperature, the microstructure of the cement paste is less homogeneous, resulting in a higher mercury intrusion porosity. Consequently, the long-term mechanical properties of cement paste are weaker in such concrete [14, 15]. More details of the studies about late-age properties of concrete cured at high temperature are summarized in Table 1. The conclusions of these studies are inconsistent and even contradictory mainly because of the different curing regimes.

In most studies, the specimens were cured at 60°C or above immediately after preparation without precuring, which differs from the practical situation. In many engineering practices, the section size of the concrete member is not so large and normal strength concrete is commonly used, so the

Table 1: Results of late-age properties of concrete cured at high temperature.

Ref. number	Cementitious system	Curing regimes: Precuring time	Curing regimes: Maximum temperature	Curing regimes: Duration at the maximum temperature	Late-age properties
[7]	Type I, V cement, FA	24 h	10, 23, 35, 50°C	CT: until testing ages	Lower late-age strength and elastic modulus of the concrete cured at 50°C.
[20]	Type I, V cement	24 h	10, 23, 50°C	CT: until testing ages	Lower 28-day strength of the concrete cured at 50°C
[21]	PC	24 h	5, 20, 30, 40°C	CT: until testing ages	Lower strength and coarser porosity of the samples cured at 40°C.
[22]	PC	24 h	4, 22, 40, 85°C	CT: until testing ages	Lower strength and coarser porosity of the samples cured at 40°C and 85°C
[23]	PC, GGBS	24 h	25, 35, 45, 55, 100°C	CT: until testing ages	Lower strength of plain cement concrete cured at 45 up to 100°C, while higher strength of samples containing GGBS cured at elevated temperature
[24]	PC GGBS LP	24 h	20, 30, 40, 50°C	CT: until testing ages	Lower 28-day strength of all samples cured at 40 and 50°C
[25]	PC	0, 1, 2, 6 d	40°C	VT: 1 d; CT: until testing ages	Almost no negative effects on late-age strength of samples cured at 40°C with precuring time.
[26]	PC, NP	0 h	40, 60°C	VT: 1, 3, 7 d; CT: until testing ages	Lower strength of the plain cement concrete and higher strength of the concrete containing natural pozzolan cured at 40°C and 60°C (CT) at 28 and 90 days Lower strength of the concrete containing natural pozzolan cured at 40°C and 60°C (VT) at 28 and 90 days
[27]	PC, LP, NP, GGBS	24 h	20, 30, 40, 50°C	CT: until testing ages; VT: 1, 3, 7, 28 d	Decreasing strength of all samples with temperature rise, and lower decreasing ratio of the samples containing mineral admixtures
[28]	PC, FA	1 d	27, 34, 42, 50°C	VT: 7 d	Lower late-age strength of OPC concrete cured at 42 and 50°C, while higher for OPC-FA concrete
[29]	PC	4, 7 h	42, 46, 85°C	VT: 4, 12, 14 h	Similar microstructure of samples cured at 46 and 42°C with that at 20°C, while coarser microstructure of samples cured at 85°C
[30]	PC, PFA, SF	24 h	20, 60°C	VT: 3 d	Higher strength of the mixes containing pulverized fuel ash and silica fume cured at 60°C, while lower strength of Portland cement mixes cured at 60°C than those under normal curing
[31]	PC, FA, GGBS	3.5 h	70°C	VT: 4 h	Higher 28-day strength of the concrete containing mineral admixtures cured at 70°C

Note. PC: Portland cement; FA: Fly ash; PFA: pulverized fuel ash; NP: natural pozzolan; SF: silica fume; GGBS: ground granulate blast furnace slag; LP: limestone powder. CT: constant temperature; VT: variable temperature.

actual internal temperature of the concrete is usually lower than 60°C. Faria et al. [16] monitored and simulated the temperature rise of a slab of 143 m long, 41 m wide, and 0.35 m thick. The results showed that the maximum temperature was about 35°C and the time of temperature rising was 1 day. Kuriakose et al. [17] obtained similar simulation results with Faria. Chu et al. [18] measured the temperature development of a dam block of 1 m thick from both site and laboratory. The results showed that the maximum temperature was about 35°C and the time of temperature rising was 2.5 day. Wu and Luna [19] obtained the temperature curve at the central point of the concrete block of 30 m long, 4 m wide and 4 m thick. The results showed that the temperature reached a maximum point (about 40°C) at about 8 days after casting. Kuriakose et al. [17] simulated the temperature evolutions at different points of a circular raft of 12 m in diameter and 3 m thick. The results showed that the maximum temperature was about 50°C and the time of temperature rising was 1 to 4 days. Moreover, it usually takes two or even more days for the internal temperature of concrete structure to reach peak value after casting of concrete in engineering practices. So precuring is an essential condition for the high-temperature curing regime in order to get close to the engineering practices.

Table 2: Chemical compositions of raw materials w/%.

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O_{eq}	LOI
Cement	21.96	4.73	3.68	62.30	2.59	1.82	0.64	1.99
Fly ash	53.33	27.65	6.04	2.86	1.35	0.45	1.54	3.46
GGBS	31.76	14.84	0.60	36.44	9.08	0	0.56	1.20

Note. $Na_2O_{eq} = Na_2O + 0.658K_2O$.

Table 3: Chemical compositions of the silica fume w/%.

Composition	SiO_2	K	Na	H_2O	LOI
DSF	92.30	0.06	0.02	1.1	2.5

In this study, the early age curing temperature was set at 45°C and different precuring times were set. The aim of this study was to investigate the influence of high curing temperature of 45°C on the late-age properties of different concretes.

2. Experimental

2.1. Raw Materials. The cement used was Portland cement with a strength grade of 42.5, conforming to the Chinese National Standard GB8076-2008. The specific surface area of the cement was 350 m^2/kg. The low-calcium fly ash used in this study conformed to the Chinese National Standard GBT1596-2005. The specific surface area of the fly ash was 356 m^2/kg. The GGBS complied with the Chinese National Standard GBT18046-2008. The specific surface area of the GGBS was 409 m^2/kg. Densified silica fume (DSF) with a density of 650 kg/m^3 was used in this study. The coarse aggregates were crushed limestone aggregates with a size of 5–25 mm. The fine aggregates were natural river sands with a fineness modulus of 2.8. Polycarboxylic superplasticizer (PS) was used to improve the workability of the concrete. The chemical compositions of the cement, fly ash, and GGBS are given in Table 2. The chemical compositions of the silica fume are given in Table 3.

2.2. Test Methods. Four concretes with different binders were designed: plain cement concrete, concrete containing 45% fly ash, concrete containing 45% GGBS, and concrete containing 12% silica fume. The replacement ratio of mineral admixture was adopted in order to design concrete with high-volume mineral admixture. However, in consideration of engineering application, the replacement ratio was not excessively large. The mix proportions of the concretes are shown in Table 4. The water-to-binder ratio (W/B) of each concrete was 0.43. Four pastes corresponding to the four concrete were also prepared.

Five different curing conditions were designed (Figure 1). For curing condition "A," the specimens were cured under standard curing condition (20 ± 1°C and relative humidity higher than 95%) for the whole age after preparation. For the other four conditions, the specimens were first cured under standard curing condition for 0.5 or 1 day after preparation and then cured in a steam curing chamber at 45°C for 3 or 7 days and thereafter cured under standard condition until testing age. It takes 2 hours for the temperature to rise from 20°C to 45°C or to decline from 45°C to 20°C.

Concretes of 100 × 100 × 100 mm were prepared for the compressive strength and permeability tests. The compressive strength of the concrete was tested at the ages of 90 and 360 days.

The chloride ion permeability of concrete at the ages of 90 and 360 days was tested according to ASTM C1202 "Standard test method for electrical indication of concrete's ability to resist chloride ion penetration."

Concrete of 100 × 100 × 25 mm was prepared for the connected porosity tests at the age of 360 days. "Saturating-drying" method was used for the test: the volume (V) of the specimen and the mass of the specimen after vacuum saturation (m_1) and drying at 80°C for 14 days (m_2) were measured. The connected porosity (p) was calculated by

$$p = \frac{m_1 - m_2}{\rho V} \times 100\%, \quad (1)$$

where ρ is the density of water.

Nonevaporable water content (w_n) was obtained by calculating the mass difference between the samples heated at 105°C and 1000°C normalized by the mass after heating at 105°C and correcting for the loss on ignition of unhydrated samples [32]. The nonevaporable water content (w_n) was calculated by the following equation [33]:

$$w_n = \frac{m_{105} - m_{1000}}{m_{105}} - \left(f_c \cdot \mathrm{LOI}_c + f_m \cdot \mathrm{LOI}_m\right), \quad (2)$$

where m_{105} is the mass of the sample at 105°C, m_{1000} is the mass of the sample at 1000°C, f_c is the mass fraction of cement, f_m is the mass fraction of the corresponding mineral admixture, LOI_c is the ignition loss of cement, and LOI_m is the ignition loss of the corresponding mineral admixture.

3. Results and Discussion

3.1. Nonevaporable Water (w_n) Content. The amount of hydration products is proportional to the nonevaporable water content of paste. For pastes with similar hydration products, the w_n content can be used as an index for the comparison of binder's hydration degree. The w_n content of four different binders at the ages of 90 and 360 days is shown in Figures 2 and 3, respectively.

It can be seen that at the ages of both 90 and 360 days, the w_n content of plain cement sample cured at 45°C for 3 d without precuring is lower than that cured under standard condition, which agrees with the previous studies including those at even higher temperature. Kantro et al. [34] reported a

Table 4: Mix proportion of concretes/(kg/m^3).

Samples	Cement	Fly ash	GGBS	Silica fume	Water	Fine aggregates	Coarse aggregates
(a) in Figures 2–8	360	0	0	0	155	808	1070
(b) in Figures 2–8	198	162	0	0	155	808	1070
(c) in Figures 2–8	198	0	162	0	155	808	1070
(d) in Figures 2–8	316.8	0	0	43.2	155	808	1070

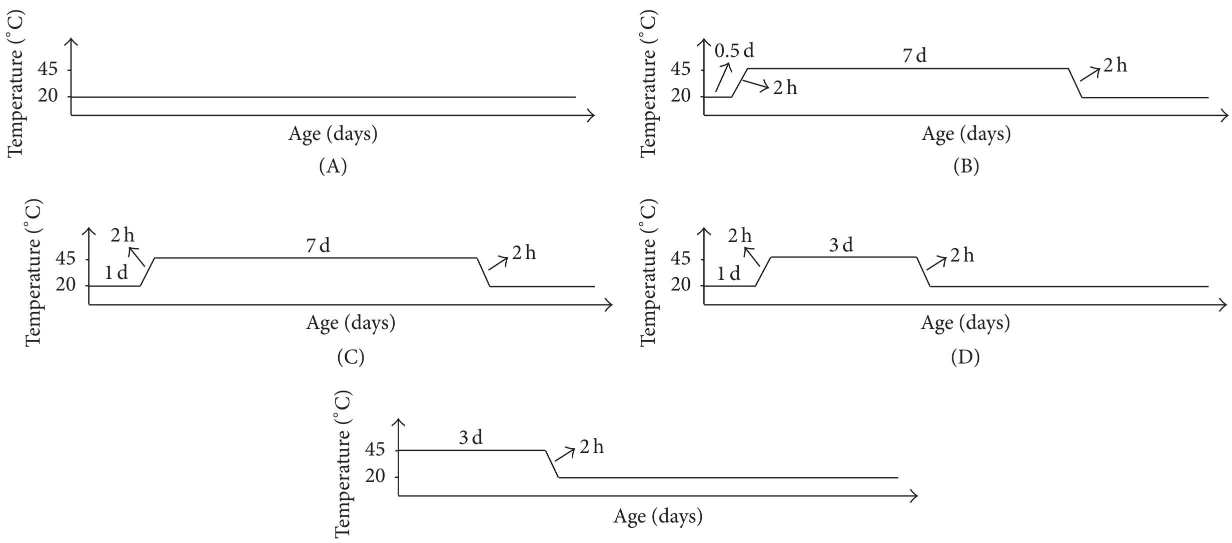

Figure 1: Five different curing conditions. Conditions: (A) under standard curing condition for the whole age; (B) under standard curing condition for the initial 0.5 d, then at 45°C for 7 d, and thereafter under standard curing condition until testing age; (C) under standard curing condition for the initial 1 d, then at 45°C for 7 d, and thereafter under standard curing condition until testing age; (D) under standard curing condition for the initial 1 d, then at 45°C for 3 d, and thereafter under standard curing condition until testing age; (E) immediately at 45°C for 3 d and then under standard curing condition until testing age.

decreased hydration degree of alite and belite at 50°C. Kjellsen and Detwiler [35] reported the lowest ultimate hydration degree at 50°C by the determination of the w_n content. Escalante-García and Sharp [36] reported that the amount of $Ca(OH)_2$ of samples cured at 40 and 60°C determined by thermogravimetric analysis (TGA) was less than that cured at 20°C, which indicated a lower hydration degree at higher temperature. The decrease of cement's hydration degree is due to the rapid hydration rate at 45°C at the initial age, which allows the dissolved ions less time for diffusion before the hydrates precipitate and builds up a denser inner C-S-H layer within the original boundaries of cement grains (as opposed to the outer C-S-H formed in the initially water-filled space), thereby retarding subsequent hydration [21, 29, 35, 36]. However, the w_n content of the plain cement samples under curing conditions "B," "C," and "D" (precured at 20°C for 0.5 or 1 d and then cured at 45°C) was higher than that under standard curing condition at the ages of both 90 and 360 days. It is an indication that, at late ages, the hydration degree of cement hydrated at increased temperature of 45°C after a period of precuring is elevated compared to that hydrated at 20°C.

For cement-fly ash paste and cement-GGBS paste, the w_n content cured at 45°C without precuring is higher than that under standard curing condition at the ages of 90 and 360 days. These results are quite different from those of the plain cement pastes. It is believed that less dense C-S-H is formed for the composite binder at the initial age due to the high replacement of cement. Moreover, high-temperature curing at an early age can enhance the activity of fly ash and GGBS, with a corresponding increase in the nonevaporable water content. Ma et al. [37] reported that mineral admixtures such as natural pozzolan, fly ash, and GGBS could modify the kinetic of cement hydration and produce additional C-S-H. At high curing temperature, these admixtures can reduce the negative effect of the temperature because of their pozzolanic reaction and great activation energies [26]. Additionally, the coating layer on fly ash particles and GGBS particles is weaker than that on cement particles, leading to correspondingly weaker inhibitory effects on the late-age reactions of fly ash and GGBS. Similar to the results of plain cement sample, the w_n content of cement-fly ash sample and cement-GGBS sample cured at 45°C with a period of precuring is increased compared to that cured under standard curing at the ages of 90 and 360 days. However, it is notable that the increments of w_n content of these two composite pastes are much greater than that of the plain cement paste, which is believed to be

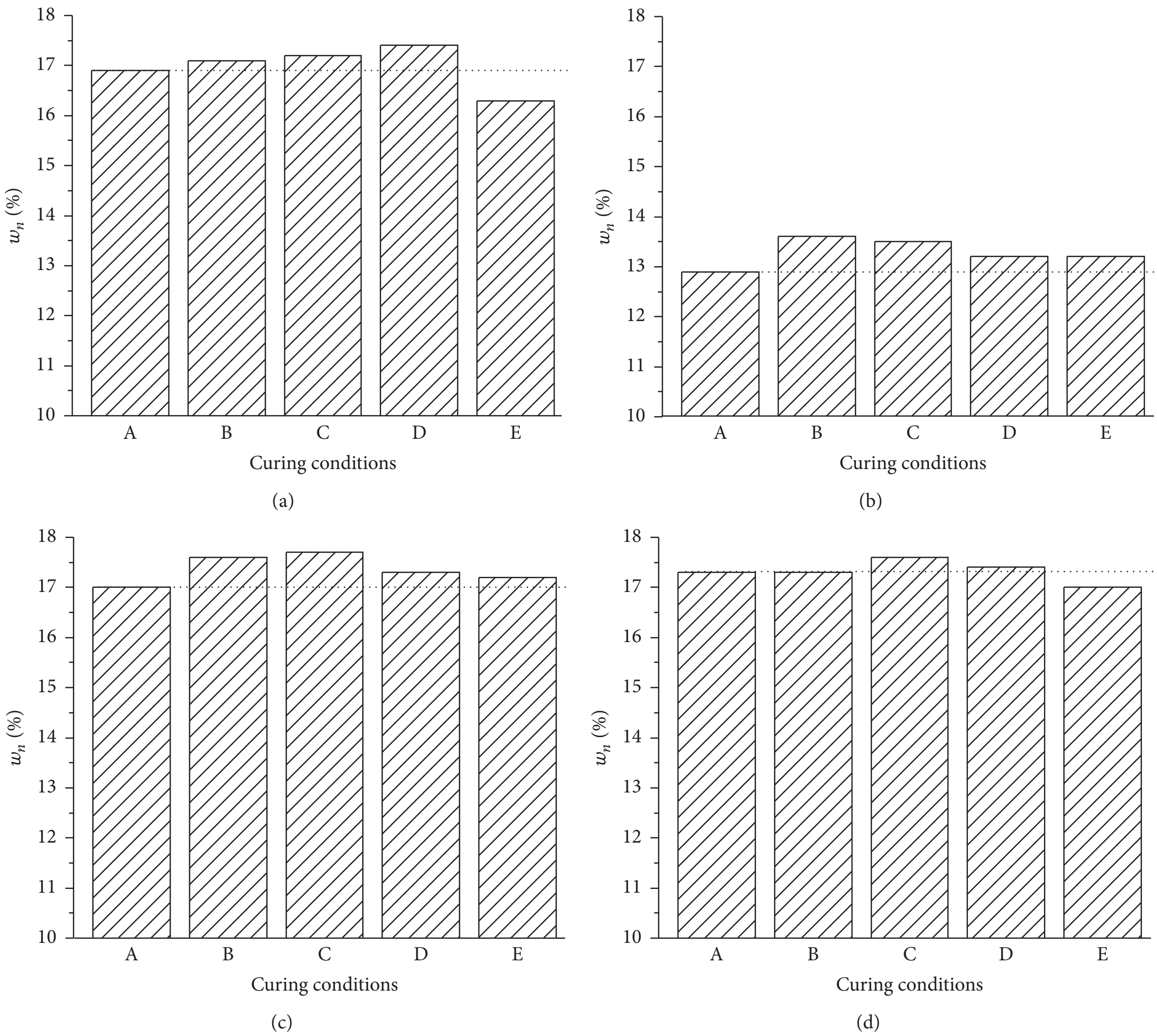

FIGURE 2: The 90 d nonevaporable water content of pastes under different curing conditions. Samples: (a) plain cement, (b) 45% FA, (c) 45% GGBS, and (d) 12% DSF.

due to the significant increment of reaction degree of fly ash and GGBS.

The performances of cement-silica fume pastes are similar to those of plain cement pastes: the samples immediately cured at 45°C without precuring display some adverse effects of high-temperature curing, while precured samples display no such adverse effects. This is because silica fume has a relatively high activity [26, 37, 38] and the degree of reaction at early age is high at elevated temperature, thereby producing a denser C-S-H layer on the particles. It can also be observed that at increased curing temperature the cement-silica fume composite binder exhibits limited or a very small improvement in the w_n content at the ages of 90 and 360 days, even though precuring time is set. This is because elevated curing temperature has little influence on the reaction degree of silica fume within 90 and 360 days, which is different from the effect of elevated temperature on fly ash and GGBS.

For each cementitious system, there was little or a very small difference in the nonevaporable water contents under curing conditions "B," "C," and "D," which indicates that the late-age hydration degree changes little by extending the precuring time from 0.5 to 1 day and the high-temperature curing time from 3 to 7 days.

3.2. Compressive Strength. The compressive strengths of four different types of concrete at the ages of 90 and 360 days are shown in Figures 4 and 5, respectively. It can be seen that the strengths of plain cement concrete and cement-silica fume concrete cured at 45°C immediately after preparation (without precuring) are lower than those of samples subjected to standard curing conditions, which is consistent with the trend of w_n content and the results of other studies [26]. It is worth noting that the w_n content of cement-GGBS paste cured under condition "E" is higher than that cured under

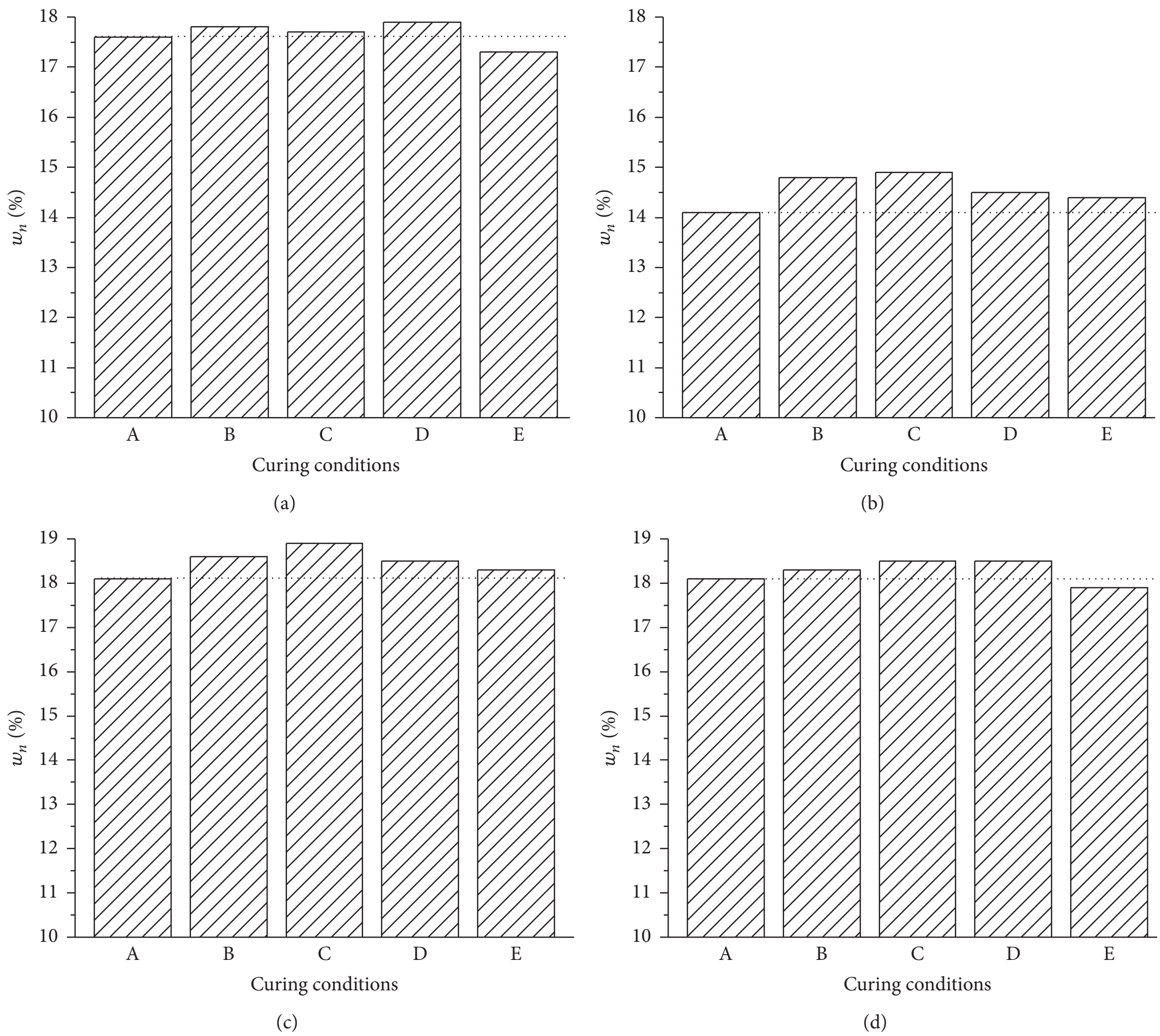

FIGURE 3: The 360 d nonevaporable water content of pastes under different curing conditions. Samples: (a) plain cement, (b) 45% FA, (c) 45% GGBS, and (d)12% DSF.

condition "A," but cement-GGBS concrete cured under condition "E" shows lower compressive strength than that cured under condition "A." This indicates that the volume percentage of large pores in the pore system increases, which is caused by nonuniform hydration products [21, 22, 26] when the samples are cured at a high temperature of 45°C. Unlike the cement-GGBS concrete, the cement-fly ash concrete cured under condition "E" shows a little higher compressive strength than that cured under condition "A." This is because the w_n content of cement-fly ash paste increases much greater than that of cement-GGBS paste at elevated temperature (Figures 2 and 3). However, it is notable that the enhancement of compressive strength of cement-fly ash concrete is far less than that of the w_n content of cement-fly ash paste, which further proves that elevated temperature tends to cause nonuniform hydration products.

Overall, the compressive strengths of all the concrete samples under curing conditions "B," "C," and "D" are close to or above that under curing condition "A" at the age of 90 or 360 days, which conflicts with the conclusions of many other studies that reveal the considerable negative effect of elevated curing temperature on the late strength (Table 1). The difference may be due to the different curing regimes: a period of precuring tends to reduce the negative effect of high curing temperature on the pore structure and hydration degree; the early hydration of binder cured at 45°C is not accelerated so sharply compared to that cured at 20°C. As expected, under the condition of elevated temperature curing with a period of precuring, the strength increments of the cement-fly ash concrete and cement-GGBS concrete are larger than those of the plain cement concrete and cement-silica fume concrete.

It can be concluded that high-temperature curing at 45°C for 3 to 7 days does not have negative effects on the late-age compressive strength of concrete, provided that precuring for 0.5 day or more is set. In many engineering practices, the heat released by the hydration of binder is not too much,

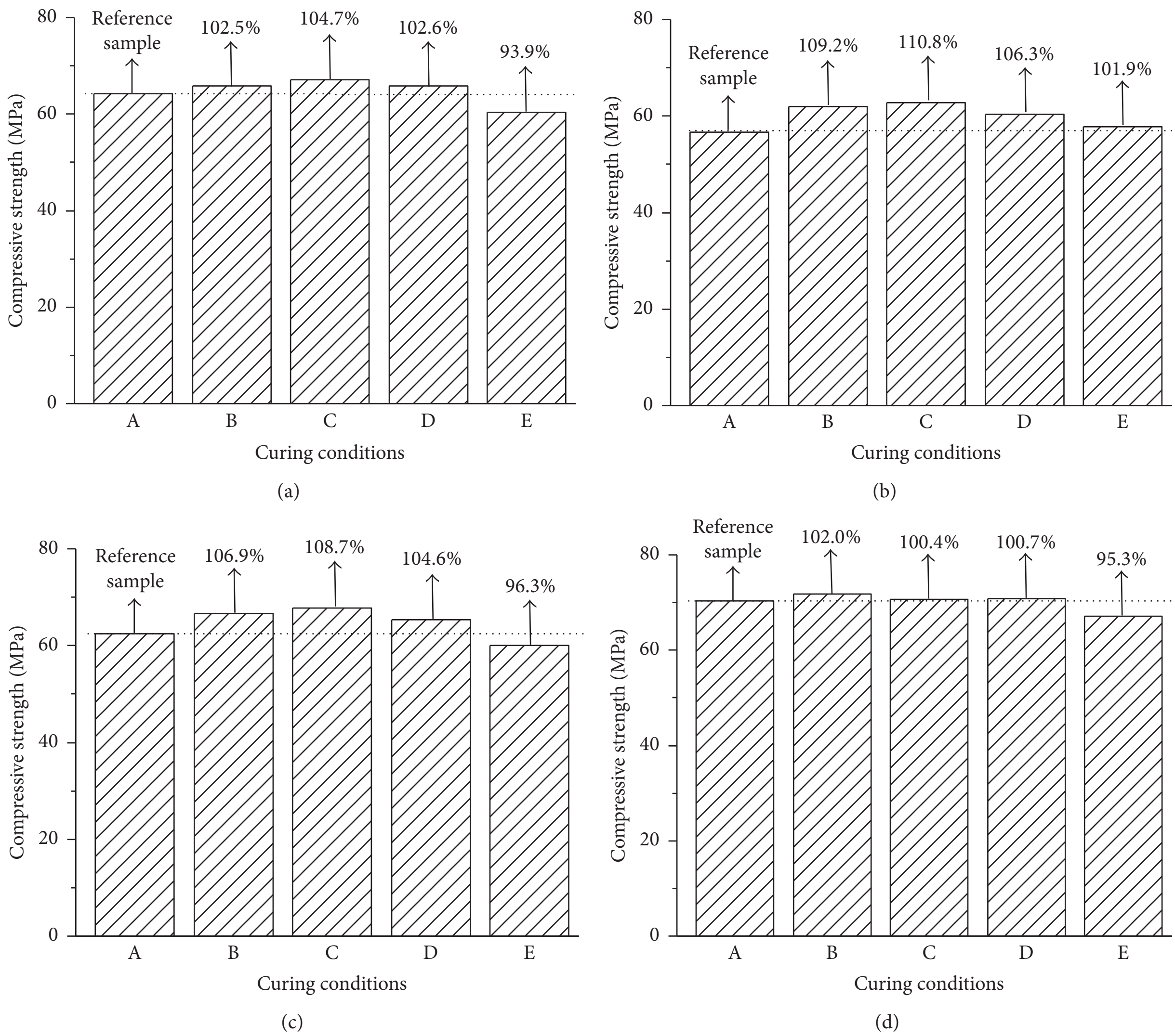

FIGURE 4: Compressive strengths of the concretes cured under different curing conditions at the age of 90 days. Samples: (a) plain cement, (b) 45% FA, (c) 45% GGBS, and (d) 12% DSF.

and it normally takes two or even more days for the concrete to reach its peak temperature from the casting temperature. Therefore, when the peak temperature inside the concrete is not too high (near or slightly higher than 45°C), the late-age compressive strength of the concrete will not be lower, even possibly higher if fly ash or GGBS is added, than that obtained under standard curing.

3.3. Chloride Ion Permeability. The charge passed values of four different types of concrete at the ages of 90 and 360 days are shown in Figures 6 and 7, respectively. According to ASTM C1202, concretes have the same grade of chloride ion permeability if the charge passed values fall in the same interval. Figure 6 shows that for each concrete, the permeability levels under the five different curing conditions are the same at the age of 90 days. This indicates that for each concrete increasing curing temperature up to 45°C has little influence on its 90 days' chloride ion permeability regardless of the precuring time and high-temperature curing time. The charge passed values of plain cement concrete and cement-silica fume concrete under curing condition "E" (high-temperature curing immediately after preparation) are obviously higher than those obtained under standard curing conditions, although the values correspond to the same permeability level.

Figure 7 shows that for plain cement concrete, cement-fly ash concrete, and cement-silica fume concrete, the chloride ion permeability levels under all the five curing conditions are the same at the age of 360 days. Though the charge passed values of the cement-silica fume concrete under curing conditions "B," "C," and "D" are a little higher than that under curing condition "A," the difference of chloride ion permeability can be neglected because the difference of charge passed value has very little influence on the permeability grade. For cement-GGBS concrete, the permeability level of concrete under curing condition "E" is the same with that under

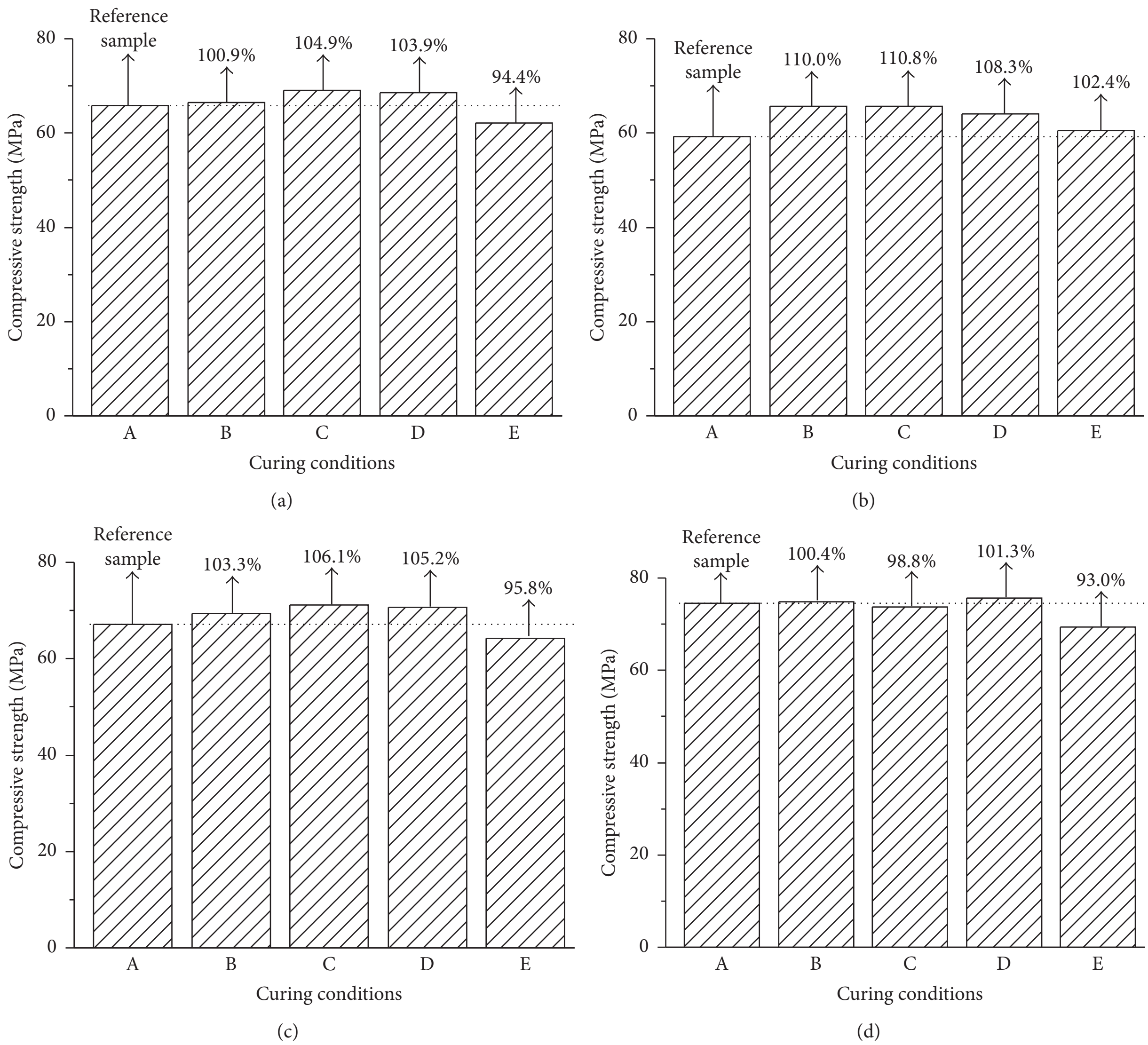

Figure 5: Compressive strength of the concretes cured under different curing conditions at the age of 360 days. Samples: (a) plain cement, (b) 45% FA, (c) 45% GGBS, and (d) 12% DSF.

curing condition "A" at the age of 360 days. However, the permeability level of the concrete under curing conditions "B," "C," and "D" is lower than that under curing condition "A." It is an indication that increasing curing temperature up to 45°C after a period of precuring tends to decrease permeability of cement-GGBS concrete at the age of 360 days. It can be inferred that for the cement-GGBS composite binder, the promoting effect of elevated temperate of 45°C on the reaction degree of GGBS is more significant than its negative effect on the hydration of cement.

It is widely accepted that the chloride ion permeability of concrete is closely related to its porosity and pore connectivity. The pozzolanic reaction of fly ash, GGBS, and silica fume can improve the pore structure at late ages (silica fume can improve the pore structure at an early age) [39]. From a comparison of the four concretes with different binders under standard curing condition, it is clear that the chloride ion permeability of the concrete containing mineral admixture is lower than that of the plain cement concrete. High-temperature curing at an early age can activate the activity of mineral admixtures and increase the reaction degree, thereby increasing the contribution to pore structure improvement. Figures 6 and 7 suggest that high-temperature curing at 45°C has no adverse effect on the chloride ion permeability of plain cement concrete at late ages, let alone on that of the concrete containing mineral admixtures. Moreover, high-temperature curing at 45°C can decrease the chloride ion permeability of concrete containing GGBS.

It can be concluded from Figures 6 and 7 that, for the concrete component used in engineering practices with an inner temperature near or slightly higher than 45°C, temperature rise at an early age has no negative effect on its

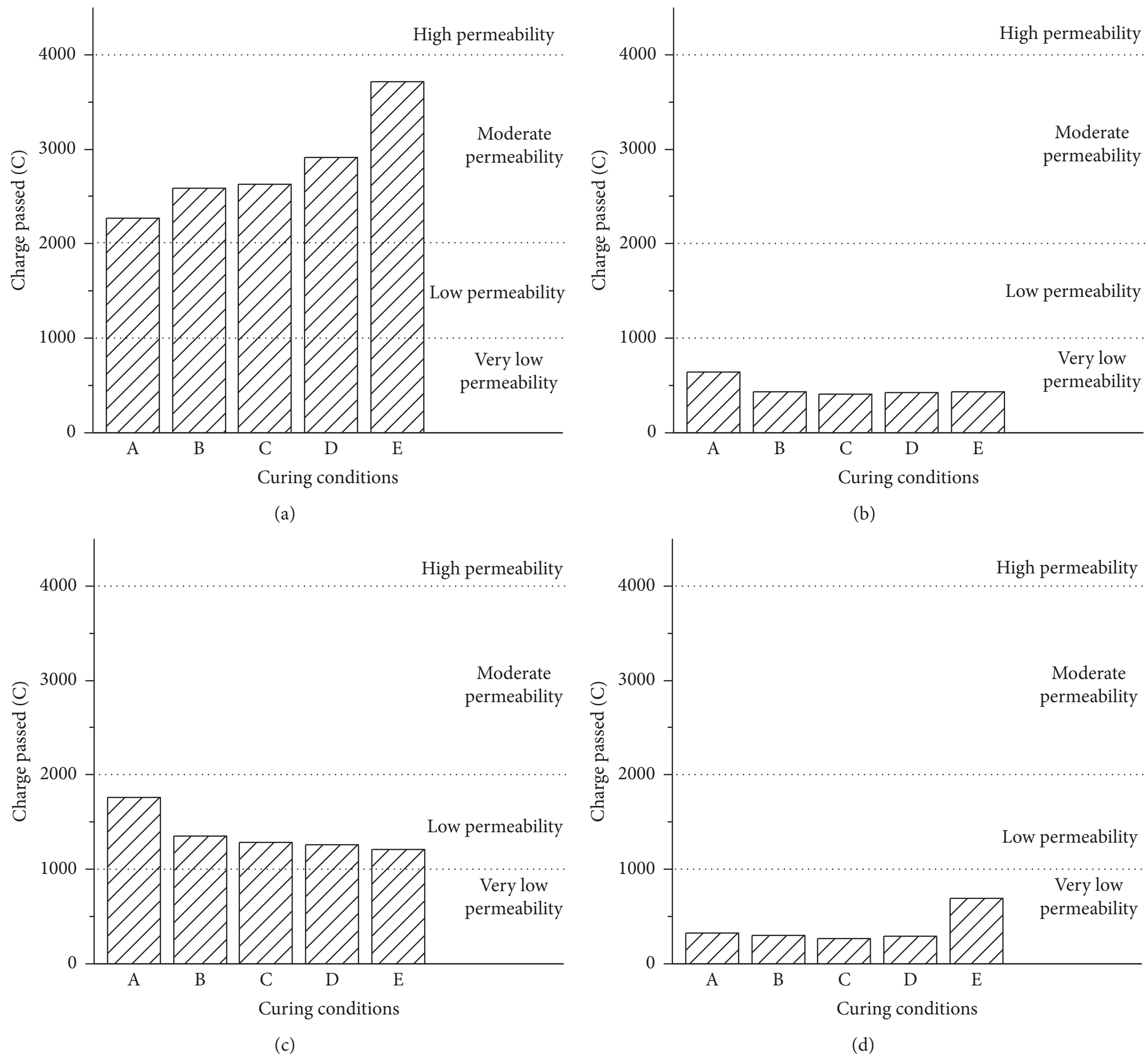

Figure 6: The chloride ion permeability of concrete at the age of 90 days. Samples: (a) plain cement, (b) 45% FA, (c) 45% GGBS, and (d) 12% DSF.

resistance to chloride ion permeability and may even have a positive effect on the concrete containing GGBS.

3.4. Connected Porosity. The connected porosity is one of the principal factors determining the penetration of aggressive agents and therefore the durability of concrete. It is an effective method to improve the resistance of concrete to chemical attack by reducing its connected porosity. The connected porosities of four concretes with different binders at the ages of 360 days are shown in Figure 8. It can be seen that mineral admixtures (fly ash, GGBS, and silica fume) can improve the pore structure of concrete at late ages, which is consistent with the results of the chloride ion permeability of concrete. For each sample, the connected porosity at elevated temperature without precuring is increased. It is an indication that elevated temperature of 45°C without precuring leads to a more heterogeneous distribution of the hydration products [29, 40, 41], which tends to increase the connected porosity. Wang et al. [42] found that the negative effect of elevated curing temperature on the concrete was greater than the hardened paste due to the deterioration of interfacial transition zone (ITZ) between hardened paste and aggregate. It is believed that the increase of porosity of ITZ due to the heterogeneous distribution of the hydration products contributes to the connected porosity variation of concrete at elevated curing temperature. However, if a period of precuring is set (whether 0.5 or 1 day), increasing the curing temperature up to 45°C tends to decrease the late-age connected porosity of each concrete. On the one hand, the negative effect of fast hydration

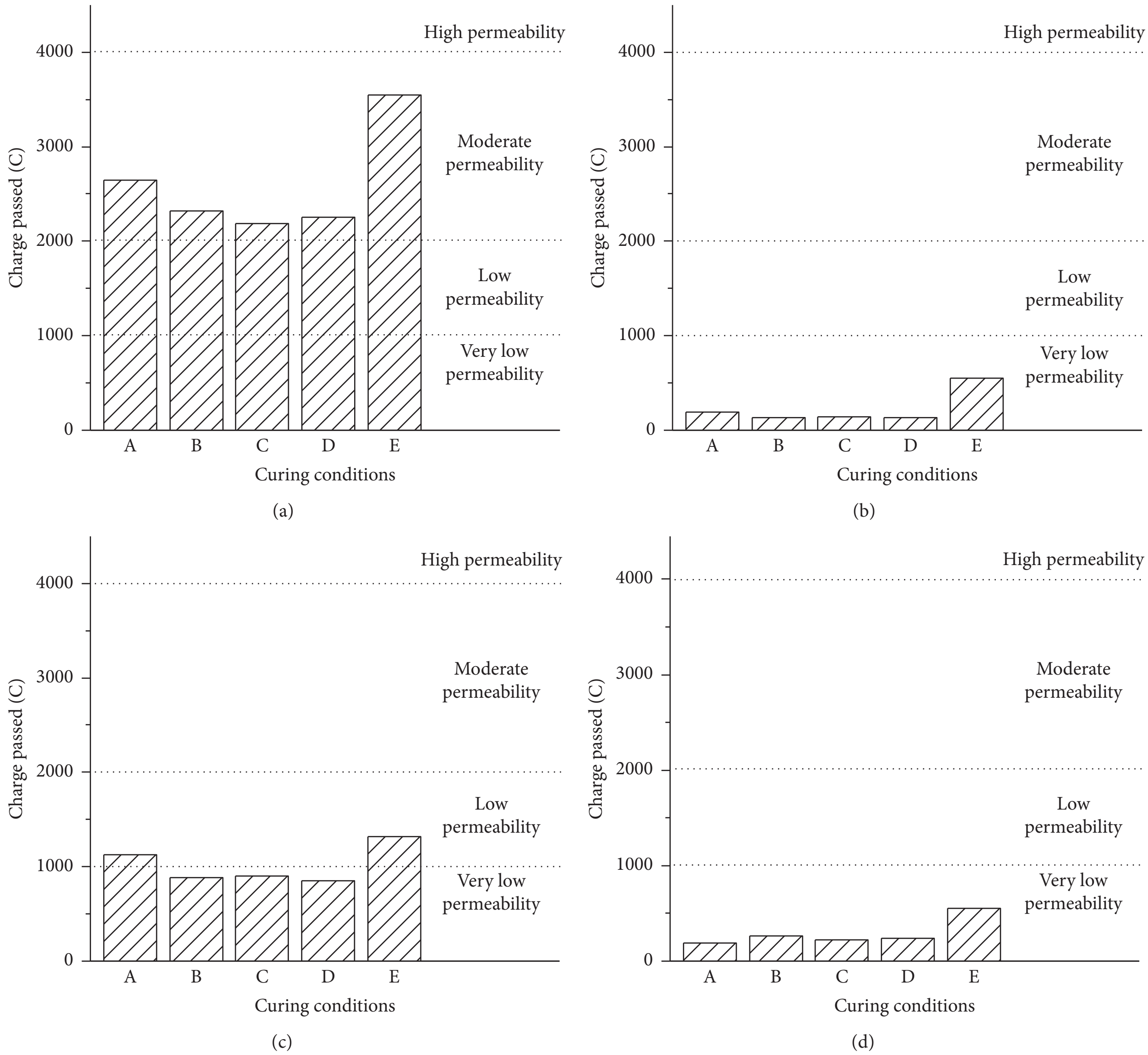

Figure 7: The chloride ion permeability of concrete at the age of 360 days. Samples: (a) plain cement, (b) 45% FA, (c) 45% GGBS, and (d) 12% DSF.

on the pore structure due to elevated curing temperature is reduced if a period of precuring is set; on the other hand, elevated curing temperature of 45°C tends to increase the late-age hydration degree of binder (Figures 2 and 3).

It can be concluded from Figure 8 that for the concrete component used in engineering practices with an inner temperature near or slightly higher than 45°C, temperature rise at an early age can improve the pore structure of concrete, especially for the concrete containing mineral admixtures. Generally speaking, it will enhance the durability of concrete.

4. Conclusions

(1) At late ages, the negative effect of elevated curing temperature of 45°C without precuring on cement hydration, pore structure, and strength of concrete cannot be neglected.

(2) With a period of precuring, increasing curing temperature up to 45°C has no negative effect on the late-age properties of concrete, and it may even enhance the strength and durability of concrete, especially the concrete containing fly ash and GGBS.

(3) For concrete components that are in engineering practices, in which the temperature rise is not very rapid and the peak temperature is near or slightly higher than 45°C, the temperature rise at an early age does not have any adverse effects on the late-age properties of concrete.

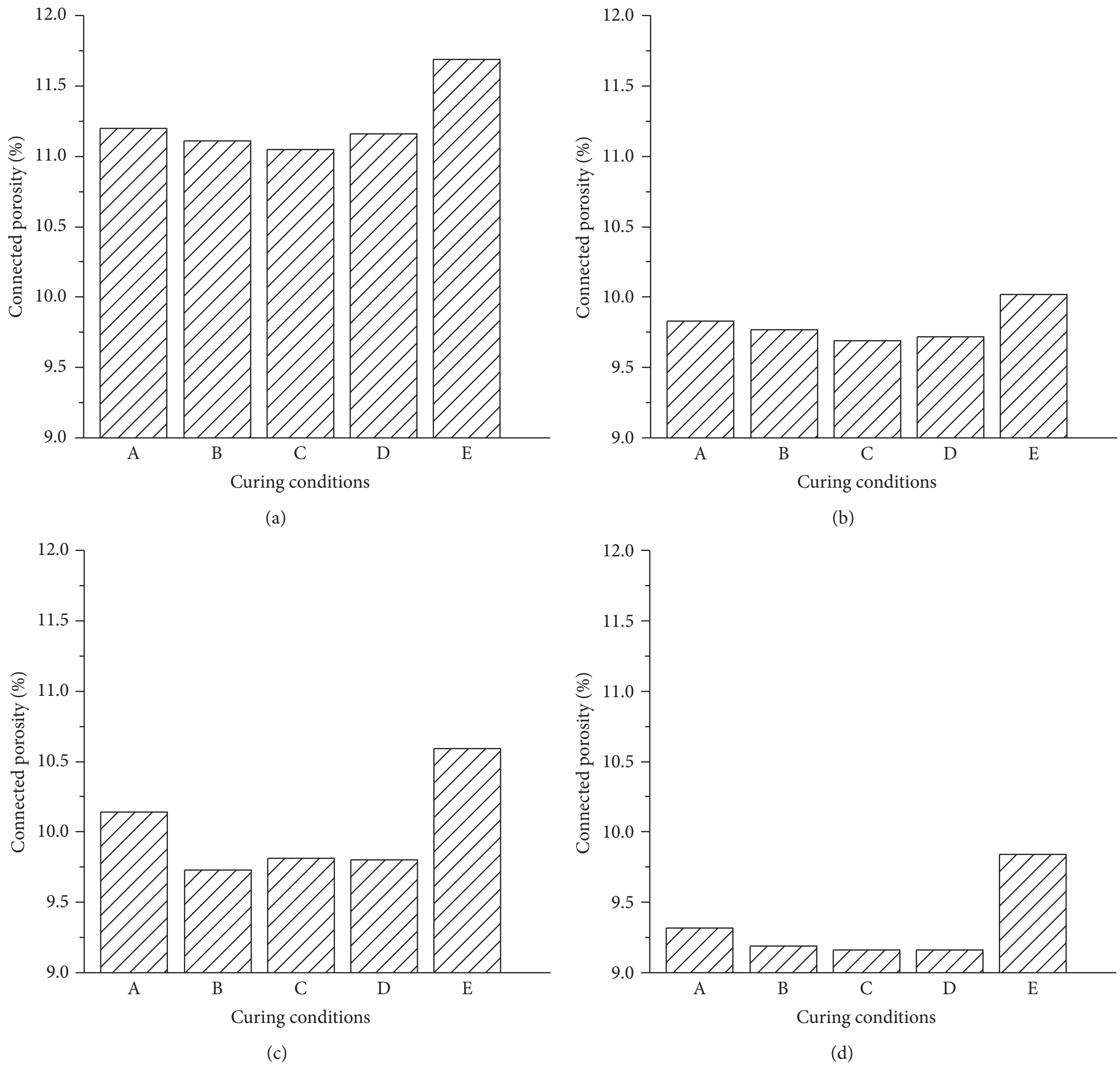

Figure 8: The connected porosity of concrete at the age of 360 days. Samples: (a) plain cement, (b) 45% FA, (c) 45% GGBS, and (d) 12% DSF.

Competing Interests

The author declares that he has no competing interests.

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