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Properties and microstructure of alkali-activated slag cement cured at below- and about-normal temperature

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HIGHLIGHTS

- Alkali activated slag paste and mortar specimens were cured at 7–30 °C.
- Curing at below normal temperature retards the setting and shrinkage of AAS.
- Curing at below normal temperature has little influence on later strength.
- Paste becomes more compact with the increases of the curing temperature.
- AAS paste cured at below-normal temperature for long age can develop to highly compact structure.

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ABSTRACT

This work experimentally investigated the effects of curing temperatures (7, 15, 20 and 30 °C) on the setting time, strength development, shrinkage, and microstructure of alkali activated slag (AAS). The results indicated that the impact of the curing temperature was much more significant on the setting time of AAS than on that of ordinary Portland cement (OPC), and 7 °C curing could prolong the initial and final setting times of AAS without significant decrease of later strength, and increase the long-term strengths. The shrinkage of AAS were slightly reduced or retarded by curing at below normal temperature. There is no significant difference in the types of hydration products in the AAS pastes cured at 7–30 °C for 28 days, whereas the paste cured for same ages became more compact with lower porosity and finer pore size distribution as the curing temperature was increased.

1. Introduction

Alkali-activated slag (AAS) is characterized by high strength and high durability [1–7]. Using industrial by-products as the main raw material, AAS is not only conductive to environmental protection and energy saving, but also to improving the performance of concrete. However, as a cementitious material, its over-short setting time and high chemical and drying shrinkage seriously impede its application in civil engineering [1,8–10]. Besides, the retarding admixtures used for ordinary Portland cement are not effective or practicable for AAS, due to inefficient retarding or shrinkage-reducing effects, or severe adverse effect on strengths [11–16].

It is well known that temperature significantly influences the setting and hardening properties of cementitious materials [17]. Several researchers have studied the effects of elevated temperature curing on the properties of AAS. The results showed that the mechanical properties and durability of AAS concrete were significantly dependent on the curing conditions. Heat treatment is very effective to promote the early strength, while the strength at later ages will be reduced, especially when the curing temperature exceeds 80 °C [18–19]. Superior performance AAS concrete can be prepared when the concrete is cured at relative humidity of 80% and temperature of 60 °C [20], while mortars with the strength as high as 70 MPa can be produced with very low alkali content (2% Na_2O) under autoclave curing [21]. Similar work has also been done on other alkali activated cementitious materials [22–24].

Contrast to elevated temperature curing, it has been proved that curing at below-normal temperature retards the setting and hydration, and the strength development of ordinary Portland cement (OPC) [17,25]. However, little work has been done regarding the effect of curing at below-normal temperature on the properties of AAS. Brough [3] compared the strength of alkali-activated slag mortars using water–glass as the activator and cured at 5, 20 and 40 °C, and found that there was a strong influence of mixing/curing

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temperature upon strength, with significant retardation in strength development at lower temperature curing (5 °C) and significant acceleration at higher temperature (40 °C) curing. The variation was much larger than that seen for a typical OPC concrete. The sample cured at 5 °C was too weak to be demoulded at 1-day, although at 28-day it still gave reasonable strength of 41.0 MPa. The similar result was obtained by Altan [21], which indicated that when the AAS mortar was cured at 5 °C, the ultimate strength is limited to about a third of that cured at room temperature or above, although it can still gain strengths comparable to those of Portland cement mortars after be cured for several weeks. However, no more details have been reported. This suggests that there is not yet a full understanding of the impact of curing temperatures on the properties and microstructure of alkali-activated slag paste and mortar. Since temperature affects both setting and hardening properties of AAS, preparing and curing AAS at below-normal temperature is possible to retard the setting of AAS, which will improve its workability in practice; meanwhile, it may also has adverse effect on the strength development of AAS and other properties such as shrinkage. Works are still necessary to be done to ascertain whether casting and curing at below-normal temperature will significantly influence the mechanical properties of AAS. The present paper focuses on the effect of curing temperature, about and below normal, on the setting time, strength, shrinkage, hydrates, pore and microstructure of alkali-activated slag cement.

2. Experimental procedure

2.1. Materials

Grade S95 ground granulated blast-furnace slag (GGBS) according with GB/T 18046-2008, Grade P·II 42.5 ordinary Portland cement (OPC) conforming to the requirements of GB/175-2007, water–glass with the SiO_2/Na_2O modulus of 1.42, and sand conforming to ISO standard were used in the experiment. The chemical composition and physical properties of GGBS and OPC are shown in Table 1.

2.2. Setting time and strength test

Alkali-activated slag (AAS) was prepared with GGBS and water–glass, with the dosage of water–glass equivalent to Na_2O being 6% of the mass of GGBS. AAS and OPC pastes were prepared with the water-to-binder ratios of 0.3 for AAS and 0.27 for OPC, respectively. The initial and final setting times were tested, according to a method adapted from GB1346-2001, with the only modification in the preparing and curing temperatures. Cubic paste specimens of 40 × 40 × 40 mm and prismatic mortar specimens of 40 × 40 × 160 mm were prepared for strength test, with the binder-to-sand ratio for the mortars being 1:3. The specimens were cured with the moulds at designed temperatures of 7, 15, 20, 30 °C, respectively, with the relative humidity higher than 95% and demoulded after 24 h, and then cured in the same conditions mentioned above, respectively. The flexural and compressive strengths of the paste and mortar specimens were tested at different ages up to 90 days.

Table 1
Chemical composition and physical properties of GGBS and OPC.

Composition/property	GGBS	OPC
Chemical composition/wt.%		
SiO_2	33.70	21.44
Fe_2O_3	1.25	3.54
Al_2O_3	14.63	4.95
CaO	36.58	64.3
MgO	1.38	1.39
TiO_2	0.48	0.22
Na_2O	0.36	0.29
K_2O	0.58	0.69
SO_3	1.71	2.36
Loss on ignition	2.49	1.59
Density/g cm^{-3}	2.89	3.12
Blaine specific area/m^2 kg^{-1}	455	325
28-Day compressive strength/MPa	N/A	56.8
28-Day flexural strength/MPa	N/A	9.6

2.3. Shrinkage test

The mortar specimens with the binder-to-sand ratio of 1:2 and the dimension 25 × 25 × 280 mm were prepared for shrinkage test. The specimens were cured in the same conditions as those for strength tests and were demoulded at 2-day to ensure that the specimens cured at 7 °C would have got strong enough to be demoulded without fear of being broken. As soon as the specimens were demoulded, the lengths were measured by a comparator with the scale division of 0.01 mm, which were taken as the initial length of the specimens. The specimens were cured in drying chambers with the relative humidity of (50 ± 4)% and the temperatures of 7, 15, 20, and 30 °C, respectively, and the lengths were measured at scheduled ages. The shrinkages at each age were calculated according to,

$$\text{Shrinkage}\ (\%) = \frac{L_{initial} - L_t}{250} \times 100\%$$

where $L_{initial}$ (mm) is the length of the specimen when demoulded at 2-day, L_t (mm) is the length at t-day, and 250 (mm) is the effective length.

2.4. Hydrates and microstructure analyses

The paste specimens cured for 28 days were sampled and immersed in absolute alcohol for 48 h to terminate the hydration, then dried at 60 °C for 10 h. The hydrates and microstructure were characterized by means of X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), differential scanning calorimetry coupled with thermogravimetric analysis (DSC/TG), scanning electron microscope (SEM), and mercury intrusion porosimetry (MIP).

3. Results and discussion

3.1. Setting times

The initial and finial setting times of AAS and OPC pastes prepared and cured at different temperatures are shown in Fig. 1. As the curing temperature increased, the initial and finial times of both the AAS and OPC decreased. However, the significance of the influence on AAS and OPC is quite different. At normal temperatures, that is, at between 15 and 30 °C in this experiment, both the initial and final setting times of AAS are much shorter than those of OPC, and the initial times of AAS at 20 and 30 °C are shorter than 45 min, the lower limit required for ordinary Portland cement at 20 °C by GB 175-2007. The intervals between the initial and final setting times are very short, between 16 and 42 min, also much shorter than those of OPC at the same temperature range, which are between 40 and 100 min. When the pastes were cured at 7 °C, the setting time of both the two cements were significantly increased, and the degree of the increase is also much greater for that of AAS than for OPC. The initial setting time for AAS cured at 7 °C increased to 420 min, 10 times as long as that cured at 20 °C. The final setting time and the difference between the initial and final setting times also increased, of which the later expanded to 100 min.

In contrast, although the initial and final setting times of OPC also increased when cured at 7 °C, the initial setting is 290 min,

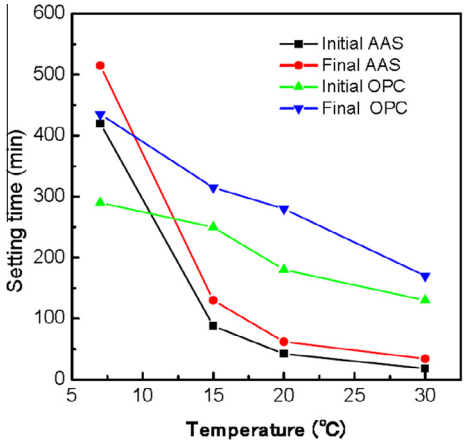

Fig. 1. Setting time of AAS and OPC cured at different temperatures.