



# Effect of cement mineralogy on the effectiveness of chloride-based accelerator



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## ABSTRACT

Chloride-based accelerators are widely used for non-reinforced concrete; however, their effectiveness varies when used with different cement types. The goal of this study was to investigate the effect of cement mineralogy on the accelerating efficiency of pure  $\text{CaCl}_2$  and commercial chloride-based accelerator. The accelerating efficiency was measured on three commercial cements (with similar  $\text{C}_3\text{S}$  content but variable mineralogy) in terms of heat flow and compressive strength development. Chlorides were determined to be less effective with high- $\text{C}_3\text{A}$ /high-alkali cement. One of the cements with normal acceleration by chlorides was selected for doping with  $\text{C}_3\text{A}$ , sulfates and alkalis in order to reproduce the reduction in chloride performance. Alkalis, specifically  $\text{K}_2\text{SO}_4$ , were determined to have a significant effect on chloride-based accelerator performance, whether pure  $\text{CaCl}_2$  or commercial admixture. Increasing  $\text{C}_3\text{A}$  or gypsum content alone did not appear to significantly affect acceleration, but in combination with high alkali content these phases can potentially reduce accelerator effectiveness.

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## 1. Introduction

Calcium chloride has been used for decades to accelerate cement hydration. Although its effect on setting and strength development has been studied at length, the mechanism of acceleration of cement hydration by  $\text{CaCl}_2$  is still not well-established. As such, it is difficult to extend the conclusions of existing phenomenological studies to cements of varying physical and chemical characteristics.

Cheung et al. [1] point out that behavior of accelerators should be separated into two effects: “time to the end of the induction period and the hydration rate after the onset of hydration.” Addition of  $\text{CaCl}_2$  accelerates both the end of the induction period and the rate of hydration of portland cement during the acceleration stage due to the acceleration of the  $\text{C}_3\text{S}$  reaction [1,2]. Consequently, setting time and early-age compressive strength gain are accelerated as well. Since it was concluded early on that the main effect of  $\text{CaCl}_2$  is on the  $\text{C}_3\text{S}$  hydration, a number of studies have been conducted on pure  $\text{C}_3\text{S}$  pastes, which do not take into account the complex interactions between different ionic species occurring during cement hydration as was pointed out by Jupe et al. [3].

There are indications in the literature that  $\text{CaCl}_2$  does not have the same effect on cements of different mineralogies, although this topic has not received much attention. Shideler [4] reported in 1952 that  $\text{CaCl}_2$  is considerably less effective in accelerating Type III cement hydration compared to other cement types. Price also stated that  $\text{CaCl}_2$  “reacts differently with different cements” [5]. Oey et al. [6] observed that generally  $\text{CaCl}_2$  is “more efficient at accelerating set in the Type II/V OPC compared to the Type I/II OPC.” Suryavanshi et al. [7] compared the effect of  $\text{CaCl}_2$  on OPC and sulfate resistant portland cement (SRPC) mortars. The authors attributed greater effect of chlorides on SRPC mortars to lower chloride binding capacity of SRPC due to its lower  $\text{C}_3\text{A}$  content. Jupe et al. [3] compared performance of  $\text{CaCl}_2$  on two oil well cements, Class H and A, of similar  $\text{C}_3\text{S}$  content and variable  $\text{C}_3\text{A}/\text{C}_4\text{AF}$  composition (0.65/17.00% and 11.00/7.00% respectively) and observed that the accelerating effect was greater for cement with low  $\text{C}_3\text{A}$  content. The authors attributed this to the possible formation of “ferrihydrite gel” in the Class H slurries, as  $\text{CaCl}_2$  has been reported to accelerate the reaction of  $\text{C}_4\text{AF}$  with gypsum [2]. This study points to a more complex interaction between phases in the presence of  $\text{CaCl}_2$  rather than merely acceleration of  $\text{C}_3\text{S}$  hydration. Although it was not remarked on by the authors, another major difference between the two cements investigated by Jupe et al. [3] was their alkali, more specifically  $\text{K}_2\text{O}$  content, which for Class A cement was almost double that of Class H. Alkalis are known to

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modify cement hydration [8], and their impact on CaCl<sub>2</sub> efficiency deserves attention.

The purpose of this study was to investigate the influence of cement mineralogy, more specifically C<sub>3</sub>A and alkali content, on the accelerating efficiency of pure CaCl<sub>2</sub> and commercial chloride-based accelerator. Identification of specific cement compounds affecting CaCl<sub>2</sub> performance can enable the engineers to make informed decisions on the appropriate materials selection for concrete mixtures in different construction applications.

**2. Materials and methods**

Three cements of similar fineness (in terms of Blaine fineness and mean particle size) and similar C<sub>3</sub>S content, but variable C<sub>3</sub>A, C<sub>4</sub>AF composition and variable alkali content were selected for this study. Their specific gravity was determined following ASTM C188, while the fineness was measured using a Blaine air-permeability apparatus in accordance with ASTM C204. Particle size distribution (see Fig. 1) was determined using LA-950 laser scattering particle size analyzer manufactured by Horiba which was then used to calculate the mean particle size (MPS). MPS has been shown to have a better correlation with heat of hydration of portland cements compared to Blaine fineness at 1, 3 and 7 days [9].

The chemical oxide composition was determined by X-ray fluorescence (XRF) in accordance with ASTM C114 and mineralogical composition was obtained from Rietveld refinement performed with HighScore Plus software 3.0 from Panalytical. X-ray diffraction scans for Rietveld refinement were collected using X’Pert 3040Pro diffractometer with Cu K $\alpha$  radiation manufactured by Panalytical. The tension and current were set to 45 kV and 40 mA respectively. The following optics settings were used: 1° divergence slit, 0.2 mm receiving slit, and 1° anti-scatter slit.

Two accelerators were used in this study: a pure reagent grade CaCl<sub>2</sub> and a commercial CaCl<sub>2</sub>-based Type E accelerating admixture (ASTM C494). The composition of the Type E admixture was obtained from the materials safety data sheet provided by the manufacturer and is listed in Table 1. A 4% volumetric solution was prepared with pure CaCl<sub>2</sub> and was used in place of the mixing water. This corresponded to 2% CaCl<sub>2</sub> addition by mass of cement. The commercial accelerator was used at the dosage that is typically used for rapid-repair concrete mixtures for jointed plain concrete pavement [10]. This dosage corresponded to 1–2% CaCl<sub>2</sub> addition by mass of cement based on the ranges listed in Table 1.

Acceleration of early-age hydration by CaCl<sub>2</sub> and commercial

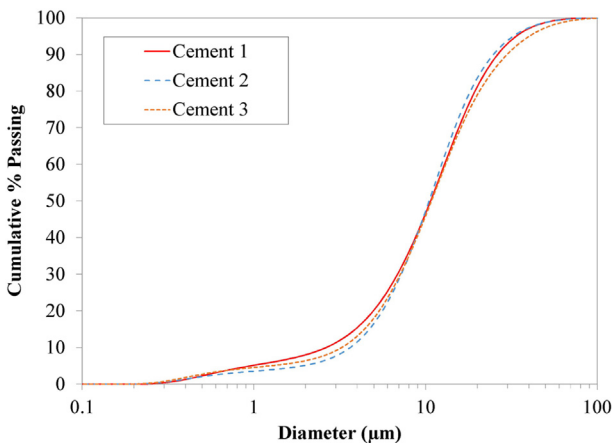


Fig. 1. Particle size distribution of as-received cements used in this study.

**Table 1**  
Commercial accelerator composition.

Component	Percentage
Calcium Chloride	25–50
Potassium Chloride	1–10
Sodium Chloride	1–10
Triethanolamine	1–10

accelerator was evaluated by measuring the heat of hydration (HOH) and compressive strength of mortar cubes for each cement with and without addition of CaCl<sub>2</sub>/accelerator. TAMAIR isothermal conduction calorimeter manufactured by TA Instruments was used to measure the heat flow and the total heat evolved following ASTM C1702, Method A, internal mixing. Compressive strength testing of the as-received cements, with and without calcium chloride/Type E admixture addition, was performed in accordance with ASTM C109 and C305. 50 mm mortar cubes were tested at the ages of 4, 8, 12 and 24 h.

As-received Cement 2 was doped with C<sub>3</sub>A, gypsum and alkali sulfates and alkali hydroxides to the levels of Cement 3 in order to identify the compounds responsible for the reduced acceleration with addition of CaCl<sub>2</sub>/Type E admixture. C<sub>3</sub>A (purity of 97.6% cubic based on Rietveld refinement) was obtained from CTL. C<sub>3</sub>A was weighed out, added to Cement 2 and mixed by hand to a homogeneous appearance prior to conducting isothermal calorimetry measurements. The rest of the doping compounds were reagent-grade chemicals. Gypsum and alkalis were also added to cement in the form of dry powder and mixed by hand. When cement was doped with more than one compound, the same procedure was adopted. The weights for each of the mixtures tested in the study are listed in Table 2.

The degree of hydration  $\alpha(t)$  was calculated based on the total heat evolution measured by isothermal calorimetry according to:

$$\alpha(t) = \frac{H(t)}{H_u} \tag{1}$$

where  $H(t)$  is the total heat released at time  $t$  and  $H_u$  is the ultimate heat of the mixture, which is calculated based on cementitious content of the mixture [11,12]. Since the mixtures in this study did not contain any supplementary cementitious materials (SCMs),  $H_u$  was equal to the total heat of hydration of cement  $H_{cem}$  which is a function of cement composition.

$$H_{cem} = 500P_{C3S} + 260P_{C2S} + 866P_{C3A} + 420P_{C4AF} + 624P_{SO3} + 1186P_{FreeCaO} + 850P_{MgO} \tag{2}$$

where  $P$  is the mass of  $i$ th component to total cement content ratio.

The accelerating ability of CaCl<sub>2</sub> and commercial accelerator was evaluated by comparing the time of occurrence and the magnitude of the main hydration peak of the accelerated sample to its counterpart with no accelerator. For example, in order to evaluate the accelerating ability of CaCl<sub>2</sub> with increasing potassium content, the time of the main hydration peak in the Cement 2 + K<sub>2</sub>SO<sub>4</sub> sample was subtracted from the time of the peak in the Cement 2 + K<sub>2</sub>SO<sub>4</sub>+CaCl<sub>2</sub> sample. The same procedure was applied to evaluate the accelerating ability of CaCl<sub>2</sub> on the magnitude of the main hydration peak.

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