



Cementitious materials exposed to high concentration of sodium chloride solution: Formation of a deleterious chemical phase change

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HIGHLIGHTS

- A damage development for concrete exposed to high concentrated NaCl solution and a cooling-heating cycle was investigated.
- High concentrated NaCl solution can interact with concrete at low temperatures, producing a detrimental chemical phase.
- The observed chemical phase can cause damage in concrete.
- A more pronounced damage was perceived in concrete made using Type I OPC.

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ABSTRACT

This paper investigates deleterious chemical interactions between cementitious materials and high concentrations of sodium chloride (NaCl) solution (i.e., salt concentration >12% by mass). The behavior of mortar specimens made using Type I and Type V ordinary portland cements was investigated as the temperature varied in the presence of NaCl solution with concentrations of 15% and 20% (by mass). A low-temperature longitudinal guarded comparative calorimeter (LGCC) in conjunction with an acoustic-ultrasonic technique were used to monitor the damage development in mortar samples. Cooling-heating cycles were applied with a minimum temperature above the freezing temperature of the solution to avoid ice formation. Nevertheless, damage was observed in specimens. Calcium aluminate phases, mainly tricalcium aluminate (C_3A), seem to be the source of damage as the magnitude of damage was relatively minor when Type V cement ($C_3A < 5\%$ by mass) was used. To further investigate the source of the damage, a low-temperature differential scanning calorimeter (LT-DSC) was used to study chemical phase transitions during temperature change in three types of powder specimens exposed to different concentrations of NaCl solution: (1) hydrated cement pastes, made using Type I and Type V cements, (2) cement pure phases, consisting of C_3A and gypsum, and (3) cement hydration products, consisting of calcium hydroxide (CH) and calcium sulfoaluminate phases (monosulfate (AF_m) and ettringite (AF_t)). A formation of a chemical phase at a temperature range between 0 °C and 10 °C was observed that may be the source of damage in cementitious materials exposed to high concentration of NaCl solutions. It was found that the interaction of high concentrations of NaCl solution with C_3A and calcium sulfoaluminate phases in the cementitious system are the origin of the chemical phase formation.

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1. Introduction

Sodium chloride (NaCl) deicing salt is widely used on concrete pavements and bridge decks to melt ice and snow, thereby improving the safety of transportation in cold-climate regions. NaCl represents over 55% of the total volume of deicers applied for anti-icing

in U.S. states and Canadian provinces each year because of its low cost and availability compared to other chloride-based deicing salts (e.g., $CaCl_2$, and $MgCl_2$) [1]. The addition of NaCl salt on the surface of concrete has been found to cause deterioration of concrete through chemical and physical interactions [2–5]. The physical damage may include: freeze-thaw damage in concrete with high degrees of saturation [4,6,7], cracks resulting from internal stresses due to salt crystallization inside concrete pores [8–12], corrosion of reinforcement [13–16], and/or scaling of concrete surface [17–19]. The chemical damage may include deleterious

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Table 1
Summary of the experimental program.

Experiment	Type of sample	Concentration of NaCl solution (% by mass)	Purpose
LGCC & AE	Mortars samples: – Type I cement – Type V cement	15% and 20%	Study damage development due to chemical interactions between NaCl salt and cementitious materials. The temperature in the sample was kept above freezing temperature of NaCl solutions.
LT-DSC	Powder samples: – Hydrated cement pastes made using Type I and Type V cement – Cement pure phases, consisting of C ₃ A and gypsum – Cement hydration products, consisting of CH, AF _m and AF _t	0%, 5%, 10%, 12%, 15%, 18%, 21%, and 24%	Investigates potential sources that result in chemical reactions between cementitious materials and NaCl solution

reactions between salts and cement paste, reinforcing steel, or aggregates [20–24].

When deicing salts are applied on the surface of concrete to melt ice/snow, salt solutions are usually absorbed into the concrete joints, cracks, or pores, and thus saturate the concrete. As water evaporates during times of low precipitation/humidity, the concentration of salt in the pore solution increases [25–27]. The concentrated salt solution may result in internal distresses in the concrete. For example, it can create osmotic pressure in the pores (water in low concentration solution diffuses towards a higher concentration solution) [28–31]. In case of additional drying, it may produce salt crystals which result in crystallization pressures [12,32]. Concentrated salt solution can also alter the pore solution properties, change the chemistry and microstructure of cement hydration products, and/or results in formations of new chemical phases that can lead to internal cracks in concrete [33–37].

While research studies suggest that NaCl deicer can be considered less detrimental than other deicer agents [38–40], there are studies reporting that NaCl can chemically react with cement paste and influence its chemistry resulting in damage in the concrete [2,3,36]. It appears that the chemical interaction between NaCl and cementitious materials is temperature dependent and the chemical interaction may not be detected using conventional experimental setup when performed at room temperature [2,41,42].

This study is built on previous works [2,43] where exposing concrete to high concentration of NaCl solution was observed to be detrimental even if the concrete did not experience temperatures that freeze the solution. The damage was rationalized to be the result of a formation of an unexpected chemical phase change within the cementitious system. This paper aims to provide further understanding of the detrimental influence of high concentrations of NaCl solution in concrete as well as to investigate the source of damage. In the present study, the authors investigate (1) the behavior of mortar specimens in the presence of high concentration of NaCl solutions (salt concentrations of 15% and 20% by mass) under thermal cycling, while keeping the applied temperature range above the freezing point of the NaCl solution in order to eliminate the ice formation and its associated damage, and (2) the effect of NaCl solution on the chemical behavior of pure phases and hydration products of the cement as the temperature varies in order to find the source of the deterioration in concrete exposed to NaCl solution.

A low-temperature longitudinal guarded comparative calorimeter (LGCC) was used to evaluate the thermal response of cementitious materials during thermal cycling and to detect phase changes. In addition, an acousto-ultrasonic technique was used to monitor damage development in cementitious materials due to chemical interactions between the NaCl solution and cementitious materials. These techniques have been proven to be capable

to detect and quantify the damage in concrete caused by chemical reactions [2,44–46]. Furthermore, a low temperature differential scanning calorimetry (LT-DSC) was employed to investigate potential sources of chemical reactions that occur between NaCl solution and cementitious materials.

2. Experimental procedures

Table 1 summarizes the experiments conducted in this study. This section also describes the preparation of powder samples, the process of material synthesizing, mix proportions of mortar samples, and sample conditioning.

2.1. Preparation of powder materials and synthesizing process

Anhydrous sodium chloride (NaCl), calcium sulfate dihydrate, (gypsum, CaSO₄·2H₂O), and calcium hydroxide (Ca(OH)₂) were obtained from Sigma Aldrich Corp. All chemicals were ACS analytical reagent grade (99% purity). The tricalcium aluminate (C₃A, 3CaO·Al₂O₃) and the ettringite (AF_t) powder samples were received from Purdue University along with their XRD test results. The C₃A was synthesized by a stoichiometric sintering of calcium carbonate (CaCO₃) with alumina oxide (Al₂O₃) at a 3:1 M ratio at 1350 °C in a muffle kiln for ~12 h. The AF_t was synthesized from two reactant solutions containing 6.65 g of alumina sulfate Al₂(SO₄)₃·18H₂O and 4.44 g of calcium hydroxide (Ca(OH)₂) were agitated for 48 h at room temperature under N₂ purge for 30 min to minimize carbonation. The suspensions were centrifuged and the solids were washed with 1 N NaOH solution to maintain a range of pH values from 10 to 13. The final solution was filtered and the solid residual was washed with diethyl ether to remove any residual aqueous phases. The formed ettringite was then dried and stored at room temperature and a relative humidity of approximately 30%.

Fig. 1 shows XRD patterns and Rietveld refinement patterns of the synthesized C₃A and AF_t phases. The Rietveld refinements were carried out by using graphical user interface Profex 3.11.1 with the program BGMN [47]. The XRD results for C₃A showed diffraction peaks characteristic for cubic C₃A, aragonite (CaCO₃) and gibbsite (Al(OH)₃) whereas the results for AF_t detected some characteristic peaks for AF_t, monosulfate (AF_m), calcite (CaCO₃) and gypsum. The XRD patterns decompositions and the quantity of calculated contributions of the phases, expressed in wt.%, are also shown in Fig. 1.

2.2. Paste and mortar samples

Samples were prepared according to ASTM C305-12 [48] using a standard mixer for both mortar and paste. Two types of portland cement were used in this study, Type I (with 10.5% C₃A) and Type V (with almost no C₃A). The chemical compositions of the cements

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