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Effect of Calcium Silicate Hydrate Seeds on Hydration and Mechanical Properties of Cement

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Abstract: C-S-H series are synthesized at different temperatures and ages by pozzolanic reaction. The change of particle size distribution, phase composition, morphology and nanostructure of C-S-H with temperatures and ages, and the effects of C-S-H seeds and seeds parameters on the hydration behavior and mechanical properties development of cement were investigated by DLS, XRD, SEM, ²⁹Si NMR, TAM-air isothermal calorimeter and mechanical properties test. The results show that the particle size, crystallinity, basal spacing and Q^2/Q^1 ratio of C-S-H increases with the increase of synthesis temperature and age. The addition of synthesized C-S-H seeds to cement pastes results in the strong acceleration effect on cement hydration and significant improvement of the early strength of cement paste and mortar. The 1 day-C-S-H seeds synthesized at room temperature can increase the strength of cement paste by about 30 MPa at 12 h. The effect does not show a very regular change with the increase of the temperature and age of seeds synthesis. Considering the effect of C-S-H seeds on the hydration and mechanical properties of cement, and economy and short cycle of seeds synthesis, the C-S-H seeds synthesized at room temperature for 1 day or 55 °C water bath for 12 hours is recommended.

Key words: calcium silicate hydrate (C-S-H); seeds; hydration; mechanical property; temperature

1 Introduction

The development of green cements containing supplementary cementitious materials (SCMs), with the aim of reducing CO₂ emissions, often results in reduced hydration activity, especially during the first hours and days. Calcium silicate hydrate (C-S-H), as the major hydration product of ordinary Portland cement, is the main source of strength of cement matrix^[1]. C-S-H seeds can directly be acted as nucleation sites for hydration products, resulting in the acceleration of early hydration of cement, which has great potential to compensate for the above-mentioned effect^[2-5]. The growth rate of C-S-H determines the exothermic rate during

the accelerated period of cement hydration. Formed C-S-H gels bond cement and hydration products together, which is of great significance to the strength improvement of cement-based materials.

Generally speaking, it is difficult to obtain C-S-H phase from cement paste, because in addition to C-S-H gels, cement paste contains other hydration products like ettringite (AFt) and calcium hydroxide (CH), and unhydrated cement clinker minerals. A variety of synthetic methods of C-S-H phase have been mentioned in the literature, such as the co precipitation of alkali silicate and calcium salt, pozzolanic reaction of calcium oxide, amorphous silica and water, and hydration of β -C₂S or C₃S^[6-8]. The coprecipitation of alkali silicate and calcium salt has the advantages of fast reaction and convenient synthesis. However, the synthesized C-S-H usually contains alkali metal ions. When it is added into concrete as seeds, the alkali metal ions have a negative impact on the durability of concrete. The C-S-H obtained by direct hydration of C₂S or C₃S is most similar to C-S-H formed by cement hydration, but the C-S-H coexists with CH. The pozzolanic reaction seems to be a suitable method to prepare C-S-H seed.

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The composition and structure of C-S-H are very complicated^[9-13]. Cong reported C-S-H shows continuity and diversity in both composition and structure and forms a continuous structural series^[14]. The Ca/Si ratio of most C-S-H in pure concrete is about 1.7, and that of C-S-H in concrete containing SCMs is lower^[13]. C-S-H with low Ca/Si ratio has longer silicate chain length and higher Q^2/Q^1 ratio than C-S-H with high Ca/Si ratio^[13,15]. Under a Ca/Si of 0.7, amorphous silica will be present in synthetic C-S-H, while by exceeding a Ca/Si of 1.45-1.6, calcium hydroxide occurs^[16].

Different C-S-H seeds may have different effects on the hydration and mechanical properties of cement, which is important for the optimal preparation of C-S-H seed and its application in concrete. However, there are few studies on C-S-H seeds with different synthesis temperatures and ages. The main aim of this paper is to characterize the change of particle size distribution and nano microstructure of C-S-H seed with synthesis temperature and age, and the effects of these parameters on the hydration behavior and mechanical properties development of cement.

2 Experimental

2.1 Raw material

Table 1 Oxide compositions of the Portland cement/wt%

Oxide	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	SO ₃	MgO	Na ₂ O	K ₂ O
Cement	18.4	5.17	64.5	3.48	4.18	1.81	0.198	0.931

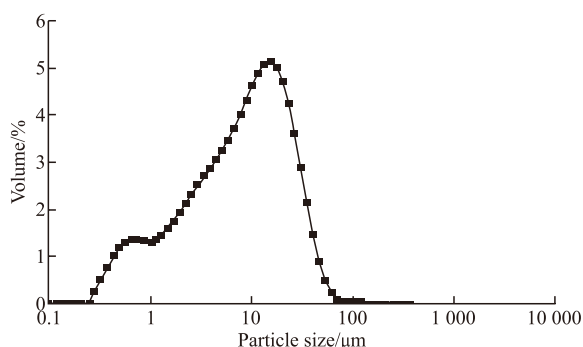


Fig.1 Particle size distribution of cement

Cement used is Type I Portland cement produced by Huaxin Cement Co., Ltd., China. The strength grade of cement is P-I 52.5 and the measured 28 d compressive strength is 67.7 MPa. Its chemical composition and particle size distribution are given in Table 1 and Fig.1, respectively. The fine aggregate is the standard sand produced by Xiamen ISO Standard Sand Co., Ltd, China. The calcium carbonate is analytical pure and from Sinopharm Group Chemical Reagent Co., Ltd.,

China. The neutral silica sol (VK-S01N) is a very fine material with an average particle size of 10 nm and its solid content is 25% (provided by Hangzhou Zhitai Purification Technology Co., Ltd., China).

2.2 Specimens preparation

C-S-H samples were synthesized by pozzolanic reaction of calcium oxide, neutral silica sol, and water at the mass ratio of water to solid of 20 and molar ratio of Ca/Si of 1.3. The reason why the Ca/Si molar ratio is set at 1.3 is to prepare single -phase C-S-H, and its structure is similar to the most common dimer dominated C-S-H structure in concrete. The calcium oxide was freshly calcined from reagent grade CaCO₃ at 1 100 °C for 2 h. The water was deionized, freshly boiled for 15 min to remove the dissolved CO₂ and then topped off with N₂ gas to reduce carbonation^[15,17]. Suspensions were stirred continuously to target ages at room temperature and by water bath at 55 °C. Synthesized C-S-H samples at room temperature are marked with RT seeds, whereas those synthesized by 55 °C water bath are marked with WB seeds. The age of seeds is indicated after the letters RT or WB. For example, RT 1 d seeds and WB 12 h seeds are 1-day seed synthesized at room temperature and 12-hours seeds by 55 °C water bath, respectively.

The newly synthesized C-S-H suspension was added into the cement at the solid dosage of 1 wt% to prepare 40 mm × 40 mm × 40 mm cement paste specimens and 40 mm × 40 mm × 160 mm mortar specimens for mechanical properties test. The water /cement mass ratio (w/c) for cement paste is 0.35. For mortar, w/c ratio is 0.5 and the mass ratio of standard sand to cement is 3. Blank specimens without C-S-H seeds were also prepared for comparative investigation. All specimens were cured to test age under standard curing conditions after demoulding.

2.3 Characterization methods

C-S-H powders were used for X ray diffraction (XRD). Scanning electron microscope (SEM) and ²⁹Si magic angle spinning nuclear magnetic resonance (²⁹Si MAS NMR) measurement were obtained by vacuum filtration of C-S-H suspension and subsequent vacuum drying and grinding. The XRD data was recorded by D8 advance powder diffractometer, using Cu K α radiation at 35 kV and 40 mA. The 2-theta value ranges from 5° to 55° with scanning speed of 5°/min. The SEM was carried out by a QUANTA FEG 450 ESEM fitted with a BSE detector with the accelerating voltage of 15 KV. The ²⁹Si MAS NMR spectra were obtained from solid-state NMR spectrometer (AVANCE III 400

MHz WB, Bruker Co.) with magnetic intensity of 9.4 T. Samples were packed into 7-mm zirconia rotors, sealed at each end with Teflon plugs. The spinning speed is 5 kHz. The chemical shift was externally referenced to tetramethylsilane direct polarization (DP). MAS spectra were obtained and adjusted by Bruker Topspin 3.2.

The synthesized C-S-H suspension was diluted to the concentration of 0.1 g/L and then placed in the ultrasonic cleaner for 10 - 15 min to disperse the particles. Its particle size distribution was measured via the NANO-ZS-ZEN-3600 dynamic light scattering instrument (DLS) produced by Malvinpaco Instrument Co., Ltd.

The hydration heat was determined by an 8-channel isothermal calorimeter (TAM AIR, Netzsch, Germany) at a temperature of 20 °C with a ± 20 mW test precision. A 100 g mix with w/c ratio of 0.35 and C-S-H seed content of 1 wt% of cement was prepared. C-S-H seed is added with newly synthesized C-S-H suspension, and its solid content is approximately calculated as 5%. The mix was then manually stirred for about 5 min to create a flowable and homogeneous paste. A sample of about 10 g was quickly sucked into a pipette and then pushed into a plastic vial to minimize water evaporation. The capped vial containing the sample was placed into channel using a tweezer to minimize heat transfer from the hand. Sample loading was completed within 2 min after mixing. The hydration heat for 168 hours was recorded. Blank cement paste without C-S-H seeds was also investigated.

3 Results and discussion

3.1 Nano microstructure of C-S-H seeds

The XRD patterns of C-S-H samples synthesized at room temperature (RT seeds) are shown in Fig.2. The C-S-H characteristic peaks near 0.307, 0.280 and 0.17-0.18 nm (about 29°, 32°, 48°) are detected in RT 1 d seeds. These peaks are broad and no basal peak is detected, indicating long range disordered C-S-H is formed^[15]. At the same time, the peak at 0.492, 0.263 and 0.193 nm are detected, indicating the presence of unreacted CH. A small amount of calcite characteristic peak appears, which is caused by partial carbonation of CH. For RT 3 d seeds to RT 7 d seeds, basal peak of C-S-H is also detected, and except for the diffraction peaks of C-S-H, no obvious other peaks are detected. The basal peak at approximately 0.11 nm (about 8°) attributed to the (002) planes, which is the characteristic of the layered stacking. As the increase of the age, the

intensities of C-S-H peaks increase, the full width at half maximum (FWHM) decrease, and the basal peak seems to shift to a lower angle, which indicates that the crystallinity of C-S-H increases with the increase of the age, and the basal spacing of C-S-H also seems to increase slightly.

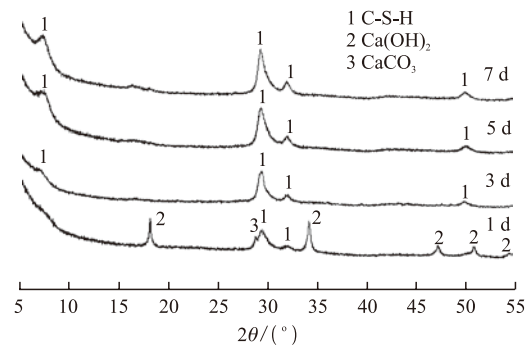


Fig.2 XRD patterns of C-S-H samples synthesized at room temperature (RT seeds)

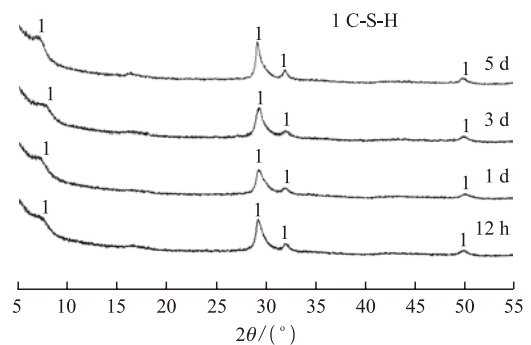


Fig.3 XRD pattern of C-S-H sample synthesized by water bath at 55 °C (WB seeds)

The XRD patterns of C-S-H samples synthesized at 55 °C in water bath (WB seeds) are shown in Fig.3. The characteristic peaks of C-S-H are detected in all the samples, and no obvious other peaks were observed, indicating the samples are phase-pure C-S-H. There is no unreacted phase in RT 12 hours seeds, because the reaction at 55 °C is much faster. And the peaks of C-S-H synthesized at 55 °C are a little stronger and narrower than those of RT seeds at the same age.

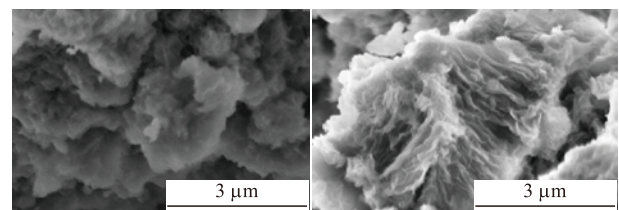


Fig.4 SEM images of C-S-H samples

The SEM images of RT 7 d seeds and WB 5 d seeds are shown Fig.4. The C-S-H exhibited dominant

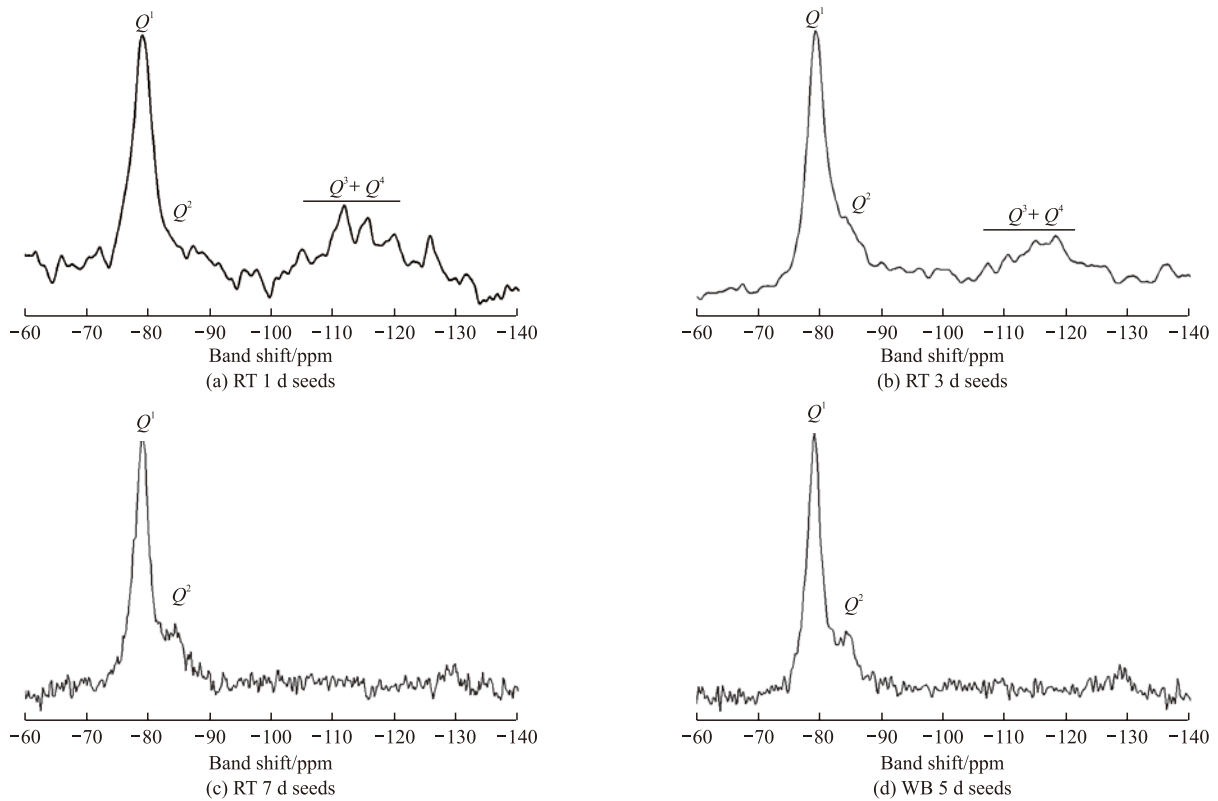


Fig.5 The ^{29}Si MAS NMR spectra of C-S-H samples

granular agglomerations, which seems to be a reticular structure composed of fibers or foils. The morphology of RT 7 d seeds seem to be finer, implying the C-S-H seeds are more amorphous.

The ^{29}Si MAS NMR spectra of C-S-H samples are shown in Fig.5. The spectrum of each sample is dominated by a peak at approximately -79 ppm assigned to Q^1 site, which is typical signal from C-S-H and has been reported by many researchers^[14,15]. A small peak between -82 to -88 ppm assigned to Q^2 site is also observed, and its intensity increases with the age of C-S-H from 1 to 7 days. It implies that silica tetrahedron of the structure of synthesized C-S-H is dominated by dimer, and average silicate chain length increases with the increase of the age. The small broad silicon Q^3 and Q^4 resonances between -100 to -120 ppm of the spectra of RT 1 d seeds and RT 3 d seeds are attributed to unreacted amorphous silica, indicating the C-S-H seeds with the age of 1 day and 3 days synthesized at room temperature are not phase pure but rather a mixture of C-S-H with a small amount of unreacted amorphous silica. Unreacted amorphous silica was not detected in XRD because it was amorphous and its content is low^[15]. In fact, there is also a small amount of unreacted CH in RT 1 d seeds as shown in Fig.2. The ^{29}Si NMR spectrum of WB 5 d seeds is quite similar to that of RT 7 d seeds, but the peak of the former is a little

sharper, indicating 55°C water bath is more favorable to the formation and crystallization of C-S-H.

3.2 Particle size distribution of C-S-H seeds

The particle size distribution of C-S-H samples synthesized in different conditions is shown in Fig.6. It can be found that temperature and the age of synthesis have a significant effect on the particle size distribution of C-S-H. For C-S-H seeds synthesized at room temperature (RT seeds), the size of particle increases and the particle size distribution widens with the increase of age. The narrower the FWHM, the more uniform the particle size. The most probable particle size of RT 1 d seeds and RT 3 d seeds is only 24 and 37 nm, respectively, whereas that of RT 5 d seeds and RT 7 d seeds is 220 and 340 nm, respectively. J Plank^[18] indicated smaller C-S-H are superior seeds materials which can strongly accelerate cement hydration. The full width at half maximum (FWHM) of RT 1 d seeds and RT 3 d seeds is only 12 and 19 nm, whereas that of RT 5 d seeds and RT 7 d seeds is 80 and 186 nm, respectively. The C-S-H synthesized in 55°C water bath (WB seeds) also showed a similar characteristic. With the increase of age from 12 hours to 5 days, the most probable size increased from 255 to 396 nm, and FWHM increased from 119 to 211 nm. However, the size of WB seeds is significantly much larger than that of RT seeds with the same age.

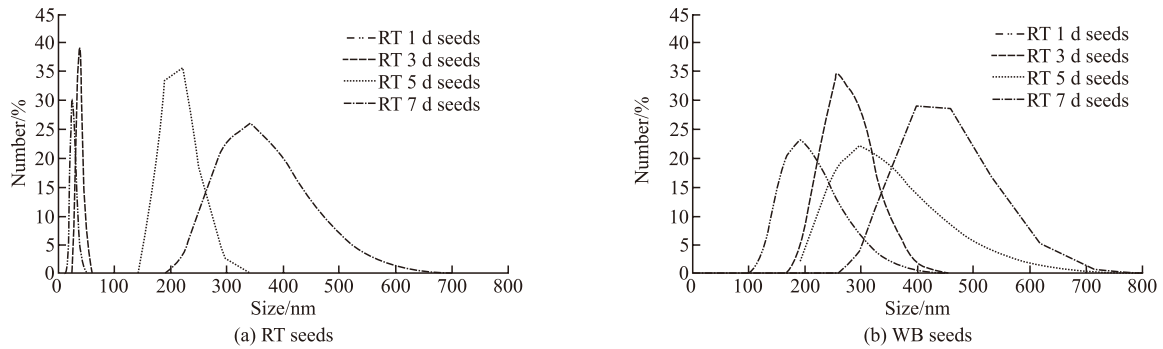


Fig.6 Particle size distribution of C-S-H seeds

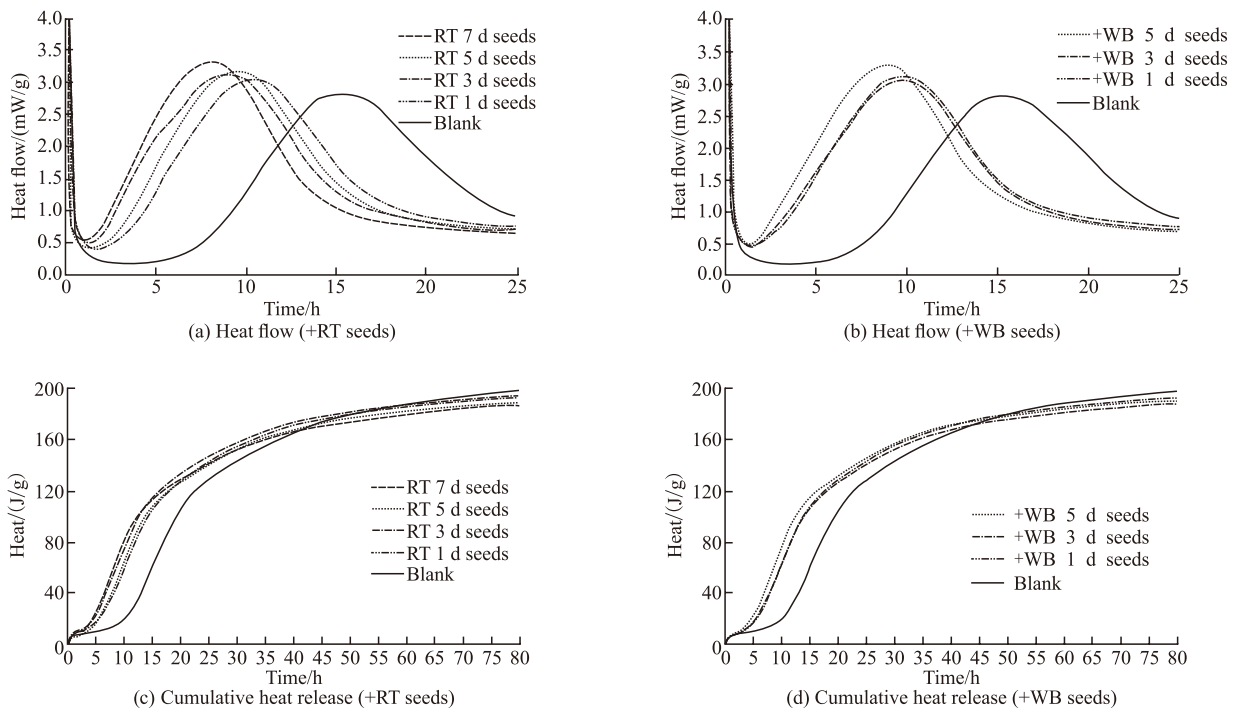


Fig.7 Heat flow and cumulative heat release of cement paste mixed with C-S-H seeds

3.3 Hydration heat of cement seeded with C-S-H

The 7-day isothermal calorimetry data of hydrated Portland cement paste with different C-S-H seeds were obtained. In order to better observe the differences among the data, only the heat flow data of the first 25 hours and the accumulated heat release data of the first 80 hours are shown in Fig.7. The hydration of cement seeded with C-S-H is also divided into five stages: pre-induction period, induction period, acceleration period, deceleration period and continuous slow hydration, which is similar to that of control cement without seeds^[19]. However, C-S-H seeds have a pronounced effect on the beginning time and length of induction period, and exothermic peak.

Fig.7 and Table 2 clearly show a time shift to earlier times and an increase of the maximum of hydration rate due to the addition of C-S-H seeds, indicating the

Table 2 Parameters in the process of hydration and exothermic

Group	The end time of induction period/h	Second exothermic peak		Heat release of 168 hours (J·g ⁻¹)
		Time of appearance/h	Peak (mW·g ⁻¹)	
Blank	2.7	15.2	2.82	214.19
+RT7 d seeds	1.2	8.1	3.32	200.80
+RT5 d seeds	1.5	9.7	3.17	202.71
+RT3 d seeds	1.3	9.1	3.14	207.87
+RT1 d seeds	1.7	10.7	3.05	206.45
+WB5 d seeds	1.4	8.9	3.28	202.32
+WB3 d seeds	1.5	9.8	3.06	200.91
+WB1 d seeds	1.5	9.7	3.13	206.31

accelerating effect of C-S-H seeds on cement hydration. The second exothermic peak of hydration rate appeared 5-7 hours earlier. The second exothermic peak value of cement added with RT 7 d seeds was 3.32 J/(g·h), which is 17.7% higher than that of blank sample.

The peak value of other cement seeded with the C-S-H ranged from 3.05 to 3.28 J/(g·h), which is 8.2%-12.4% higher than of blank sample. Compared with the cement paste without C-S-H, the hydration heat release of cement paste mixed with synthetic seeds under different conditions decreased by 2.95% - 6.25%. It is possible that the C-S-H seeds are wrapped on the surface of the unhydrated cement particles in the early stage of hydration, which makes a part of the cement particles not fully hydrated.

Kanchanason *et al.*^[20] have prepared C-S-H/PCE composites with particle sizes ranging from 35 to 535 nm and compared their acceleration behavior. Acceleration was enhanced when equal masses of smaller seeds were used. Combined with the observation that a larger amount of C-S-H enhances acceleration, this indicates that the total number of seeds added, or the total surface provided, might be crucial factors. Nicoleau *et al.*^[21] have thoroughly investigated the correlation of seeds quantity and acceleration with respect to cement mineralogy. They found 4 areas of accelerating behavior, depending on the amount of seeds added to different cement pastes. For overly small quantities in some cements, an annihilation of acceleration was observed, leading to an initial plateau, followed by quasi linear accelerating behavior with rising seeds concentration.

Comparative analysis of the literature shows that the C-S-H seeds synthesized in different conditions and ages have different quantity, so that the concentration of C-S-H suspension is different, and the degree of acceleration of Portland cement is also different under hydration reaction. At the time of 12 h hydration, the order of hydration rate (from large to small) was RT1 d > WB1 d > WB3 d > RT5 d > RT3 d > WB5 d > RT7 d > control.

3.4 Effect of C-S-H seeds on the mechanical properties of Portland cement

The compressive strengths of cement paste mixed with different C-S-H seeds are shown in Fig.8. After mixing with C-S-H seeds, the compressive strength of each paste increases significantly before the age of 28 days, but there is no significant difference at 28 days. G Land^[22] found that the strength of 12 h was doubled by adding 0.1 wt% C-S-H, which may be improved by increasing the concentration of C-S-H seeds. The improvement of compressive strength of cement paste in this work is more obviously. RT 1 d seeds and RT 3 d seeds have the most obvious improvement on the strength at 12 hours and 1 days, which increase about 30 and 27 MPa respectively. The room temperature

synthesis conditions seem to be more effective to the improvement of compressive strength of cement paste at early age than 55 °C water bath. However, the effect of seeds on the strength of cement paste does not change regularly with the synthesis temperature or age.

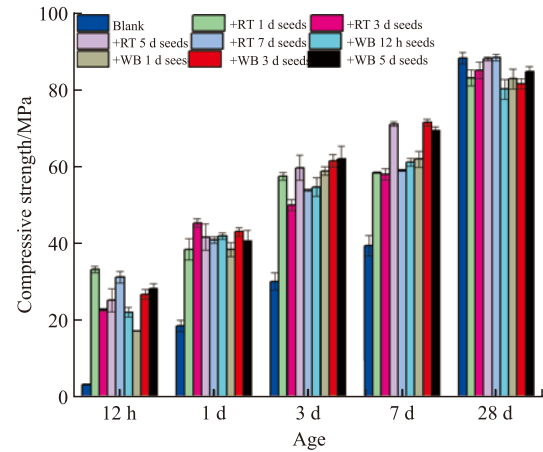


Fig.8 Compressive strength development of cement paste mixed with C-S-H seeds

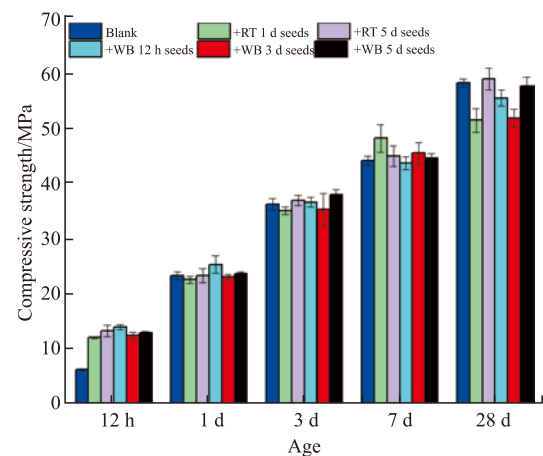


Fig.9 Compressive strength development of cement mortar mixed with C-S-H seeds

Fig.9 and Fig.10 show the mechanical properties of cement mortar mixed with C-S-H seeds of different synthesis temperatures. The 12 hours strength of all the mortar added with C-S-H seeds is more than double that of blank sample. Combined with the results in Fig. 8, it can be found that the increase range of the mortar strength due to the addition of C-S-H seeds is smaller than that of the paste. With the increase of curing age, the strength increases step by step. Similar to the results of cement paste, C-S-H seeds synthesized at room temperature seems to be more effective to the improvement of the flexural strength of cement mortar at 12 hours than that synthesized at 55 °C water bath. X F Wang^[23] proposed that the thermo mechanical properties of C-S-H composites varied with the porosity and compo-

ment ratio within a certain range. The volume, shear and Young's modulus of C-S-H composites decrease with the increase of temperature.

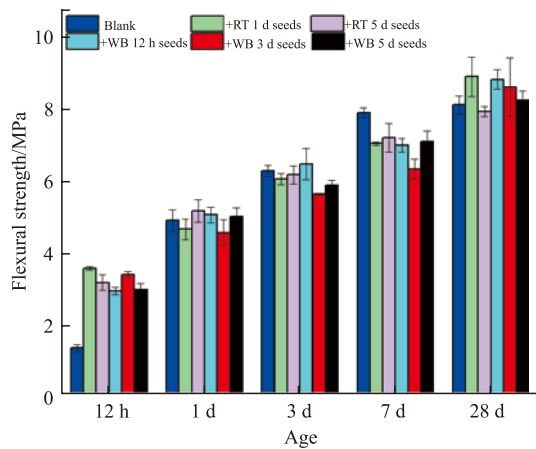


Fig.10 Flexural strength development of cement mortar mixed with C-S-H seeds

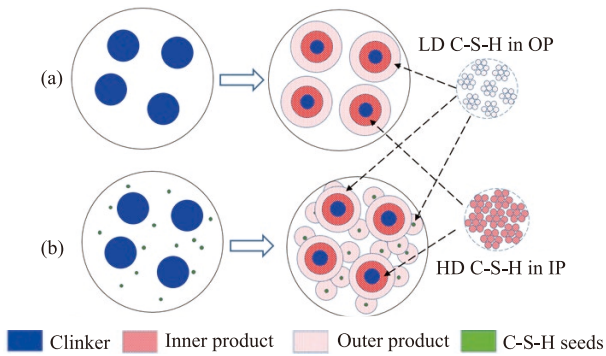


Fig.11 Schematic diagram of cement hydration:
(a) control cement; (b) cement mixed with C-S-H seeds

The acceleration of hydration (Fig.7) and significant improvement of early strength (Fig.8-10) of cement by C-S-H seeds are related to the nucleation effect, which decreases the apparent activation energy of the paste. As shown in Fig.11, C-S-H seeds become secondary nucleation for hydration products, which reduces the inhibition of clinker dissolution. In addition, nucleation in the pore space removes Ca^{2+} and OH^- from the solution, which further accelerates clinker dissolution. At early age, more formation of outer products with low density due to the increase of precipitation spots induced by the C-S-H seeds reduced the porosity of cement-based material, which increased significantly the early strength. However, with the increase of the age, whether the cement paste with or without C-S-H seeds, more inner products (IP) with high density are also formed due to the further hydration of clinker. High density C-S-H (HD C-S-H) has higher nano-mechanical property than low density C-S-H (LD C-S-H). Therefore, with the increase of age, the improvement of

cement strength by C-S-H seeds is not obvious as that in the early stage, and sometimes it is slightly lower.

It is very complicated of the influence of the synthesis temperature and age of C-S-H seeds on the hydration and mechanical properties of cement. C-S-H seeds with shorter age has smaller particle size and larger specific surface area. At the same content, there are more seeds particles and nucleation sites per unit volume for C-S-H seeds with shorter age, which can promote the formation of more hydration products. The acceleration of cement hydration and the improvement of cement strength are more obvious than those of seeds with longer age. However, the seeds with longer age are phase-pure C-S-H, whereas C-S-H seeds with shorter age may not be pure C-S-H, such as CH or amorphous silica residue. Although the nano CH or SiO_2 also have some acceleration effect on cement hydration, the acceleration effect is not as good as C-S-H seeds [2]. In addition, the crystallinity and Q^2/Q^1 value of C-S-H seeds increase with age (Fig.5), and the nano-mechanical properties may be higher, which may result in higher nano properties of C-S-H precipitation induced by C-S-H seeds. The above effects may make the effect of C-S-H seeds on cement hydration and mechanical properties not show a very regular change with synthesis age of seeds.

Similarly, the temperature of C-S-H seed synthesis affects its growth rate, particle size distribution and nanostructure. Compared with the room temperature condition, the water bath accelerates the growth rate of the seeds, which makes the particle size of the seeds increase in a short period of time, and the crystallinity and Q^2/Q^1 value also increase. Figs.8-10 show WB 12 h seeds also significantly improve the early mechanical properties of cement.

Considering the effect of C-S-H seeds on the acceleration of cement hydration, the improvement of cement mechanical properties, and economy and short cycle of seeds synthesis, the C-S-H seeds synthesized at room temperature for 1 day and 55 °C water bath for 12 hours are recommended.

4 Conclusions

a) C-S-H series are synthesized at different temperatures (room temperature and 55 °C) and ages (12 hours - 7 days) by pozzolanic reaction of fresh calcium oxide, nano silica gel, and deionized water. A small amount of unreacted CH or amorphous silica are found in the C-S-H seeds synthesized less than 3 days at room

temperature, and other seeds are pure-phase C-S-H.

b) Particle size distribution, and nano microstructure of C-S-H change regularly with the temperature and age of C-S-H synthesis. The growth and multiplication of C-S-H seeds can be accelerated by water bath at 55 °C. The particle size, crystallinity, basal spacing and Q^2/Q^1 ratio of C-S-H increases with the increase of the temperature and age of synthesis. The most probable particle size of C-S-H seeds synthesized less than 3 days at room temperature is less than 40 nm, whereas that of other C-S-H seeds is more than 200 nm.

c) The addition of synthesized C-S-H seeds to cement pastes results in the strong acceleration effect on cement hydration and significant improvement of the early strength of cement paste and mortar. The maximum value of hydration rate increases about 10%-20%, and the maximum appears 5-7 hours earlier. In the early stage, more low-density outer products were formed due to the increase of precipitation sites induced by C-S-H seeds, which reduced the porosity of cement-based materials and significantly improved the early strength. The 1 day-C-S-H seeds synthesized at room temperature can increase the strength of cement paste by about 30 MPa at 12 h.

d) The effect of C-S-H seeds on the acceleration of cement hydration and the improvement of mechanical properties does not show a very regular change with the increase of the temperature and age of seeds synthesis. Considering the effect of C-S-H seeds on the acceleration of cement hydration, the improvement of cement mechanical properties, and economy and short cycle of seeds synthesis, the C-S-H seeds synthesized at room temperature for 1 day or 55 °C water bath for 12 hours is recommended.

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