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ARTICLE

# Effects of Curing Methods on the Permeability and Mechanism of Cover Concrete

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## ABSTRACT

Curing methods are one of the most important factors in determining the quality and compactness of cover concrete. The effect of curing methods on the water absorption and sorptivity coefficient of cover concrete with the substitution ratio of fly ash (FA) and ground granulated blast slag (GGBS) for cement between 30 wt% and 40 wt% was studied by capillary water absorption test. The vacuum saturation test and mercury intrusion test were employed to characterize these differences in the pore structure of cover concrete under different curing methods. With further analysis of the compactness of microstructure by SEM, the mechanism of the impact of curing methods on the permeability of cover concrete was revealed. The results obtained indicate that the effect of curing methods on the water absorption, sorptivity coefficient and porosity of cover concrete shows the trend of natural curing > cover curing > water curing > standard curing. It is also shown that reasonable curing is advantageous to reduce the porosity and permeability of cover concrete. In natural curing conditions, the appearance of porosity increasing and pore structure coarsening is more critical for covering concrete with mineral admixtures than for pure cement concrete. Therefore, the permeability of cover concrete; Permeability; Capillary water absorption; Porosity

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# **1. Introduction**

Concrete curing is defined as the process of concrete conducting adequate hydration and continuous hardening by providing a suitable temperature and moisture <sup>[1]</sup>. Wet curing is critical to both strength and durability of concrete <sup>[2]</sup>, but the strength of concrete has not grown alongside durability. The test results obtained by Dinku and Reinhardt <sup>[3]</sup> showed that the sorptivity coefficient has a very good relevance to gas permeability.

The permeability is widely used as an index for the durability characterization of concrete, and is mainly affected by the combination of curing methods and mineral admixtures <sup>[4]</sup>. The application results show that the chloride-resistant permeability, carbonizing-resistant capability and freezing-resistant property of wet-cured concrete are superior to that of dry-cured concrete <sup>[5-7]</sup>. Liu indicated that the gas permeability of the mixtures prepared by replacing cement with 0%-50% by weight of slag is lower than pure cement mixtures <sup>[8]</sup>. Basheer and Nolan <sup>[9]</sup> reported that the moisture distribution within 30 mm of the concrete surface is highly susceptible to environmental changes. With premature exposure to wind and sun, the moisture contained in the cover concrete will evaporate rapidly, forming interconnected capillary channels, which reduces the impermeability of concrete <sup>[10]</sup>. Additionally, the interconnected pores and micro-cracks of cover concrete provide passage for moisture and corrosive media into the internal concrete, which is the chief cause of concrete cracks and steel corrosion [11,12].

Recent research on early-age curing regimes mainly focuses on the effects of curing method, curing temperature and curing humidity on the mechanical and durability of concrete <sup>[2,13-15]</sup>. Jiang <sup>[16]</sup> studied the effect of early-age curing temperature and thermal insulation on the 28 d compressive strength and microscopic properties of ultrahigh performance concrete (UHPC), the results have shown that the compressive strength and matrix density increase due to early curing temperature increment, and the thermal insulation curing could be used instead of the steam curing <sup>[17]</sup>. It has been observed that the early

strength of concrete increases significantly with the curing temperature <sup>[18,19]</sup>. It has been verified by some researchers that heat damage from steam curing has a disadvantageous influence on the durability evaluation of concrete <sup>[20,21]</sup>. Bai <sup>[22]</sup> investigated the linkage between capillary water absorption, strength and carbonization depth of composite cementitious concrete under dry curing and water curing conditions. It can be seen that the capillary water absorption and carbonization depth of dry-cured concrete increased and the strength decreased in comparison with concrete under water curing <sup>[23]</sup>. But this difference between the dry curing and water curing decreased with the metakaolin content, and increased with the fly ash content <sup>[24]</sup>. Capillary water absorption is able to characterize the ease of porous materials to transfer water through capillaries. Considering results showed of capillary transport of water as a function of curing methods, water-binder ratio and composition of materials, water absorption is correlated with the pores on concrete, especially for cover concrete <sup>[25,26]</sup>. Khatib and Mangat<sup>[27]</sup> reported the differences in water absorption of the concrete between the surface and the interior, and the results have shown that the water absorption of the top of the concrete cube is several times greater than that of the inside <sup>[28]</sup>. Nevertheless, the study of quantitative characterization of the effects of curing methods on the water absorption and the pore distribution of cover concrete has been reported rarely.

Against the above background, the study is dedicated to exploring and characterizing the effects of curing methods on the permeability of cover concrete with high-volume mineral admixture. Four curing methods including natural curing, cover curing, standard curing and water curing (various humidity conditions) and four mixtures (different cementing materials systems) were designed in this research. Water absorption and sorptivity coefficient measured by the capillary water absorption test were applied to quantitatively characterize the effect of curing methods on the permeability of cover concrete. Meanwhile, simultaneous total porosity and pore structure were analyzed to investigate how both can affect the permeability of cover concrete. Moreover, to further mechanism study, the thermo-gravimetric analysis (TGA) and scanning electron microscopy (SEM) were adopted to describe the hydration degree and microstructure of cover concrete. This plays an important role in providing data support for the durability evaluation of cover concrete prepared by highly adding a number of mineral admixtures.

# 2. Experimental programme

## 2.1 Raw materials and mix ratio

The cement used in this study is  $P \cdot O$  42.5 grade ordinary Portland cement (OPC) obtained from Shan Dong Wanhua Chemical Group Co., Ltd, China. The density of cement is about 3.15 g/cm<sup>3</sup>, and its specific surface area is 3370 cm<sup>2</sup>/g. Table 1 gives cement's physical properties. The fly ash (FA) of class I and ground granulated blast slag (GGBS) of grade S95 were used as replacement materials. The density and specific surface area of FA are 2.25 g/cm<sup>3</sup> and 3800  $\text{cm}^2/\text{g}$ , and that of GGBS is 2.89 g/cm<sup>3</sup> and 4400  $\text{cm}^2/\text{g}$ . Table 2 presents the chemical components of cement and mineral admixtures. Fine aggregate (S) is ISO Standard Sand that complied with ISO 679 Standard.

Four mixtures containing pure cement (marked as OPC), fly ash (30% by mass of cementitious materials, marked as FA 30), ground granulated blast slag (40% by mass of cementitious materials, marked as S40), and a combination of fly ash and slag (15% and 20% by mass of cementitious materials respectively, marked as FA15S20) were evaluated in the study. Table 3 presents the detailed proportions of four different mixtures. The binder-to-sand ratio (B/S) in all mixtures was the same and fixed at 0.5. As shown in Table 3, water-binder mass ratios (W/B) of 0.35 and 0.45 were designed for each mixture.

#### 2.2 Sample preparation and curing

According to the predesignated mix ratio, the fresh mixture was prepared and then cast into wood molds with dimensions of 200 mm  $\times$  200 mm  $\times$ 20 mm. The mold is made of wood plywood. The moisture and continuous casting do not affect the mold quality. After compacting, the slab samples were cured till the test timing of 7 days. Thanks to the addition of mineral admixture and relatively low water binder ratio, reduced bleeding tendency was presented. Immediately after demolding, the slab

Standard consistency water consumption		Setting time (min)		Fineness (%)		oundness	Flexural strength (MPa)		•	Compressive strength (MPa)	
(%)		Initial Final setting		80 μm		oununess	3 d 28 d		3 d	28 d	
28.4		205 2	260	0.5	Q	ualified	4.8	6.8	22.6	44.8	
			Table	2. Chemic	al compo	osition of bind	lers.				
Material	CaO	SiO <sub>2</sub>	Al	${}_{2}\mathbf{O}_{3}$	Fe <sub>2</sub> O <sub>3</sub>	<b>K</b> <sub>2</sub> <b>O</b>		SO <sub>3</sub>	Na <sub>2</sub> O	MgO	
Cement	60.00	23.20		90	2.58	0.88		3.97	0.28	1.74	
Fly ash	2.71	47.20	37	.62	4.55	1.15		2.06	0.49	2.59	
Slag	39.59	33.89	14	.22	0.91	0.64		2.96	0.36	6.43	
			Table	e <b>3.</b> The m	ixture pr	oportion desig	gn.				
Mix code	W/B	Cement (kg/m <sup>3</sup> ) F		Fly ash (kg/m <sup>3</sup> )		GGBS (kg/	'm <sup>3</sup> )	Sand (kg/m <sup>3</sup> )	Wat	ter (kg/m <sup>3</sup> )	
OPC	2636		0			0		5273	923		
FA30	0.35 or 0.45	1845		791		0		5273	923		
S40	0.55 01 0.45	1582 0		0		1054		5273	923		
FA15S20		1714		395		527		5273	3 923		

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specimens from every mixture were subjected to the following four typical curing methods: (i) Natural curing (lack of moisture curing, samples were placed outside at the average temperature of 26.7 °C and the average relative humidity of 60%); (ii) cover curing (samples were sealed with plastic film to prevent moisture evaporation and placed indoors at the average temperature of 20 °C); (iii) standard curing (samples were cured in a curing room with the temperature at  $20 \pm 2$  °C and relative humidity at 98%); (iv) water curing (the samples were demolded and then performed in a water tank filled tap water at the temperature of 20 °C).

#### 2.3 Experimental methods

#### Capillary water absorption test

The quality of curing has a direct impact on the permeability of covered concrete. The specimens with a dimension of 200 mm  $\times$  200 mm  $\times$  20 mm were employed to simulate the surface of concrete for the test of capillary water absorption and characterization of permeability. According to ASTM C 1585-04<sup>[29]</sup>, the capillary water absorption test was carried out as shown in Figure 1. At first, the samples of the specified age were dried at 105 °C until constant weight. Then, the lower surface of 200 mm  $\times$ 200 mm of samples was contacted with water, and the bottom of the sample was exposed to water at approximately 3 mm, to keep a constant height of water level. Meanwhile, the mass of the specimens was recorded every 5 min during the initial process of water absorption, and later recorded every 30 min for 1660 min.

The water absorption *I* and sorptivity coefficient *S* were calculated as:

$$I = \frac{(i_i - i_1)}{\rho_w A} = S\sqrt{t}$$
(1)

where *I* is water absorption, mm;  $(i_t - i_1)$  is the mass of water adsorbed in, g;  $P_w$  is the density of water, and the value is 0.001 g/mm<sup>3</sup>; *A* is the cross-section of the sample exposed to water, mm<sup>2</sup>; *S* is a sorptivity coefficient, mm/min<sup>0.5</sup>; *t* is time, min.

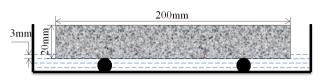


Figure 1. Schematic diagram of capillary water absorption test.

#### Vacuum saturation test

After measuring the water absorption, the vacuum saturation test was adopted immediately to determine the total porosity of cover concrete. Due to the significant impact of curing methods on the porosity of cover concrete <sup>[30]</sup>, the plate-type specimen was directly used for testing to better characterize the actual structure. The specimens conducted vacuum accelerated saturated water and marked the mass of the specimen after vacuum water saturation as  $m_s$ . Then, the specimens were dried at 105 °C for 12 h, and marked the mass of the completely dry sample as  $m_d$ . The value of total porosity *P* can be calculated by following:

$$P = \frac{m_s - m_d}{\rho_w V} \tag{2}$$

where  $(m_s - m_d)$  is the mass change of sample, g;  $\rho_w$  is the density of water; *V* is the volume of the specimen, mm<sup>3</sup>.

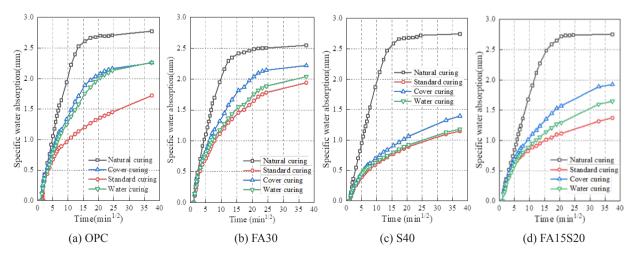
#### Microscopic test

After the specimen was broken, specimens of the surface layer were selected, soaked in absolute anhydrous ethanol for 24 h to stop further hydration. Then thermo-gravimetric analysis (TGA), mercury intrusion porosimetry (MIP) and scanning electron microscopy (SEM) were conducted for the microscopic test, respectively. Fresh particles were heated from a setting temperature of 20 °C to 1000 °C with a rate of 10 °C/min by the TGA-DSC I type thermal analyzer. The Auto Pore IV9500 mercury porosimeter was used to measure the pore structure of cover concrete with a maximum pressure of 228 MPa and a contact angle of 130°. The micro-morphology of samples was observed by Nova Nano SEM-50 scanning electron microscope at a scanning frequency of 50/60 Hz.

# **3** Results and discussion

## **3.1 Influence of curing methods on water ab**sorption of cover concrete

The changes in water absorption of cover concrete with capillary absorption time, curing method and water binder ratio are shown in Figure 2 and Figure 3. As the absorbing water time increases, the water absorption of cover concrete increases significantly, but the later growth decreases slowly, which is in accordance with the process followed by a bilinear change of capillary water absorption described by Liu's results <sup>[31]</sup>. The capillary water absorption of all samples with wet-cured methods such as standard curing and water curing is higher than that of drycured methods like natural curing and cover curing, which shows that the permeability of cover concrete to a large extent is governed by its curing method. Under natural conditions, in a low humidity environment (below 60% RH), the free water in covered concrete is easy to be evaporated, resulting in water moving into the air and forming water loss channels. The water absorption of standard-cured concrete is minimum, followed by water curing, and the water absorption of cover curing is less than that of natural curing. After de-moulding, unhydrated cement particles will further hydration, through timely and effective wet curing, replenishing rapidly the water consumed by hydration in the matrix, which is beneficial to improve the hydration degree and compactness of cover concrete. This indicated that a suitable curing method is detrimental to the permeability of cover concrete. It is worth noting that the water absorption of samples with water curing keeps higher than that with standard curing. The hydrate is alkaline, and the cement hydration products are dissolved because of the corrosion of water. Especially for Ca(OH)<sub>2</sub> with large solubility, it is dissolved first, resulting in the formation of more pores, thereby reducing the impermeability of cover concrete <sup>[32,33]</sup>. Furthermore, water absorption of samples tends to have results affected by the water binder ratio and the type of cementing material. Comparing Figure 2 and Figure 3, it can be observed that the water absorption of the 0.45 water-binder ratio is greater than that of the 0.35 water-binder ratio. The curing methods affect the water absorption to different degrees for diverse types of cementing material. For S40 samples, water absorption of natural curing is increased by 37%-60% as compared to that of standard curing. For OPC samples, water absorption of natural curing is 15%-40% higher than that of standard curing. The extent to which different cementing materials endure in the erosion process varies, and cementing material system with high-volume slag has a high resistance to water erosion <sup>[34]</sup>. Owe to high pozzolanic reactivity, slag reacts with Ca(OH)<sub>2</sub> in the matrix to generate C-S-H, which results in a significant decrease in the content of Ca(OH)<sub>2</sub> in the matrix, avoiding a large amount of Ca(OH)<sub>2</sub> dissolution.



It is found that the curve of water absorption for

Figure 2. Influence of the curing methods on the 7-day water absorption of specimens when water-binder ratio is 0.35.

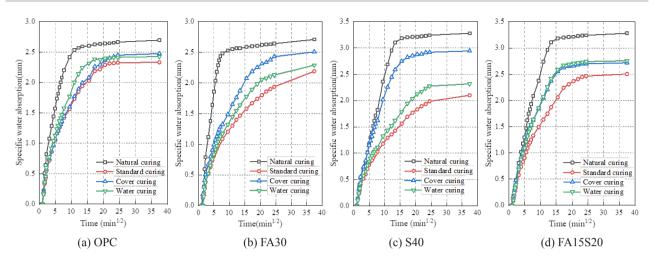


Figure 3. Influence of the curing methods on the 7-day water absorption of specimens when water-binder ratio is 0.45.

all samples shows a two-stage change. Therefore, the sorptivity coefficient of the samples is calculated using the slopes of the water absorption/square root of time  $(t^{0.5})$  curves in the early stage to characterize the strength of permeability <sup>[35]</sup>. Figure 4 gives the sorptivity coefficient variation of cover concrete with different curing methods and water-binder ratios. As is shown in the picture, the sorptivity coefficients of cover concrete with a water binder ratio fixed at 0.45 are all greater than that of a water binder ratio fixed at 0.35. This phenomenon can be explained by the high water-binder ratio mixtures having a larger initial water-filled space, namely a large amount of porosity, which provides more paths for water to penetrate into the matrix. The changes in sorptivity coefficients of different curing methods are the same as the water absorption, which manifests as

the trend of standard curing < water curing < cover curing < natural curing. Covering concrete with different curing methods has a great influence on the sorptivity coefficient, particularly in FA30, and the average value of the sorptivity coefficient of FA30 with standard curing is 1/2-3/5 of that with natural curing conditions. However, compared with pure cement, the cover concrete of FA30 has a higher sorptivity coefficient under all curing methods. When the water-binder ratio is 0.35, the addition of slag can significantly reduce the sorptivity coefficient of cover concrete, while when the water-binder ratio is 0.45, there is no significant difference in sorptivity coefficient between the OPC and the S40. Since the sorptivity coefficient of cover concrete is greatly impacted by its hydration degree and water-binder ratio, according to the mixture ratio, TGA measure-

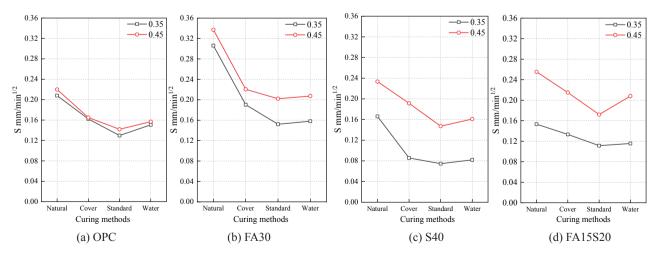


Figure 4. Influence of the curing methods on the 7-day sorptivity coefficient.

ment was applied to test the hydration properties, and the results are shown in **Figure 5**.

The DTG curves of the samples at 7 days are presented in Figure 5, and several typical peaks can be observed in DTG curves. The first endothermic peak existing at about 60-300 °C is attributed to the water loss and disintegration of ettringite and C-S-H gel. One peak occurring between 400-550 °C is caused by the decomposition of  $Ca(OH)_2$ , and the weight loss of a small amount of calcite appeared at 700 °C <sup>[10]</sup>. As can be seen in Figure 5a cement blended with slag has more hydration products, and results in a higher degree of hydration, so the hydrated body is more compact. That explains why the sorptivity coefficient of cement mixed with slag is half that of pure cement. In contrast, the content of hydration products of cement mixed with fly ash is lower due to the poor reactivity of fly ash <sup>[36]</sup>, which

FA15S20

416.89

leads to showing a higher water absorption and low impermeability in the capillary water absorption test. However, research has shown that the sorptivity coefficient of cement blended with fly ash continues to decrease with the prolongation of curing time and the development of the pozzolanic reaction <sup>[37]</sup>. In addition, according to the calculated Ca(OH)<sub>2</sub> content data in Table 4, it can be observed that with the increase of the water binder ratio, the amount of Ca(OH)<sub>2</sub> increases. At a constant water binder ratio, different cementing material systems display various permeability characteristics, and the main difference comes from the different hydration degrees. But it is difficult to explain the change of water absorption between samples with various water-binder ratios, so it needs to be described through the pore structure for further analysis.

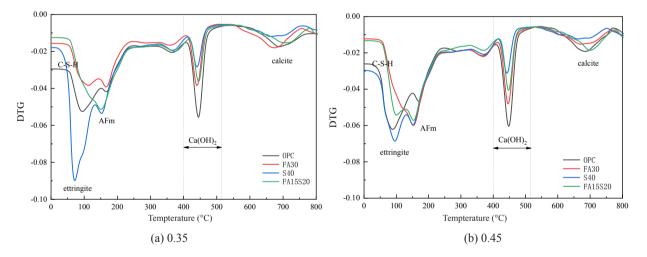


Figure 5. DTG curves of specimen at 7 days.

W/C		Endotherr	Endothermic peak/°C		ding weight /%	Weight loss(H <sub>2</sub> O)/%	CH/%
	code	Inital	Final	$W_1$	$W_2$	$W_1 - W_2$	$W_{\mathrm{CH}}$
0.35	OPC	407.95	504.63	89.97	87.61	2.36	9.70
	FA30	407.78	504.67	91.97	90.34	1.63	6.70
	S40	407.78	504.43	87.56	86.23	1.33	5.47
	FA15S20	417.95	514.55	90.40	88.87	1.53	6.29
	OPC	408.10	543.31	88.73	85.88	2.85	11.76
0.45	FA30	408.26	533.86	90.38	88.24	2.14	9.04
	S40	415.33	523.96	87.73	86.36	1.37	5.63

89.63

523.92

Table 4. Calculation value of Ca(OH)<sub>2</sub> content for different specimens.

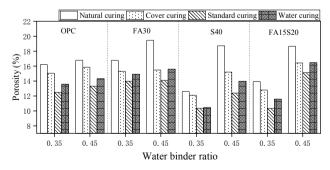
87.92

1.72

7.07

#### **3.2 Influence of curing methods on pore struc**ture of cover concrete

Some studies have shown that the permeability of cover concrete is directly related to its pore structure <sup>[38,39]</sup>. The 7-day total porosity from the exposed surface of the specimens measured by the vacuum saturation test under different curing methods is shown in Figure 6. It is easy to find that the porosity of the mixtures with a 0.45 water-binder ratio is significantly greater than that with a 0.35 water-binder ratio. This explains why increasing the water-binder ratio from 0.35 to 0.45 results in the water absorption and sorptivity coefficient of cover concrete increases. Besides, the curing method has a significant effect on the porosity of the specimens. The standard-cured cover concrete is the lowest, followed by the cover curing and water curing, and natural-cured cover concrete has the highest porosity, which indicates that proper curing methods can reduce the total porosity of cover concrete, thereby reducing permeability. However, the experimental results also show that the influential level of the curing method is distinct from the total porosity of different cementing material systems. For cover concrete of pure cement, the total porosity of natural curing is about 17%, which is higher than that of wet curing by 25%; for cement blended with fly ash, the total porosity of natural curing is higher than that of wet curing by 20%-40%; for slag, it is higher about 20%-50%. Thus, the pore structure development of concrete with high-volume mineral admixtures is more sensitive to the curing methods.



**Figure 6.** The 7-day total porosity of specimens with different curing methods.

The pore size distribution of cover concrete with

different curing methods is characterized by MIP and is presented in Figure 7. As shown in Figure 7, the pore size is mostly distributed below 50 nm, which is defined as harmless pores and less harmful pores, having little impact on the permeability of the cover concrete. It is proved that the addition of high-volume mineral admixtures tends to increase the maximum pore size of the sample. The pore size of pure cement is mainly distributed in 7-14 nm, and that of cement blended with fly ash and slag is mainly distributed in 11-17 nm and 6-32 nm. Comparing the pore structure of specimens under standard curing and natural curing methods, it can be seen that the proportion of harmless pores and less harmful pores decreases. The pore size distribution curves of natural curing shift right, and as the proportion of macropore of the sample increases, the internal pore tends to be connected. This indicates that the loss of water in the sample of natural curing makes the pore structure of cover concrete much coarser, which is not conducive to the development of compactness. Especially for the cement mixed with slag, the pore structure coarsening of natural curing is the most significant.

### **3.3 Mechanism discussion on the permeabili**ty of cover concrete based on SEM

The curing method has a crucial influence on the microstructure development of cover concrete. For further study on the mechanism of the permeability of cover concrete, the microstructure of cover concrete under natural curing and standard curing was tested by SEM. Typical morphologies of cement main hydration products such as ettringite and Ca(OH)<sub>2</sub> cannot be identified from SEM images <sup>[40]</sup>, but it can find out the difference in the effect of curing methods on microscopic morphology. As can be seen from Figure 8, the cover concrete is loose and porous under natural curing, and the hydrate has lots of pores and micro-cracks, which are related directly to the process of water loss under the condition of low humidity. Compared with natural curing, the microstructure of standard-cured cover concrete is relatively compact and has no cracks, which explains

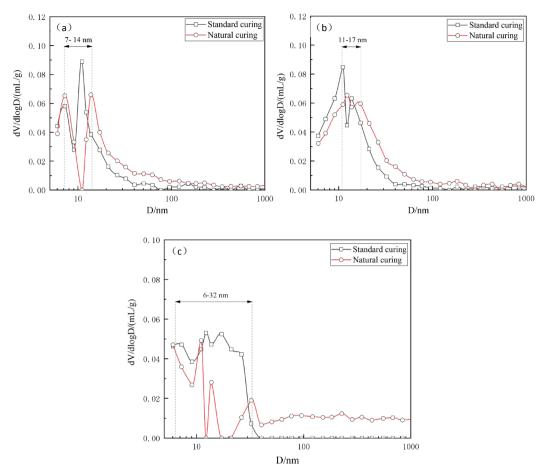


Figure 7. The pore structure of specimens with different curing methods: (a) OPC; (b) FA30; (c) S40.

why standard curing is beneficial to the improvement of impermeability at a micro level. Besides, cementing material type influences microscopic morphology. From the distribution of hydration products, the arrangement of hydration products of cement blended with mineral additive is more uniform and orderly due to the decrease of  $Ca(OH)_2$  content. From both images in **Figures 8b and 9b**, the hydrate of cement blended with fly ash has lots of micro-pores. In comparison, the micro morphologies of pure cement pastes and pastes containing slag are more compact, which is in accordance with the result of the higher sorptivity coefficient of the mixture with fly ash.

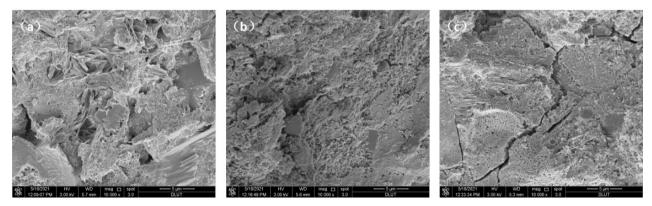


Figure 8. Morphologies of cover concrete with natural curing: (a) OPC; (b) FA30; (c) S40.

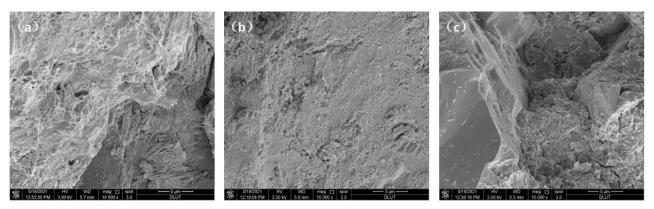


Figure 9. Morphologies of cover concrete with standard curing: (a) OPC; (b) FA30; (c) S40.

# 4. Conclusions

Based on the results and the discussion of water absorption, sorptivity coefficient, and pore structure of the cover concrete, the following conclusions are drawn:

(1) Strengthening curing has an obvious positive effect on water absorption of cover concrete. For four different curing methods, the water absorption and sorptivity coefficient of cover concrete with natural curing is the maximum, followed by cover curing and water curing, and standard curing is the maximum.

(2) There is a big difference in the water absorption property among the various cementing materials system. Under identical curing conditions, the FA30 performs higher with sorptivity coefficient when compared to the S40. The results obtained by TGA show that the early-age permeability of FA30 is the maximum, which is caused by the low pozzolanic reactivity of fly ash. As the activity of slag is higher than that of fly ash, the anti-permeability of cover concrete of S40 is considerably higher than that of FA30.

(3) There is a clear correlation between the pore structure and water absorption property. The porosity of cover concrete with natural curing is the maximum because of the increase in interconnected pores caused by moisture exchange, and the water absorption is correspondingly the highest. From SEM images, compared to natural curing, the matrix of standard curing is denser.

(4) Proper curing methods can improve the im-

permeability of cover concrete, and cement with mineral admixtures is more sensitive to curing methods. The reason is that the porosity and pore structure of samples mixed with mineral admixtures are more susceptible to curing methods than pure cement samples.

# **Conflict of Interest**

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/ or publication of this article.

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