



Evaluating the use of supplementary cementitious materials to mitigate damage in cementitious materials exposed to calcium chloride deicing salt



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ARTICLE INFO

Article history:

Received 16 May 2016

Received in revised form

21 March 2017

Accepted 5 May 2017

Available online 6 May 2017

Keywords:

Calcium chloride

Calcium oxychloride

Chemical reaction

Concrete

Damage

Supplementary cementitious material

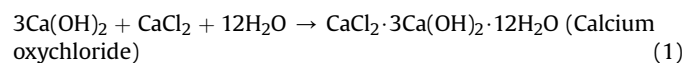
ABSTRACT

This paper discusses the role of supplementary cementitious materials (SCM) in reducing damage caused by calcium oxychloride formation. Calcium oxychloride is a destructive product of a reaction between calcium hydroxide (CH) that exists in a cementitious matrix and CaCl₂ that can enter the pores of the matrix when it is used as a deicing salt. Paste samples were prepared where a percentage of ordinary portland cement was replaced with various types of SCM (including fly ash, slag, and silica fume). This paper examined the amount of calcium oxychloride that formed using low-temperature differential scanning calorimetry, and damage development detected using acoustic emission. Thermogravimetric analysis was also performed to determine the relationship between the amount of CH in cementitious materials and the amount of calcium oxychloride formation. The results show that the use of SCM is effective in reducing the calcium oxychloride formation and resulting damage when cementitious materials are exposed to various compositions of solution containing CaCl₂. The explanation of the benefit of using SCM is that it can reduce the calcium oxychloride formation due to a reduction in the amount of CH in the cementitious materials through pozzolanic reaction and dilution of cement. As a result, cementitious materials with SCM exposed to CaCl₂ may experience less damage and have a longer service life.

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1. Introduction

Concrete pavements exposed to CaCl₂ deicing salt have in some cases shown damage at the joints. It has been shown that this damage may be due to a destructive chemical reaction between cementitious matrix and CaCl₂ [1–7]. The formation of calcium oxychloride is one source of damage in concrete exposed to CaCl₂ [4,5,8] and it is described as a rapid reaction resulting in damage and degradation in concrete at early ages. Calcium oxychloride forms as calcium hydroxide (CH) from a hydrated cementitious matrix reacts with CaCl₂ solution as described in Equation (1) [3,4].



Calcium oxychloride forms at temperatures between 0 and 50 °C (i.e., a temperature higher than the freezing point of water) [3,4,9–12]. The formation of calcium oxychloride can expand during the phase transformation from solid to liquid in cementitious materials resulting in damage and cracking even if concrete does not experience any freezing cycles [3–5,13–15].

The chemical reaction between CaCl₂ and the cementitious materials can be mitigated through the use of supplementary cementitious materials (SCM) [3,4,16–20]. SCM can reduce the amount of CH in cementitious materials by diluting ordinary portland cement (OPC) and also consuming CH through pozzolanic reaction [18,21]. Therefore, the potential for calcium oxychloride formation (the expansive phase causing damage) is reduced in concrete made using SCM as it is exposed to CaCl₂; resulting in the concrete being less susceptible to damage. The objective of this

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paper is to provide data to illustrate the beneficial contributions of various SCMs (including fly ash, slag cement, and silica fume) in reducing the amount of calcium oxychloride formation in cementitious materials exposed to varying concentrations of CaCl_2 . This paper discusses the effect of using SCM on damage development in cementitious materials exposed to CaCl_2 deicing salt.

2. Experimental program

An experimental program was developed that consisted of preparing cementitious paste samples using various types of SCM and exposing them to various concentrations of solution containing CaCl_2 . After exposure, the amount of calcium oxychloride, damage development, and microstructural changes were evaluated in the samples using low-temperature differential scanning calorimetry (LT-DSC), and acoustic emission (AE), respectively. Thermogravimetric analysis (TGA) was also used to quantify the effect of SCM in reducing the amount of CH in cementitious systems.

2.1. Materials

Type I cement (OPC I), Type V cement (OPC V), silica fume (SF), class F fly ash (F-FA), and slag (SL) were used in this study and their properties are described in Table 1. Calcium chloride dihydrate with a formula weight of 147.01 g/mol and de-ionized (DI) water were used to prepare CaCl_2 solutions. CH powder used in this study had a CH purity greater than 98.2% and a formula weight of 74.09 g/mol.

2.2. Sample preparation, conditioning, and testing procedure

Paste samples were prepared using OPC I and OPC V with a water-to-cement ratio (w/c) of 0.42 by mass. To prepare SCM paste samples, OPC I was used with mass replacement of 7% and 20% silica fume, 20% class F fly ash, and 45% slag. An additional paste sample was prepared using only OPC I with a w/c of 0.48 to evaluate the effect of w/c on damage development in cementitious materials exposed to CaCl_2 solution. The mixing procedure was performed following ASTM C305-14 [23]. After mixing, the paste was placed and consolidated in several cylindrical molds. The containers were sealed and cured for 3 months at a temperature of 23 ± 1 °C. Different types of specimens were prepared for LT-DSC, TGA, and AE as described in Table 2.

2.2.1. Low temperature differential scanning calorimetry (LT-DSC)

Calcium oxychloride formation is a phase change at a temperature above 0 °C, therefore, LT-DSC has been suggested to be used to quantify the amount of calcium oxychloride [6,18]. A powder was prepared from the middle third of the cast cylindrical paste samples. A milling machine was used to grind the paste sample to produce the powder. The powder was then passed through a 75 μm sieve (no. 200). After the powder is ground, it was placed in a vacuum oven at 55 ± 1 °C for 24 h. The powder was then stored in a sealed container to minimize the potential for additional hydration or carbonation before exposure to CaCl_2 solution. The calcium chloride solution was prepared by mixing granular reagent grade dehydrate calcium chloride and deionized water. CaCl_2 concentrations of 0, 5, 10, 15, 20, 25, 29.8, and 35% by mass of solution were used. Paste powder specimens consisting of 10 mg \pm 0.5 mg of ground powder were mixed with 10 mg \pm 0.5 mg of solution having a powder-to-solution mass ratio of 1 to 1. Powder specimens were blended with solutions in a high-volume stainless steel pan, and they were then tested in the LT-DSC immediately after mixing.

To synthesize calcium oxychloride, CH, CaCl_2 , and H_2O with molar ratios equal to 3:1:12 (CH: CaCl_2 : H_2O) were prepared. Since calcium oxychloride with 3:1:12 M ratio can form a solid in the temperature below 50 °C, blending was performed at 70 °C at which the calcium oxychloride is in liquid phase to produce a homogenous calcium oxychloride solution. Several specimens (10 mg \pm 0.5 mg) were then taken from synthesized calcium oxychloride and placed in the stainless steel high volume pans and tested in LT-DSC to quantify the specific latent heat associated with calcium oxychloride formation.

For LT-DSC testing, the initial temperature of the test was set to equilibrate at 25 °C [6,18]. After the initial temperature became stable, the LT-DSC cell was cooled to -90 °C with a cooling rate of 5 °C/min. The specimen temperature was kept constant at this temperature (-90 °C) for 5 min to allow the specimen to equilibrate. A cycle of heating and cooling was established between -90 °C and -70 °C. The specimen equilibrated again at -90 °C for 5 min; and the temperature of the specimen was increased to 70 °C at a rate of 5 °C/min.

2.2.2. Thermogravimetric analysis (TGA)

A portion of powder that was prepared for LT-DSC experiment before CaCl_2 solution exposure was kept for TGA experiment. To

Table 1
Properties of cementitious materials used in this study.

Item	Percent by mass (%)				
	Type I Cement (OPC I)	Type V Cement (OPC V)	Silica Fume (SF)	Class F Fly Ash (F-FA)	Slag ^b (SL)
Silicon Dioxide (SiO_2)	19.43	21.3	94.73	48.58	–
Aluminum Oxide (Al_2O_3)	5.39	2.6	–	22.17	–
Ferric Oxide (Fe_2O_3)	3.18	4.2	–	19.43	–
Calcium Oxide (CaO)	63.45	63.2	–	3.70	–
Magnesium Oxide (MgO)	2.97	4.5	–	0.92	–
Sulfur Trioxide (SO_3)	3.38	2.8	–	1.13	0.27
Loss on Ignition	0.88	1.20	3.41	0.95	–
Sodium Oxide	0.35	–	–	0.57	–
Potassium Oxide	0.77	–	–	2.37	–
Free Lime	– ^a	0.38	–	–	–
Insoluble Residue	0.25	0.18	–	–	–
Total Equivalent Alkali as Na_2O	0.86	0.21	–	2.13	0.83
Tricalcium Silicate (C_3S)	60	64	–	–	–
Dicalcium Silicate (C_2S)	10	13	–	–	–
Tricalcium Aluminate (C_3A)	9	0	–	–	–
Tetracalcium Aluminoferrite (C_4AF)	10	13	–	–	–

^a Not available.

^b The slag used in this study meets the requirements of ASTM C989-14 [22] with slag activity indices of 94 and 124% for 7 d and 28 d, respectively.

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