



Freezing characteristics of air-entrained concrete in the presence of deicing salt

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ABSTRACT

The interaction between a salt solution and ice formation in capillary pores might provide new insight into the salt frost scaling process. To accomplish this small prismatic specimens (10 mm × 10 mm × 90 mm) were cut from air-entrained concrete, dried at 50 °C until constant weight, then immersed in sodium chloride (NaCl) solutions of different concentrations (0%, 3%, 9% and 12%) until they reached full capillary saturation. They were then subjected to a freeze–thaw (F–T) cycle in a high resolution low temperature dilatometer (LTD) with the surface temperature and uniaxial length-change continuously monitored. Internal saturation of capillary pores with increasing salt solutions is found to have a profound effect on the F–T response including the temperature rise and instant dilation associated with ice nucleation. It is concluded that salt ions retard ice formation and the effect of ice-growth on pore expansion during an F–T cycle. The presence of salt ions in the concrete pores and surface liquid has a counter-balancing effect on specimen length-change associated with ice-growth, which may provide one possible explanation for the pessimum salt concentration effect. The extent of surface scaling is also affected by the intrinsic capillary transport property (i.e., sorptivity) of the porous cementitious binder. Salt scaling is exacerbated in concretes with increasing sorptivity.

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1. Introduction

Concrete frost durability in general and in particular the frost durability in the presence of deicing salt which is used extensively by highway agencies for snow melting purposes is a major technological problem with many unanswered questions [1]. Studies on frost durability mechanisms typically separate the problem into internal frost damage, a bulk cracking problem, and the surface scaling/flaking problem associated with a salt exposure condition [2]. The latter case is complicated by the influence of salt on the freezing behavior of water in concrete pores and on concrete surface, which leads to many different theories trying to explain why scaling occurs when exposed to a salt solution, while practically no scaling develops when the surface is exposed to pure water [3–13].

Another major complicating factor in the understanding of this process is the so-called pessimum salt concentration effect. Scaling is the worst in a low to moderate concentration range (typically 1.0%–6.0%) [3,7,13–15] and more surprisingly even a 0.5% concentration is reported to cause severe damage [13,15]. Several studies turned to simultaneous measurement of the uniaxial length change and the temperature profile

during an F–T test to investigate how the presence of salts affects ice formation and thus pore pressure build-up [5,10,16–20]. However, the majority only considered internal pore solution [5,10,16,17,20]. In some cases the specimens were harshly pre-saturated prior to F–T test [5,10,20] and thus these findings may be clouded by the high degree of pore saturation in which the air-void system may be compromised.

In the present study, prismatic air-entrained specimens (10 mm by 10 mm by 90 mm) sawn from concrete samples were pre-dried at 50 °C, followed by immersion in water or sodium chloride (NaCl) solutions for capillary saturation only. The effect of salt solutions of different concentrations (0%, 3%, 9% and 12%) inside concrete pores and on concrete surface was investigated on the length- and temperature-change behavior and the associated ice nucleation and growth. Results of this study will hopefully shed more light on the mechanisms for salt frost scaling and the associated pessimum salt concentration effect in cementitious materials.

2. Materials and methods

2.1. Materials

Type I Portland cement and grade 120 slag cement were used as cementitious materials and their characteristics are shown in Table 1. Fine aggregate was silica sand with a fineness modulus of 2.43. Coarse aggregate was lime stone with a 25 mm nominal maximum size.

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Table 1
Characteristics of Portland cement and slag cement.

	Portland cement	Slag cement
CaO, %	62.4	38.0
SiO ₂ , %	20.4	37.5
Al ₂ O ₃ , %	5.04	7.77
Fe ₂ O ₃ , %	2.51	0.43
SO ₃ , %	2.75	3.21
Na ₂ O, %	0.25	0.28
K ₂ O, %	0.67	0.46
MgO, %	3.43	10.7
C ₃ S	53.66	–
C ₂ S	18.01	–
C ₃ A	9.11	–
C ₄ AF	7.64	–
Blaine fineness, cm ² /g	3920	5508

2.2. Mix characteristics and specimen preparation

Concrete mixes with two water-binder (w/b) ratios (0.33 and 0.45, denoted as 033-OS and 045-OS respectively) and one replacement level with slag cement at 0.45 w/b ratio (50% by weight of total cementitious materials, denoted as 045-50S) were prepared. Cylindrical specimens (150 mm in diameter and 300 mm in height) for 045-OS and 045-50S mixes were cast in the laboratory according to ASTM C192 [21], while concrete beams (150 mm × 150 mm × 533 mm) for 033-OS mix were made in a ready-mix plant and delivered to the laboratory after one-day curing. For mixes made in the laboratory, coarse aggregate, part of the mixing water along with the liquid superplasticizer were added to the mixer before it started rotation. This was followed by the addition of fine aggregate, cement and the rest of the mixing water with the air entrainer after the mixer started rotation. The mixing procedure consisted of 3-minute mixing, 3-minute rest, followed by 2-minute final mixing. A slump of 80–100 mm was achieved with a superplasticizer dosage of 4.0 ml/kg cementitious material. The dosage of air entrainer varied from 0.9–1.5 ml/kg cementitious material to achieve an air content of 3.0–8.1%. One non-air entrained concrete mix (045-OS) was prepared as well. All the specimens were removed from the mold after one day and then submerged in tap water at 20 °C for another 27 days before air curing commenced. Mix design and air void properties on hardened concrete specimens according to ASTM C457 [22] are listed in Table 2. The contents of fine and coarse aggregates were 775 kg/m³ and 1115 kg/m³ for the 045-OS and 045-50S mixes and 672 kg/m³ and 1068 kg/m³ for the 033-OS mixes.

Table 2
Mix design and air void properties of hardened concretes.

Mix	w/b ratio	Cementitious material (kg/m ³)	Curing age	Air content (%)		Powers' spacing factor (μm)
				Total	Entrained (<0.5 mm)	
045-OS-1.9%	0.45	290	>1 year	1.88	0.97	265
045-OS-3.0%	0.45	290	28 d, 56 d, >1 year	3.02	2.49	135
045-OS-5.1%	0.45	290	28 d, 56 d	5.12	4.37	104
045-OS-8.1%	0.45	290	28 d, 56 d	8.08	7.16	64
045-50S-3.0%	0.45	290	28 d	3.04	2.08	158
045-50S-4.5%	0.45	290	28 d	4.51	3.43	114
045-50S-7.0%	0.45	290	28 d	7.04	5.79	81
045-50S-3.8%	0.45	290	28 d	3.83	3.01	148
045-50S-7.7%	0.45	290	28 d	7.74	5.63	105
033-OS-5.1%	0.33	390	28 d	5.10	3.44	138
033-OS-5.7%	0.33	390	28 d	5.70	3.53	117
033-OS-8.7%	0.33	390	28 d	8.65	7.18	61
033-OS-9.3%	0.33	390	28 d	9.32	6.19	88

2.3. Testing procedures

2.3.1. Low temperature dilatometer (LTD) test

In this test, a high-resolution horizontal pushrod dilatometer was used to investigate the length-change profile along with the temperature signal associated with ice nucleation and growth in concrete pores, as seen in Fig. 1(a). The LTD has a length-change resolution of 1.25 nm/digit and a temperature precision of 0.1 K. The sample holder was customized to accommodate longer specimens up to 100 mm (Fig. 1(b)) and a liquid nitrogen dewar was equipped for low temperature control.

Concrete specimens sawn from the 150 mm × 300 mm cylinder had a typical dimension of 10 mm × 10 mm × 90 mm and a continuous matrix embedded with coarse aggregate particles (Fig. 1(c)). The prism was pre-dried in the oven at 50 °C to a near-constant weight and then pre-saturated with a test solution (demineralized water or a NaCl solution, denoted as “w” or “x%”, respectively, where “x%” is the salt concentration by mass ranging from 3%–12%) for 1–2 h, until the rapid rise in moisture uptake ended (Fig. 2) such that capillary pores were saturated while air-void filling was prevented. When the effect of surface liquid was studied, several sponge segments pre-saturated with the exposure liquid were placed on the top surface to retain a liquid reservoir while preventing the formation of continuous ice sheets (Fig. 1(c)). The prism was then wrapped in a plastic foil to avoid moisture loss during the test.

The preconditioned specimen was placed in the sample holder such that the pushrod was positioned directly against the center of the sample end and the thermocouple touched one of the lateral surfaces of the specimen (Fig. 1(c)). The specimen was then exposed to a specific temperature program fluctuating between 20 °C and –20 °C in 12 or 15 h for each cycle with a cooling/heating rate of 10 °C/h (Fig. 3). Two profiles were selected and the only difference was the 3-hour isothermal stage

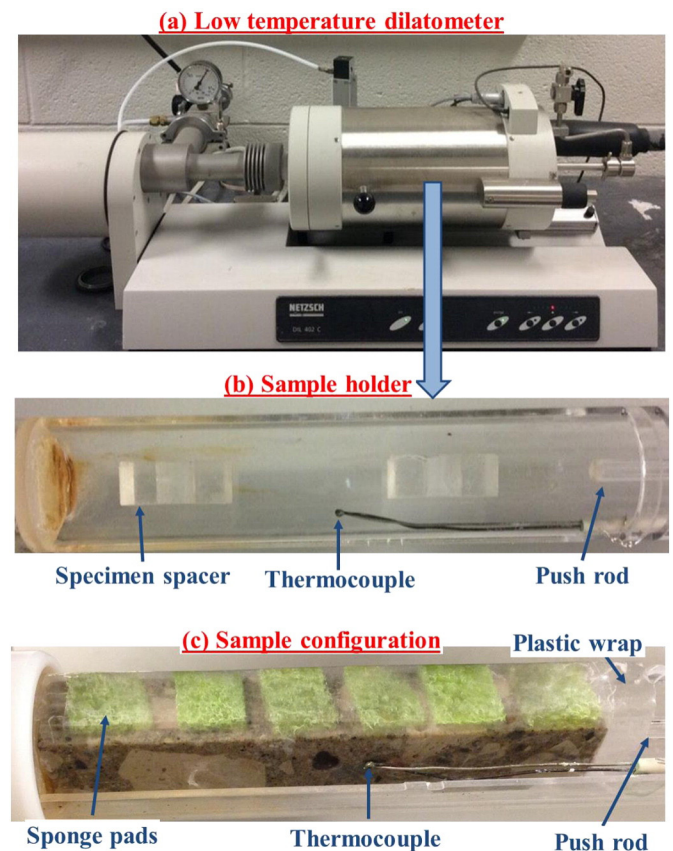


Fig. 1. Low temperature dilatometer configuration.

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