



Freeze thaw and deicer salt scaling resistance of concrete prepared with alkali aluminosilicate cement



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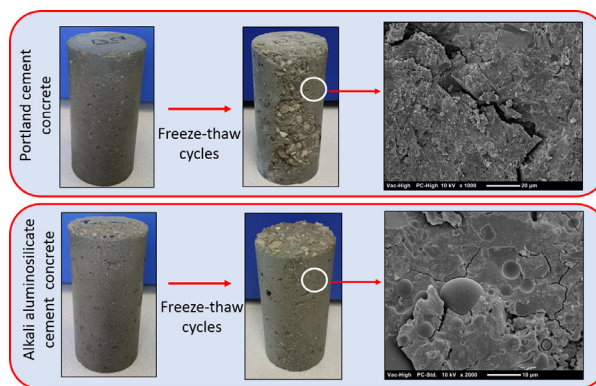
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HIGHLIGHTS

- Alkali aluminosilicate binder produced excellent resistance to freeze-thaw cycles.
- Deicer salt scaling can be a drawback of concrete prepared with alkali aluminosilicate cement.
- Chemically modified alkali aluminosilicate binder produced improved deicer salt scaling resistance.

GRAPHICAL ABSTRACT



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ABSTRACT

Concrete materials were prepared with a cement based primarily on the alkali aluminosilicate chemistry. Two aspects of concrete performance were emphasized and compared against those of ordinary Portland cement (OPC) concrete: freeze-thaw durability, and deicer salt scaling resistance. Test results indicated that the concrete prepared with the alkali aluminosilicate cement (AAC) produced excellent freeze-thaw durability; its deicer salt scaling resistance, however, was lower than that provided by the OPC concrete. Efforts were made to improve the deicer salt scaling resistance of the AAC concrete through refinement of the AAC composition. The use of an air-entraining agent was found to enhance the deicer salt scaling resistance of the AAC concrete. Modification of the AAC chemistry with polyethylene glycol, tartaric acid, or a combination of sodium benzoate and triisopropanolamine was found to also improve the AAC concrete resistance to deicer salt scaling with minimal effect on compressive strength.

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1. Introduction

In cold climates, concrete faces two primary durability challenges that involve physical deterioration mechanisms: (i) general deterioration of concrete exposed to freeze-thaw cycles in the

presence of moisture; and (ii) scaling of the concrete surface exposed to deicer salt. The freeze-thaw damage is usually manifested as cracking and spalling of concrete, which are caused by progressive expansion of the cement paste under repeated cycles of freezing and thawing [1]. The combination of freeze-thaw cycles and deicer salt application has particularly damaging effects on concrete [2,3]. The cumulative damage to concrete under these two effects compromises the moisture barrier qualities of concrete, and magnifies the susceptibility of concrete to freeze-thaw attack

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and other mechanisms of deterioration (e.g., corrosion of reinforcing steel) [4,5].

Alkali activated aluminosilicate binders have been found to experience minor deterioration when exposed to freeze-thaw cycles [6]; their resistance to chloride ion diffusion is also found to be high [7]. There is, however, very limited literature on the deicer salt scaling resistance of these binders. Alkali aluminosilicate hydrates are highly complex, and encompass diverse structural qualities which are poorly understood. The availability of limited data on the long-term durability of alkali-activated binders, including their deicer salt scaling resistance, is an important factor hindering their commercial success in spite of their favorable performance, cost and sustainability attributes [8–10]. The work reported herein was inspired by our background field investigations which pointed at the susceptibility of the conventional (two-part) alkali-activated binders to deicer salt scaling. Fig. 1 shows the extensive deicer salt scaling experienced by a sidewalk after six months (covering one full winter) of exposure to freeze-thaw cycles and deicer salt in mid-Michigan (USA). The damage to this sidewalk seemed to be limited to its surface.

2. Materials and methods

2.1. Materials

The AAC used in this investigation was developed via mechanochemical processing described in the previous work of the same authors [11]. The aluminosilicate precursors used in the mechanochemical process were coal fly ash, ground granulated blast furnace slag, and albitite. The (dry) alkalis used as raw materials were sodium hydroxide, sodium silicate, and calcium oxide. Sodium tetra-borate (Borax) was also added to the cement formulation to increase its set time. Portland cement (Lafarge Type I/ASTM C150) was used as control for comparative assessment of the new hydraulic cement. The chemical compositions and the Blaine fineness of the OPC and AAC are presented in Table 1. When compared with Portland cement, the AAC has lower calcium and higher silicon, aluminum and alkali metal (Na & K) contents.

The particle size distributions of the OPC and AAC, measured using laser granulometry, are presented in Fig. 2. The median particle sizes were 9.8 and 7.4 μm for the OPC and AAC, respectively.

Natural sand with maximum particle size of 4.75 mm was used as fine aggregate. Limestone with a maximum particle size of 12.5 mm was used as coarse aggregate. The particle size distributions of the coarse and fine aggregates are presented in Fig. 3. The fineness modulus of the blend of coarse and fine aggregates used for proportioning of concrete was 4.3.

In order to improve the resistance of concrete to deicer salt scaling, several additives were used in concrete. The additives and their dosages are summarized below.

Commercial air-entraining agent (MasterAir AE 90) was used at a dosage of 1.5 ml per kg of cement. This dosage was used to produce a fresh concrete mixture with air content of 5–7 percent measured using the pressure method per ASTM C231. It should be noted that, at the same dosage of air entraining agent, the air contents of the OPC concrete and the AAC concrete were 7.2 and 5.2%, respectively. The difference could be due to the absorption of the air-entraining agent by the organic carbon that was present in fly ash and survived the mechanochemical process of transforming fly ash and other raw materials into hydraulic cement [12]. The differences in the chemistry of the OPC versus the AAC could have also influenced the

performance of the admixture that has been developed for use with OPC. Polyethylene glycol (PEG) powder (purchased from sigma Aldrich) with molecular weight of 600 was used at 2.5% by weight of cement. It has been reported that the presence of Polyethylene glycol increases the fraction of pores within the 0.1–1.0 μm size range for which the capillary force is much lower than in smaller capillaries [13]. A combination of sodium benzoate and triisopropanolamine was added to concrete as recommended in the literature at 2 and 0.4%, respectively, by weight of cement [14].

Tartaric acid (polyvalent organic oxyacid) powder (purchased from sigma Aldrich) was used at 0.5% by weight of cement. Tartaric acid is a common generic foaming agent used for plaster and cement compositions [15]. This dosage was used after trial and adjustment to achieve a viable balance of compressive strength and set time. Calcined clay with chemical compositions comprising mainly SiO_2 (52 wt%) and Al_2O_3 (46 wt%) was used at 5 wt% by weight of cement to reduce the Si-to-Al ratio of the hydrates of the AAC. Kaolin (purchased from sigma Aldrich) was calcined at 750 $^\circ\text{C}$ in a laboratory furnace (Neytech Benchtop Muffle Furnaces) for 12 h with a heating rate of 15 $^\circ\text{C}/\text{min}$.

2.2. Methods

A 19 L planetary mixer (Hobart A-200, Lombard) was used to prepare the concrete mixture presented in Table 2. With the mixer operating, the hydraulic cement was added first followed by water, and mixing was continued for 2 min. Any additives were then added, and mixing was continued for an extra 2 min. Fine and coarse aggregates were then added, and 3 more minutes of mixing produced a homogeneous fresh concrete mixture. The fresh mix was placed inside molds, and consolidated via vibration at medium intensity for 2 min. The molded specimens were sealed and stored at room temperature for 24 h; they were then demolded and kept sealed at room temperature and $95 \pm 2\%$ relative humidity for the required period of curing (typically 7 days, as described for each test procedure).

The ASTM C1585 sorptivity test was used as a simple and rapid method of determining the tendency of concrete to absorb water by capillary suction. Duplicate cylindrical specimens (cured in sealed condition for 7 days) with 100 mm diameter and 50 mm height were used for this purpose. The concrete specimens were dried in an oven at $105 \pm 2^\circ\text{C}$ to reach a constant mass prior to starting the test. Density, void content, and water absorption capacity of concrete specimens were also measured following the ASTM C642 test procedures. The void space in concrete largely results from the use of water beyond that needed for the hydration process; evaporation of this excess water leaves the capillary pore space. There are also entrapped air voids in concrete due to its imperfect consolidation.

Freeze-thaw tests were conducted per ASTM C666 (Procedure A: rapid freeze-thaw under wet conditions, as shown in Fig. 4a). Freeze-thaw tests were performed on cylindrical specimens of 75 mm diameter and 150 mm height, that had been cured for 7 days in sealed condition at room temperature. One freeze-thaw cycle involves lowering the temperature of the specimen from 6 to -16°C , followed by heating from -16 to 6°C . One cycle lasts 3.2 h, which comprises 1.8 h of freezing 1.4 h of thawing as shown in Fig. 4b; approximately 7 cycles of freezing and thawing were completed per day.

Weight loss and ultrasound pulse velocity were measured after exposure of concrete specimens to repeated freeze-thaw cycles. E-METER (manufactured by James Instrument) resonance frequency tester was also performed for measurement of the resonant frequency and the dynamic modulus of elasticity. The percent change in dynamic elastic modulus (P_c) was determined after application of freeze-thaw cycles:

$$P_c = \frac{n_c^2}{n^2} \times 100$$

where, c is the number of cycles of freezing and thawing, n_c is the resonant frequency after c cycles, and n is the initial resonant frequency (at zero cycles).



Fig. 1. (a) Picture of the sidewalk cast in summer; and (b) the extensive deicer salt scaling experienced after six months.

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