

HYDRATION CHARACTERISTICS OF ALKALI-ACTIVATED SLAG CEMENT

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Hydration Characteristics of alkali-activated slag cement (AASC) are different of those of ordinary Portland cement. Effects of activator type, modulus of water glass (WG), and Na₂O equivalent on setting time, compressive strength, hydration heat and hydration products were studied. The results show that with the increase of modulus in the range of 0.8-2.0, initial and final setting time of AASC extends gradually. As for hydration heat, When WG modulus increases from 0.8 to 2.0, the first combined peak of AASC grows lower and grows a bigger diffusion; accelerated hydration peak appears later; induction period prolongs and cumulative hydration heat reduces. When Na₂O equivalent increases from 3% to 6%, for WG-activated slag, the first peak and accelerated hydration peak appear earlier and higher, induction period becomes shorter and setting time of paste is somewhat longer. Besides, for NaOH (NH)-activated slag, initial peak and accelerated hydration peak appear earlier and higher and setting time of paste is shorter. Cumulative hydration heat of both AASC increases with the increase of Na₂O equivalent. At the same Na₂O equivalent, setting time of slag activated by NaOH is longer than WG-activated slag cement, and compressive strength of the former is lower than the later. In the range of 0.5-2.2, with the increase of modulus, compressive strength of AASC first increases, then was followed by a decrease. The main hydration product is C-A-S-H gel.

Keywords: Alkali-activated slag cement, Activator, Hydration heat

1. Introduction

As a kind of environment friendly cementitious material, alkali-activated slag cement (AASC) is different in mineral composition from ordinary Portland cement [1, 2]. The hydration and hardening speed of AASC is fast as well as the strength is high [3-5]. Different activator has different destruction and decomposition action on slag structure and also has different effect on the hydration process and strength development. Sodium hydroxide (NaOH) and water glass (WG) are the most commonly used activator because of their easily available and low cost [6]. Scholars [7, 8] studied setting time and mechanical properties of the slag cement activated with water glass. According to Bakharev, when the modulus of water glass was 1.25, slag cement can have the greatest strength [3]. But Fernando points out that higher modulus of water glass can determine higher hydration level [4]. Vladimír Živica studied the effect of activator on setting time and workability of alkali-activated slag cement system; the experimental results show that setting time of the sodium silicate-activated slag cement is the shortest [8]. Compared with that, setting time of slag cement activated by sodium carbonate and sodium hydroxide is a little longer. Setting time and mechanical properties appear different with different activator, which indicates that slag possesses a selectivity for

activator, and different slags have their suitable activator.

In this study, blast furnace slag from Chongqing area was selected. The effect of activator type, modulus of water glass and alkali equivalent content on setting time and hydration heat was studied. Also compressive strength and hydration products were studied. So it is expected to put forward a theoretical reference for the appropriate selectivity of activator type, modulus of water glass and alkali equivalent content.

2. Experimental

2.1. Materials

The ground granulated blast furnace slag was obtained from Chongqing Steel and Iron Group with the specific gravity of 2.85g/cm³ and specific surface area of 453m²/kg. The main chemical composition of the slag is shown in Table 1. The quality index $((CaO+MgO+Al_2O_3)/(SiO_2+MnO+TiO_2))$, basic index $((CaO+MgO)/(SiO_2+Al_2O_3))$, and activity index (Al_2O_3/SiO_2) are: 1.83, 1.07 and 0.37 respectively.

Used activators were water glass (WG) and sodium hydroxide (NaOH) solution. WG is a kind of industrial product, its chemical composition and physical properties is shown in Table 2. NaOH is caustic soda flake came from Deyang, Sichuan Province, with the purity of 99%. NaOH was

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Table 1

Chemical compositions of slag								
SiO ₂ (%)	Al ₂ O ₃ (%)	CaO(%)	Fe ₂ O ₃ (%)	MgO(%)	SO ₃ (%)	K ₂ O(%)	Na ₂ O(%)	Loss of ignition(%)
30.97	11.40	37.59	1.79	7.60	0.22	0.66	0.37	1.01

Table 2

Chemical composition and physical properties of water glass				
SiO ₂ (%)	Na ₂ O(%)	Ms	Baume degree(°Bé)	density(g/cm ³)
26.49	9.95	2.75	40.0	1.405

blended to water glass to adjust the modulus (Ms) of WG solution (molar ratio of SiO₂ to Na₂O). A NaOH solution with the concentration of 30% was prepared beforehand. Tap water was used in our experiments.

2.2. Experimental methods

According to China's national standard GB/T1346-2011 (Test methods for water requirement of normal consistency, setting time and soundness of the Portland cements), setting time of AASC was tested. 0.25 water/binder ratio (mass ratio) and 3%, 4%, 5%, 6% Na₂O equivalent content of the mass of slag were used to prepare cement pastes. The modulus of water glass was: 0.5, 0.8, 1.0, 1.2, 1.5, 1.8, 2.0 and 2.2 respectively. Paste specimens of 20 mm×20 mm×20 mm were prepared and cured in standard conditions for compressive strengths determinations.

The hydration heat testing equipment (TAM air and its supporting experimental method) was adopted to test the hydration heat of AASC. Pastes from 5 g slag powder and 0.45 water/binder ratio (mass ratio) were prepared; NaOH was added in the form of powder.

A Rigaku D/Max-5A 12kW diffractometer was used to analyze the hydration products. At 28 days standard curing, specimens were soaked in absolute ethyl alcohol and dried to constant weight at 60°C, to stop hydration. Before XRD testing, specimens were ground to a fineness corresponding to pass by sieve of 0.08mm.

3. Results and discussion

3.1. Setting time and compressive strength

3.1.1. Effect of WG modulus on setting time and compressive strength

Figure 1 presents the setting time of AASC activated with WG at 4% Na₂O equivalent. In the range of 0.8-2.0, with the increase of modulus, initial and final setting time of paste gradually increases. When modulus is greater than 2.0, the setting time increases considerably, this is similar to Ma Bao-guo's test results [9]. His results indicate that when modulus of WG increased between the range 1.0-1.75, there was no obvious retarding effect. While the modulus increased beyond 2.25, setting time increased greatly.

Compared with the AASC activated with modulus of 0.8, initial and final setting time of slag

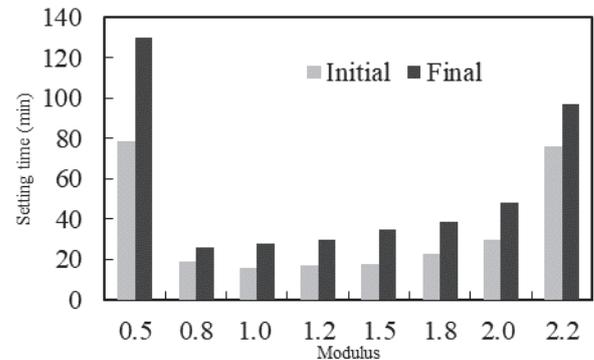


Fig.1 - Influence of WG modulus on setting times of AASC (4%Na₂O).

cement activated with modulus of 0.5 increases more, which do not meet the above rule. [SiO₄]⁴⁻ groups type and their quantity are different when the modulus of WG is different. While modulus is smaller, liquid alkalinity becomes higher and [SiO₄]⁴⁻ groups are less. At the early hydration stage, the main reaction is between the [SiO₄]⁴⁻ group from WG and Ca²⁺ dissolved from the surface of slag grains [10, 11], so less [SiO₄]⁴⁻ group is unfavorable to the formation of the C-A-S-H. Also, the higher the liquid alkalinity, the more stable is the WG solution. On this account, initial and final setting time of slag cement activated with modulus of 0.5 water glass is longer. When modulus is higher than 0.8, liquid alkalinity becomes lower, which is unfavorable to the hydration of slag. With the increase of modulus of WG, its monomeric [SiO₄]⁴⁻ reduces, and polymers [SiO₄]⁴⁻ increases, so the quantity of silica-oxygen group activation point reduces, thereby setting time is prolonged [12-14].

From Figure 2, we can see that in the range of 0.5-2.2, with the increase of modulus of WG, compressive strength of 3 days, 7 days, and 28 days of AASC all first increases, then followed by a decrease. This is corresponding to the results of setting time which first decreases, and then increases. So the effect of modulus of WG on setting time and compressive strength of AASC is consistent. As mentioned above, more individual groups of [SiO₄]⁴⁻ are introduced with the increase of modulus, so strength increases. But too big modulus (>2.0) causes low alkalinity of WG and slows hydration of slag, which can delay the compressive strength growth of AASC.

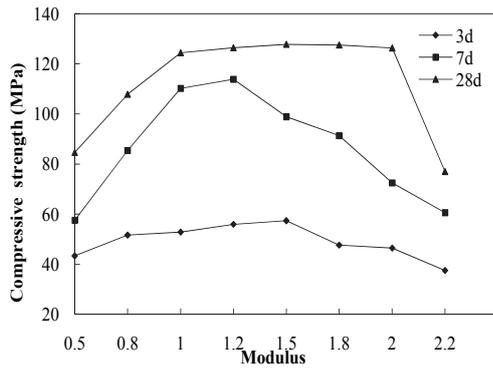


Fig.2 - Influence of WG modulus on compressive strength of AASC (4%Na₂O).

Therefore, it can be assumed that as an activator, reasonable modulus of WG is between 1.0 and 2.0. For a given WG-slag-water system, in the condition of the limited strength, the modulus of water glass can choose a larger value, so as to retard the rapid setting.

3.1.2. Effect of activator type and Na₂O equivalent on setting time and compressive strength

Setting time of AASC activated with WG (modulus is 1.5) and NaOH are presented in Figure 3. It can be seen that in the range of 3%-6% Na₂O equivalent, initial and final setting time of AASC activated with WG present a small increase with the increase of Na₂O equivalent. For example, at the dosage of 3% Na₂O equivalent, initial and final setting time are 17min and 21min respectively, while at the dosage of 6% Na₂O equivalent, initial and final setting time are 26min and 36min respectively. Also it can be seen that the interval between initial and final setting is between 3-5min, which is small.

The results of setting times are similar to FU Xing-hua's test results [15], but incompatible with other scholars' [5, 7, 8], reflecting the complex effect of alkali content on [SiO₄]⁴⁻ and [AlO₄]⁵⁻ polymerization rate. In FU's test [15], when modulus of WG is 1.3, and when the alkali content increased from 6% to 12%, initial setting time increased from 160min to 342min, and final setting time increased from 199min to 389min. Cengiz et al. [5] concluded that the initial and final setting of water glass and sodium hydroxide activated slag paste occurred much earlier than for OPC paste. The final setting times of AAS paste reduced with an increase in the sodium concentration of the activator. Vladimír Živica [8] reported that AAS cement mortar activated with sodium silicate showed decreased setting time as the dosage of sodium silicate was increased.

From previous results [5, 7, 8], we can say

that increasing of water glass addition results in a higher concentration of [SiO₄]⁴⁻ and consequently an increase of the reaction rate (a higher concentration of reactants induces a higher reaction rate). On the other hand, a higher concentration of OH⁻ helps the dissolution of Ca²⁺ from the slag grains and consequently increases the reaction rate. But in this experiment, we observed that with the increase of Na₂O equivalent, the fluidity of the cement paste increased a little, which may be attributed to the fluidify action of activator. And that fluidify action diluted the activator concentration and the activation was weakened for the present. Therefore, the hydration of slag is a little slower with the increase of Na₂O equivalent.

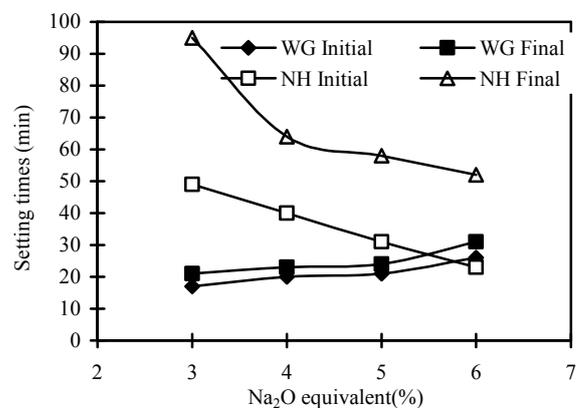


Fig.3 - Influence of activator and Na₂O equivalent on setting times of AASC (Ms=1.5).

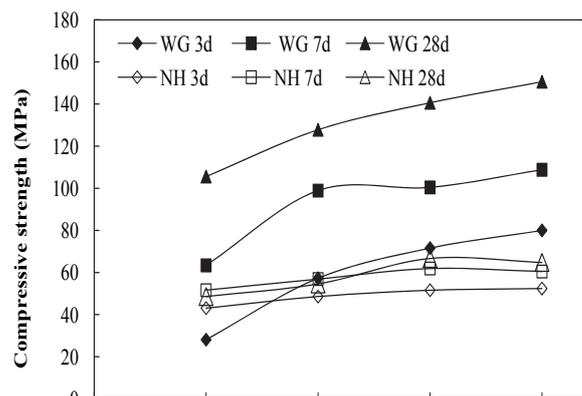


Fig.4 - Influence of activator and Na₂O equivalent on compressive strength of AASC (Ms=1.5).

It can also be seen in Figure 3 that when the activator used is NaOH, increasing of the activator dosage reduces the setting times, both initial and final. At the dosage of 3% Na₂O, initial time is 49min., while at 6% Na₂O, initial time is reduced to 23min.

From the above discussion, it can be concluded that compared with WG-activated slag cement, setting time of slag activated by NaOH is

longer. NaOH solution has a high concentration of OH⁻, which can break not only Ca-O and Mg-O bonds, but also a significant number of Si-O and Al-O bonds. So a very thin layer consisting of C-A-S-H gel with low Ca/Si ratio, having a very low solubility, precipitates very quickly out of the solution. While for the slag activated by water glass, in the early hydration stage, the main reaction is between the [SiO₄]⁴⁻ groups from water glass and Ca²⁺ dissolved from the surface of slag. In view of this, setting time of the two kinds of alkali-activated slag cement is different [11, 16, 17]. More research should be carried out further.

In Figure 4, it can be seen that in the range of 3%-6% Na₂O equivalent, compressive strength of AASC shows a small increase with the increase of Na₂O equivalent. Compared with WG-activated slag cement, compressive strength of slag cement activated by NaOH is lower. Besides, with an increase of hydration age and Na₂O equivalent, compressive strength presents more increase in WG-activated slag cement than in NH-activated slag cement. In other words, hydration age and Na₂O equivalent has little effect on compressive strength of NH-activated slag cement. This is because that a faster hydration with a higher strength development in the early stages of hydration. And a strained structure is formed, so there is very small increase of strength in time.

3.2. Hydration heat

3.2.1 Effect of WG modulus on hydration heat

The hydration of AASC can be described by three models [7, 18-20]. The heat evolution curve of slag activated with WG belongs to type III. In type III, there are two peaks (one initial and one addition initial) before induction period and one accelerated hydration peak appears after the induction period.

Because the initial and the additional initial peaks appear very close, and the two peaks often merge into one in measurement. The initial peak corresponds to the wetting and dissolution of slag grains and adsorption of some ions onto the surface of slag grains. The additional initial peak is mainly due to the formation of the "primary C-A-S-H" [7]. The precipitation of "primary C-A-S-H" results in a very long induction period. It can be seen from Figure 5a, there is a merged peak before accelerated hydration peak.

Figure 5 illustrates heat evolution rate of activated slag cement with WG having different modulus with 4% Na₂O equivalent. The first combined peak appears after 4-5 min. With modulus varying from 0.8 to 2.0, the first combined peak grows lower and a bigger peak corresponding to accelerated hydration appears later. The accelerated hydration peak appears at 29h, 35h and 48h respectively when modulus of WG is 0.8, 1.5 and 2.0. This can be explained by that some [SiO₄]⁴⁻ in the solution may polymerize and no enough Ca²⁺ is available to react when higher modulus WG is used. In previous setting time experiment, higher modulus leads to slower setting, which is consistent with the heat evolution rate. Just for slag activated by WG with modulus 0.5, the situation is somewhat different, that is possibly due to the too large alkalinity of activator solution and seriously different [SiO₄]⁴⁻ anion structure as described previously.

The effect of modulus of WG on cumulative hydration heat of binder with 4% Na₂O equivalent is illustrated in Figure 6. The increase of WG modulus determines a decrease in cumulative hydration heat. The reason mentioned above and it is unnecessary to go into details here.

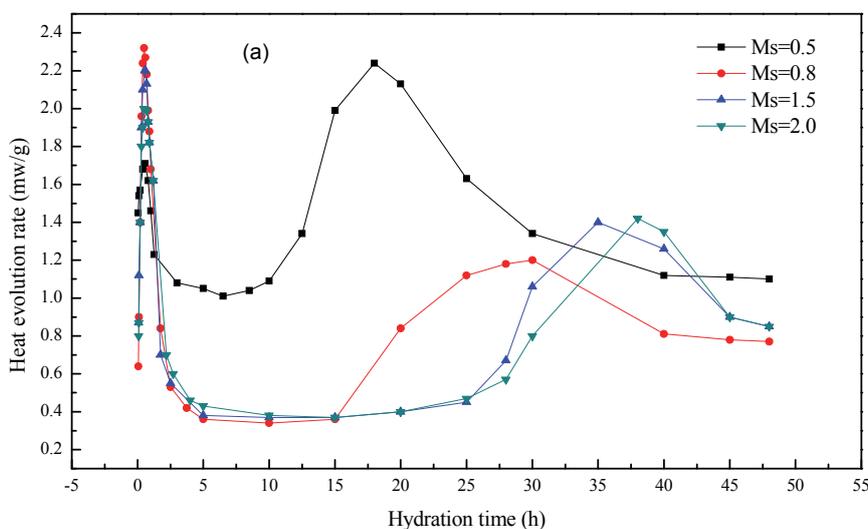


Fig. 5 continues on next page

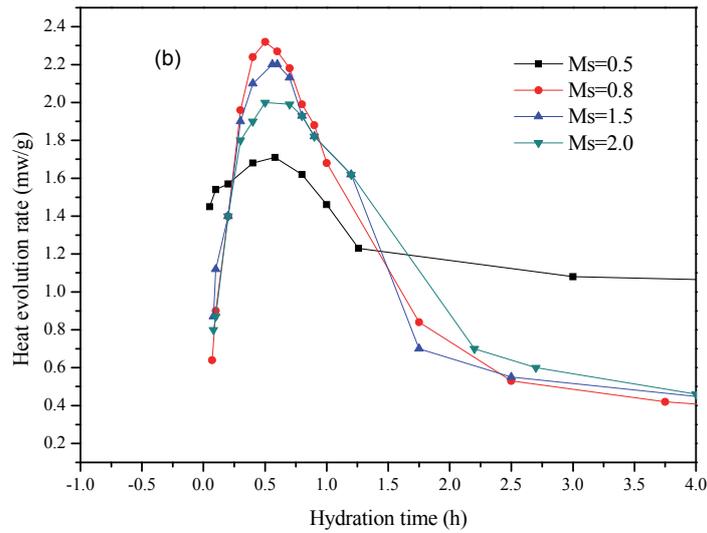


Fig.5 - Influence of modulus of WG on hydration heat evolution rate of AASC (4% Na₂O equivalent), a- hydration heat evolution rate in 48 hours; b- hydration heat evolution rate in 4 hours.

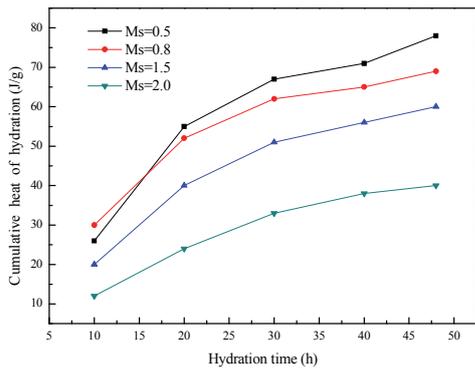


Fig.6. - Influence of modulus on cumulative heat of hydration of AASC (4% Na₂O equivalent).

3.2.2 Effect of Na₂O equivalent of WG-activator on hydration heat

Figures 7 and 8 illustrate heat evolution rate and cumulative hydration heat of WG-activated slag cement with different Na₂O equivalent and modulus of 1.5. The precipitation of C-A-S-H gel results in a very long induction effect. The long induction period, varying from about 5 to 24 h, is observed before the appearance of accelerated hydration peak. The higher the Na₂O equivalent is, the higher are both the merged peaks and accelerated hydration peaks, and the shorter is the induction period. The Na₂O equivalent below 6% has a significant effect on the length of induction period. The magnitude of accelerated hydration peak of slag with 6% Na₂O is higher than with 3%, 4% and 5%. It may be possible that the “primary C-A-S-H” promotes the further hydration of slag.

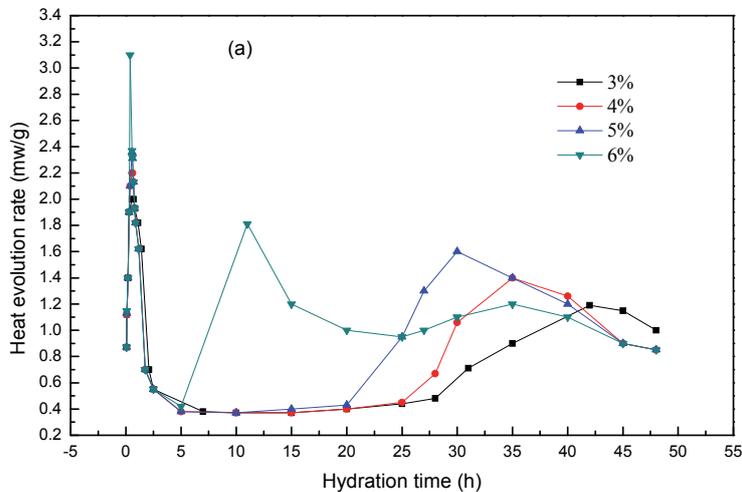


Fig. 7 continues on next page

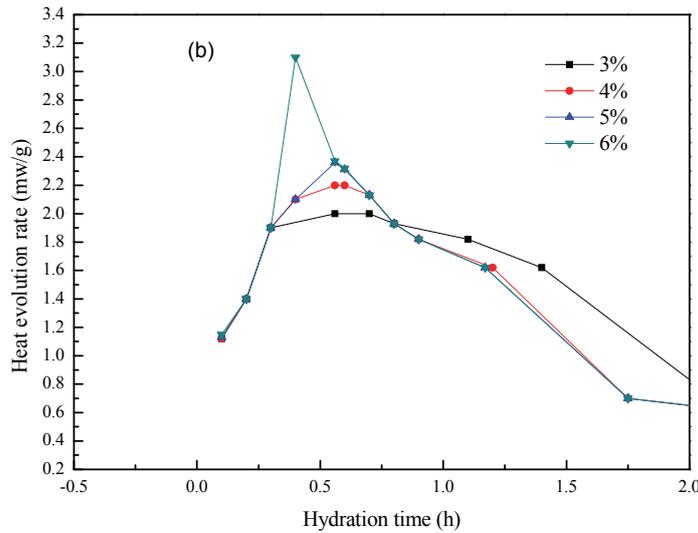


Fig.7 - Influence of Na₂O equivalent on heat evolution rate of AASC (Ms=1.5), a- hydration heat evolution rate in 48 hours; b- hydration heat evolution rate in 2 hours.

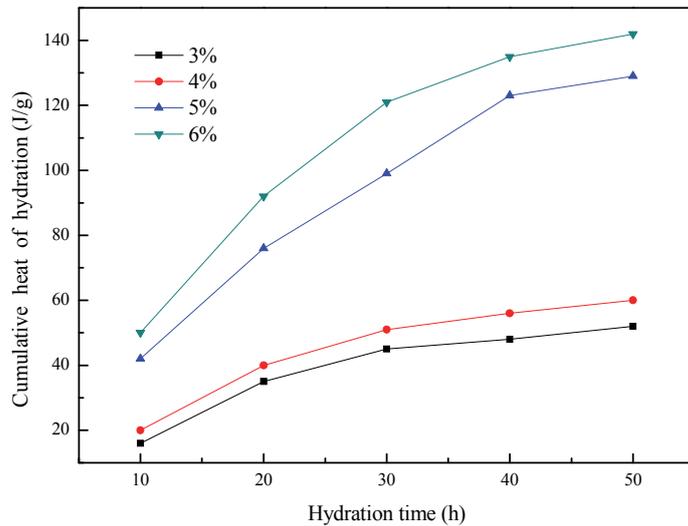


Fig.8 - Influence of Na₂O equivalent on cumulative heat of hydration of AASC (Ms=1.5).

Increasing activator dosage shows an increase in cumulative hydration heat especially above 5% Na₂O equivalent. When the activator dosage is 6%, accelerated hydration peak advanced to the 12 h or so and hydration heat of the first 48h reaches up to 150J/g.

3.2.3. Effect of Na₂O equivalent of NaOH - activator on hydration heat

The effect of Na₂O equivalent on heat evolution rate and cumulative hydration heat of alkali-activated slag cement with NaOH is illustrated in Figures 9 and 10. The heat evolution curve of the slag activated with NaOH belongs to type II—one initial peak appears before the induction period and one accelerated hydration peak appears after the induction period. The first initial peak on heat evolution diagram appears within 4-6min immediately after addition of water

and accelerated hydration peak following the induction period appears after 4 to 8 h. Increasing Na₂O equivalent increases the released heat and shortens the two peaks (first initial peak and accelerated hydration peak) interval slightly. Especially when Na₂O equivalent varies from 5% to 6%, heat evolution rate obviously increases from 86.3mw/g to 129mw/g. And when alkali equivalent varies from 3% to 6%, cumulative hydration heat increases from 174.9 J/g to 293.8J/g.

In contrast to slag activated with WG, for slag activated with NaOH, the initial peak is higher and the accelerated hydration peak appears earlier; also, the induction period appears shorter and cumulative hydration heat is more. This determines a faster hydration with a high strength development earlier. But a strained structure is formed, which explain the very small increase of

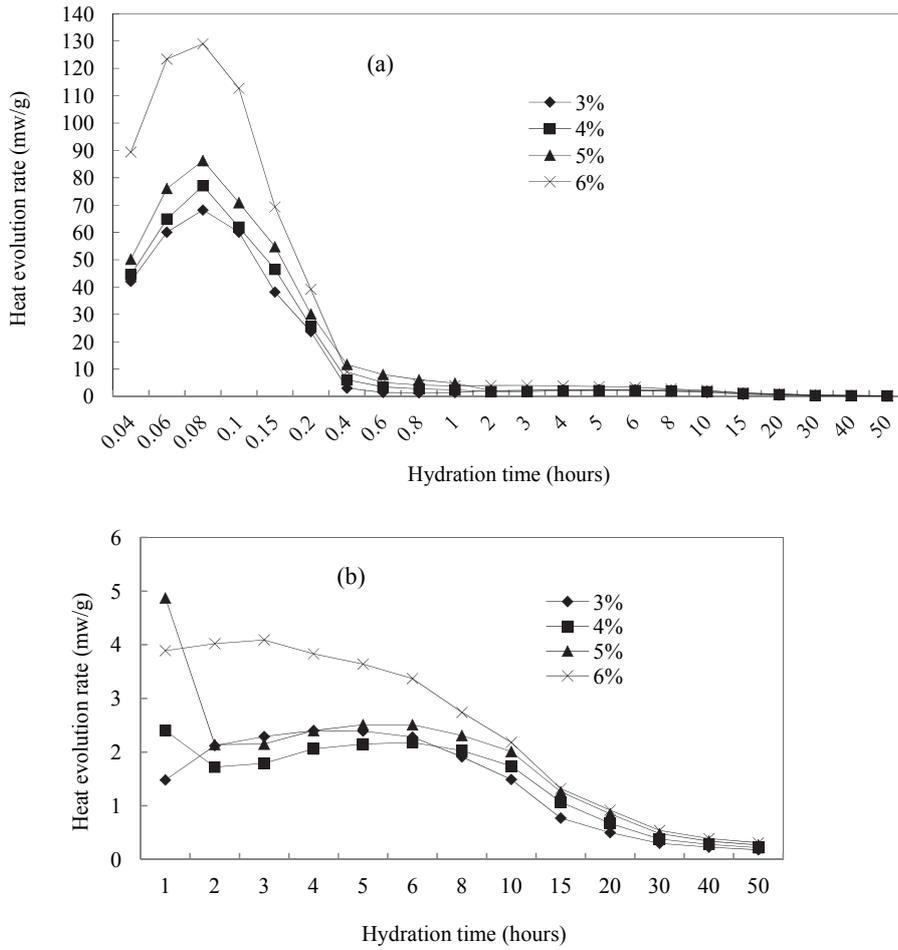


Fig.9 - Influence of Na₂O equivalent on heat evolution rate of AASC (NH)
 a- hydration heat evolution rate in 50 hours; b- hydration heat evolution rate from 1 to 50 hours

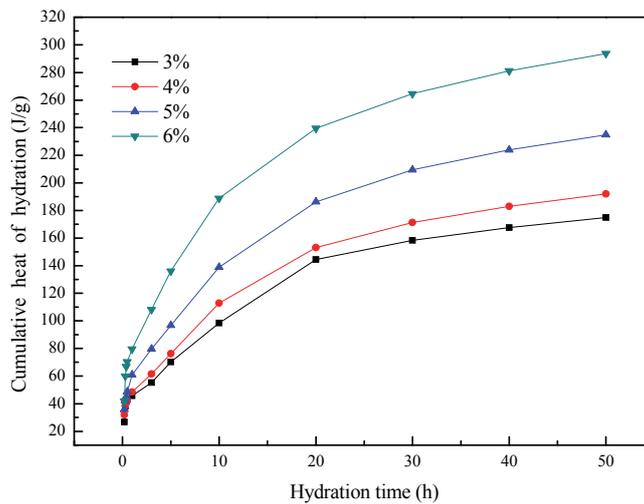


Fig.10 - Influence of Na₂O equivalent on cumulative hydration heat of AASC (NH)

strength in time. In addition, the very high initial peak of slag activated with NaOH is mostly due to the dissolution of powder NaOH. Also, it can increase the cumulative heat of hydration.

Alkali-activated slag cement hydration process

could be considered as having the following mechanism: the dissolution and destruction of the slag structure, coagulation of hydration products, and condensation of hydration products [10, 21, 22]. In alkaline solution, high concentration of OH⁻

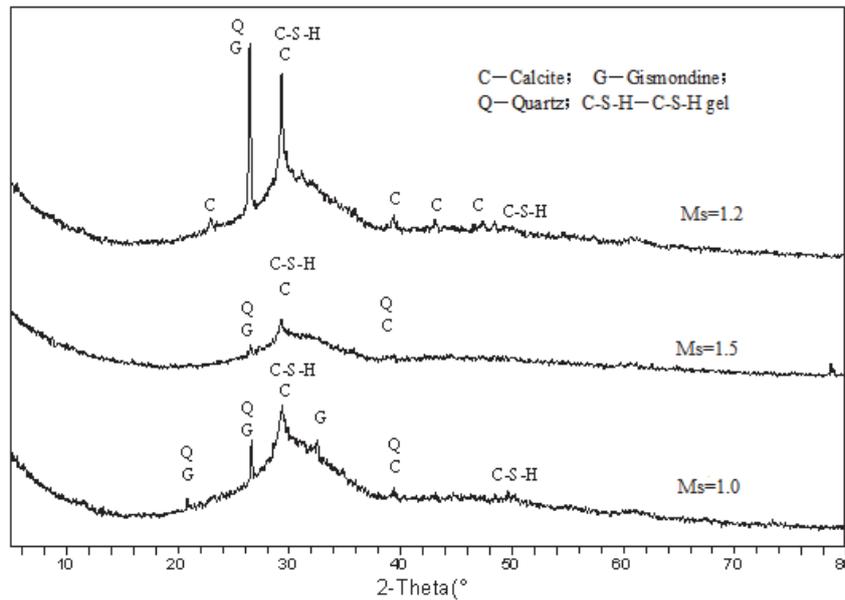


Fig.11 - XRD patterns of AASC activated with WG after 28days of hardening (4% Na₂O equivalent).

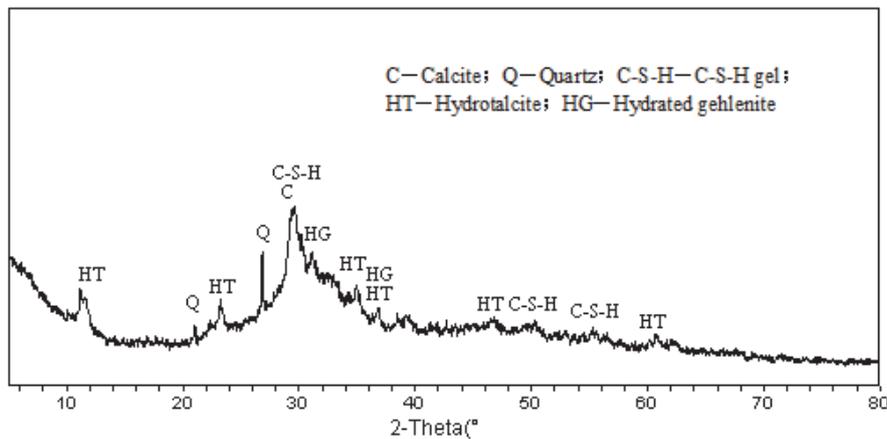


Fig.12 - XRD pattern of AASS activated with NaOH after 28 days of hardening (4% Na₂O equivalent).

breaks Ca-O, Si-O and Al-O covalent bonds, then vitreous slag forms the free end containing non-bridging oxygen and $[\text{SiO}_4]^{4-}$ with hydroxyl. In a short period of time, Ca^{2+} and $[\text{SiO}_4]^{4-}$ enter into the solution and combine to form a compact low Ca/Si C-S-H. Therefore, compared with Portland cement, alkali-activated slag cement set more quickly, so its initial peak appears earlier and early compressive strength progress higher. In slag activated with different activator, at early period, the anion or anions group reacting with Ca^{2+} can come from activator (slag activated with water glass, for instance) or slag (slag activated with NaOH, for instance), so hydration heat, setting time and strength progress are different.

3.3. Hydration products

XRD patterns of AASC activated with WG after 28days of hardening in standard conditions is shown in Figure 11. A wide diffuse diffraction peak for C-S-H (C-A-S-H) gel is seen between 0.28 nm-

0.31 nm; this has nothing to do with modulus of WG. In addition, the characteristic peaks of gismondine appear. At about 30°, when modulus of WG is 1.2, the peak of AASC is high and sharp, showing the characteristic peak of CaCO_3 , which indicates that the AASC sample is slightly carbonized. Modulus of WG has no significant influence on the hydration products of AASC. But when modulus is different, the basicity, and amount and polymerization status of $[\text{SiO}_4]^{4-}$ groups is different. These will affect the disintegration of slag and affect the depolymerization and polymerization process of $[\text{SiO}_4]^{4-}$ groups.

Figure 12 shows the XRD patterns of AASC activated with NaOH after 28 days of hardening in standard conditions. The main hydration product is still C-S-H (C-A-S-H) gel. Similar results has been reported in references [23, 24]. However, in hydration products, there is no gismondine, while there are some characteristic peaks of hydrotalcite and hydrated gehlenite.

4. Conclusion

1) In the range of 0.8-2.0, with the increase of WG modulus, initial and final setting time of WG-activated slag cement increases and compressive strength first increases, then followed by a decrease. Reasonable modulus of water glass is between 1.0 and 2.0. With the increase of Na₂O equivalent in the range of 3%-6%, initial and final setting time and compressive strength increase for AASC activated with water glass, while for AASC activated with NaOH, initial and final setting time decreases and compressive strength increases slightly.

2) The heat evolution curve of the slag activated with water glass belongs to type III that two peaks (one initial and one addition initial) appear before induction period and one accelerated hydration peak appears after the induction period. The heat evolution curve of the slag activated with NaOH belongs to type II—one initial peak appears before the induction period and one accelerated hydration peak appears after the induction period. In contrast to slag activated with water glass, for slag activated with NaOH, initial peak is higher and accelerated hydration peak appears earlier, also the induction period is shorter and cumulative heat of hydration is more.

3) With modulus varying from 0.8 to 2.0, the first combined peak of slag activated with water glass grows lower and a bigger diffusion and accelerated hydration peak appears later. Increasing Na₂O equivalent determines an increase in cumulative heat of hydration no matter what activator used. With Na₂O equivalent varying from 3% to 6%, both the first combined and accelerated hydration peaks are higher and earlier, and the induction period is shorter when water glass is used. This is the same to NaOH.

4) The main hydration product is C-A-S-H gel. In WG-activated slag cement the characteristic peaks of gismondine appears. In NaOH-activated slag cement there are some characteristic peaks of hydrotalcite and hydrated gehlenite.

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