

# Improvement of workability and early strength of calcium sulphoaluminate cement at various temperature by chemical admixtures

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## HIGHLIGHTS

- Workability and early strength of CSA at 0 °C were improved by LC and LC-AS.
- Workability and early strength of CSA at 40 °C were improved by B and SG-B.
- The formation of CSA hydration products was impacted by chemical admixtures.

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## ABSTRACT

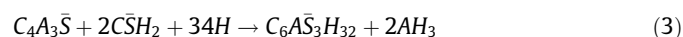
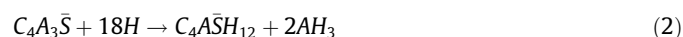
Effects of chemical admixtures on the workability and strength development of calcium sulphoaluminate cement (CSA) at various temperature were researched, of which mechanisms were analyzed by XRD and TG. The setting time and early strength of CSA were significantly improved by lithium carbonate-aluminum sulfate (LC-AS) at 0 °C and sodium gluconate-borax (SG-B) at 40 °C. LC-AS not only enhanced ettringite formation, but also accelerated belite hydration, and the amounts of ettringite was significantly increased at 0 °C. However, the formation of ettringite was retarded by SG-B, and the reduction of the amounts of ettringite can be achieved at 40 °C.

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## 1. Introduction

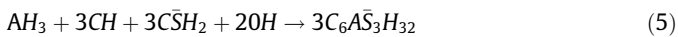
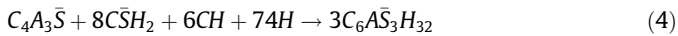
Calcium sulphoaluminate cement (CSA) has been used and standardized in China about 40 years, and it obtained increasing attention because of releasing lower amounts carbon dioxide than ordinary Portland cement (OPC) in its production process [1–3]. Above all, its vital properties, including high early compressive strength, micro-expansion, low temperature adaptability, and poor permeability, etc., led to its wide application for rapid repair engineering [4–9]. The cement nomenclature has been used: C = CaO, S = SiO<sub>2</sub>,  $\bar{S}$  = SO<sub>3</sub>, F = Fe<sub>2</sub>O<sub>3</sub>, and H = H<sub>2</sub>O [10]. The main component of CSA is ye'elimite (C<sub>4</sub>A<sub>3</sub> $\bar{S}$ ), and the secondary phases include belite (C<sub>2</sub>S), tetracalcium aluminoferrite (C<sub>4</sub>AF), gehlenite (C<sub>2</sub>AS), tricalcium aluminate (C<sub>3</sub>A), and anhydrite (C $\bar{S}$ ) or gypsum

(C $\bar{S}$ H<sub>2</sub>), etc. The primary hydration products of CSA are ettringite (AFt), monosulphoaluminate (AFm), and aluminum hydroxide (AH<sub>3</sub>), meanwhile, the minor phases calcium silicate hydrate gel (C-S-H), strätlingite (C<sub>2</sub>ASH<sub>8</sub>), C<sub>4</sub>(A,F)X<sub>2</sub>·yH<sub>2</sub>O, and calcium aluminate hydrate (C<sub>3</sub>AH<sub>6</sub>) can be formed in hydration process [11,12]. Eqs. (1)–(5) show the common hydration reactions in CSA. In early period, ye'elimite hydrates to form AFm and AH<sub>3</sub> in the absence of calcium sulfate (2), but AFt is formed in the presence of anhydrite or gypsum (3). In later stage, the continuous hydration of C<sub>2</sub>S can guarantee the development of later strength of CSA, as well as accelerate the generating of AFt (4) and (5).



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As is well known, the short setting time and high early strength of CSA have been attributed to the fast hydration of ye'elimite, then the optimum setting time, strength development, and volume stability of CSA have been guaranteed by suitable calcium sulfate [13,14]. However, the execution conditions of rapid repair construction encountered, especially temperature, have great influence on repair efficiency and effectiveness by means of accelerating or delaying the CSA hydration process. For instance, the development of CSA early strength is significantly prolonged at low or negative temperature, which results in a long construction period. The setting time of CSA is quickly shortened at high temperature, which leads to a demanding construction requirement [15–17]. It can be observed that the contradiction between suitable workability and high early strength of CSA caused by temperature is urgent to be solved, thus its range of application can be largely extended, possibly.

At low temperature or negative temperature, the slow hydration of CSA, as well as OPC, results in too long setting time and too poor early strength. From raw material design, high calcium sulfate contents exert an accelerate effect on CSA hydration at early age. On the contrary, low calcium sulfate contents tend to promote the development of CSA strength at later age [18–21]. Further, various nanomaterials such as nano-SiO<sub>2</sub>, nano-CaCO<sub>3</sub>, nano-Fe<sub>2</sub>O<sub>3</sub>, and nano-TiO<sub>2</sub> are used in OPC or CSA, on account of “filler” effect and “crystal nucleus” effect for voids, to shorten the setting time and increase the early strength, and the highest early strength and shortest setting time are observed by containing 10 wt% nano-TiO<sub>2</sub> in CSA [22–24]. On the other hand, the compounds containing lithium, aluminum and calcium are used as hardening accelerator admixtures in CSA. For instance, Péra, J. reported that the setting of CSA is accelerated by an addition of 0.05 wt% LiCl or 0.05 wt% Li<sub>2</sub>CO<sub>3</sub> at 25 °C, thanks to the fast precipitation of Li-containing aluminum hydroxide [25–27]. Based on above, reference [28] revealed that CaAl-layered double hydroxides (CaAl-LDHs) as a hardening accelerator admixture in OPC enhance the early compressive strength by 61% on the condition of standard curing (20 ± 2 °C in water). The addition of LiAl-layered double hydroxides (LiAl-LDHs), at room temperature (20 ± 1 °C), not only accelerate the hydration of CSA at early age, resulted in the setting time being shortened, but also increase the amount of hydration products, which promotes the structure more compact, resulting in the compressive strength of the CSA at 1, 3 and 7 days being increased [29]. Until now, effects of hardening accelerator admixtures such as lithium and aluminum on CSA hydration at low even negative temperature have seldom been reported in the literatures.

At high temperature, the faster reaction and greater hydration heat of CSA in the first 12 h require the use of retarders to obtain a sufficient setting time [30–31]. Organic retarders such as polycarboxylate, sugars, carbamide, citric or tartaric acids can affect both the workability and compressive strength of CSA at 20 °C [32–35]. For instance, the use of polycarboxylate is observed by a decreased compressive strength at early stage and an increased compressive strength at 28 days (71 and 84 MPa at 3 and 28 days, respectively). Carbamide extends initial setting time of CSA to more than 25 min, decreases its early compressive strength but has a tiny effect on its later compressive strength [36]. General inorganic retarders used consist of phosphoric acid, borax, and chloride salts. Specifically, phosphoric acid has the optimal effect on delaying setting time. Borax, at 25 °C, can be precipitated with Ca<sup>2+</sup> of cement grains to adsorb onto the surface of the cement, thereby a calcium-based borate layer is formed, which is responsible for its retarding effect [37–41]. The effects of these retarders on the hydration of CSA and

OPC are qualitatively comparable. However, chloride salts, which are powerful accelerators in OPC, have retarding effects in CSA, somewhat depending on dosage and temperature [42]. Unfortunately, effects of retarders such as borax on CSA hydration at high temperature have been not observed.

Therefore, five types of specific chemical admixtures chosen had been incorporated into CSA containing powder naphthalene based superplasticizer (NS) to improve its workability and early strength at –10 °C, 0 °C, 20 °C, and 40 °C. The effects of chemical admixture kinds and contents on setting time, fluidity, and compressive strength had been researched at various temperature. Specially, the suitable kind and content of chemical admixtures at specific temperature had been summarized, when its workability and early strength can be harmonized. Further, the kinds of hydration products of CSA with and without chemical admixtures were examined by X-ray diffraction (XRD), and their amounts were examined by Thermogravimetric analysis (TGA).

## 2. Material and methods

### 2.1. Material and mix proportions

The chemical and mineralogical composition of the low alkalinity calcium sulphoaluminate cement (CSA, 42.5) used in this study were presented in Tables 1 and 2. The chemical composition was determined by X-ray fluorescence (XRF), and its mineralogical composition was calculated by using Rietveld refinement from the X-ray diffraction pattern in Fig. 1. Lithium carbonate (LC), aluminum sulfate (AS), calcium nitrate (CN), sodium gluconate (SG), and borax (B) were used as chemical admixtures, of which purity are above 95%, together with powder naphthalene based superplasticizer (NS) being used. Moreover, the quartz sand (S) was used with the maximum diameter of 0.25 mm.

Four specific temperature were chosen for research, –10 °C, 0 °C, 20 °C, and 40 °C, respectively. The paste and mortar specimens of CSA were prepared at above specific temperatures, and the ratio of water to cement was 0.29 for paste specimens, yet 0.26 for mortar specimens, of which the mass ratio of quartz sand to cement is 1. At 0 °C, a certain amount of LC, AS, and LC-AS used as hardening accelerator admixtures were incorporated into CSA containing 1.0 wt% NS with and without sand to perform mortars and pastes respectively. At –10 °C, a certain amount of LC, and LC-CN used as hardening accelerator admixtures were added into CSA containing 1.0 wt% NS with sand to perform mortar specimens. At 20 °C, a certain amount of NS used as superplasticizer were mixed into CSA with and without sand to perform mortar and paste specimens, respectively. At 40 °C, a certain amount of SG, B, and SG-B used as retarders were doped into CSA containing 1.0 wt% NS with and without sand to perform mortar and paste specimens, respectively. The details of mix proportions were shown in Table 3.

Water, powder and sand were stored at the respective temperatures (–10 °C, 0 °C, 20 °C, and 40 °C) for 1 day before mixing. Specially, the chemical admixtures would be dissolved in tap water before being stored in order to avoid the freeze of water at negative temperature. Specimens were performed for 4 min using a laboratory mixer equipped with an anchor stirrer, and then cured at the constant temperature and humidity curing tank (respective temperatures, above 90% relative humidity) before the test. Paste specimens were prepared for the setting time test, fluidity test and mechanism analysis, Mortar specimens were prepared for compressive strength test.

### 2.2. Test methods

#### 2.2.1. Setting time

The setting time test on CSA pastes with and without chemical admixtures were measured in accordance with the Chinese National Standard GB/T 1346-2011 [29]. Pastes were prepared with 500 g CSA, 145 g water (W/C = 0.29), and predetermined mass of NS and chemical admixtures at 0 °C, 20 °C, and 40 °C with relatively humidity more than 90%. Vicat apparatus was used to measure the setting time.

#### 2.2.2. Fluidity

The fluidity test on CSA pastes with and without chemical admixtures were measured in accordance with the Chinese National Standard GB/T 8077-2012 [29]. Pastes were prepared with 300 g CSA, 87 g water (W/C = 0.29) and predetermined mass of NS and chemical admixtures at 0 °C, 20 °C, and 40 °C with relatively humidity more than 90%, then pastes were casted into a conical mold (height of 60 mm, top diameter of 36 mm, and bottom diameter of 60 mm) and the conical mold was lifted up to let the paste flow freely for 30 s on the glass plate. The fluidity was determined by average value of diameters of the pastes at two vertical directions. In order to determine the change of fluidity, the pastes fluidity were measured repeatedly at 5 min, 10 min, and 15 min.

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