

Comparison Study of Hydration Characteristics Between Cements with 50% and 85% Slag

Junwei SONG¹, Lu WANG^{1*}, Shuhua LIU^{1,2}, Jieli ZHU¹, Yong OUYANG¹

¹Nanchang Key Laboratory of Material and Structure Detection, Jiangxi University of Technology, Nanchang, 330098, China

²State Key Laboratory of Water Resources and Hydropower Engineering Science, Wuhan University, Wuhan, 430072, China

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This study aims at comparing the hydration characteristics between two kinds of cements, the Portland slag cement (PSC) containing 50 wt.% ground granulated blast furnace slag (GGBFS), and the supersulfated cement (SSC) containing 85 wt.% GGBFS. The reason caused the different hydration properties with the same type raw materials were analyzed by determining the compressive strength of the pastes, the hydration heat release and the hydration products at different curing ages. The results show that in PSC system, alkali activation was predominating and it mainly generated hydrated calcium silicate (C-S-H gels) and calcium hydroxide (CH). While in SSC system, the hydration mechanism was the combination of sulfate and alkali excitation, it mainly generated C-S-H gels and ettringite. The compressive strength of PSC was higher than that of SSC at every curing age. However, the difference of the compressive strength between PSC and SSC narrowed gradually as curing age went on. The early compressive strength of PSC was high while at late hydration age it increased slowly. The early compressive strength of SSC was low, while the later compressive strength increased quickly. Compared with PSC, the hydration heat evolution rate of SSC was slower and the hydration heat of SSC was relatively less low. Moreover, the second exothermic peak of SSC delayed.

Keywords: Portland slag cement, supersulfated cement, strength, microstructure, hydration characteristic.

1. INTRODUCTION

Ground granulated blast furnace slag (GGBFS) is a kind of mineral admixture with potential active, which is widely used in common cement to improve its physical and mechanical properties. As we know, after water quenching, slag remains in metastable state, which is a kind of amorphous glassy, with the composite of crystallization and glass phase. Portland Slag cement (PSC) containing 50% slag is popular used in modern concrete [1], while it also contains about 30%–40% cement clinker. Slag utilization of PSC is very high, while it still consumes large natural resources. The supersulfated cement (SSC) is roughly consists of 75%–85% slag, 5%–20% gypsum and less than 5% cement clinker [2]. The content of slag and gypsum in SSC are much higher than those in PSC, while the clinker content in SSC is far lower. The raw materials of the two cements are almost the same in chemical composition kinds, but the contents of each component are different, which might lead to some differences in hydration [3–4].

For SSC, the slag content is high up to 85%, which can recycle substantial by-products water-quenched slag produced by more steel industrials. At the same time, the sulfate content of SSC is very high, which can make large use of chemical industrial by-product phosphogypsum, to alleviate phosphogypsum stocking problem. Thus the environmental pollution and land occupation problem can be alleviated, which is demonstrate that SSC is superior to

PSC in the use of waste residue. As it is known, cement is one of indispensable cementitious materials in construction, as well as a kind of material with high energy consumption and high pollution. Each year, the emission of CO₂ on account of cement production is about 6%–7% of total carbon emissions, which brings serious environmental pollution problems [5]. Reducing the use of traditional cement or adopting new cementitious materials to replace clinker can alleviate this serious situation [6]. Cement clinker dosage in SSC is relatively low. Thus the preparation of SSC can reduce the use of cement clinker to a great extent, which is conducive to reduce greenhouse gas CO₂ emission and save the non-renewable resources coal mine.

The slag in PSC and SSC is identified as an alkali slag, which can provide active oxide and continue the reaction in later hydration. The hydration degree of PSC decreases with the increase of slag dosage, and the early hydration will consume more calcium hydroxide (CH) to maintain the reaction [7]. There is a variety of forms to stimulate the activity of alkali slag, and different activators will cause different hydration products and hydration processes [8–10]. The application of PSC is very common in China, and many researchers have studied on its hydration characteristics [11–14]. SSC has been applied in some parts in China in the 1950s and 1960s, but the related research and application are very rare [15–18]. Although some scholars have carried on the research and application in this area in recent years. For example, Zhao Qinglin has put forward the cement concrete development abroad [19] and used it in pavement concrete [20]. Some domestic and foreign scholars have studied in the hydration process of

* Corresponding author. Tel.: +86-791-88136778.
E-mail address: luwang@whu.edu.cn (Lu Wang)

SSC with different fineness [21, 22]. However, the hydration characteristics of SSC are still unclear, especially the differences between SSC and PSC. In this paper, the comparison of hydration characteristics between PSC and SSC is studied, from macroscopic to microcosmic, including strength, early hydration heat release characteristics, and hydration products, to reveal the difference of hydration properties between SSC and PSC, hoping to give a reference for the wide application of SSC in the future.

2. EXPERIMENTAL

2.1. Constituent materials

In this experiment, PSC containing 50 wt.% slag was produced by Huaxin cement plant, and SSC containing 85 wt.% slag, 12 wt.% gypsum and 3 wt.% clinker, which was prepared in laboratory. Among the raw materials, the XRD pattern of slag (Fig. 1), did not include any characteristic peaks.

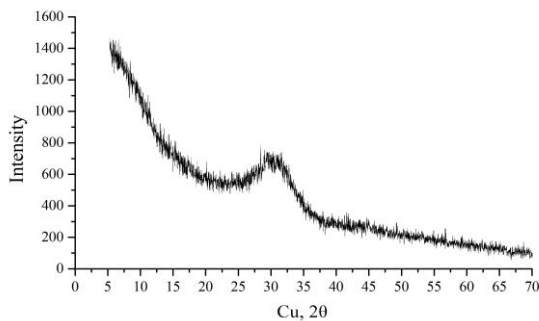


Fig. 1. XRD pattern of slag

Gypsum was adopted to neutralization phosphogypsum, which was neutralized with 4 % lime, hereinafter referred to as the phosphogypsum (PG). X-ray fluorescence (XRF) method was taken to test chemical compositions of PSC, SSC and PG. The results are shown in Table 1. From Table 1, the SO_3 content of PSC is 3.33 % (not more than 4.00 % control standard), the content of MgO is 4.25 % (not more than 6.0 % control standard), which is in accord with GB175-2007 about the relevant requirement of PSC. SO_3 content of SSC reaches up to 12.48 %, which is far beyond the standard of SO_3 content in PSC, and the CaO content is lower than that of PSC. For common cement, gypsum is used to control hydration rate in the hydration process and regulate setting time; while for SSC, gypsum is an important excitation

Table 1. Chemical composition of PSC, SSC and PG (mass %)

Samples	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	Fe ₂ O ₃	LOSS
PSC	0.21	4.25	10.21	26.47	0.07	3.33	0.87	51.27	2.05	1.27
SSC	0.24	5.38	11.63	25.81	0.10	12.48	0.57	41.25	1.18	1.36
PG	0.05	0.27	0.55	5.31	1.03	48.91	0.22	42.3	0.47	0.89

Table 2. Physical properties of PSC and SSC

Samples	Initial setting time, min	Final setting time, min	Fineness, m ² /kg	Water requirement of normal consistency, mass %
PSC	124	580	382	27.6
SSC	360	1680	375	26.5

agent. Basic properties of PSC and SSC are listed in Table 2. From Table 2, the fineness and water requirement of normal consistency are similar, while the initial and final setting time of PSC are earlier than those of SSC, which can explain why early concretion harden of SSC is slow.

2.2. Specimens preparation

Paste specimens (40 mm × 40 mm × 40 mm) from the two cements were molded with water to binder ratio of 0.4 under the standard test conditions with temperature control in 20 ± 1 °C, and relative humidity not less than 90 %, respectively. Then all specimens were placed in a standard curing box to maintain for some days. When curing to stipulated age of 3, 7, 28, and 90 days, the paste specimen respectively were moved to a series of tests, such as compressive strength and microscopic measurements.

2.3. Test methods

When curing to stipulated age, compressive strength of specimens was determined by electric hydraulic pressure testing machine with the type WAY-2000, and the specific test procedure is referred to GB/T17671-2005, method of testing cement- determination of strength.

Then the paste specimens were broken and the small samples in the central part of the specimens were selected to soke in anhydrous ethanol to terminate the hydration for microscopic measurements including X-ray diffraction technique (XRD) and scanning electron microscopy analysis (SEM). Among them, XRD method was used to determine crystal type and relative quantity of hydration products. The equipment used was D/MAX-RB model, using copper target and continuous scan. The samples were scanned from 5° to 70°(2θ range) at the rate of 0.02° per second. The samples used for the analysis were ground to produce particles below 80 μm size. SEM method was used to observe fractured surface of the hydration products, and the equipment is a scanning electron microscope model JSM-5610LV. The samples were fractured to expose the fresh surface after the test of compressive strength.

Early hydration heat and heat release rate of PSC and SSC with water to binder ratio of 0.4 were measured by a TAM Air isothermal micro-calorimeter with eight channels. For each measurement 10 g of dry sample were mixed by hand with a spatula for 1 min with 4 g of distilled water.

The weight accuracy was 0.001 g. The hydration reaction was carried out in a 20 mL ampere bottle at constant temperature of 20 °C.

3. RESULTS AND DISCUSSION

3.1. Hydration heat evolution

The hydration heat evolution and cumulative hydration heat of PSC and SSC within 72 h is shown in Fig. 2. Like the typical hydration heat evolution for common cement, those of both PSC and SSC exhibited five distinctive periods: (i) dissolution period; (ii) induction period; (iii) acceleration period; (iv) deceleration period; (v) steady period. Comparing to SSC, the induction period of PSC was relatively short, which might ascribe to the dissolution of calcium-rich phase, CH, silicate-rich phase and gypsum from cement clinker. This dissolution process happened within a short period of time. SSC contained slag of up to 85 % in weight, and slag was a kind of amorphous glassy, which was the composite of crystallization and glass phase. Among them, glass phase was the active component, and its content and structure played an important role in the activity of slag. Vitreous content in slag was about 90 %, composed by the network forming agent (mainly for SiO₂) and network change agent (mainly for K₂O, Na₂O, CaO and MgO), to balance the charge. Because of the silicon oxygen tetrahedron network, slag had the potential activity [23]. In common environment, it was hard for slag to react with water. So the induction period of SSC at early hydration age was very long. In the acceleration period, massive hydration products such as C-S-H gels and ettringite are generated, begins instantaneously once SO₄²⁻ and SiO₄⁴⁻ in the solution reach a high concentration. The second exothermic peak of PSC was a lot in advance compared with SSC, especially for its higher heat release rate within 30 h. At the same time, total heat release quantity of PSC is higher than that of SSC at any time.

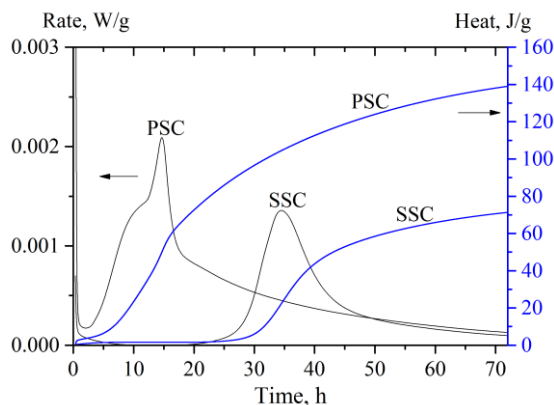


Fig. 2. Hydration heat and rate evolutions of PSC and SSC at 20 °C

From Fig. 2 the hydration heat release parameters of SSC and PSC can be obtained and the results are presented in Table 3 and Table 4. The ending time of induction period of PSC was 2.08 h, which was far earlier than 19.62 h of SSC. Because of the extreme low hydration heat release rate of PSC and SSC at the ending time of the

induction period, their cumulative heat quantity are both very low, as 3.69 J/g and 3.92 J/g. At the same time, the second exothermic peak time of PSC with 14.65 h was coming earlier than SSC with 25.44 h, and the corresponding heat release rate of PSC was higher than that of SSC with the corresponding heat quantity of PSC with 49.85 J/g twice of SSC with 22.78 J/g. The key influence factor on hydration heat rate of cement was the component content but not the type of raw materials. The lower the content of clinker, the lower heat release rate, so as the less heat. Table 4 shows the hydration heat of PSC and SSC within 24, 48 and 72 h, which represent the exact early hydration heat of SSC were lower than that of PSC all the time. During the period from 24 to 48 h, the increase of hydration heat of SSC was rapid, maybe as a period of increasing gradually and at last reach an acceleration period. While from 48 to 72 h its growth gradually reduces. Most of heat quantity of PSC was released within 24 h, the later hydration exothermic rate was smaller due to the second exothermic peak appeared within 24 h. Based on the analysis of the early hydration heat of two kinds of cement, SSC had the lower hydration heat than PSC all the time, and early hydration exothermic is slow. Compared with PSC, the early hydration rate of SSC was extremely low which results to less hydration products and incompact structure, and leading to the lower early strength.

Table 3. Hydration heat at the characteristic time

Samples	Ending time of induction period, h	Heat, J/g	The second exothermic peak, h	Heat, J/g
PSC	2.08	3.69	14.65	49.85
SSC	19.62	3.92	25.44	22.78

Table 4. Hydration heat at the early age (J/g)

Samples	Hydration heat at 24 h	Hydration heat at 48 h	Hydration heat at 72 h
PSC	83.51	121.99	139.04
SSC	11.53	77.43	91.14

3.2. Compressive strength

Fig. 3 shows the compressive strength contrast between PSC and SSC at the age of 3, 7, 28 and 90 days. At the age of 3 days, the compressive strength of PSC was 27 MPa, which was much higher than that of SSC and which made clear that strength of PSC is higher and SSC is relatively low, which can be explained that setting times of SSC are much longer than those of PSC in two cements' physical properties in Table 2. The slope line between two curing ages (such as from 3 to 7 days) in Fig. 3 represents the compressive strength increasing rate of cement during the curing age, which is the ratio between strength difference and age difference, the unit is MPa/d. Compressive strength increasing rate of PSC from 3 to 7 days was 3.68 MPa/d, which was a little lower than that of SSC with 3.78 MPa/d. Although the compressive strength of SSC at 3 days was relatively low, the increasing rate of compressive strength was accelerated as the growth of the curing ages. From 7 to 28 days, the strength growth rate of SSC was 0.77 MPa/d, which was also higher than that of

PSC with 0.59 MPa/d. During the curing age from 28 to 90 days, the strength increasing rate of SSC was 0.11 MPa/d, which was much higher than that of PSC with 0.05 MPa/d. At the curing age of 90 days, PSC strength was 57 MPa and SSC strength was 55 MPa, which indicates that with the growth of curing age, the strength difference was narrowing gradually.

The compressive strength of PSC and SSC both increased with the growth of curing age. Although the compressive strength of PSC was higher than that of SSC at every age, the strength difference between PSC and SSC was narrowing gradually. Although the early strength of SSC was very low and the growth rate was slow, the increasing rate of compressive strength was relatively fast in late hydration, which could maintain enough compressive strength, even mounting to the level of PSC at 90 days.

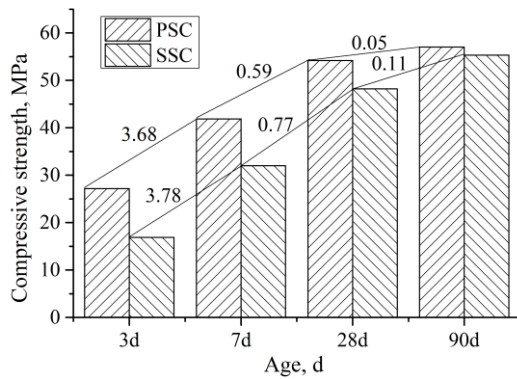


Fig. 3. Compressive strength of PSC and SSC at every curing age

3.3. Hydration products

Fig. 4 shows the XRD patterns of hydration products of PSC and SSC at every curing age. It could see that the hydration products crystal types of PSC were composed mainly by calcium hydroxide (CH) and a small amount of ettringite. While the hydration products of SSC were ettringite and some gypsum. At the hydration age of 3 days for PSC specimens in Fig. 4 a, the characteristic diffraction peak of CH was the strongest, at the same time a little ettringite, hydrogarnet and tetracalcium aluminate hydrate could be observed. With the increase of hydration, the diffraction peaks of all hydration products reduce. At early hydration more CH generates from the dissolution of cement clinker, while its diffraction peak reduced a lot after 3 days. The reason was the hydration reaction which needed to consume more CH to generate C-S-H gel [21, 23]. And the consumption rate of OH^- was much quicker than the generation from cement clinker. The reaction from 28 to 90 days there was little change of CH diffraction peak because the reaction mainly relied on internal materials of slag in the middle and late hydration reaction. There was some weak ettringite diffraction peaks can be observed at the age of 3 days, while weaken gradually as the curing age growing, little could be found in 90 days. Because of the instability of ettringite in cement system with low concentration of SO_4^{2-} and OH^- . In Fig. 4 b, strong ettringite diffraction peak of SSC could be observed at every age. There was a small amount of gypsum diffraction peak, while ettringite diffraction peak

was the strongest in 3 days. With the increase of curing age reaching up to 90 days, the diffraction peaks of ettringite and gypsum both weakened, which shows that alkalinity of paste solution decreases with the increase of curing age, while only in a certain alkaline environment ettringite can remain stable. Meanwhile, the further dissolution of gypsum can make SO_4^{2-} more in the system, which can increase the amount of ettringite to complement the ettringite's decomposition, while the formation rate was relatively slow and the total quantity of ettringite decreased. Although the ettringite diffraction peak after 90 days weakened at some extent, the characteristic peak was still strong, which indicated that ettringite can still exist stable. At the same time, gypsum crystal diffraction peak was weakening gradually with the increasing curing age from 3 to 90 days. Probably most gypsum crystal were dissolving in cement system as ions to participate in the reaction to generate C-S-H gels. As the reaction progress, more hydration products generated and filled in the pore structure gradually which could make the strength of specimens increase along with the age.

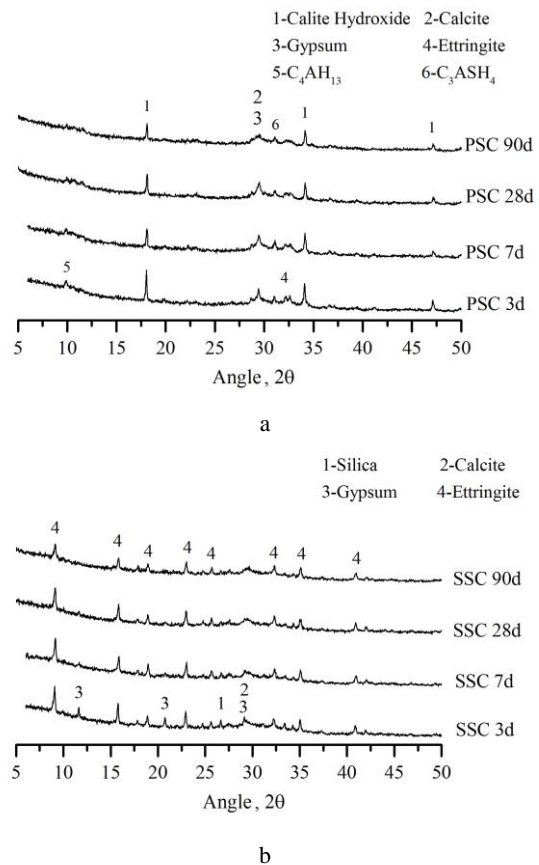


Fig. 4. Hydration products' XRD patterns of PSC and SSC in curing age: a – XRD patterns of PSC; b – XRD patterns of SSC

Fig. 5 a illustrates the micrographs of specimen PSC in 28 and 90 days. Hexagonal-platelet morphology of CH could be obviously observed at the age of 28 days. While only a little needle bar ettringite could be found. In 90 days, much of hydrated calcium silicate(C-S-H) granular or fibrous existed and it's difficult to observe CH, the structure became uniform and dense. From 28 to 90 days, CH crystal decreased and hydration product C-S-

H increases gradually, which are winding and filling in pores each other to make structure compact and strength increase, which is consistent to the conclusions of strength test results and XRD test of CH diffraction peak intensity. Fig. 5 b shows the micrographs of specimen SSC in 28 and 90 days. At the age of 28 days some needle bar ettringite growing rules existed in the specimen's cracks or holes. In 90 days, there was still some needle sticks crisscrossed the growth of ettringite, filling in the hole, at the same time granular C-S-H also could be found. As the growth of hydration age, micrograph of ettringite did not change obviously, which can exist in the system stable, and the C-S-H appearing gelatiniform ultimately, existing in the hole or as the growth bed of ettringite.

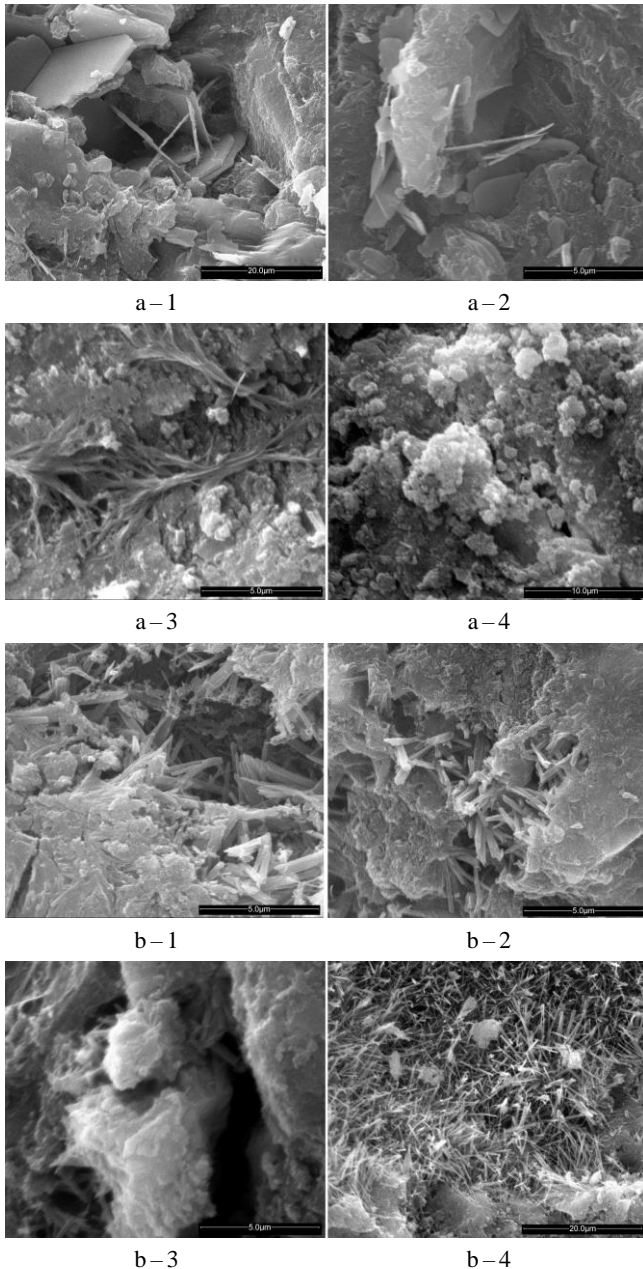


Fig. 5. SEM micrographs: a–PSC; b–SSC at the age of 1 and 2–28 d; 3 and 4–90 d

3.4. Discussion of hydration characteristics

The types of raw materials of PSC and SSC were the same, and the mix proportion and curing conditions were

consistent. However the only difference was the content proportion of raw materials. As shown in Table 1, the biggest difference of contents were SO_3 and CaO , while the content of CaO was too high to consume up over the hydration period, so the point is SO_3 , which may have important effect on their hydration characteristics. From the chemical compositions of the two cements in Table 1, SO_3 content of PSC was 3.33 %, while that of SSC was 12.48 %, which was above over 9.15 % than PSC. So SO_4^{2-} is the start point why the hydration products type of the two cements are different, and also the role of CaO in hydration process, which will give out the difference of the two cements' hydration.

K^+ , Na^+ and Ca^{2+} ions can destroy Si-O keys, break down the oxygen bridge, and at the same time reduce the viscosity of glass and destroy the vitreous body structure, which will make slag activity [24, 25]. When the ratio of $(\text{K}_2\text{O}+\text{Na}_2\text{O}+\text{CaO}):\text{SiO}_2$ reaches to 2:1, the network is broken gradually. The bigger the ratio is, the faster the nucleation and crystallization rate of glass, so as the higher the activity of slag. According to the data in Table 1, the ratio of PSC is 1.98 by chemical analysis calculation, and that of SSC is 1.63, which means that the activity of SSC is lower than that of PSC on the chemical composition. From the early hydration heat release rate and heat release (Fig. 2) and early strength (Fig. 3), PSC are higher than those of SSC all the time, which indicates that both hydration heat release rate and strength development are associated with the potential activity of slag. The higher the activity is, the faster the reaction is and as the strength increases more quickly. The XRD test analysis also shows the hydration products crystal quantity of PSC in 3 days is relatively higher than that of SSC. From the early experimental results, the strength, hydration heat and the quantity of early hydration products for SSC, which has a higher content of slag are lower than those of PSC. Hydration of slag are mainly caused by the network modifier of K_2O , Na_2O , CaO and so on. Slag and water will not react under normal temperature or the reaction would be very slowly, only when added with the alkaline-earth metals, which can change the PH value of the paste, then dissolving out K_2O , Na_2O and CaO , etc. The research done by Runzhang Yuan indicates that [25] only when the pH value is beyond 12, the slag paste can have hydration activity. It also specifies the basic solution conditions of slag hydration.

The surface of slag shows faint hydration reaction after mixed with water, some part substances dissolved in the particle surface to generate C-S-H gels, thus reducing the permeability of slag vitreous surface vitreous, which makes the water molecules unable to seep into the internal and internal ions cannot come out either. That is why it has a lower activity and needs exciting agent to destroy the slag vitreous structure to promote hydration reaction of the slag at room temperature [26, 27]. From Fig. 4 and Fig. 5, the hydration products of PSC are mainly for CH and C-S-H gels, while those of SSC are mainly for ettringite, C-S-H and a small amount of gypsum incomplete hydration. Hydration process of PSC is mainly for alkali excitation. After mixing with water, the clinker mineral C3S, C3A, C4AF hydrated with H_2O rapidly, mainly generate CH, hydrated calcium aluminate (CAH) and hydrated calcium

ferrite (CFH), then OH⁻ in hydration system combining with Ca²⁺ and Mg²⁺ existing in the surface of slag vitreous generated CH and magnesium hydroxide (MH) to destroy the vitreous body surface, and make the hydration develop process of internal and hydrate with Ca²⁺ and Mg²⁺ to generate CH and MH, which lead to the destruction, decomposition and dissolved of the slag vitreous network structure. CH and the active SiO₂ dissolved from the system hydrated to generate C-S-H. As a continuation of the hydration reaction, CH crystal dissolved continually to react, and C-S-H continually precipitated, making the paste thicken and harden gradually. Due to less than 5 % of the clinker in SSC, after mixing with water, clinker reacted with water first to form little C-S-H and CH. Research indicated that adding sulfate only in a certain alkaline environment, the activity of slag can be activated and got high gel strength [25]. Alkaline environment was created by the reaction of clinker and water, many OH⁻ ions exist in cement system, which will destroy the keys of slag's silica polymer chains and dissolved active Al₂O₃ and active SiO₂ into the system to form C-S-H and CAH, which can accelerate the dispersion and dissolvent. At the same time, SO₄²⁻ and Ca²⁺ dissolving from phosphogypsum reacted with active Al₂O₃ from slag and hydration product CAH, which will consume a large number of Al³⁺ and Ca²⁺ to generate ettringite, which in turn to accelerate slag hydration. Starting from the essence, the stimulation of SSC is the combination of sulfate and alkali excitation, but the action period is different. Alkali excitation is mainly to create an alkaline condition, which is better for the process of sulfate excitation. So the hydration products of XRD pattern (Fig. 4 b) are mainly ettringite, and CH cannot be found.

4. CONCLUSIONS

1. The compressive strength of PSC was higher than that of SSC at every curing age. However, the difference of the compressive strength between PSC and SSC narrowed gradually during curing age. The early compressive strength of PSC was high while at late hydration age it increased slowly. The early compressive strength of SSC was low, while the later compressive strength increased quickly.
2. Compared with PSC, both hydration heat evolution rate and hydration heat of SSC were relatively low, and its second exothermic peak delays too.
3. Hydration products of PSC are mainly hydrated calcium silicate (C-S-H) and calcium hydroxide (CH). In this system, OH⁻ destroys the vitreous body surface and reacts with network modifier to destruct, breakdown and dissolve the vitreous network structure, which is predominantly alkali activation.
4. Hydration products of SSC are mainly C-S-H gels and ettringite. Many OH⁻ ions from the reaction of clinker and water would destroy the keys of slag's silica polymer chains and dissolved active Al₂O₃ and active SiO₂ into the system to form C-S-H and CAH to accelerate the hydration, which is the combination of sulfate and alkali excitation.

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