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Effect of temperature on the hydration process and strength development in blends of Portland cement and activated coal gangue or fly ash*

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Abstract: This paper describes the results of an investigation into the effect of the variation of curing temperatures between 0 and 60 °C on the hydration process, pore structure variation, and compressive strength development of activated coal gangue-cement blend (ACGC). Hardened ACGC pastes cured for hydration periods from 1 to 360 d were examined using the non-evaporable water method, thermal analysis, mercury intrusion porosimetry, and mechanical testing. To evaluate the specific effect of activated coal gangue (ACG) as a supplementary cementing material (SCM), a fly ash-cement blend (FAC) was used as a control. Results show that raising the curing temperature accelerates pozzolanic reactions involving the SCMs, increasing the degree of hydration of the cement blends, and hence increasing the rate of improvement in strength. The effect of curing temperature on FAC is greater than that on ACGC. The pore structure of the hardened cement paste is improved by increasing the curing temperature up to 40 °C, but when the curing temperature reaches 60 °C, the changing nature of the pore structure leads to a decrease in strength. The correlation between compressive strength and the degree of hydration and porosity is linear in nature.

Key words: Activated coal gangue (ACG), Fly ash (FA), Blended cement, Hydration, Curing temperature, Strength
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1 Introduction

The use of industrial waste as supplementary cementing materials (SCMs) for Portland cement is increasing in popularity. These cement blends offer several advantages over pure cement, providing better properties, reducing environmental pollution, reducing production energy and CO_2 emissions. These benefits drive the search for new potential SCMs from industrial waste streams. Recent research shows that the coal gangue is a potential SCM, since it possesses a degree of pozzolanic activity after physical or chemical activation (Leng, 2000; Gong *et al*., 2005a; 2005b).

The hydration process, hydration mechanism, and the microstructure of activated coal gangue-cement blend (ACGC) have been investigated at room temperature (Hu *et al*., 2005; Wang *et al*., 2007; Liu and Wang, 2008; Zhang *et al*., 2010). The hydrate morphology variation at various curing temperatures has also been described (Liu *et al*., 2006). However, from an engineering viewpoint, the study of the use of activated coal gangue (ACG) in the manufacture of cement blends has been limited to laboratory scale due to the absence of experimental data that confirm the properties and stability of this matrix when manufactured under varying "real-life" environmental conditions. For example, there is still a lack of a clear understanding of the effect of curing temperature on the extent of the pozzolanic reaction in

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ACG and the consequent effect on strength development of ACGC.

Curing temperature is widely known to play an important role in the hydration, microstructure formation, and bulk properties of Portland and blended cements (Lothenbach *et al.*, 2007; 2008; Matschei and Glasser, 2010). Increased temperatures promote the hydration leading to high early strength development. In the later stages of curing, however, the strength of cements hydrated at increased temperatures can be lower than that of equivalent samples hydrated at room temperature. The evolution of hydrated phases at 60 °C causes microstructural changes, reducing the effect of these high temperature curings on the final performance of the cement. The relationship between porosity (total porosity, partial porosity in pore size interval between 1 and 0.1 μm, and partial porosity in pore size interval below 0.1 μm) and the degree of hydration of cement pastes containing metakaolin at 60 °C is defined less than that found at 20 °C curing regimes (Rojas and Sánchez de Rojas, 2005). It indicates that curing temperature has a direct effect on this correlation. The pozzolanic reaction involving SCMs is significantly accelerated by increasing curing temperature. The non-evaporable water (NEW) and calcium hydroxide (CH) contents show an inversion after extended curing although the levels increase with temperature in the early stages of curing (Escalante-garcía and Sharp, 2001; Haneharas *et al.*, 2001; Gómez-Zamorano and Escalante-García, 2010).

To characterize the effect of curing temperature on the final properties of ACGC and the relationship between the development of properties, degree of hydration, and microstructure, we examined the degree of hydration, structure variation, and strength development in ACGC over a range of temperatures from 0 to 60 °C for hydration periods of 1 to 360 d. A cement blend containing fly ash (FA) was used as a control.

2 Materials and methods

2.1 Materials

Portland cement was blended with the SCMs. The main SCM was ACG made by high temperature calcination from an experimental blend of coal gangue. This particular coal gangue has a high level of calcium achieved by the addition of certain proportions of limestone, gypsum, and fluorite (Li *et al.*, 2006). The ACG was then cooled and ground into powder. FA was used as a control SCM. The two SCMs were incorporated at 30% (w/w) with Portland cement, respectively. The two blended cements prepared were ACGC and FA-cement blend (FAC). The chemical compositions and particle size distributions of the two cement blends are given in Table 1 and Fig. 1. The physical properties of the two cement blends are given in Table 2.

2.2 Experimental procedures

The cement pastes were prepared with a water: binder ratio of 0.3. Cube shaped samples (20 mm×20 mm×20 mm) were cast in steel molds, and cured

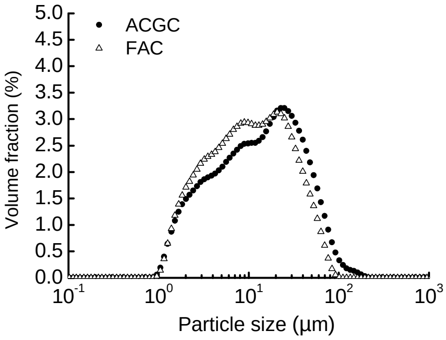

Fig. 1 Particle size distributions of ACGC and FAC

Table 2 Physical properties of ACGC and FAC

Material	Density (g/cm^3)	Specific surface area (m^2/kg)	Free-CaO (%, w/w)
ACGC	2.98	440	0.52
FAC	2.86	400	0.06

Table 1 Chemical compositions of ACGC and FAC

Material	Chemical composition (%, w/w)									LOI*
	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	SO_3	Na_2O	TiO_2	
ACGC	47.5	33.7	7.98	2.37	0.95	0.79	2.00	0.20	0.29	1.82
FAC	41.3	32.4	16.80	2.21	0.79	0.47	1.71	0.14	0.54	1.99

LOI: loss on ignition

in airtight sealed containers at 0, 20, 40, and 60 °C. The samples were removed from the molds after 1 d, and then cured at the experimental temperatures in airtight sealed containers. The compressive strength of the samples was determined at preset intervals.

Pieces of approximately 10 mm nominal size obtained after fracturing during the compressive strength test were subjected to drying treatment by washing with ethanol and then being placed in a vacuum oven at 45 °C for 24 h to stop the hydration reaction. To determine the porosity and pore size distributions using a mercury intrusion porosimeter (MIP), the dried pieces were reduced to approximately 2 mm nominal size. To determine the degree of hydration by the NEW method and the CH content with differential scanning calorimetry (DSC), the dried pieces were ground to pass 80 μm sieve.

The fully hydrated cement samples were prepared with an excess of water and extended curing after ultra-fine grinding. They were cured in airtight sealed containers at 0, 20, 40, and 60 °C, respectively. After 360 d, the hydration of these fully hydrated samples was stopped as described above. An X-ray diffraction (XRD) analysis showed that there were no clinker minerals remaining in the samples, demonstrating that fully hydrated samples at various curing temperatures had been obtained. The powder was then used to determine the NEW level by high temperature calcination.

The content of NEW at a certain curing time, relative to that in a fully hydrated paste of the same cement at the same curing temperature, was used as a measurement of the degree of hydration. The degree of hydration was hence defined as the degree of reaction of the blended cements at a certain curing time in comparison with the maximum extent of reaction under the same curing conditions.

3 Results and discussion

3.1 Degree of hydration

Fig. 2 shows the degree of hydration of ACGC and FAC in relation to the hydration time at various curing temperatures. The higher the curing temperature, the higher the degree of hydration. However, the difference in the degree of hydration between various curing temperatures is very small in the later stages of curing, having declined from the level of difference observed in the early stages of curing. This indicates that increasing curing temperature accelerates the reactions in the blended cements, especially the early hydration of cement clinker phases.

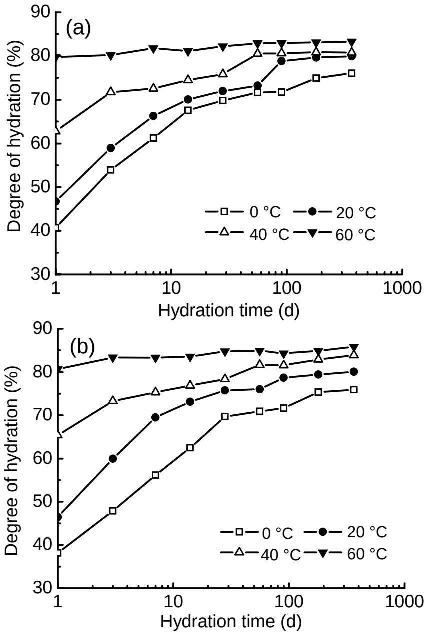

Fig. 2 Degree of hydration of ACGC (a) and FAC (b) over a range of curing temperatures

Since the pozzolanic reaction occurs in the later hydration period of ACGC and FAC, a secondary stage showing a fast increase in the degree of hydration, beginning at 90, 56, 28, and 14 d at curing temperatures of 0, 20, 40, and 60 °C, respectively. It can be seen that the onset of the secondary fast increase stage occurs earlier with the increase of curing temperatures, which indicates that higher curing temperatures accelerate the pozzolanic reactions in ACG and FA in the later stages of hydration.

When the curing temperature is low, the degree of hydration of FAC early in the curing process is lower than that of ACGC at the equivalent stage, whereas when the curing temperature is high the opposite occurs. This phenomenon is particularly evident at temperatures of 0 and 60 °C, indicating that the degree of hydration of FAC is affected by the curing temperature to a greater extent than that of ACGC. In summary, low temperatures delay, and high temperatures accelerate the degree of hydration of FAC to a greater extent than that observed in ACGC.

3.2 Calcium hydroxide content

In general, the CH found in the cement pastes is generated by the hydration of calcium silicate. CH is consumed by pozzolanic reactions involving SCMs such as ACG or FA. Hence, the CH content variation observed in the cement pastes is a combined effect of the hydration of cement clinker phases and the pozzolanic reactions involving SCMs. The CH content of the cement pastes decreases when the amount consumed is more than that generated. Therefore, the hydration process of cement clinker phases and the extent of the pozzolanic reactions of the SCMs can be investigated by examining the variation in the CH content.

As shown in Fig. 3, at 20 °C, the CH content in ACGC and FAC from 1 to 7 d increases with the hydration time, and then goes into a gradual decline. At 7 d, the CH content of ACGC and FAC are greater than 8% (w/w) and near 10% (w/w), and after 360 d are 7% (w/w) and 6% (w/w), respectively. It is clear that the increase in the CH content of ACGC up to 7 d is slower than that observed in FAC over the same period, which indicates that the pozzolanic reaction in ACG occurs earlier, and the reaction extent is higher than in FA. However, later in the curing, the pozzolanic effect of FA is more significant than that of ACG resulting in a lower CH content.

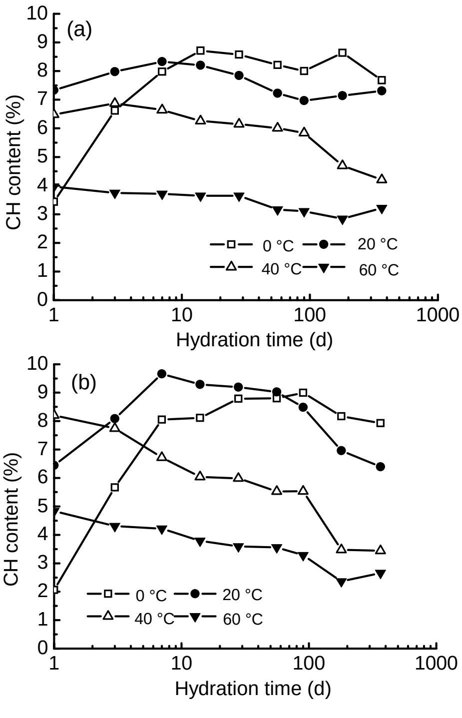

Fig. 3 CH content of ACGC (a) and FAC (b) over a range of curing temperatures

At 40 and 60 °C, the CH content of ACGC is lower than at 20 °C: the higher the curing temperature, the lower the CH content. In addition, raising the curing temperature brings forward the time at which the amount of CH generated by cement clinker hydration is less than that consumed by the pozzolanic reaction, that is, 7 d at 20 °C, 3 d at 40 °C, and 1 d at 60 °C. The accelerated consumption of CH indicates that the pozzolanic reaction of ACG is accelerated by increasing the temperature. The effect is not so marked in the clinker phases.

At 40 and 60 °C, the CH content of FAC hydrated for 1 d reaches a maximum, however, at 20 °C, the content is lower than that at 40 °C, and higher than that at 60 °C. That is because the pozzolanic reactivity of FA is lower than that of ACG during the early stages of curing, a curing temperature of 40 °C accelerates the hydration of cement clinker phases more than it does to the pozzolanic reaction of FA, but at a higher curing temperature (60 °C) the acceleration effect is more significant on the rate of reaction in FA. After 1 d, the CH content decreases gradually with the hydration time, below the level observed at 20 °C. The final CH contents of FAC at 40 and 60 °C are lower than those of ACGC. The higher the curing temperature, the more CH has been consumed. Therefore, raising the curing temperature accelerates the pozzolanic reaction of both ACG and FA, but the accelerating effect is more significant to the latter than to the former.

At 0 °C, the time for the CH content of ACGC to reach its maximum value of approximately 9% is 14 d, whereas for FAC it is 90 d. Compared with samples cured at 20 °C, this low curing temperature delays both the hydration of cement clinker phases and the pozzolanic reactions of ACG and FA. The effect on the latter is more significant than that on the former. This extends the time before the amount of CH generated by cement hydration is less than that consumed by pozzolanic reaction. The final CH content of ACGC is lower than that of FAC, which indicates that the pozzolanic reactivity of FA is delayed to a greater extent than that of ACG.

We can conclude that raising the curing temperature accelerates both the hydration of cement clinker phases and the pozzolanic reaction involving SCMs, and the effect is more significant on the latter than on the former. In addition, FAC is more significantly affected by curing temperature than ACGC. The low curing temperature will further delay and the high curing temperature will further accelerate the pozzolanic reaction of FA.

3.3 Pore structure

Mehta and Monteiro (1993) and Taylor (1997) suggested that pore sizes observed in hydrated cement pastes can be divided into four classes, and these pore size ranges can be correlated to paste properties (Table 3).

Table 3 Classification of pore size in hydrated cement paste and properties affected (Mehta and Monteiro, 1993; Taylor, 1997)

Designation	Pore diameter (nm)	Properties affected
Hydrated phases (gel) porosity	<10	Shrinkage and creep
Medium capillaries	10–50	Strength, permeability, creep and shrinkage at high relative humidity
Large capillaries	$50–10^3$	Strength and permeability
Large capillaries and entrained air	$>10^3$	Strength

Figs. 4–6 show the total porosities, mean pore diameters, and pore size distributions of ACGC and FAC after 360 d of hydration at various curing temperatures. The results show that in general, the total porosities and the mean pore diameters of ACGC and FAC decrease with an increase in curing temperature with the exception of observations made at 60 °C. This is because the proportion of gel pores to capillary pores increases with the degree of hydration as the curing temperature is increased. At 60 °C, however, the total porosities and the mean pore diameters of ACGC and FAC increase enormously as the proportion of gel pores decreases and that of large capillary pores increases to some critical value.

The increase in curing temperature accelerates the hydration of cement clinker phases and promotes the formation of more hydrates, filling the space

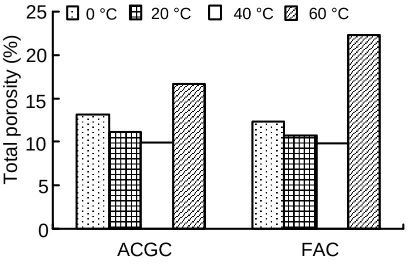

Fig. 4 Total porosities of ACGC and FAC over a range of curing temperatures

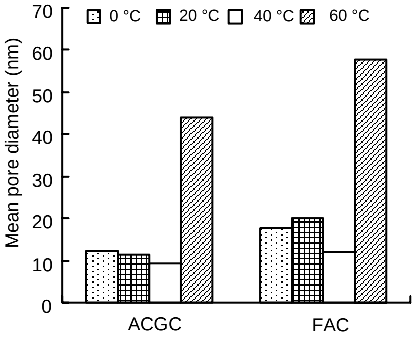

Fig. 5 Mean pore diameters of ACGC and FAC over a range of curing temperatures

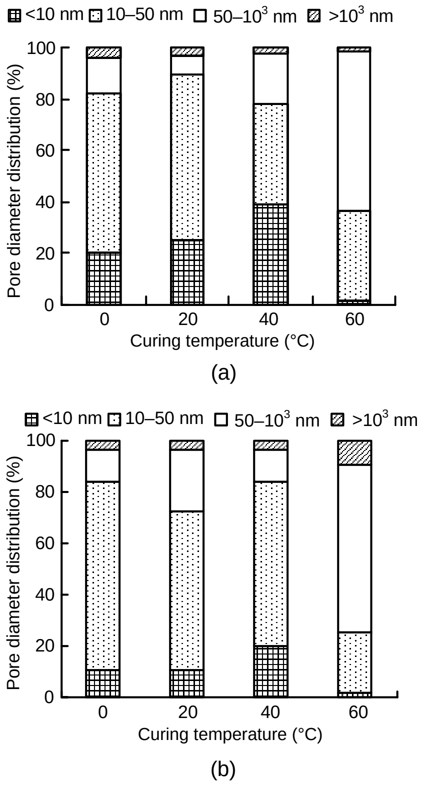

Fig. 6 Pore diameter distributions of ACGC (a) and FAC (b) over a range of curing temperatures

originally occupied by water. The reactions involving SCMs are accelerated by the increase in curing temperature to a greater extent than those of the clinker phases (Section 3.2), resulting in the formation of pozzolanic reaction products that fill the pores. In addition, the residue from the SCMs will act as microaggregates. Thus, the pore structure of the hardened cement paste is refined. However, if the curing temperature is too high, the hydrates grow too quickly, and consume a great amount of free water, resulting in an increase of large capillary pores and consequent pore structure deterioration.

Fig. 6 also shows that, except for the deterioration of the pore structure at 60 °C, the pore structure of ACGC is finer than that of FAC at 0, 20, and 40 °C. This indicates that the ACG modifies the pore structure of hardened cement paste to a greater extent than FA when cured below 40 °C.

3.4 Compressive strength

Fig. 7 shows the compressive strength of ACGC and FAC over a range of curing temperatures in relation to the hydration time. Basically, the higher the curing temperature, the higher the compressive strength, especially during the early stages of curing. The increased rate of compressive strength with hydration time varies over a range of curing temperatures. Note that in the later stages at 60 °C, the compressive strength is reduced.

The lower the curing temperature, the lower the early compressive strength of ACGC and FAC, but the faster the later compressive strength increases, which gradually narrows the gap between low temperature and high temperature. There is a reduction of compressive strength for ACGC at 60 °C after 56 d, and that for FAC at 60 °C is after 14 d. The compressive strengths of ACGC after 360 d in descending order are 40 °C (98 MPa), 20 °C (97 MPa), 0 °C (96 MPa) and 60 °C (85 MPa), and those of FAC are 20 °C (102 MPa), 40 °C (101 MPa), 0 °C (101 MPa), and 60 °C (93 MPa).

At 0, 20, and 40 °C, the early compressive strength of ACGC increases with increasing curing temperature, which is due to the accelerating effect of curing temperature on the hydration of blended cement. In addition, there is a secondary stage of fast increase of compressive strength due to the products formed by reaction between the active SiO_2 and Al_2O_3 in ACG with the CH. The higher the curing temperature, the sooner the secondary stage of fast increase occurs: after 14 d at 0 °C, 7 d at 20 °C, and 7 d at 40 °C. With the increase of curing temperature, the reactivity of ACG increases, and the time for onset of the pozzolanic reaction is advanced. A 60 °C curing temperature simultaneously accelerates the hydration of cement clinker phases and the pozzolanic reaction of ACG even in the early stages of curing, and the accelerating effect is more significant on the latter than on the former, resulting in no secondary stage of fast increase in compressive strength. As for FAC, the secondary stage of fast increase in compressive strength occurs at 0 and 20 °C between 14 and 56 d.

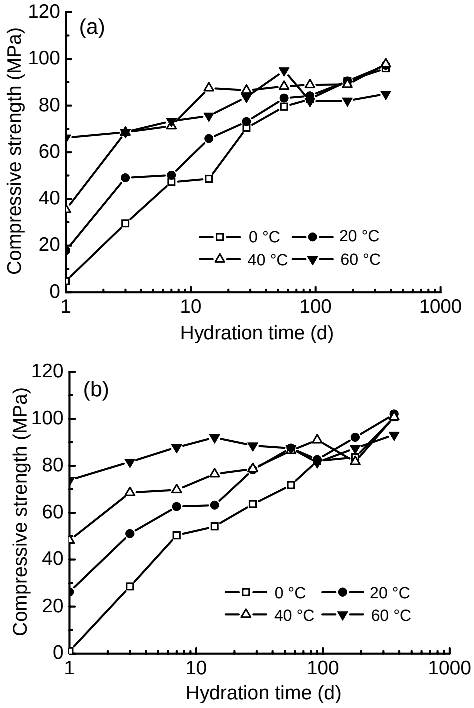

Fig. 7 Compressive strength development of ACGC (a) and FAC (b) over a range of curing temperatures

We can conclude that raising curing temperature accelerates the early reaction of cement clinker phases and SCMs and thus increases early strength, but probably at the expense of final strength. However, although the initial compressive strength is low at low curing temperatures, the final compressive strength may be high. The differences in strength development between ACGC and FAC are probably due to the degree of hydration and the microstructure. Since the low temperature delays, while the high temperature accelerates the hydration of FAC to a greater extent than in ACGC, the early compressive strength of FAC is lower than that of ACGC at 0 °C, but higher than that of ACGC at 20, 40, and 60 °C. Because the final extent of pozzolanic reaction of FA is higher than that of ACG (Section 3.2), and FA acts as a micro-aggregate more effectively than ACG in the hardened cement paste, then the final compressive strength of FAC is higher than that of ACGC.

3.5 Relationship between compressive strength and the degree of hydration

The hardened cement paste consists of hydrates with various morphologies and densities. The intrinsic strength of the dense, crystalline particles, and the bonding properties of ill-crystallized material generate bulk strength. Hence, the amount of hydrates plays a very important role in the strength development of hardened cement paste. The relative amount of hydrates to that in a fully hydrated paste of the same cement at various curing temperatures is expressed as the degree of hydration.

Using the values of the degree of hydration in conjunction with the values of compressive strength, it is possible to find a valid statistical equation that describes the relationship between the two parameters.

Fig. 8 shows the linear correlation between the degree of hydration and compressive strength over a range of curing temperatures for hydration periods from 1 to 360 d. The correlation coefficients for the curing temperatures of 0, 20, and 40 °C are all above 0.94, whereas the correlation coefficient for 60 °C is relatively low. Therefore, it can be inferred that at the curing temperature of about 40 °C and below, the compressive strength depends on the hydration degree. However, a curing temperature of 60 °C may affect the microstructure and damage the later compressive strength even though the degree of hydration is high.

3.6 Relationship between compressive strength and the porosity

Using the total, capillary, and gel porosity values in conjunction with the values of compressive strength, it is possible to find a valid statistical equation that describes the relationship between the two parameters.

Fig. 9 shows the linear correlation between total, capillary, and gel pore porosity versus compressive strength at various curing temperatures at a hydration time of 360 d. The correlation coefficients for the total, capillary, and gel porosity are near 0.8 and in some cases even above 0.9. The main adverse factor influencing strength was the formation of pores with a radius greater than 10 nm, while pores with a radius less than 10 nm were advantageous since gel pore formation is related to a certain amount of hydrates.

The effect of curing temperature on the final strength of hardened cement paste is related to a variety of physical factors. In addition to the amount of hydrate, the variety, morphology, and distribution of the hydrates, the thermal expansion coefficient and the relative strength of different phases, and the interface structure, etc., would also affect the bulk strength of the material in varying degrees. In various ways, the factors listed above are determined by the degree of hydration, phase compositions, and microstructure, which together, in turn, determine physical properties, including strength.

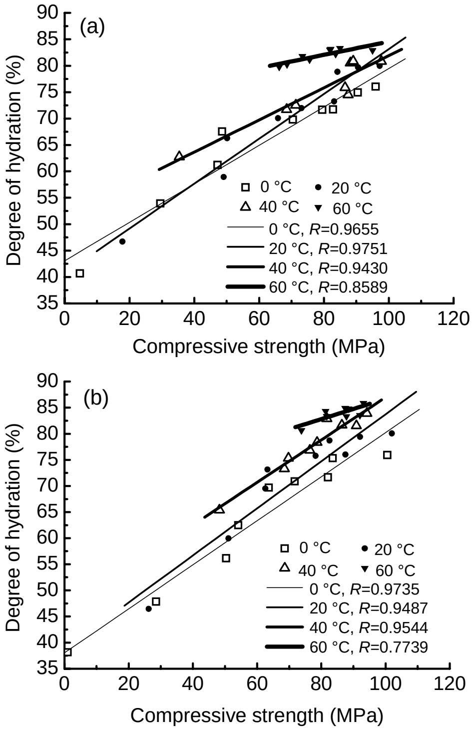

Fig. 8 Relationship between compressive strength and the degree of hydration over a range of temperatures for ACGC (a) and FAC (b)

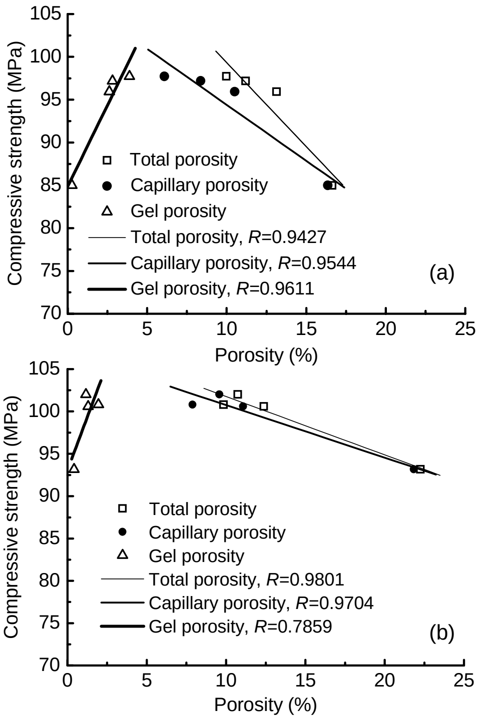

Fig. 9 Relationship between compressive strength and the porosity over a range of temperatures for ACGC (a) and FAC (b)

Raising the curing temperature to a certain extent has a beneficial effect on strength development due to the acceleration of the hydration of cement clinker phases and the pozzolanic reactions involving the SCMs, which results in an increased amount of hydrates and modified pore structure. However, the high curing temperature also has an adverse effect on the strength development due to the fast growth, large size, and inhomogeneous distribution of the hydrates, as well as the pore structure deterioration. The combined effects of both determine the final strength of hardened cement paste. At curing temperatures of approximately 40 °C and below, the beneficial effect is dominant. Hence, the compressive strength increases with increasing curing temperature. At a curing temperature of 60 °C, the situation is reversed.

4 Conclusions

1. Raising the curing temperature accelerates the hydration of cement clinker phases and the pozzolanic reaction of SCMs, hence increasing the overall hydration degree of the cement blends. The acceleration effect is more significant on SCMs than on cement clinker phases. The hydration of FAC is affected by curing temperature greater than that of ACGC.

2. The pore structure of ACGC and FAC is modified by raising the curing temperature except at a curing temperature level of 60 °C. The modification effect of ACG is more significant than that of FA. At 60 °C, the pore structure of both cements deteriorates.

3. Raising curing temperature increases early strength of the cement blends, but tends to decrease the final strength. However, although the initial compressive strength is low at a low curing temperature, the final compressive strength may be high.

4. Linear correlations between compressive strength and the degree of hydration, as well as between compressive strength and the porosity have been found. The increase of the degree of hydration with the increase in curing temperature increases the strength, but the increase in capillary porosity of the hardened cement paste decreases the strength.

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