

## Research Article

# Influence of Lithium Carbonate on C<sub>3</sub>A Hydration

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Lithium salts, known to ameliorate the effects of alkali-silica reaction, can make significant effects on cement setting. However, the mechanism of effects on cement hydration, especially the hydration of C<sub>3</sub>A which is critical for initial setting time of cement, is rarely reported. In this study, the development of pH value of pore solution, conductivity, thermodynamics, and mineralogical composition during hydration of C<sub>3</sub>A with or without Li<sub>2</sub>CO<sub>3</sub> are investigated. The results demonstrate that Li<sub>2</sub>CO<sub>3</sub> promotes C<sub>3</sub>A hydration through high alkalinity, due to higher activity of lithium ion than that of calcium ion in the solution and carbonation of C<sub>3</sub>A hydration products resulted from Li<sub>2</sub>CO<sub>3</sub>. Li<sub>2</sub>CO<sub>3</sub> favors the C<sub>3</sub>A hydration in C<sub>3</sub>A-CaSO<sub>4</sub>·2H<sub>2</sub>O-Ca(OH)<sub>2</sub>-H<sub>2</sub>O hydration system and affects the mineralogical variation of the ettringite phase(s).

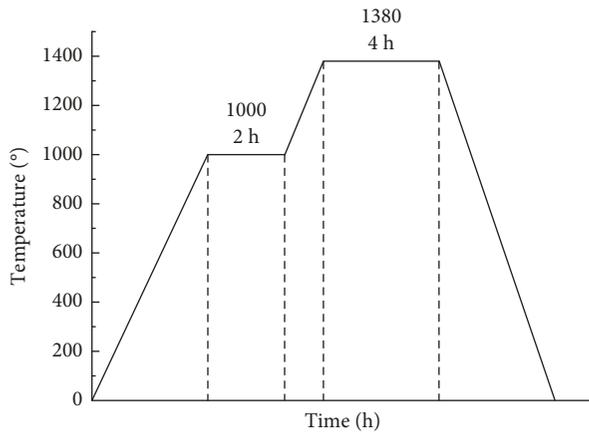
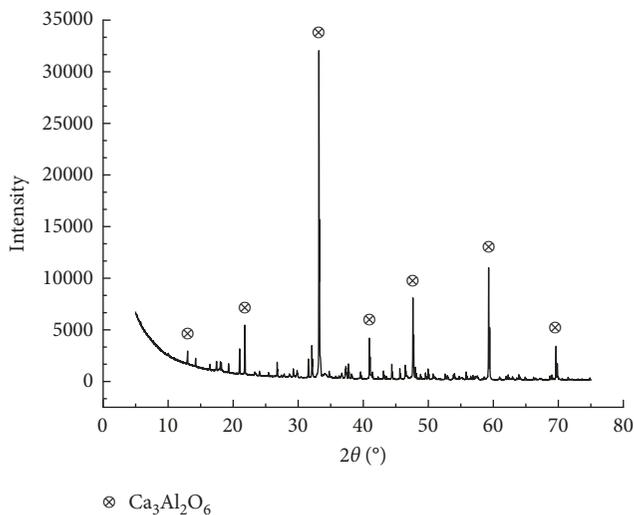
## 1. Introduction

Lithium, one of the most active alkali elements, has high charge density and stable electric double layer. Li<sup>+</sup> has small radius, strong polarization effect, and a bigger hydration radius, which provide a higher chemical activity for lithium salts compared to other salts usually used in the cement paste, such as NaCl and CaCl<sub>2</sub>. Lithium salts have been known to ameliorate the effects of alkali-silica reaction (ASR) for many years. When lithium salts are added to the cement paste or concrete, they significantly affects the properties, especially the setting time [1], expansion caused by the ASR [2], long-term strength [3], and the amount of hydration products [1]. However, most researches focus on the inhibition of alkali-aggregate reaction (AAR), and there are few reports on the cement hydration. There are ongoing debates on the mechanism of lithium salts, especially Li<sup>+</sup>, on the hydration and properties of cement paste and concrete. Ong [4] points out that lithium salts with lower solubility can restrain cement hydration, which is contrary to the fact that lithium ion has the function to accelerate cement hydration, according to the research from Millard and Kurtis [5]. Some researches suggest that the effects of lithium salts on the

properties of cement paste or concrete vary with the categories of cement and lithium salts [6–11] and even the content of lithium salts in cement paste [12]. Some other research shows that lithium salts can react with cement hydration products and form lithium aluminate hydrate, which acts as seeds for more stable hydration products, resulting in the improvement of properties of cement or concrete. Nevertheless, Mo [13] points out that the content of C-S-H produced by cement hydration is reduced for lithium ion intervening AAR. Therefore, it is necessary to investigate the mechanism of effects of lithium ions on cement hydration.

Tricalcium aluminate (C<sub>3</sub>A) is the most important and reactive major component contributing to the early properties of cement or cementitious products and plays a critical role in the early stages of hydration process of Ordinary Portland Cement (OPC) and Calcium Aluminate Cements (CAC). C<sub>3</sub>A reacts with water to form calcium hydroaluminates (i.e., AFt-type phase and C<sub>3</sub>AH<sub>6</sub>), which induces a stiffening to the hardened paste.

In this study, the influence of lithium carbonate on the hydration of C<sub>3</sub>A with or without CaSO<sub>4</sub>·2H<sub>2</sub>O is investigated. A range of analytical techniques, such as conductimetry,

FIGURE 1: Process of  $C_3A$  synthesis.FIGURE 2: XRD patterns of  $C_3A$  phase.

isothermal calorimetry, X-ray diffraction (XRD), and Fourier transformation infrared spectrometer (FTIR), are performed to characterize the hydration process and reveal the mechanism behind.

## 2. Materials and Methods

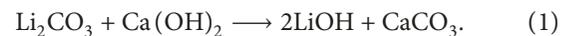
Tricalcium aluminate used in this study is synthesized by heating highly pure limestone (99.0%  $CaCO_3$ ) and technically pure  $Al_2O_3$  (99.0%) with molar ratio 3 : 1 at 1380°C. The mixture of limestone and  $Al_2O_3$  is first mixed for 2 hours and pressed as a pellet with a diameter of about 40 mm and maximum press of 100 kN under a rate of 4 kN/s. The pellets are then calcined at 1000°C for 2 hours before cooling to room temperature and being crushed and ground in absolute ethanol to achieve homogeneity. Then the powders are remoulded and heated at 1380°C for 4 hours. The process (Figure 1) is repeated for three times, and finally the specimens are crushed, finely ground, and passed 80  $\mu m$  sieve. The synthesized powder is analysed by XRD, and almost pure cubic  $Ca_3Al_2O_6$  ( $C_3A$ ) phase is identified (Figure 2), with its

specific surface area of 350  $m^2/kg$  (Blaine's method). The contents of its free lime and insoluble residue are 0.27% and 0.10%, respectively.

$C_3A$  hydration is conducted in a saturated calcium hydroxide solution (namely CC paste) in order to mimic the pore solution and avoid carbonation during early cement hydration. The  $C_3A$  hydration with different additives,  $Li_2CO_3$  (analytical reagent, 1.5% by weight related to the  $C_3A$ , CCL paste) or/and  $CaSO_4 \cdot 2H_2O$  (analytical reagent, 20% by weight related to the  $C_3A$ , CCLS paste) and the control paste (20% by weight related to the  $C_3A$ ) (Figure 3), is also studied based on CC paste proportion. Conductimetry and isothermal microcalorimetry of hydration system are carried out based on the literature [14]. Meanwhile, the paste is dried at 55°C and then ground to a particle size smaller than 45  $\mu m$ , and the mineralogical compositions are performed by XRD (D8, Bruker AXS Corporation, Germany) with a scan rate of 1.000° per min and FTIR (Nicolet 6700, Thermo Electron Scientific Instruments, USA) with a frequency range of 4000–399  $cm^{-1}$ .

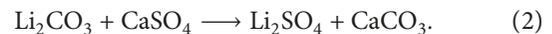
## 3. Results and Discussions

**3.1. Conductivity and pH Value.** The pH value and conductivity of pastes are shown in Figures 4 and 5, respectively. When  $Li_2CO_3$  is added to the CC hydration system, the following reaction takes place [15]:



With a higher activity compared to calcium ion, the addition of  $Li_2CO_3$  improves the alkalinity of  $C_3A$  hydration environment. The dissolution of aluminum ion or its group is promoted as a result of the water molecules around  $Al^{3+}$  being replaced by  $OH^-$ , which favors the  $C_3A$  hydration [12, 16, 17]. Consequently, the pH value and conductivity are increased due to a higher alkalinity and ion concentration in the hydration system.

When  $CaSO_4 \cdot 2H_2O$  is added to the CCL paste, the hydration of CCL paste is retarded or delayed by sulfate ion [18], and the ion concentration is significantly reduced due to the lower solubility of  $CaCO_3$ , formed as follows:



Consequently, CCLS hydration system resulted in lower pH value and conductivity than those of CCL paste.

**3.2. Heat Evolution during Hydration.** According to the results from calorimetry (Figure 6), the addition of lithium carbonate and  $CaSO_4 \cdot 2H_2O$  significantly affects the heat evolution rate (heat flow) of pastes during the first 48 hours of hydration. The peak positions and heat evolution rates of pastes are shown in Table 1. And the heats evolved during hydration in three time ranges are shown in Table 2.

When  $C_3A$  is added into saturated  $Ca(OH)_2$  solution, the first peak of heat evolution rate is generated from the dissolution of  $C_3A$  grains and precipitation of  $3CaO \cdot Al_2O_3 \cdot 6H_2O$  ( $C_3AH_6$ ) at 0.18 hour (3). As the hydration continues, the heat evolution rate decreases,

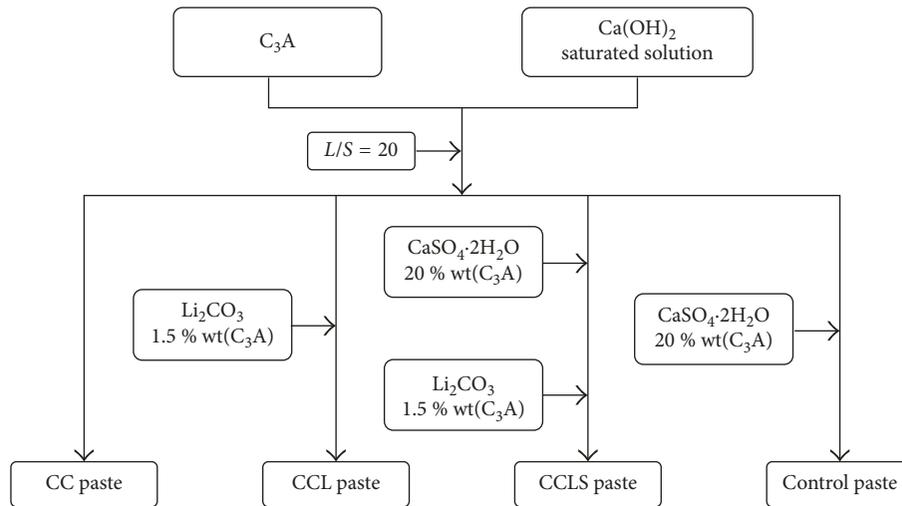


FIGURE 3: The flowchart of pastes preparation.

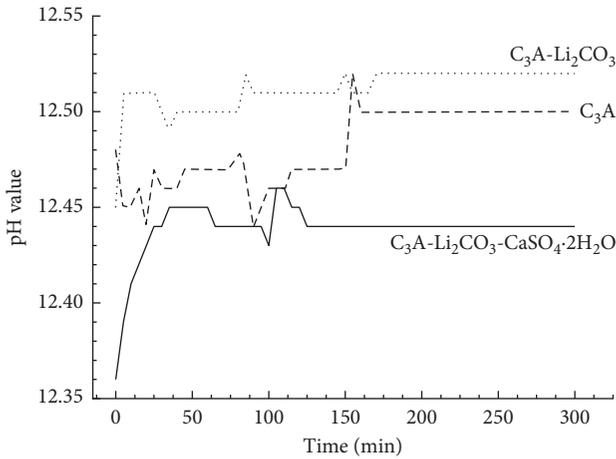


FIGURE 4: pH value of the pastes.

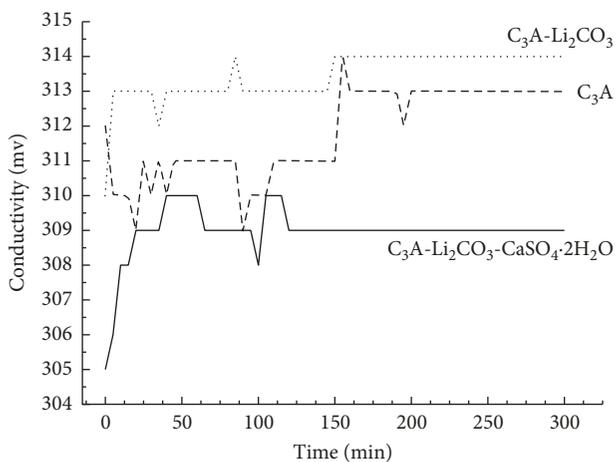
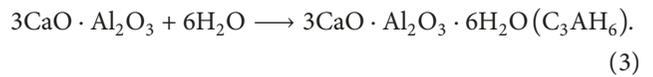
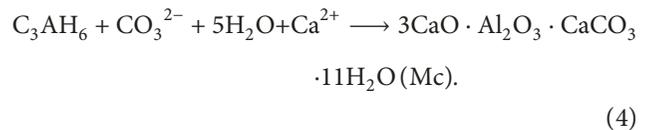


FIGURE 5: Conductivity of the pastes.

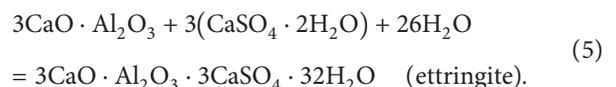
and the hydration progress is gradually controlled by ion diffusion.



As for the hydration of CCL paste,  $\text{C}_3\text{A}$  grains are quickly dissolved, and a large amount of heat is released by the positive effect of  $\text{Li}_2\text{CO}_3$ . However, the first maximum heat evolution rate is reduced because a large amount of microcrystalline hydration products are formed and precipitated on the surfaces of  $\text{C}_3\text{A}$  grains [19], resulting in an earlier but smaller peak than that of CC paste. With the increase of hydration time, the second peak is generated which may be related to the carbonation of  $\text{C}_3\text{A}$  hydration products (4) [19, 20]. After 0.85 hour, the heat flow of CCL paste is higher than that of CC paste at the same time due to  $\text{Li}_2\text{CO}_3$  and carbonation.



When  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  is added to CCL paste, the first maximum heat evolution rate is reduced further due to the retardation of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  on the  $\text{C}_3\text{A}$  hydration despite the positive effect from  $\text{Li}_2\text{CO}_3$ . Then, a broad and lower peak of heat evolution rate is observed, attributing to the formation of  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$  (ettringite) (5), which results in a higher heat flow than that of CCL paste after 4.5 hours at the same time.



As the hydration continues, the total heat evolved of three pastes is shown in Figure 7. The order of total heat evolved changes with the time of hydration but eventually reaches an order (by 48 hours): CCL paste is greater than

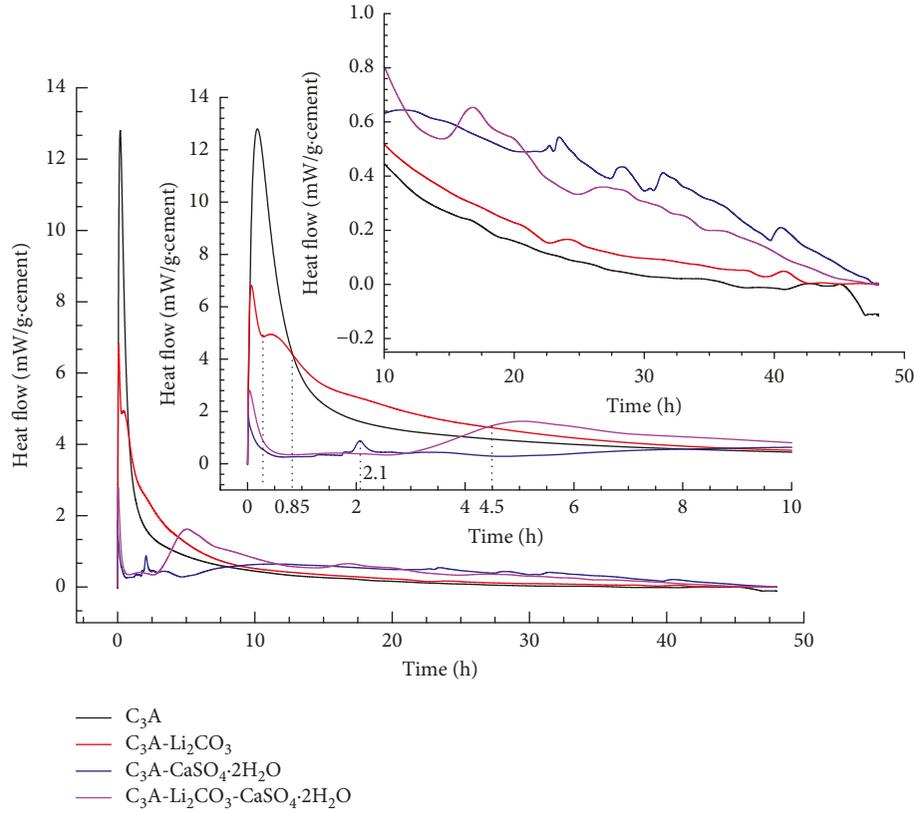


FIGURE 6: Heat flow over time during cement hydration.

TABLE 1: Maximum heat evolution rate and peak position.

Paste	First peak	Second peak
CC	12.80 mW/g-cement at 0.18 h	—
CCL	6.82 mW/g-cement at 0.08 h	4.95 mW/g-cement at 0.48 h
Control	1.86 mW/g-cement at 0.02 h	0.88 mW/g-cement at 2.07 h
CCLS	2.80 mW/g-cement at 0.04 h	1.63 mW/g-cement at 5.06 h

TABLE 2: The comparison about the heat evolution rate of four pastes.

Range	0~0.85 h	0.85~4.5 h	4.5~48 h
CC paste	1st	2nd	4th
CCL paste	2nd	1st	3rd
Control paste	4th	3rd	2nd
CCLS paste	3rd	4th	1st

CCLS paste, CCLS paste is greater than CC paste, and CC paste is greater than control paste. These indicate that  $\text{Li}_2\text{CO}_3$  improves the degree of  $\text{C}_3\text{A}$  hydration.

**3.3. Phase Analysis.** In order to obtain better understanding on the differences of heat flow and heat evolution rate of three pastes, the mineralogical compositions of pastes at 0.85 hour, 2.1 hours, and 4.5 hours are characterized by XRD (Figure 8).

The diffraction patterns of CC paste, CCL paste, and CCLS paste at various ages are shown in Figure 8. According to the methods reported in literature [21], the content of hydration products in these specimens is calculated and shown in Table 3. The content of  $\text{C}_3\text{A}$  in CCL paste is lower than that of in CC paste at the same age, indicating that  $\text{Li}_2\text{CO}_3$  has a positive effect on  $\text{C}_3\text{A}$  hydration. The content of calcium hydroxide (CH) in CCL paste is lower than that of CH in CC paste at the same age, which agrees with the explanation for the pH value and conductivity, so as to suggest that  $\text{Li}_2\text{CO}_3$  improves the  $\text{C}_3\text{A}$  hydration. Combined with the full width at half maximum (FWHM) during the calculation process as reported in literature [21, 22], the results suggest that the content of  $\text{C}_3\text{AH}_6$  in CCL paste is lower than that of in CC paste at 0.85 hour, which is consistent with the result from calorimetry. For CC paste and CCL paste, the content of  $\text{C}_3\text{AH}_6$  at 4.5 hours is smaller than that of  $\text{C}_3\text{AH}_6$  at 2.1 hours, which may be related to the carbonation of  $\text{C}_3\text{AH}_6$  (4).

As for CCLS paste (Figure 8(c)), the main crystalline phases are anhydrous  $\text{C}_3\text{A}$ , ettringite, Mc, Ms, and

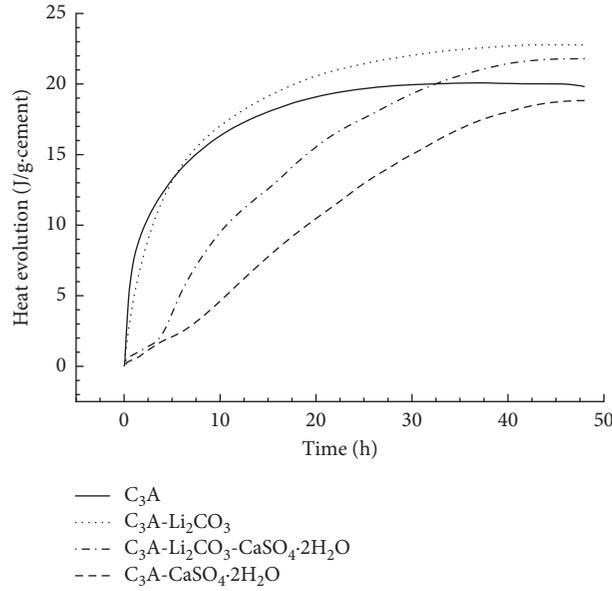


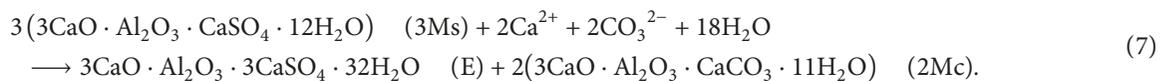
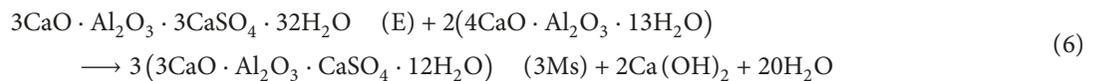
FIGURE 7: Heat evolution over time during cement hydration.

TABLE 3: The content of hydration products in the pastes at various ages (wt.%).

Number	Anhydrous C <sub>3</sub> A	C <sub>3</sub> AH <sub>6</sub>	Mc	Ms	E	G	CH
CC-0.85 h	56.52	40.56	—	—	—	—	2.92
CC-2.1 h	54.62	42.42	—	—	—	—	2.91
CC-4.5 h	51.21	45.92	—	—	—	—	2.87
CCL-0.85 h	47.75	41.94	8.65	—	—	—	1.67
CCL-2.1 h	44.66	44.47	9.32	—	—	—	1.55
CCL-4.5 h	45.68	41.97	11.02	—	—	—	1.33
CCLS-0.85 h	63.16	—	15.23	3.94	10.80	4.01	2.86
CCLS-2.1 h	61.72	—	17.54	4.68	9.52	3.78	2.76
CCLS-4.5 h	58.56	—	20.67	3.26	13.53	1.25	2.73

CaSO<sub>4</sub>·2H<sub>2</sub>O, which are different from CC and CCL pastes but similar to C<sub>3</sub>A-CaSO<sub>4</sub>·2H<sub>2</sub>O hydration system [14]. In Table 3, as for CCL paste, the ratio of Mc to C<sub>3</sub>A of CCLS paste is higher, indicating that Li<sub>2</sub>CO<sub>3</sub> can partly promote the C<sub>3</sub>A hydration in the presence of CaSO<sub>4</sub>·2H<sub>2</sub>O, which is consistent with the result of heat evolution (Figure 7). The contents of Mc and CaSO<sub>4</sub>·2H<sub>2</sub>O show the conventional change with the increase of age, but those of Ms

and E have a different trend, which is slightly different from previous research on C<sub>3</sub>A-CaSO<sub>4</sub>·2H<sub>2</sub>O hydration system [14]. This is mainly due to that Li<sub>2</sub>CO<sub>3</sub> affects the amount of ettringite (E) and Ms ((6) and (7)) [20]. For C<sub>3</sub>A-CaSO<sub>4</sub>·2H<sub>2</sub>O-Ca(OH)<sub>2</sub>-H<sub>2</sub>O hydration system, with the addition of Li<sub>2</sub>CO<sub>3</sub>, the hydration of C<sub>3</sub>A is promoted and the amount of ettringite (E) and Ms is affected.



FTIR is performed to characterize the phases of three pastes at 0.85 hour, 2.1 hours, and 4.5 hours (Figure 9).

The bands around 3662 cm<sup>-1</sup> (OH<sub>free</sub>) and at 530 cm<sup>-1</sup> (v-AlO<sub>6</sub>) are observed in the spectra of CC (Figure 9(a)) and

CCL (Figure 9(b)) specimens, indicating the presence of C<sub>3</sub>AH<sub>6</sub> in CC and CCL pastes [23–27]. The spectra of CC and CCL pastes present absorption bands near 789 cm<sup>-1</sup> and 804 cm<sup>-1</sup> indicating the presence of C<sub>3</sub>A. However, the

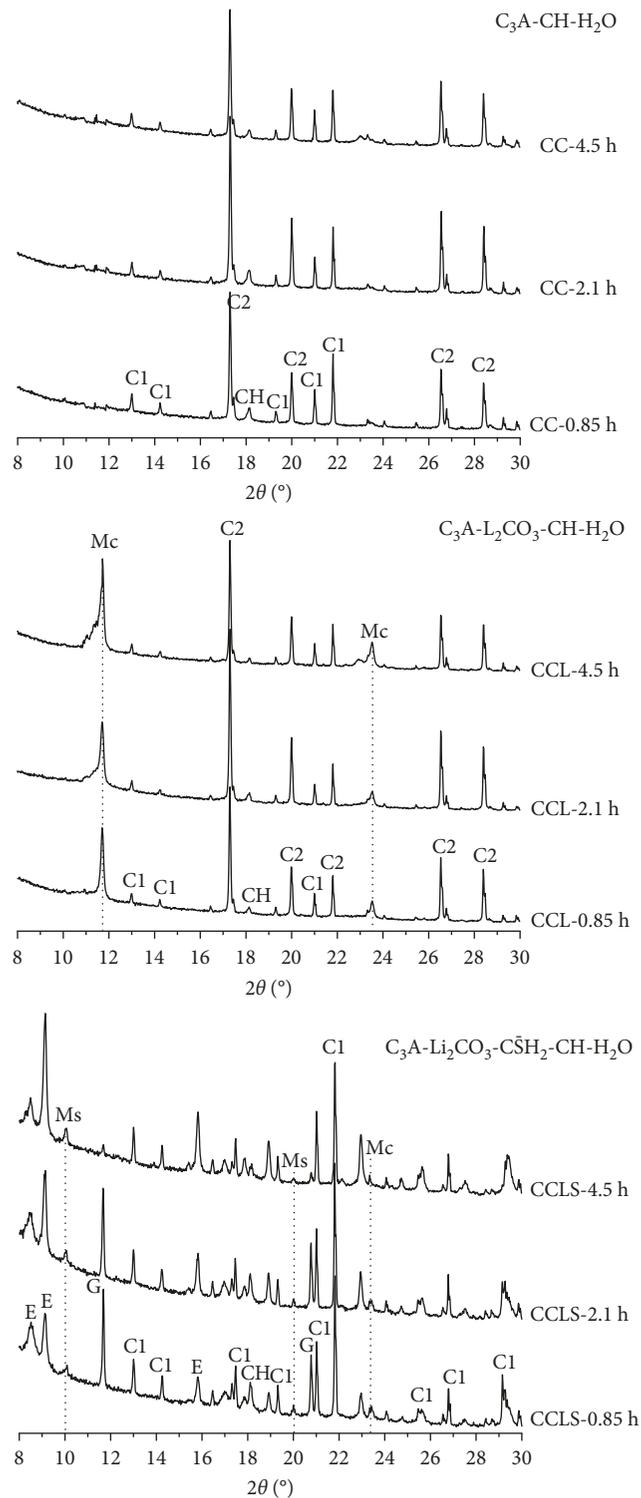


FIGURE 8: XRD patterns of three pastes (C1:  $C_3A$ ; C2:  $C_3AH_6$ ; E: ettringite; Ms: monosulfoaluminate; G: gypsum; Mc: monocarboaluminate; Hc: hemicarboaluminate; CH: calcium hydroxide).

spectrum of CCL paste presents an absorption band around  $3540\text{ cm}^{-1}$ , probably due to the carbonation of hydration products [23], which is different from that of CC paste. Combined with the XRD results of the CCL paste, it is proposed that the products from carbonation could be Mc,

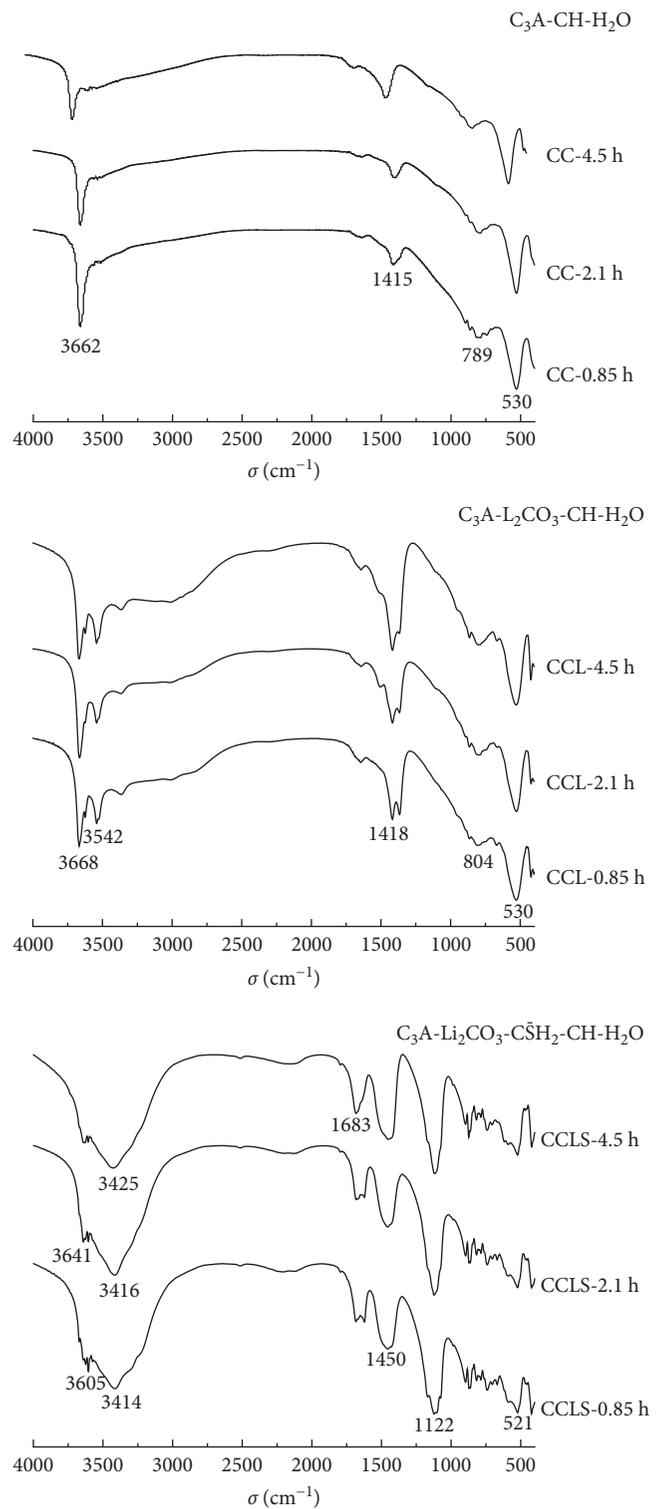


FIGURE 9: FTIR spectra of three pastes.

suggesting that addition of  $Li_2CO_3$  changes the hydration products of  $C_3A$  hydration system. As for CCLS paste (Figure 9(c)), the bands at  $3605\text{ cm}^{-1}$ ,  $3641\text{ cm}^{-1}$ , and  $1683\text{ cm}^{-1}$  are observed, attributing to Aft and  $CaSO_4 \cdot 2H_2O$ . The absorption band around  $3410\text{ cm}^{-1}$ , indicating the presence of carbonation, suggests that Mc is also the hydration product of CCLS paste. According to the results of

FTIR on CC, CCL, and CCLS pastes, the addition of  $\text{Li}_2\text{CO}_3$  significantly affects the types of hydration products of  $\text{C}_3\text{A}$  hydration system, which coincides with the XRD results.

#### 4. Conclusions

In this study, the effect of  $\text{Li}_2\text{CO}_3$  and  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  on the hydration of  $\text{C}_3\text{A}$  is investigated. A range of analytical techniques, such as conductimetry, isothermal calorimetry, XRD, and FTIR, are performed to characterize the hydration process. According to the results obtained, the following conclusions can be drawn:

- (1)  $\text{Li}_2\text{CO}_3$  promotes the hydration of  $\text{C}_3\text{A}$ , and the retarding of  $\text{C}_3\text{A}$  hydration by  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  can be reduced by  $\text{Li}_2\text{CO}_3$  addition.
- (2) The positive effects of  $\text{Li}_2\text{CO}_3$  on  $\text{C}_3\text{A}$  hydration are mainly due to a high pH value resulted from lithium ion and carbonate released from  $\text{Li}_2\text{CO}_3$ , which promotes the formation of hydration products.
- (3)  $\text{Li}_2\text{CO}_3$  can promote the  $\text{C}_3\text{A}$  hydration in  $\text{C}_3\text{A}$ - $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ - $\text{Ca}(\text{OH})_2$ - $\text{H}_2\text{O}$  hydration system and affect types and amount of hydration products.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest regarding the publication of this study.

#### Acknowledgments

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