



Influence of lithium salt on the performance of calcium sulfoaluminate cement

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Abstract

This work explores the effect of lithium salt on the properties of calcium sulfoaluminate (CSA) cements where different proportions of gypsum are added. Experiments comprising hydration heat release, setting time, mechanical strength and hydration products are studied. The results showed that the addition of Li_2CO_3 significantly accelerates the early hydration and decreases the setting time of CSA cement whatever the gypsum content added to cement. When the content of Li_2CO_3 increases, the setting time decreases accordingly. When gypsum is incorporated, the compressive strength development of the mortars with Li_2CO_3 suffers a decrease, especially after one day of hydration. When the amount of gypsum increases, the later strength of the mortars with Li_2CO_3 exhibits no obvious decrease. The addition of Li_2CO_3 inhibits $\text{C}_4\text{A}_3\bar{\text{S}}$ hydration of CSA cement comprising 10% gypsum at 1 day but promotes ettringite formation after 28 days of hydration. In contrast, the presence of Li_2CO_3 promotes $\text{C}_4\text{A}_3\bar{\text{S}}$ hydration of CSA cement without gypsum addition.

Keywords Calcium sulfoaluminate cement · Lithium salt · Setting time · Compressive strength · Hydration

Introduction

Calcium sulfoaluminate (CSA) cement is receiving great attention due to their environmental benefits in contrast with Portland cement (PC) [1, 2]. CSA cement consists ye'elimite ($\text{C}_4\text{A}_3\bar{\text{S}}$) and different proportions of belite (C_2S), calcium sulfate ($\bar{\text{C}}\text{S}$) and aluminoferrite (C_4AF). CSA cement has excellent properties, such as high early strength, rapid setting, low permeability and shrinkage compensating, which are highly dependent on the hydration of $\text{C}_4\text{A}_3\bar{\text{S}}$ with calcium sulfate [3–5]. For those reasons, CSA cement represents a sustainable alternative to PC. Currently, CSA cement is mainly applied in winter construction projects, precast products and emergency repairs [6]. Although CSA cement is receiving increasing attention, many barriers prevent their industrial-scale production and widespread usage [7].

The hydration mechanism of CSA cement is well characterized. Due to the reaction of $\text{C}_4\text{A}_3\bar{\text{S}}$ with calcium sulfate, ettringite and aluminum hydroxide form as the primary early age hydration products. As soon as calcium sulfate

is deficient, monosulfate forms together with aluminum hydroxide [8–10]. The quantities of precipitated ettringite and monosulfate are thus closely related to the type and amount of added calcium sulfate. Moreover, the content of added calcium sulfate significantly affects the distribution of hydration products and the properties of CSA cement-based materials [9]. It is found that calcium sulfate speeds up the hydration kinetics of $\text{C}_4\text{A}_3\bar{\text{S}}$ at early ages [11]. Additionally, the strength development and dimensional stability of CSA cement are also sensitive to the content of calcium sulfate. If properly formulated, a great deal of CSA cement-based materials can be manufactured [12].

CSA cement is mostly used in small-scale construction engineering due to lack of formalized design guidance. When CSA cement is applied in quick repairs, accelerators are necessary to meet the requirements of rapid setting and hardening in quick repairs. It is known that lithium salts can speed up the hydration of calcium aluminate cement as well as CSA cement [13–15]. The acceleration may be related to the fast emergence of Li-containing aluminum hydroxide. Its occurrence provides seeds for promoting the formation of amorphous aluminum hydroxide, which conversely accelerates the hydration process of cements [16]. Some work has been done to discuss the effect of lithium ions on the hydration of CSA cement at early ages [16]. The results showed

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that the hydration is sped up by a rise in the lithium concentration up to 30 $\mu\text{mol Li/g}$ of the CSA cement and then levels off. Therefore, a small amount of lithium is enough for the acceleration of CSA cement. The acceleration of cement hydration can cause the changes of the macro-properties, like setting, hardening and the mechanical properties. However, the deep study on the development of the properties of CSA cement with lithium salts is very limited. Thus, it is important to incorporate the appropriate amount of lithium salts to improve the performance of CSA cement.

As calcium sulfates and lithium salts affect the early hydration of CSA cement by different mechanisms, the presence of sulfate ions may change the hydration process accelerated by lithium ions. Consequently, the objective of this article is to discuss the effect of lithium salt on the hydration and properties of CSA cement with variable gypsum contents. The early hydration of cement pastes was characterized by the heat evolution and setting time. Moreover, the mechanical strength, hydrates assemblage and microstructure were also investigated. This study will provide new insight for the application of CSA cements in building technologies.

Experimental

Materials

A CSA cement clinker and natural gypsum (NG) were utilized in this work to prepare the cements. The CSA cement clinker was manufactured by Liujiu Cement Co., Ltd (China). The chemical compositions and phase contents of CSA cement clinker and NG are listed in Table 1. The main compounds of the CSA cement clinker are $\text{C}_4\text{A}_3\bar{\text{S}}$ and C_2S . In addition, the CSA cement clinker contains other minor compounds including C_4AF , mayenite (C_{12}A_7) and $\bar{\text{C}}\bar{\text{S}}$. It

has a Blaine fineness of $3500 \text{ cm}^2 \text{ g}^{-1}$. Analytical grade lithium carbonate (Li_2CO_3) was used as the accelerator.

Mix design and methods

Four binder compositions were investigated. The CSA cement clinker was replaced by an increased amount of NG from 5%, 10% and 15 mass%, and marked with the symbol of C1, C2 and C3, respectively. The CSA cement clinker was considered as a reference (marked as C0). The amount of Li_2CO_3 was by mass of clinker-gypsum combinations, e.g., 0.00%, 0.03%, 0.07% and 0.1%. The lithium salt was dissolved in advance into the mixing water to ensure a uniform dispersion of the accelerator in the sample.

The setting time was measured by a standard Vicat apparatus according to Chinese standard GB/T 1346-2001. The standard mortars consisted of the cement, sand and water with the proportion of 1:3:0.5. The mortars were then cast in the molds (40 mm \times 40 mm \times 160 mm). The molds were transferred to the curing room where the temperature and relative humidity (RH) were 20 °C and 95%, respectively. After one day of curing, the mortars were demolded and continuously cured in water at 20 °C. At the specific ages, the mortars were utilized for mechanical strength test on the basis of Chinese standard GB/T 17671-1999. The value of the compressive strength was the average of six samples. With regard to the volume stability measurements, the mortars were also composed of the cement, sand and water with the proportion of 1:3:0.5. The mortars were cast in the molds (25 mm \times 25 mm \times 280 mm). Then, the molds were cured by using the same procedure described above. After one day of curing, the mortars were demolded, and the initial length was measured immediately. The mortars were subsequently transferred to cure in air and water, respectively, and the length changes were measured at the specific testing ages.

The hydration heat release was examined by isothermal conductive calorimetry. Experiments were conducted at 20 °C by employing an eight channel TAM AIR conductive calorimeter. The water-cement ratio was 0.5. The measurements were conducted at the atmosphere of air.

The cement pastes were prepared at a constant water-cement ratio of 0.4. At the specific period, the cement pastes were crushed and immersed in ethanol for 48 h in order to stop the cement hydration. Then, the samples were transferred to a vacuum desiccator with a temperature of 40 °C. Finally, the samples were ground to pass through a 200 mesh sieve for X-ray diffraction (XRD) analysis.

The XRD patterns were recorded on an X-ray diffractometer (Bruker D8 Advance diffractometer; Cu K α radiation). The X-ray tube was conducted at 40 kV and 40 mA. Step scans were carried out in the range of 5–65° with a step size of 0.02°. TOPAS 4.2 software was applied for the

Table 1 Chemical compositions and phase contents of CSA cement clinker and NG/%

Oxide	CSA clinker		NG		Minerals	
	CSA clinker	NG	CSA clinker	NG	CSA clinker	NG
CaO	42.1	32.3	$\text{C}_4\text{A}_3\bar{\text{S}}$	65.0	–	–
SiO ₂	10.5	2.0	C_2S	18.7	–	–
Al ₂ O ₃	32.0	1.0	C_4AF	5.5	–	–
Fe ₂ O ₃	1.9	0.5	C_{12}A_7	6.5	–	–
MgO	1.4	1.3	$\bar{\text{C}}\bar{\text{S}}$	4.3	–	–
Na ₂ O	0.1	0.6	$\text{C}\bar{\text{S}}\text{H}_2$	–	–	96.5
K ₂ O	0.3	1.2	$\bar{\text{C}}\bar{\text{S}}$	–	–	2.9
SO ₃	9.0	39.5	SiO ₂	–	–	0.6

Rietveld quantitative analysis of the samples. Microstructure and morphology were identified by scanning electron microscopy (SEM).

Results

Hydration heat release

Figure 1 depicts the hydration heat release of CSA cements with the addition of Li_2CO_3 . The initial peak which occurs after the cement gets in touch with the water is ascribed to the wetting and dissolution of the samples as well as the occurrence of the hydration reactions [17–19]. The first hydration heat release is possibly ascribed to the dissolution of $\text{C}_4\text{A}_3\bar{\text{S}}$ and the initial formation of hydrates [20–22]. Sample C0 shows a short dormant period after the initial peak until about 1 h. Afterward, sample C0 is characterized by two peaks which occur at about 1.9 h and 4 h, respectively. The first hydration peak is corresponding to the consumption of gypsum and the formation of ettringite together with aluminum hydroxide [23, 24]. The second peak presents the period of a continuous hydration reaction after the gypsum depletion and monosulfate and more ettringite form as the main hydration products [25]. When Li_2CO_3 is added to sample C0, the dormant period disappears and the heat flow reaches the first maximum around 15 min of hydration, indicating a stronger acceleration of hydration. Moreover, this heat peak is more intense than the corresponding one at about 1.9 h in the case of sample without Li_2CO_3 addition. The subsequent hydration peak occurs at about 2.5 h, which is earlier than that of sample without Li_2CO_3 (4 h). This reflects the trend of Li_2CO_3 to accelerate the formation of sulfoaluminate hydrates. Besides, the third heat peak of sample C0 with Li_2CO_3 occurs after 5 h of hydration. When

10% gypsum is added to CSA clinkers, the initial peak is more intense and the dormant period is significantly shortened. After the dormant period, sample C2 is characterized by two peaks with their maxima at 0.7 h and 1.6 h with a shoulder at 2.1 h, respectively. It can be found that the presence of gypsum remarkably accelerates the hydration of CSA cements. In comparison, the hydration peaks of sample C2 with Li_2CO_3 occur at 0.4 h and 1.5 h with a shoulder at 2.6 h, respectively, which are also earlier than these of sample without Li_2CO_3 . It can be seen that the addition of Li_2CO_3 increases the heat flow of the first maximum but decreases the heat flow of the subsequent peaks for sample C2, while for sample C0, the presence of Li_2CO_3 enhances the heat flow of all hydration peaks.

Setting time

The setting time of CSA cements with different concentrations of Li_2CO_3 is demonstrated in Fig. 2. The addition of Li_2CO_3 significantly affects the setting time of CSA cements. The incorporation of Li_2CO_3 results in a prominent decrease in the setting time. This indicates that Li_2CO_3 accelerates the setting process of CSA cements. The phenomena agree with the findings of the heat release of CSA cements (Fig. 1). The setting time of CSA cement was observed to be corresponding to the time of the peak of hydration heat [26]. It is observed that the first heat peak for samples with Li_2CO_3 is higher and occurs faster than that of samples without Li_2CO_3 . Therefore, the earlier precipitation of the hydrates causes the setting time of CSA cements with Li_2CO_3 to become shorter. Moreover, whatever the gypsum content added to the cements (0%, 5%, 10% or 15%), the setting time decreases with the increased amount of Li_2CO_3 . When the dosage of Li_2CO_3 increases to 0.07%, there is a slight decrease in the setting time. The initial setting time

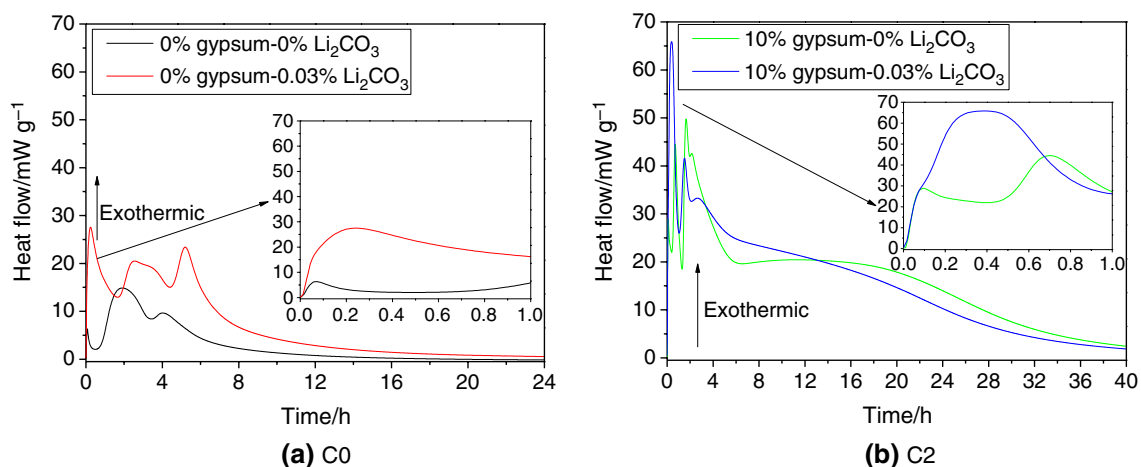
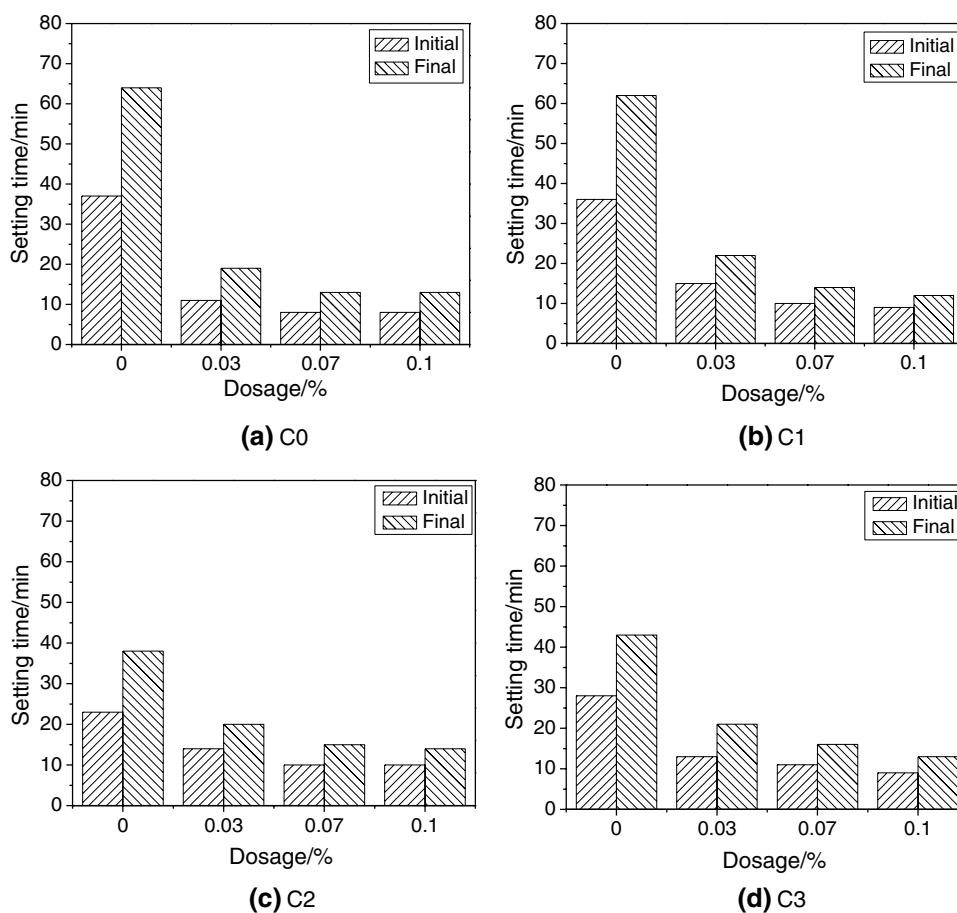


Fig. 1 Influence of Li_2CO_3 on the hydration heat release of CSA cements

Fig. 2 Influence of Li_2CO_3 on the setting time of CSA cements with different gypsum contents



and final setting time of sample C0 are shortened to 8 min and 13 min in the presence of 0.1% Li_2CO_3 , respectively. The presence of gypsum causes a decrease on the setting time of CSA cements in the absence of Li_2CO_3 . When the content increases from 0 to 10%, the initial setting time of CSA cements decreases from 37 to 23 min, while the same content of gypsum significantly decreases the final setting time from 64 to 38 min. However, the setting time of CSA cements is slightly prolonged when the cement contains 15% gypsum.

Mechanical strength

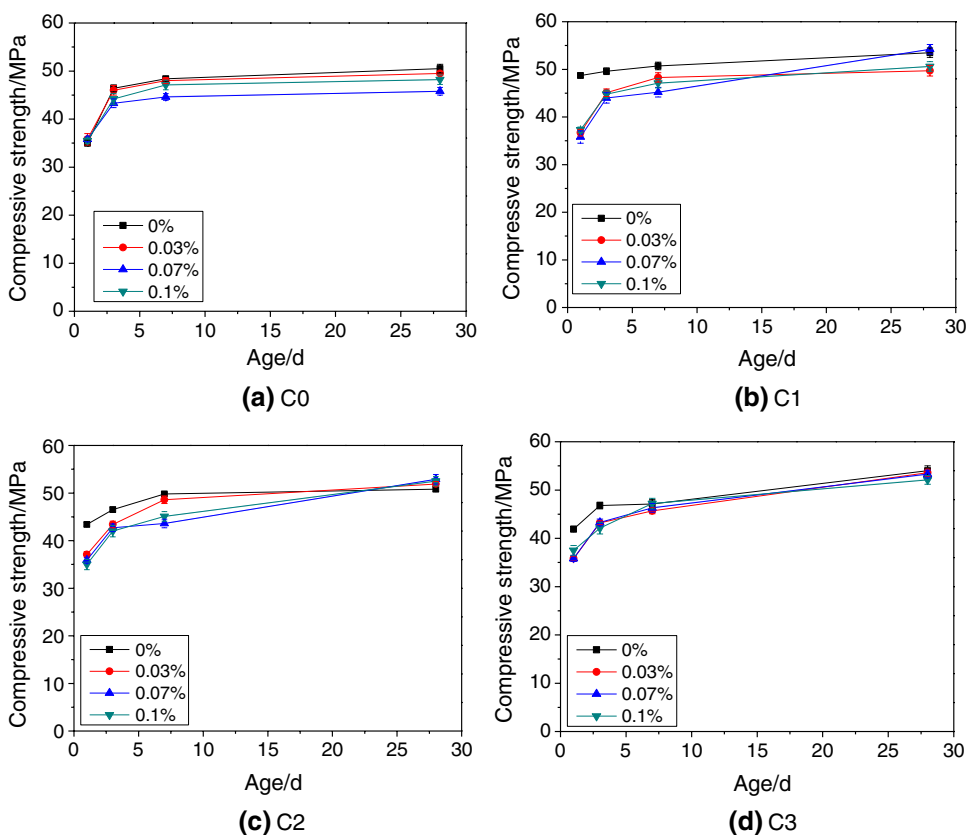
The effects of Li_2CO_3 on the compressive strength development of CSA cements are demonstrated in Fig. 3. The main strength increase is observed after 7 days of hydration. The CSA cements show rapid early strength because the hydration reactions of C4A3S and gypsum initiate rapidly and thus lead to the precipitation of ettringite and aluminum hydroxide [27]. The effect of Li_2CO_3 on the compressive strength development varies for different cements. After one day of hydration, the strength development of sample C0 with the addition of Li_2CO_3 is almost equal to that of the reference cement. When the hydration proceeds from 3 to 28 days,

adding Li_2CO_3 into the CSA cements slightly decreases the strength of the mortars. The lowest strength is recorded for specimen with 0.07% Li_2CO_3 . The incorporation of gypsum enhances the compressive strength of the mortars, particularly at 1 day. It can be seen that a dosage of 5% gypsum is optimum for the compressive strength of CSA cements. In the presence of gypsum, the strength of the mortars with the addition of Li_2CO_3 suffers a decrease, especially after 1 d of hydration. With increasing Li_2CO_3 contents, it can be found that there are no obvious differences in the strength development except for sample C1 with 0.07% Li_2CO_3 at 28 d and sample C2 with 0.03% Li_2CO_3 at 7 d. The compressive strength of sample C2 with the presence of Li_2CO_3 becomes slightly higher than the reference cement at 28 d. As gypsum content increases, the later strength of the mortars with Li_2CO_3 exhibits no obvious decrease.

Volume stability

CSA cements have shown shrinkage compensation during the manufacturing process [28]. The hydration of C4A3S with calcium sulfate forms ettringite and aluminum hydroxide, which results in the expansion and early age strength development of CSA cements [29]. Moreover, if significant

Fig. 3 Compressive strength development of CSA cements comprising different gypsum contents with dosages of Li_2CO_3 varying from 0 to 0.1%



ettringite forms after hardening, cracking may occur [30–32]. Thus, it is worth to study the volume stability of CSA cements. Figures 4 and 5 display the dimensional stability of mortars for C0 and C2 system with different Li_2CO_3 contents. As can be seen from Fig. 4a, when cured in air, the Li_2CO_3 -added samples (C0) exhibit relatively lower drying shrinkage compared with the blank sample. All mortars exhibit rapid shrinkage up to 7 days, and then, the shrinkage achieves a plateau until 28 days. The mortar with 0.03% Li_2CO_3 yields the lowest shrinkage among the investigated mortars. As expected, much lower drying shrinkage is obtained for the mortars due to the incorporation of gypsum.

When gypsum is incorporated into the cement, the effect of Li_2CO_3 on the drying shrinkage is not significant. The mortar with 0.1% Li_2CO_3 always obtains the lowest drying shrinkage within the curing age. After 7 d of hydration, all the mortars exhibit similar drying shrinkage.

When cured in water, 0.03% and 0.1% of Li_2CO_3 result in much higher expansion rate of sample C0, while the expansion rate of sample with 0.07% Li_2CO_3 is similar to that of the reference mortar. The addition of gypsum results in much higher expansion of the mortars. Enhancing the content of gypsum leads to more ettringite formation, which increases the risk of expansion [32]. However, the expansion

Fig. 4 Influence of Li_2CO_3 on the dimensional changes of CSA cements cured in air

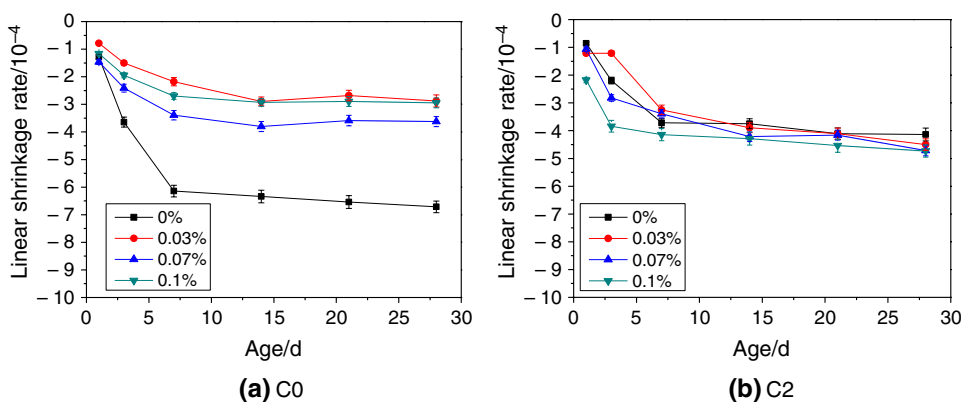


Fig. 5 Influence of Li_2CO_3 on the dimensional changes of CSA cements cured in water

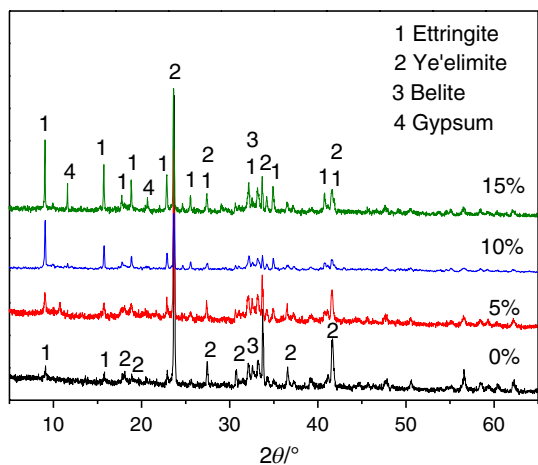
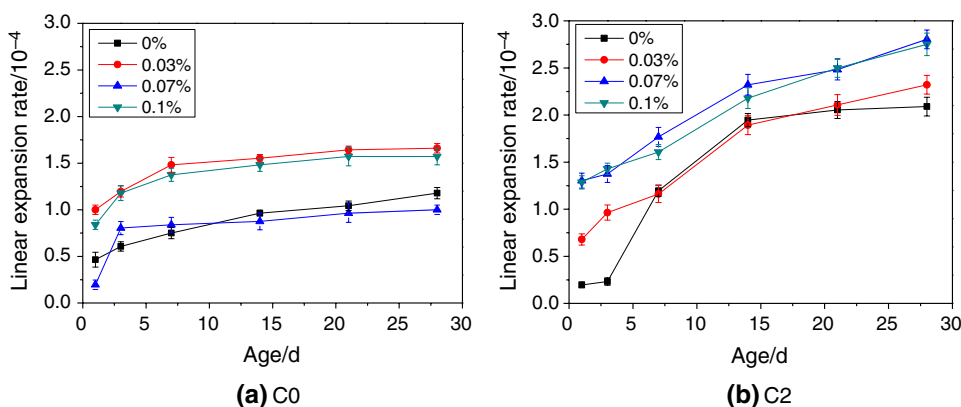


Fig. 6 XRD patterns of cements containing different gypsum contents after 1 d

rates of sample C2 are within 3×10^{-4} at 28 d which does not affect the volume stability of CSA cements. When gypsum is incorporated into the cement, 0.07% and 0.1% of Li_2CO_3 result in much higher expansion rate of sample C2, while the expansion rate of sample with 0.03% Li_2CO_3 is similar to

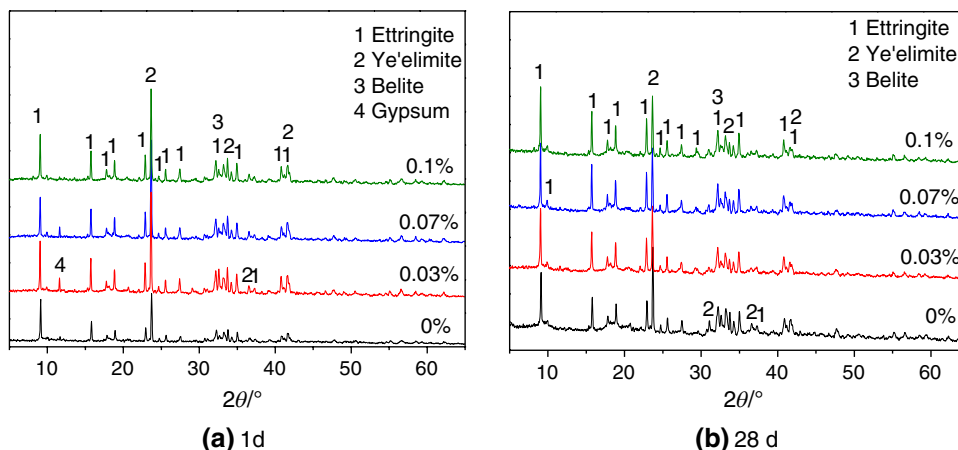
the reference cement mortar. The results in this investigation indicate that the addition of Li_2CO_3 does not have adverse effect on the volume stability of CSA cement.

Hydration products

The XRD patterns of cements containing different gypsum contents after 1 d are presented in Fig. 6. It is observed from the XRD patterns that $\text{C}_4\text{A}_3\text{S}$ and C_2S do not react completely in the pastes. All samples display the formation of ettringite even in the paste without gypsum addition. The trace of aluminum hydroxide is not found by XRD due to its poor crystallized structure [11, 33]. Due to the rise of gypsum content, the intensity of ettringite peaks increases significantly, while the peaks of $\text{C}_4\text{A}_3\text{S}$ decrease. In the cement paste free from gypsum, a small amount of ettringite has formed after 1 d. However, precipitation of calcium monosulfoaluminate hydrate is not detected. Gypsum can be clearly identified in the paste with 15% gypsum, showing that the added gypsum is excessive at early ages.

Figure 7 shows the XRD patterns of the C2 system with different Li_2CO_3 contents after 1 d and 28 d. It can be found that the dominant hydration product of the C2 system is still ettringite in the presence of Li_2CO_3 . The Li_2CO_3 dosage

Fig. 7 XRD patterns of hydrated C2 with the addition of Li_2CO_3 at **a** 1 d, **b** 28 d



affects the hydration of $C4A3\bar{S}$. After 1 day of hydration, the characteristic peaks of $C4A3\bar{S}$ appear to become strong with the addition of Li_2CO_3 . Moreover, gypsum can be clearly observed in the paste containing 0.03% and 0.07% Li_2CO_3 . The finding shows that the addition of Li_2CO_3 inhibits the hydration of $C4A3\bar{S}$ at 1 day. After 28 days of hydration, the XRD patterns show some distinctions. An increase in ettringite can be clearly observed and the addition of Li_2CO_3 promotes the ettringite formation. Traces of gypsum are not noticed.

Figure 8 shows the XRD patterns of hydrated C0 and C3 with the addition of Li_2CO_3 at 1 d. It can be found that the intensity of ettringite peaks significantly increases with the presence of Li_2CO_3 , while the intensity of $C4A3\bar{S}$ peaks decreases correspondingly for sample C0. In contrast, the

intensity of ettringite peaks slightly decreases with the addition of Li_2CO_3 for sample C3. Moreover, an increase in gypsum can be observed in the sample containing Li_2CO_3 . The results shown in Figs. 7 and 8 are identical to the compressive strength development shown in Fig. 3.

Microstructures of the cement pastes with Li_2CO_3 are presented in Figs. 9 and 10. Based on the results of hydration products, it is discovered that after one day of hydration, a small amount of ettringite has formed in the cement paste free from gypsum. The formed ettringite may be embedded in the hardened paste as given in Fig. 9. As shown in Fig. 10, needle-shaped ettringite crystals can be discovered in the cement paste with 10% gypsum. It can be found that the presence of Li_2CO_3 does not change the morphology of ettringite crystals when compared to the reference sample.

Fig. 8 XRD patterns of hydrated C0 and C3 with the addition of Li_2CO_3 at 1 d

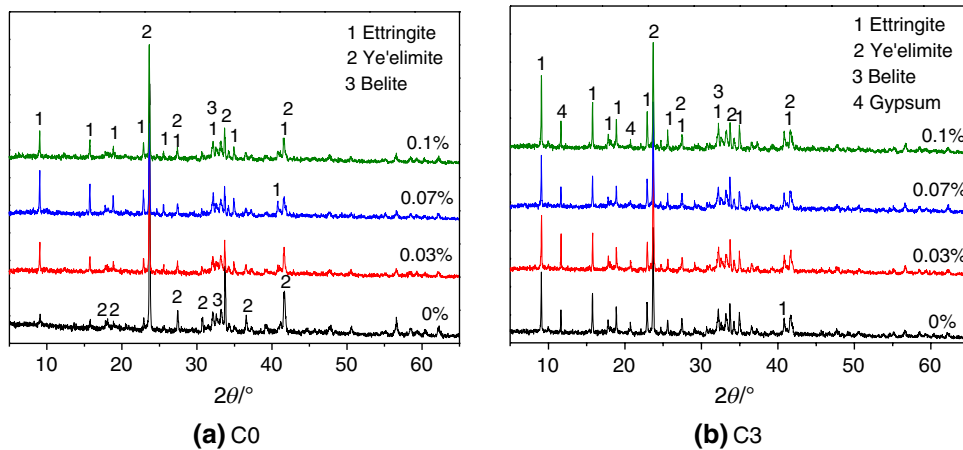


Fig. 9 SEM micrographs of hydrated C0 after 1 d

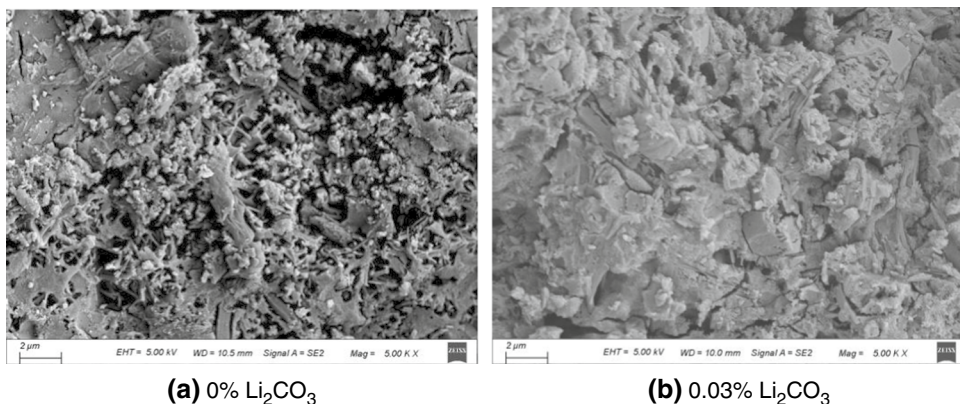
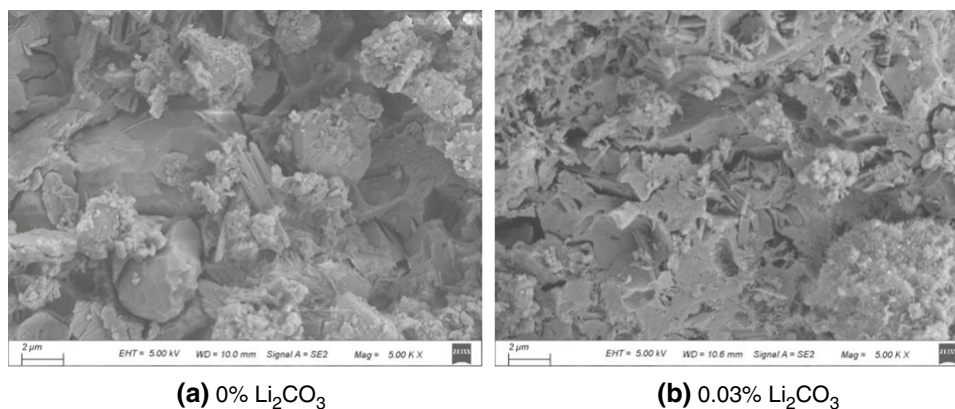


Fig. 10 SEM micrographs of hydrated C2 at 1 d



Conclusions

The influence of lithium salt on the performance of CSA cements was studied. The following conclusions are summarized based on this study:

- (1) The addition of Li_2CO_3 remarkably accelerates the hydration process of CSA cement at early ages and decreases the setting time of CSA cement. Whatever the gypsum content added to the cement, the setting time of CSA cement decreases with the increased amount of Li_2CO_3 .
- (2) In the presence of gypsum, the compressive strength development of the mortars with the incorporation of Li_2CO_3 suffers a decrease, especially after one day of hydration. As gypsum content increases, the later strength of the mortars with Li_2CO_3 exhibits no obvious decrease.
- (3) Compared with the reference mortar, the addition of Li_2CO_3 results in lower drying shrinkage and higher expansion rate of sample C0. In the presence of Li_2CO_3 , the addition of gypsum causes much lower drying shrinkage and higher expansion rate of the mortars.
- (4) The addition of Li_2CO_3 does not alter the hydration products, but inhibits the hydration of C4A3S of CSA cement containing 10% gypsum at 1 day. In contrast, the presence of Li_2CO_3 promotes the precipitation of ettringite after one day of hydration.

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