Deicer Scaling Resistance of Concrete Mixtures Containing Slag Cement

Phase 2: Evaluation of Different Laboratory Scaling Test Methods

National Concrete Pavement Technology Center

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16. Abstract

With the use of supplementary cementing materials (SCMs) in concrete mixtures, salt scaling tests such as ASTM C672 have been found to be overly aggressive and do correlate well with field scaling performance. The reasons for this are thought to be because at high replacement levels, SCM mixtures can take longer to set and to develop their properties: neither of these factors is taken into account in the standard laboratory finishing and curing procedures. As a result, these variables were studied as well as a modified scaling test, based on the Quebec BNQ scaling test that had shown promise in other research.

The experimental research focused on the evaluation of three scaling resistance tests, including the ASTM C672 test with normal curing as well as an accelerated curing regime used by VDOT for ASTM C1202 rapid chloride permeability tests and now included as an option in ASTM C1202. As well, several variations on the proposed draft ASTM WK9367 deicer scaling resistance test, based on the Quebec Ministry of Transportation BNQ test method, were evaluated for concretes containing varying amounts of slag cement. A total of 16 concrete mixtures were studied using both high alkali cement and low alkali cement, Grade 100 slag and Grade 120 slag with 0, 20, 35 and 50 percent slag replacement by mass of total cementing materials. Vinsol resin was used as the primary air entrainer and Micro Air® was used in two replicate mixes for comparison.

Based on the results of this study, a draft alternative test method to ASTM C762 is proposed.

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DEICER SCALING RESISTANCE OF CONCRETE MIXTURES CONTAINING SLAG CEMENT PHASE 2: EVALUATION OF DIFFERENT LABORATORY SCALING TEST METHODS

Final Report
July 2012

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EXECUTIVE SUMMARY

One of the durability issues with concrete pavements and parts of concrete structures exposed to deicing chemicals is deicer scaling of surfaces. Scaling of the surface paste layer from the surface is related to freezing and thawing cycles and made worse by exposure to deicing chemicals such as road salt. In most cases, well placed, finished and cured, air-entrained concrete surfaces are resistant to scaling. However, use of inappropriate concrete mixtures or improperly timed or type of finishing, poor curing practices, or premature exposure to freezing temperatures can result in localized scaling.

The standard test method for evaluating the scaling resistance of concrete mixtures is ASTM C672. This method was developed when concretes typically only contained portland cement as the cementitious binder. Now that concretes typically contain fly ash or slag cement, it has been found that these binders often perform poorly when tested by ASTM C672. In Phase 1 of this study, a number of existing concrete pavements and bridges with slag cement were investigated and very few incidences of localized scaling were observed. As well, any scaling that was found that could not be attributed to poor construction practices.

This study was undertaken to evaluate alternative test methods, including finishing and curing practices, in order to develop an alternative laboratory test method to ASTM C672 that would better represent the field performance of concretes containing slag cement. A test method already in use by the Quebec Ministry of Transportation was evaluated, and several modifications were tested. As a result, a new draft test method has been proposed.

INTRODUCTION

Ground granulated blast-furnace slag (GGBFS), simply referred to as "slag cement" or "slag" in this document, has a long history of use with portland cement in concrete. Generally, slag improves many properties of both plastic and hardened concrete. The Transportation Research Board (TRB) Committee on Chemical Additions and Admixtures for Concrete summarizes the impact of slag on the properties of portland cement concrete (PCC) as listed in Table 1 (TRB 1990). It is apparent that slag can make a significant contribution to the production of durable concrete products.

Table 1. Impact of slag cement on various properties of concrete (TRB 1990)

Concrete Property	Impact of Slag
Plastic Concrete Properties	
Air entrainment	Slag may require a slightly larger amount of air-entraining admixture (0%-20%) to reach a given air content; however, this depends on the fineness of the slag.
Water requirement	Slag has little impact on the water demand of concrete.
Workability and finishing	Slag improves the workability and finishing of concrete.
Bleeding	Slag generally reduces bleeding of concrete.
Time of setting	Slag has little influence on the setting time when ambient temperatures are above 80ºF. Below 60ºF special precautions need to be observed to avoid a delay in construction processes.
Hardened Concrete Properties	
Strength	Slag may reduce the early (1 to 7 days) strength of concrete. However, the longer-term (ultimate) strength is generally increased.
Freeze-thaw resistance	Concrete containing slag cement has good freeze-thaw performance; however, scaling may increase.
Permeability	Slag, even at modest replacements (25%), greatly reduces the permeability of concrete and improves its resistance to chloride penetration.
Alkali-silica reaction (ASR) resistance	Slag decreases the expansion caused by ASR.
Sulfate resistance	Slag increases the resistance of concrete to sulfate attack.
Temperature rise	Slag reduces the temperature rise in mass concrete when used in sufficient quantities.

Salt scaling is a prevalent phenomenon in northern climates where concrete is exposed to freezing cycles and the use of deicer salts is quite common. Several scaling tests have been developed to help determine the susceptibility of concretes exposed to deicers and freeze-thaw environments. These include the ASTM C672 test method used in much of North America, the RILEM TC 117-FDC/CDF test method developed in Germany (Setzer and Auberg 1995, Setzer et al. 1996), the Swedish Standard SS 13 72 44 or Borås test method, the MTO LS-412 used in Ontario, and the BNQ NQ 2621-900 test method used in Quebec**.**

Although there are a variety of variations of these test methods being used, their accuracy for predicting the performance of concrete exposed to deicers in a freeze-thaw environment may vary considerably. This has become especially evident with the use of supplementary cementing materials (SCMs) to replace part of portland cement content in concrete mix designs. Of interest is the use of ground granulated blast-furnace slag (referred to as "slag cement") as a replacement for portland cement in amounts higher than 25 percent. For example Bouzoubaa et al. (2008) argued that the BNQ NQ 2621-900 test provides results that better mimic field performance than the ASTM C672 test in particular when evaluating slag cements.

Due to poor performance in laboratory scaling tests, currently the Ministry of Transportation (MTO) and Federal Highway Administration (FHWA) both limit the use of slag to 25 percent replacement for bridge decks and barriers or concretes exposed to deicer salts (Bektas et al. 2010). Bektas et al. (2010) argue that this limit is inappropriate and that a limit of 50 percent slag replacement is better suited and correlates more closely to field performance of slag cements. Other factors besides slag content can significantly influence the performance of concretes to salt scaling, and these include construction practices, curing, maturity, and finishing (Taylor et al. 2004; Boyd and Hooton 2007).

With the use of SCMs such as slag cement in concrete mixtures, salt scaling tests such as ASTM C672 have been found to be overly aggressive, often providing misleading data, and don't correlate well with field scaling performance. The reasons for this are thought to be because this test, including finishing and curing procedures, was developed when concretes typically only used portland cement as the binder. At high replacement levels, SCM mixtures can take longer to set and to develop their properties; neither of these factors is taken into account in the standard laboratory finishing and curing procedures. As a result, these variables were studied as well use of a modified scaling test, based on the Quebec Ministry of Transportation BNQ scaling test that had shown promise in other research (Bouzoubaa et al. 2008, 2011). A new draft ASTM test ASTM WK 9367, based on the BNQ test, had been previously proposed for the evaluation of concrete to salt scaling, but there were several variables that needed to be evaluated before further development was possible, and that was the thrust of this research program.

The experimental research focused on the evaluation of three scaling resistance tests, including the ASTM C672 test with normal curing, as well as an accelerated curing regime used by VDOT for ASTM C1202 rapid chloride permeability tests, and now included as an option in ASTM C1202. In addition, several variations on the proposed draft ASTM WK9367 deicer scaling resistance test, based on the Quebec Ministry of Transportation BNQ test method, were evaluated for concretes containing varying amounts of slag cement. A total of 16 concrete mixtures were cast using each of a high alkali cement and low alkali cement, Grades 100 and 120 slags with 0, 20, 35 and 50 percent slag replacement by mass of total cementing materials. Vinsol resin was used as the primary air entraining admixture and Micro Air® was used in two replicate mixes for comparison.

Based on the results of this study, a draft alternative test method to ASTM C672 is proposed as provided in Appendix A.

PROBLEM STATEMENT

Concrete containing slag generally exhibits excellent long-term strength and durability. However, several authors have expressed concern about the scaling resistance of concrete containing slag, especially when the dosage of slag exceeds 50 percent of the total cementitious materials in the mixture (TRB 1990; Klieger and Isberner 1967; Marchand et al. 1994, 1995; Luther et al. 1994 ACI 233 1995). Much of the concern appears to be based on the results of laboratory scaling tests (most commonly ASTM C672), which tend to be in poor agreement with field observations (Klieger and Isberner 1967; Marchand et al. 1994, 1995; Luther et al. 1994 ACI 233 1995; Hooton et al. 1997; Hooton 2000; Bleszynski et al. 2002; Boyd et al. 2007). Others indicate that the test performs adequately for evaluating the relative scaling resistance of concrete specimens (Newlon et al. 1994). Phase I of this study (Schlorholtz et al. 2008) concluded that concretes containing slag have performed well in field pavements except when good construction practices were not followed.

RESEARCH APPROACH

The experimental research focused on the evaluation of three scaling resistance tests, including the ASTM C672 test with normal curing, as well as an accelerated curing regime used by the Virginia Department of Transportation (VDOT) for ASTM C1202 rapid chloride permeability tests. Several variations on the proposed draft ASTM WK9367 deicer scaling resistance test, based on the Quebec BNQ test method, were evaluated for concretes containing varying amounts of slag cement. A total of 16 concrete mixtures were studied using either a high alkali or low alkali portland cement, Grades 100 or 120 slag at 0, 20, 35, and 50 percent replacement by mass of total cementing materials. Vinsol resin was used as the primary air entraining admixture and Micro Air® was used in two replicate mixes for comparison.

Project Goals (Phase 2- Laboratory Study)

- Document the performance of concrete mixtures containing different cement replacement levels with slag cement when subjected to different variations of deicer scaling test methods
- From evaluation of the results obtained from different variations of the deicer test methods, develop a draft standard test method for use as an alternative to ASTM C672

EXPERIMENTAL WORK

Materials

Cementitious Materials

Two ASTM Type I portland cements types were utilized in this project incorporating low alkali (LA) and high alkali (HA) cement. The LA cement was from Lafarge, Alpena, Michigan. The HA cement was from Holcim, Mississauga, Ontario. Their analyses are presented in Table 2. The ASTM C989 Grade 100 and 120 slags were from Lafarge, near Chicago, Illinois, and their analyses are shown in Table 3.

Table 2. Chemical analysis of the low and high alkali cements

	Low Alkali Cement	**High Alkali Cement**
Blaine fineness (m^2/kg)	383	379
$S0_3$ (%)	2.48	3.97
SiO_2 (%)	20.24	19.6
Al_2O_3 (%)	4.71	5.29
TiO_2 (%)	0.22	
P_2O_5 (%)	0.11	
Fe_2O_3 (%)	2.75	2.3
CaO (%)	62.98	62.61
MgO (%)	2.65	2.4
Na_2O (%)	0.23	0.25
K_2O (%)	0.49	1.19
LOI 1000°C (%)	2.20	2.73
Total (%)	99.75	99.79
Bogue Composition		
C_3S (%)	69.4	55.8
C_2S (%)	5	14.1
C_3A (%)	7.8	10.1
C_4AF (%)	8.6	7

Table 3. Chemical analysis of the Grade 100 and 120 slags

	Grade 100	**Grade 120**
Surface Area Blaine (m^2/kg)	506	590
Cl (%)	0.056	0.094
SO_3 (%)	2.88	2.84
Sulfide (%)	1.16	1.17
Cl (%)	0.056	0.094
SiO_2 (%)	34.48	35.17
Al_2O_3 (%)	10.54	9.52
TiO_2 (%)	0.48	0.45
P_2O_5 (%)	0.01	0.01
Fe_2O_3 (%)	0.76	0.49
CaO (%)	38.4	38.96
MgO (%)	10.66	10.81
Na_2O (%)	0.26	0.27
K_2O (%)	0.41	0.36
Mn_2O_3 (%)	0.32	0.32
LOI 1000°C (%)	1.41	1.34
Total (%)	100.63	100.63

Chemical Admixtures

The MB-VR™ Vinsol-Resin and Micro Air® air-entraining admixtures were obtained from BASF. The air entraining admixtures were dispersed in the mixing water, which was gradually added to the batched materials being mixed. To achieve a range of 100-150 mm slump, Glenium® 7700, a polycarboxylate based high range water reducer was used, supplied by BASF. The high range water reducer was added as needed, based on the observed plasticity of the concrete. Usually half the water reducing admixture was added during initial mixing and the remainder following the 3-minute rest period.

Mix Water

Potable water was used throughout the concrete mixing process.

Aggregates

The 20 mm dolomitic crushed stone was obtained from Holcim's Milton Ontario quarry and the glacial sand from St Marys Sunderland pit in Ontario.

The fineness modulus (FM) for the fine aggregate used to cast mixes prior to October 12, 2011 was 2.89 and the FM used in the casting of concrete mixes after 12 October 2011 was 2.50. The relative densities for these two deliveries of fine aggregate were 2.76 and 2.72, respectively, while absorptions were 2.18 and 0.63, respectively.

The dry-rodded density of the 5-20 mm coarse aggregate was 1561 kg/m^3 for mixes cast prior to October 12, 2011 and 1538 kg/m^3 for mixes cast thereafter. The relative density and absorption for the two deliveries of coarse aggregates were 2.75 and 2.78, respectively, while absorptions were 1.86 and 1.69 percent, respectively.

Geotextile

A non-woven geotextile material, type 800R from Terrafix® Geosynthetics Inc. in Toronto was placed at the bottom of the scaling slab molds for the BNQ test to absorb bleed water. It was approximately 4 mm thick and was placed dry at the bottom of the mold in two layers to achieve the specified thickness of 7±1 mm specified in the BNQ test method. The absorption capacity of the geotextile when fully saturated was determined to be approximately 3960g/m^2. The geotextile's rate of absorption decreases with time as it becomes saturated and is reported in Figure 1.

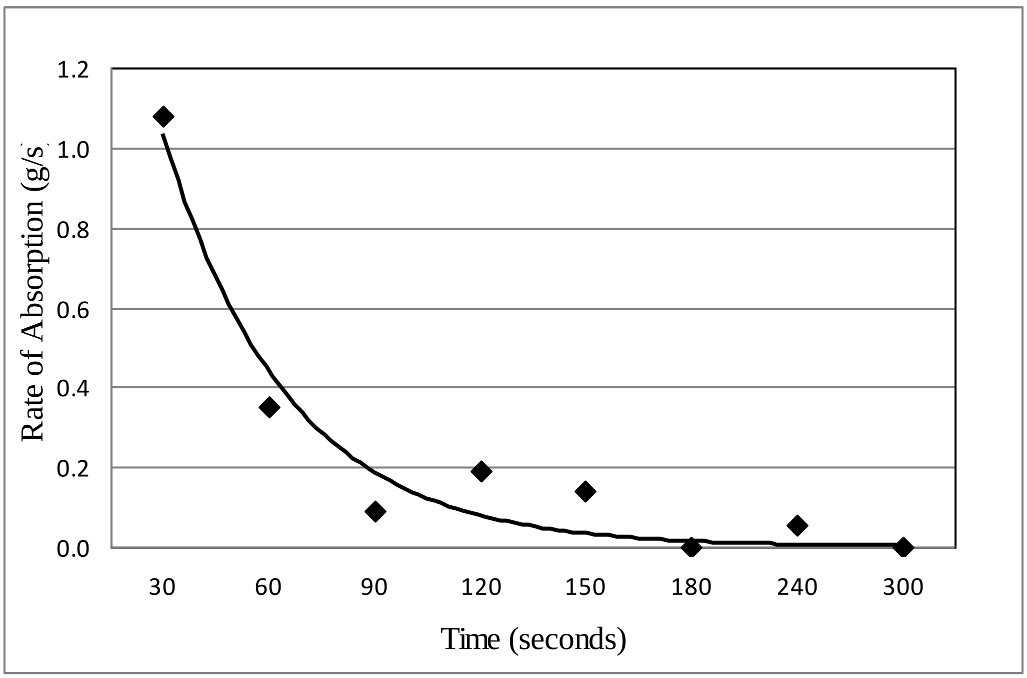

Figure 1. 8 mm thick geotextile rate of water absorption

Deicing Solutions

Two types of solutions were prepared for deicer scaling, 3wt% NaCl for the modified BNQ scaling test and slabs cured using the VDOT curing regime, and 4wt% $CaCl_2$ for the ASTM

C672 tests. Distilled water was used in the preparation of each solution. The $CaCl_2$ compound used to prepare the deicer solution was supplied in the form of $CaCl_2 \cdot 2H_2O$ with a molecular weight of 147.01g. Therefore its anhydrous equivalent was calculated to be 53.09g/L of $CaCl_2 \cdot 2H_2O$.

Concretes

Mix Designs

Using the aggregate data, mix designs were established as reported in Table 4. All mixing was performed in accordance with ASTM C192/C192M. From each mix, a minimum of six slabs measuring 200 mm x 300 mm were cast. Their thickness was either 90 mm without the geotextile or 82 mm when the geotextile was placed at the bottom of the molds. Two slabs with 90 mm thickness were tested according to ASTM C672, and the other slabs were tested according to the modified BNQ and VDOT curing procedures. A total of 15 cylinders were cast per mix for compressive strength, rapid chloride permeability testing (ASTMC1202) and for hardened air void analysis. Cylinders were also cured according to the VDOT accelerated curing regime, which requires seven days of moist curing at 23°C followed by 21days of moist curing at 38°C. For Mixes #9 and #16, eight slabs were cast to evaluate the effectiveness of one-dimensional freezing and the removal of the geotextile in the modified BNQ procedure. Mix #4 was recast due to a very low slump experienced during initial casting. Mix #5 was recast because too high an air content was obtained during initial casting. Mix #9 was recast to evaluate the effects a 14 day drying period following the accelerated VDOT curing regime, and to verify reproducibility of previous results.

Table 4. Concrete mix designs

Cast Date	Mix	Portland Cement Type	Slag Content (%)	Slag Type	Air Entraining Admixture (mL/m3)	HRWR Admixture (mL/m3)	w/cm	Total Cement Content (kg/m³)	Coarse Agg. Mass (kg/m³)	Fine Agg. Mass (kg/m³)	Mass Portland Cement (kg/m³)	Mass Slag Cement (kg/m³)	Mass Water (kg/m³)
24-Aug-11	1	High-Alkali	0	N/A	135.7	1571.4	0.42	338	971.4	902.4	338	N/A	142.0
21-Oct-11	2	High-Alkali	50	Grade 100	78.6	2000.0	0.38	338	1016.6	844.5	169	169	128.4
28-Oct-11	3	High-Alkali	50	Grade 120	128.6	2000.0	0.38	338	1016.6	844.5	169	169	128.4
26-May-11	4	Low Alkali	0	N/A	107.7	923.1	0.42	338	971.4	902.4	338	N/A	142.0
07-Nov-11	4 Redo	Low Alkali	0	N/A	105.7	1285.7	0.42	338	1016.6	855.9	338	N/A	142.0
18-Oct-11	5	Low Alkali	20	Grade 100	85.7	928.6	0.42	338	1016.6	837.6	270.4	67.6	142.0
12-Oct-11	5 Redo	Low Alkali	20	Grade 100	75.7	1214.3	0.42	338	1016.6	837.6	270.4	67.6	142.0
14-Oct-11	6	Low Alkali	35	Grade 100	75.7	1142.9	0.42	338	1016.6	841.0	219.7	118.3	142.0
15-Aug-11	7	Low Alkali	50	Grade 100	92.9	1142.9	0.42	338	971.4	890.7	169	169	142.0
31-Aug-11	8	High Alkali	20	Grade 100	107.1	642.9	0.42	338	971.4	883.7	270.4	67.6	142.0
26-Aug-11	9	High Alkali	35	Grade 100	128.6	571.4	0.42	338	971.4	887.2	219.7	118.3	142.0
06-Oct-11	9 Redo	High Alkali	35	Grade 100	126.7	1000.0	0.42	338	971.4	887.2	219.7	118.3	142.0
21-Jun-11	10	High Alkali	50	Grade 100	85.7	0.0	0.42	338	971.4	890.7	169	169	142.0
31-May-11	11	Low Alkali	20	Grade 120	100	857.1	0.42	338	971.4	883.7	270.4	67.6	142.0
01-Jun-11	12	Low Alkali	35	Grade 120	92.9	571.4	0.42	338	971.4	887.2	219.7	118.3	142.0
07-Jun-11	13	Low Alkali	50	Grade 120	90	857.1	0.42	338	971.4	890.7	169	169	142.0
23-Aug-11	14	High Alkali	20	Grade 120	113.3	533.3	0.42	338	971.4	883.7	270.4	67.6	142.0
19-Aug-11	15	High Alkali	35	Grade 120	114.3	714.3	0.42	338	971.4	887.2	219.7	118.3	142.0
17-Aug-11	16	High Alkali	50	Grade 120	126.7	1466.7	0.42	338	971.4	890.7	169	169	142.0

Batching and Mixing

All aggregates were batched at least two days prior to mixing and sealed in plastic buckets. The coarse aggregate was washed in perforated plastic buckets to allow for drainage and remained there until mixing. Moisture content of the aggregates was determined according to ASTM C566 (2004). The LA and HA cements and Grade 100 and 120 slags were kept in sealed drums until the day of mixing. The air entraining admixture was added to the mix water and stirred to allow for dispersal. All the cementing materials and aggregates were added to the mixer, dry mixed, and then the mix water was added gradually. A portion of the high-range water reducer was added during initial mixing and the remainder was added as necessary after the 3 minute rest period.

Casting and Finishing

Immediately after mixing was completed, air and slump tests were performed on the fresh concrete, according to ASTM C231 (2010) and ASTM C143 (2010). Fifteen cylinders (100 mm x 200 mm) were then cast and cured according to ASTM C192 (2007). Accelerated curing was performed on two compression test cylinders and one RCPT cylinder according to VDOT as described previously.

Two slabs (200 mm x 300 mm) were cast for each deicer scaling test being performed using wooden forms. The forms were lightly oiled prior to casting and two layers of geotextile were placed in the bottom of each slab mold, except for slabs tested according to ASTM C672. The concrete was placed in one layer, with some excess on the surface, and rodded every 1300mm^2 (2in^2) using a 16 mm diameter rod. The sides of the mold were then tapped with a rubber mallet to consolidate the concrete. The excess concrete was then removed using a wooden screed. The surface was finished immediately after initial screeding using a wooden trowel by performing two passes in the longitudinal and transverse directions. The trowel was moistened just enough (not wet/saturated) to prevent water absorption from the concrete surface to the wood. A gentle sawing motion was applied with each pass, which allowed the trowel to glide across the surface and produce a smoother finish. The trowel was cleaned after each pass to remove concrete stuck to the bottom and ensure a smooth surface. After bleeding appeared to be complete, brushing of the concrete surface was performed on the slabs tested according to ASTM C672. One pass was made in the transverse direction with a medium stiff plastic brush. The slabs and cylinders were then cured by covering them with a damp tarpaulin, and then a plastic sheet to prevent evaporation.

Curing

All the slabs and cylinders were demolded after 24 hours and placed in the moist curing room at 25°C and 100 percent relative humidity (RH), according to ASTM C511 (2009). The RCPT cylinders were immediately submerged in lime water solution with 4g/L of $Ca(OH)_2$.

The ASTM C672 and BNQ scaling slabs were moist cured for 14 days followed by a 14 day drying period at 50 percent RH. The VDOT slabs were moist cured for 7 days, then placed in plastic containers and submerged in 4g/L of $Ca(OH)_2$ solution and moist cured for an additional 21 days at 38°C. After curing the BNQ and VDOT slabs were then pre-saturated with 3wt% NaCl to limit osmotic effects.

What is referred to as the VDOT curing regime in this paper was a curing regime originally developed by Ozyildirim (1998) for the purpose of reducing permeability of mixes containing SCMs, which were evaluated according to ASTM C1202. Ozyildirim (1994) recommended specimens to be cured for 7 days at 23°C and then for 21 days at 38°C to accelerate pozzolanic reactions, and therefore reduce permeability in these mixes.

Ponding

An impervious frame or dike keeping the saline solution at the surface of the specimen was made using 25 mm wide by 60-75 mm high extruded polystyrene rigid insulation. It was bonded to the sides of the concrete slabs using clear silicone caulking. Sufficient time was allowed, according to manufacturer recommendations, for the silicone to dry before pouring in any saline solution. For the BNQ tests, a 3wt% NaCl saline solution was placed on the slab surface inside the dike to commence pre-saturation, and the slabs were covered with a plastic sheet to prevent evaporation.

Test Methods and Procedures

Compressive Strength Testing

Testing was performed on 100 mm x 200 mm concrete cylinders according to ASTM C39 The ends of the cylinders were ground flat and loading was applied at a rate of 2 kN/s until failure. Compressive strength tests were performed at 7, 14, 28 and 56 days.

Rapid Chloride Permeability Testing (RCPT)

RCPT tests were performed according to ASTM C1202, with several modifications. Instead of vacuum saturating the specimens, after demolding, the concrete cylinders were immediately placed in 4g/L $Ca(OH)_2$ solution for saturation and remained submerged until time of test. On the day of test, the 100 mm x 200 mm cylinders were cut to obtain two discs of 100 mm diameter and 50 mm thickness from the center portion of the cylinder. The circumference of each disc was then wrapped in electrical tape to seal the concrete. Each disc was then placed in the cell, with 3wt% NaCl solution at the positive terminal and 0.3N NaOH solution at the negative terminal. The charge and current passed at one minute was recorded to obtain conductivity according to ASTM C1760, and the test was allowed to run for six hours, with continuous recording of temperature, current, and charge passed. Two specimens were tested at 14, 28 and 56 days.

Deicer Scaling Tests

Three primary scaling procedures were evaluated in this study according to the ASTM C672 test, the modified BNQ (proposed ASTM WK 9367) test, and the BNQ test using the VDOT accelerated curing regime. A summary of the differences between these tests is provided in Table 5. The visual assessment used to assess the surface condition of each slab was normalized for the three methods used and is presented in Table 6.

Table 5. Comparison of the three deicer scaling methods evaluated in this project

	ASTM C672 (with MTO mass loss limits)	BNQ 2621-900	Modified BNQ 2621-900 with VADOT Accelerated Curing
Saline Solution	4% $CaCl_2$	3% NaCl	3% NaCl
Specimens	•Rectangular prisms measuring 300mm·200mm·90mm •Surface area 0.06m2 •2 specimens / mix	•Rectangular prisms measuring 300mm·200mm·82mm •Surface area 0.06m2 •2 specimens / mix	•Rectangular prisms measuring 300mm·200mm·82mm •Surface area 0.06m2 •2 specimens / mix
Finishing	Finish after the concrete has stopped bleeding and then brush with a medium-stiff brush	Only initial wood screed required, no final finishing, instead 7 ± 1mm geotextile is placed at bottom of specimen when casting to provide drainage.	Only initial wood screed, no final finishing, instead 7 ± 1mm geotextile is placed at bottom of specimen when casting to provide drainage.
Curing of Specimens	•14 days at 100% RH •14 days at 50% ± 5% RH	•14 days at 100% RH •14 days at 50% ± 5% RH •7 days of presaturation with saline solution	•21 days at 38°C; 100% RH (immersed in lime water in this study) •7 days of presaturation with saline solution without 14 day drying period (except as later modified)
Dike Placement	• 2 days after end of 14 day moist curing period	• 2 days after end of 14 day moist curing period	•after end of 28 day moist curing period (later modified when 14d drying period added)
Freezing and Thawing Cycles	•50 cycles of 24h •Freezing at -18 ± 3°C for 16 ± 1h •Thawing at 23°C ± 2°C for 8 ± 1h; 45-55% RH	•50 cycles of 24h (BNQ uses 56 cycles) •Freezing at -18 ± 3°C for 16 ± 1h of which a maximum of 12h and a minimum of 7h at -18°C •Thawing at 23°C ± 2°C for 8 ± 1h; 45-55% RH	•50 cycles of 24h •Freezing at -18 ± 3°C for 16 ± 1h of which a maximum of 12h and a minimum of 7h at -18°C •Thawing at 23°C ± 2°C for 8 ± 1h; 45-55% RH
Evaluation of Surface Quality	•After 5, 10, 15, 25 and 50 cycles •Visual assessment of surface based on 5 categories •Mass measure of cumulative scaled off material in kg/m2 (based on MTO limit)	•After 5, 10, 15, 25 and 50 cycles •Visual assessment of surface based on 5 categories •Mass measure of cumulative scaled off material in kg/m2	•After 5, 10, 15, 25 and 50 cycles •Visual assessment of surface based on 5 categories •Mass measure of cumulative scaled off material in kg/m2
Acceptance Criteria	After 50 cycles the cumulative mass of scaled off material must be <0.8 kg/m^2	After 50 cycles the cumulative mass of scaled off material must be <0.5 kg/m^2	After 50 cycles the cumulative mass of scaled off material must be <0.5 kg/m^2

Table 6. Visual assessment ratings used for scaling

Visual Assessment Rating	Characteristics of the scaling surface
0	No significant scaling observed
1	Very slight scaling 3mm (1/8") depth, max, no coarse aggregate visible and no pop-outs present
2	Slight to moderate scaling and/or presence of a few pop-outs
3	Moderate scaling with some exposed coarse aggregate
4	Moderate to severe scaling where the coarse aggregate is clearly exposed and there is scaling of the surface mortar
5	Severe scaling course aggregates are visible over the entire surface

Air Void Analysis

Air void analysis was performed on one 100 mm x 200 mm concrete cylinder for each mix that had been cured for at least 28 days. The cylinder was cut longitudinally in half so that two specimens approximately 100 mm x 75 mm could be polished. After polishing, surfaces were blackened and the voids filled with barium sulfate, then the air void parameters were measured using the RapidAir 457 image analysis equipment.

Temperature Analysis of Slab Temperatures during Freezing

One slab was instrumented with three separate thermocouples to measure the rates of freezing and thawing: (a) just above the slab surface and submerged in NaCl solution, (b) 3 mm below the slab surface, and (c) at the slab’s center of mass 42 mm below the surface. The 1st cycle was performed on the top shelf of the vertical freezer, the 2nd cycle took place in the middle portion, and the 3rd cycle was at the bottom of the freezer. Readings were automatically recorded every 5 minutes over three 24-hour cycles.

Placement of the thermocouples was achieved by molding the relatively stiff wires into shape and securing them to each other with steel wire. The ends of the thermocouples were centered in the slab. Figure 2 shows the setup of the thermocouples. Concrete was uniformly placed and consolidated around the wires to completely cover them. Excess concrete was removed from the surface with the wood screed, and the wires were gently wiggled into place, simulating vibration. Finishing of the surface was performed carefully around the single protruding thermocouple. The slab was cured according to the ASTM C672 procedure and exposed to freezing and thawing at 28 days of age in 3wt% NaCl solution.

Figure 2. Thermocouples prior to concrete placement

Depth of Carbonation and Chloride Ion Penetration

After 50 cycles of freezing and thawing, the slabs from each mix were split in two equal sections. One half of each slab interior was sprayed with a phenolphthalein solution to determine the extent of carbonation and the other half was sprayed with silver nitrate to determine the extent of Cl^- ingress. Measurements were then made using a ruler with millimeter precision to determine the average carbonation and chloride penetration depth.

RESULTS AND DISCUSSION

Compressive Strength Results

Testing was performed on 100 mm x 200 mm concrete cylinders according to ASTM C39. The cylinders were ground flat using an end grinder and loading was applied at a rate of 2 kN/s until failure. Compressive tests were performed at 7, 14, 28 and 56 days. For each mixture, two additional cylinders were tested at 28 days that had been cured using the accelerated VDOT regime consisting of 7days moist curing at 23°C (73°F) followed by 21 days moist curing at 38°C (100°F). Table 7 presents the strength results.

Table 7. Compressive strength results

Average Compressive Strengths (MPa) (1 MPa = 145 psi)					
Mix #: % Cement w/cm	**7 days**	**14 days**	**28 days**	**56 days**	**VDOT Accelerated Curing 28 days**
Mix #1: 100HA 0.42wc	34.3	39.2	43.0	48.1	48.6
Mix #2: 0.5HA 0.5SG100 0.38wc	40.7	44.0	48.0	60.7	51.0
Mix #3: 0.5HA 0.5SG120 0.38wc	44.6	49.9	50.8	60.1	54.3
Mix #4: 100LA 0.42wc	43.8	45.7	47.0	54.1	51.9
Mix #4: 100LA 0.42wc Recast*	25.5	28.2	24.5	36.9	29.0
Mix #5: 0.8LA 0.2SG100 0.42wc	37.5	40.7	44.1	38.7	49.0
Mix #5: 0.8LA 0.2SG100 0.42wc Recast	37.8	43.2	45.8	50.5	48.3
Mix #6: 0.65LA 0.35SG100 0.42wc	38.4	45.2	49.2	50.2	48.8
Mix #7: 0.5LA 0.5SG100 0.42wc	35.4	41.5	45.7	48.7	49.8
Mix #8: 0.8HA 0.2SG100 0.42wc	29.8	37.8	34.3	46.0	39.2
Mix #9: 0.65HA 0.35SG100 0.42wc	30.3	33.0	35.7	43.1	39.6
Mix #9: 0.65HA 0.35SG100 0.42wc Recast	39.8	42.1	49.3	45.2	44.3
Mix #10: 0.5HA 0.5SG100 0.42wc	23.4	28.2	29.1	33.0	32.5
Mix #11: 0.8LA 0.2SG120 0.42wc	38.2	37.5	42.0	44.8	49.6
Mix #12: 0.65LA 0.35SG120 0.42wc	37.3	41.4	48.3	48.7	51.2
Mix #13: 0.5LA 0.5SG120 0.42wc	42.5	43.0	44.3	49.4	50.4
Mix #14: 0.8HA 0.2SG120 0.42wc	32.2	38.9	37.5	42.7	37.2
Mix #15: 0.65HA 0.35SG120 0.42wc	35.2	36.5	38.5	45.0	41.8
Mix #15: 0.65HA 0.35SG120 0.42wc Micro Air®	47.4	48.3	52.2	53.3	54.8
Mix #16: 0.5HA 0.5SG120 0.42wc	36.7	39.4	38.5	45.9	42.4
Mix #16: 0.5HA 0.5SG120 0.42wc Micro Air®	35.8	38.2	38.2	43.1	43.1

* High air content resulted in low strengths

All the mixes, except for Mix #10 with high air content, had compressive strength results ranging between 40 and 50 MPa at 56 days. When the w/cm ratio was reduced from 0.42 to 0.38 (Mixes# 2 and 3), compressive strengths of 60 MPa were obtained at 56 days. Compressive strength increased with curing time except in four instances, where slight drops in compressive strength occurred at either 28 or 56 days. It is assumed this was due to cylinder defects or from the end grinding process, resulting in slightly lower compressive strengths (the end-grinder was refurbished after these tests were completed).

As expected, the low 0.38 w/cm mixes with the 50 percent HA cement achieved some of the highest compressive strengths, despite their high slag content of 50 percent. This corresponds with other results, such as those of Valenza II and Scherer (2007a). In general, the accelerated curing did not increase compressive strength significantly at 28 days. Only marginal gains of about 4 MPa were experienced due to the VDOT accelerated curing, which consisted of 7 days moist curing followed by 21 days of submerged curing in $Ca(OH)_2$ solution at 38°C (100°F). This is probably because the temperature difference between the moist curing room at 23°C and the 38°C room is small (a difference of only 15°C). Additionally, much of the hydration has taken place by the 7th day, when the accelerated curing begins.

From Table 7 it is clear that almost all the mixes achieved 70 percent of their compressive strength within 7 days. Therefore, even with mixtures containing slag, accelerated curing did not have a large impact on strength. The LA cement mixes performed better than the HA mixes in all compressive strength tests regardless of slag type and slag content used. On average, LA cement with Grade 100 slag developed 10 MPa higher compressive strengths when compared to HA cement mixes with identical slag contents. In addition, the LA cement combined with Grade 120 slag mixes experienced only an average 5 MPa higher compressive strength, compared to the HA cement mixes with identical slag contents. Overall, the LA cement mixes had higher compressive strengths than HA cement mixes at identical slag contents.

By comparing each the same cements (LA or HA) with each of the slag types, it was observed that Grade 100 slag mixes performed better than Grade 120 slag mixes in some cases, but not in others. In each case, the differences in compressive strength were not substantial. It was expected that the Grade 120 cement slag mixes would achieve higher compressive strengths than the Grade 100 slag mixes due to increased fineness of grinding. However, other variables such as cement type, entrained air content, and the dosage of high-range water reducer would also have influenced the results.

Increasing the slag content did not have a consistent impact on compressive strength in any of the slag mixes. It was expected that with increasing slag content, the concretes would exhibit lower compressive strengths at 7 days and higher gains in compressive strength over time (at 56 days). This may have been due to the high fineness of both slags, enabling them to react quickly. Table 4.4 displays air content results. Increasing the air content reduced compressive strength, as expected. A reduction of about 40 percent was observed in Mix #4 when it was initially cast with a low hardened air content of 3.4 percent and then recast with a hardened air content of 10 percent. However, very little influence of air content was observed on the compressive strength of Mix #5 that was initially cast with a high hardened air content of 7.8 percent and then recast with 5.1 percent air content.

Mixes #15 and 16 that were recast using Micro Air® air-entraining admixture performed better in terms of compressive strength when compared to the same mixes cast using Vinsol resin. This is somewhat surprising because the air contents in these mixes were much higher. It is possible that the smaller air bubbles produced using Micro Air® allow for better load distribution, and therefore higher strength, compared to concretes with the larger air bubbles produced using Vinsol resin.

In summary, the primary factors governing compressive strength results appear to be the w/cm ratio, cement type, and age. Varying the slag type produced inconclusive results. Varying the slag content did not make a significant difference in compressive strength, but increasing the air content above 9 percent reduced compressive strength.

Rapid Chloride Permeability Test (RCPT) Results

For each concrete mix two specimens were analyzed using the ASTM C1202 procedure. The RCPT results for each mix are displayed in Table 8. It was observed that reducing the w/cm ratio from 0.42 to 0.38 (Mixes #2 and #3) produced the most significant reduction in charge passed and best results in terms of resistance to current flow. It may be argued that the increase in slag content is what caused the majority of the reduction in current flow; however, when the remaining 50 percent slag mixes are compared to Mixes #2 and #3, it is clear that the charge passed is still much lower; as the w/c ratio is reduced, the concrete pores become smaller and the capillary pore network more tortuous.

Nevertheless, increasing the slag content also significantly reduced current flow in every case. For example, when 100 percent cement was used, the charge passed was >4000 coulombs at 7 days and reduced to the low 2000 coulomb range by 56 days. Adding 50 percent slag reduced the charge passed to <1400 coulombs at 7 days, and to around 700 coulombs at 56 days.

Table 8. ASTM C1202 Data

ASTM C1202 Resistance to Chloride Penetration (coulombs)				
Mix #: % Cement w/cm	**14 days**	**28 days**	**56 days**	**VDOT Accelerated**
Mix #1: 100HA 0.42wc	4514	3552	2393	1959
Mix #2: 0.5HA 0.5SG100 0.38wc	821	674	592	522
Mix #3: 0.5HA 0.5SG120 0.38wc	773	560	518	511
Mix #4: 100LA 0.42wc	4920	3664	2057	3283
Mix #4: 100LA 0.42wc Recast	4208	3124	2678	2192
Mix #5: 0.8LA 0.2SG100 0.42wc	2568	2399	1565	2044
Mix #5: 0.8LA 0.2SG100 0.42wc Recast	2055	1933	1606	1592
Mix #6: 0.65LA 0.35SG100 0.42wc	1430	1203	1002	898
Mix #7: 0.5LA 0.5SG100 0.42wc	1260	1020	764	778
Mix #8: 0.8HA 0.2SG100 0.42wc	2971	1772	1341	1299
Mix #9: 0.65HA 0.35SG100 0.42wc	1412	1258	832	930
Mix #9: 0.65HA 0.35SG100 0.42wc Recast	1430	1164	861	805
Mix #10: 0.5HA 0.5SG100 0.42wc	1238	1019	763	658
Mix #11: 0.8LA 0.2SG120 0.42wc	2743	2018	1254	1415
Mix #12: 0.65LA 0.35SG120 0.42wc	2019	1243	1147	909
Mix #13: 0.5LA 0.5SG120 0.42wc	1332	992	648	744
Mix #14: 0.8HA 0.2SG120 0.42wc	2207	1734	1209	1386
Mix #15: 0.65HA 0.35SG120 0.42wc	1463	1164	762	950
Mix #15: 0.65HA 0.35SG120 0.42wc Micro Air®	993	738	609	492
Mix #16: 0.5HA 0.5SG120 0.42wc	993	826	703	688
Mix #16: 0.5HA 0.5SG120 0.42wc Micro Air®	860	573	568	606

At 56 days and in general, HA cement-slag mixtures performed better in the RCPT test than LA cement-slag mixtures, but differences were less than 500 coulombs on average.

In general, the Grade 120 slag performed better than the Grade 100 slag, however, the differences were small (<500 coulombs). Again this is probably due to the higher fineness of the Grade 120 slag cement.

The age of the concrete had a large influence on the RCPT results. As curing age increased, the charge passed decreased significantly due to the higher degree of hydration. Accelerated curing had a marginal, but positive effect on the RCPT results (unlike the large reductions found for fly ash concretes by VDOT (Ozyildirim 1998)). This is likely because the temperature difference between the moist curing room at 25°C and the 38°C room is small (only 12°C). Additionally, unlike fly ash, some of the slag hydration has taken place by the 7th day, which is the point when accelerated curing begins.

The charge passed was reduced in Mixes #15 and #16 when Micro Air® admixture was used instead of Vinsol resin, even though the air contents were higher. This may be due to the smaller air bubbles produced, which limit ionic flow.

The main factors influencing the RCPT test appear to be w/cm ratio, age of concrete and slag content. The choice of HA cement over LA cement, Grade 120 over Grade 100 slag, and use of an accelerated curing regime all reduced coulomb values, but the differences were small. The high resistance to current flow of the slag mixtures in the ASTM C1202 test indicates that slag concretes can resist Cl^- ion ingress better than 100 percent HA and LA portland cements.

Scaling Test Results

Deicer scaling tests were conducted according to ASTM C672, the modified BNQ, and BNQ test using the accelerated VDOT curing procedure. The average cumulative scaling mass loss is presented in Table 9 for each testing procedure and corresponding mix design.

Reducing the w/cm ratio from 0.42 to 0.38 resulted in the most significant reduction in scaling, even with 50 percent slag content regardless of test method and slag grade used. All slabs with w/cm of 0.38 passed the scaling mass loss requirements. Reduction of the w/cm ratio, improves concrete durability, reduces bleeding, and decreases porosity.

Table 9. Scaling mass losses and concrete air properties

Average Scaling Mass Loss after 50 cycles (g/m2)									
Mix	Curing Regime and Average Mass Loss at 50 Cycles						Fresh Air Content (%)	Hardened Air Content (%)	Spacing Factor (mm)
Test Method	ASTM C672	BNQ	VADOT	VADOT + 14days drying	BNQ 1D Freezing	BNQ no geotextile			
Salt Solution	4% CaCl2	3% NaCl	3% NaCl	3% NaCl	3% NaCl	3% NaCl			
Preconditioning of Slabs	14dmoist/ 14d dry	14dmoist / 14d dry/7d solution	7d moist-23C/21-d wet-38C	7d moist-23C/ 21d wet-38C/14d dry	14dmoist/ 14d dry/7d solution	14dmoist/ 14d dry/7d solution			
Age when Freezing started	28 days	35 days	28 days	42 days	35 days	35 days			
Mix #1: 100HA 0.42wc	1064	95	3692				6.0	4.84	0.247
Mix #2: 0.5HA 0.5SG100 0.38wc	248	486	277				6.2	5.97	0.196
Mix #3: 0.5HA 0.5SG120 0.38wc	101	163	487				6.4	6.97	0.178
Mix #4: 100LA 0.42wc	409	104	3966				6.1	3.36	0.278
Mix #4 Recast: 100LA 0.42wc	170	163	487				9.4	10	0.122
Mix #5: 0.8LA 0.2SG100 0.42wc	268	75	78				7.5	7.77	0.165
Mix #5 Recast: 0.8LA 0.2SG100 0.42wc	205	79	116				6.8	5.09	0.275
Mix #6: 0.65LA 0.35SG100 0.42wc	527	241	197				7.0	6.7	0.253
Mix #7: 0.5LA 0.5SG100 0.42wc	580	1529	1221				6.1	4.95	0.252
Mix #8: 0.8HA 0.2SG100 0.42wc	944	986	934				6.2	4.5	0.239
Mix #9: 0.65HA 0.35SG100	897	958	1101				7.0	4.91	0.283
Mix #9 Recast: 0.65HA 0.35SG100	290	N/A	1013	553	137		6.4	5.82	0.248
Mix #10: 0.5HA 0.5SG100 0.42wc	2568	2662	1698				7.0	6.8	0.155
Mix #11: 0.8LA 0.2SG120 0.42wc	399	478	545				7.0	5.92	0.181
Mix #12: 0.65LA 0.35SG120 0.42wc	730	1342	1563				6.5	5.05	0.264
Mix #13: 0.5LA 0.5SG120 0.42wc	1574	2576	2042				6.4	5.63	0.228
Mix #14: 0.8HA 0.2SG120 0.42wc	777	236	761				6.2	5.83	0.214
Mix #15: 0.65HA 0.35SG120 0.42wc	546	1661	1018				6.0	3.22	0.358
Mix #16: 0.5HA 0.5SG120 0.42wc	637	1797	1683			1658	6.0	4.52	0.23

100% Portland Cement Mixtures

Scaling performance after exposure to the VDOT accelerated curing (but with no drying period) was much worse than with the ASTM and BNQ curing methods. The ASTM method passed the 100 percent LA cement mix, however, scaling slightly exceeded the limits for the 100 percent HA cement mix. It is possible that the 100 percent HA cement mix failed the3 MTO mass loss criteria using the ASTM C672 method because the hardened air content was found to be 4.8 percent, which is below the specified range of 6 to 7 percent air entrainment. The hardened air content of the LA cement mix was found to be very low at 3.4 percent, while the fresh concrete air content as measured by the air meter was 6.1 percent. This large discrepancy is believed to have occurred because of a longer than expected casting time, since only one technician was available for the preparation of this particular mix. This resulted in a very low slump, which made it difficult to finish and consolidate the concrete. However, these results also indicate the strong resistance of the LA cement mix to scaling even at low air entrainment contents. The BNQ method gave the best performance with very limited scaling, which was below both the ASTM <0.8 kg/m^2 and BNQ <0.5 kg/m^2 mass loss scaling limits.

20% Slag Mixtures

The LA cement mixes performed better than the HA cement mixes regardless of slag type used. All the 20 percent slag mixes passed the MTO scaling limit except for Mix #8 (80% HA 20% SG100 0.42w/c). Mix #8 failed all three scaling tests marginally (refer to Table 9), which could be explained by its lower hardened air-entrained content of 4.5 percent, compared to air contents greater than 5 percent for the remaining 20 percent slag mixes. The BNQ method gave the best performance, meeting both the MTO <0.8kg/m^2 and BNQ <0.5 kg/m^2 scaling mass loss requirements. The Grade 120 slag performed better than the Grade 100 slag in the HA cement mixtures, but the Grade 100 performed better in the LA cement mixtures.

35% Slag Mixtures

The slabs exposed to the ASTM method performed the best and passed the MTO limit of 0.8 kg/m^2. It is important to note that Mix #9 (65%HA 35% SG100 0.42w/cm) initially just failed the ASTM test at 4.9 percent hardened air entrainment, but when recast with a higher hardened air-entrained content of 5.8 percent it passed well below the specified MTO scaling limit. The results appear to be inconclusive for the BNQ standard and VDOT curing methods as they performed very well for Mix #6 (65%LA 35% SG100 0.42w/cm), but performed quite poorly in all the other 35 percent slag mixes. However, these mixtures marginally exceeded the MTO scaling limit of 0.8 $kg/m^{2,}$ and if the hardened air contents are compared it is clear that Mix #6 had better air entrainment than Mixes #9, #12, and #15. The improvement of the hardened air content for these mixes may reduce scaling damage as seen in the recasting of Mix #9. Discrepancies between the fresh air content and hardened air content will be discussed later.

Mix #9 (65%HA 35% SG100 0.42w/cm) was recast to determine reproducibility, to compare one-dimensional freezing cycles (using insulated bottom and sides of the slabs), and to add a 14 day drying cycle after the VDOT 28 day accelerated curing cycle. Slabs subjected to one-dimensional freezing experienced significantly less scaling than all other slabs at 50 cycles.

Extremely little scaling was observed and thawing cycles were noted to be longer by about two hours. By touch, the frozen surface was observed to be harder and stiffer than the frozen solution surface of other slabs. It is likely that the insulation provides lower temperature gradients reducing thermal shock, and therefore reduces scaling. One-dimensional freezing could also reduce hydraulic pressures as the concrete's pores below the surface may remain frozen and not be subjected to the continuous freeze-thaw cycling. However, further evaluation of one-dimensional freezing needs to be performed because the air content in the recasting of Mix #9 was also improved and contributed to improved scaling.

Additionally, a 14 day drying cycle was added after the VDOT accelerated curing regime and before saturating with salt solution for 7 days prior to initiating freezing cycles. It was hypothesized that during curing, slabs subjected to VDOT accelerated curing regime become more saturated, since they are submerged in $Ca(OH)_2$ solution and cured for 21days at 38°C (100°F). This saturation increases scaling when cycling commences, therefore a 14 day drying cycle was added to mimic the BNQ and ASTM conditioning procedures. The slabs were exposed to the VDOT accelerated curing and then to a 14 day drying period experienced significantly less scaling than those without a drying cycle by about 50 percent and met the MTO scaling limits. Part of the improvement in scaling can also be attributed to the better air entrainment obtained in the recasting of Mix #9. Battaglia et al. (2010) emphasized that even when carbonation is controlled and extended curing performed by submerging the specimens, extensive scaling was still observed. This was attributed to the saturation of the specimens prior to scaling. Eliminating the immersion saturation process and adding a 14 day drying period improves scaling performance, as evidenced in this study.

50% Slag Mixtures

The slabs exposed to the ASTM preconditioning cycle performed the best and in some cases met MTO mass loss scaling requirements as in Mixes #7 and #16, but not the lower BNQ scaling mass loss limit. It was observed that in general, the slabs exposed to the BNQ and VDOT curing/conditioning methods experienced low levels of scaling during the initial 15 cycles, at which point the slab surfaces deteriorated rapidly until termination of testing. It is possible that the 4 percent $CaCl_2$ solution used in the ASTM test (as found to be the pessimum concentration by Verbeck and Klieger 1957) is not as detrimental to the concrete surface at high levels of slag content, compared to the 3 percent NaCl solution used in the BNQ and VDOT testing. This was confirmed by Valenza II and Scherer (2007a) who suggested that a pessimum concentration exists at approximately 3 percent, independent of the solute used. Through the application of hand pressure it was observed that the frozen surfaces of slabs with $CaCl_2$ solution were less stiff than those ponded with NaCl solution. Therefore, it is possible that the ice bond with the concrete surface when $CaCl_2$ solution is used is weaker than the bond formed using NaCl. As the ice cracks, a lower stress is transferred to the surface resulting in less scaling. This theory may be supported by the differential in scaling observed by the VDOT and BNQ methods where large quantities of small flakes <5 mm in diameter were collected as residue, and smaller quantities of larger flakes approximately 15 mm diameter were collected from slabs exposed to the ASTM procedure. This phenomenon was consistent for all mixes tested and may be observed in the scaled surfaces of the slabs in Figures 3 and 4 where the former was exposed to ASTM and the latter to the BNQ with VDOT accelerated curing test procedure.

Figure 3. ASTM C672 exposure for Mix #13 (50%LA 50%SG120 0.42w/c) at 25 cycles where coarser sized pieces of scale were observed

Figure 4. BNQ (VDOT curing) exposure for Mix #13 50%LA 50%SG120 0.42w/c at 25 cycles where finer sized pieces of scale were observed

Additionally, eight instead of six slabs were cast for Mix #16 (50%HA 50SG120 0.42w/cm) to test the adequacy of adding the geotextile layer to the lower side of the BNQ slabs. Based on the data collected, it appears that the presence or absence of the geotextile in the forms (to allow some bleed water to be removed) did not make a difference in the scaling of the slabs as the scaling curves were almost identical. It is important to note that none of the 16 mixes cast experienced significant bleeding, probably due to their low w/cm ratio; therefore, this result is not surprising.

After the 15th freezing cycle, the BNQ and BNQ-VDOT slabs experienced an increased rate of scaling. It is possible this occurred as the cement pores become more saturated after part of the concrete surface was damaged and flaked off. Repeated freeze-thaw cycling could also damage the finer capillary pore structure typically associated with slag mixes, inducing micro cracking and allowing deeper salt penetration.

From the analysis of the above results it appears that the ASTM C672 procedure actually performed better in the evaluation of slag cements especially at high slag contents. However, as slag content increased, typically scaling did as well. Mixtures that performed extremely poorly included Mix #10 and Mix #13, which incorporated LA, HA, Grade 100 and 120 materials. The hardened air content of these mixtures was good at 6.8 percent and 5.6 percent, respectively, and spacing factors were below 0.25 mm. Corresponding Mixes #9 and #12, which incorporated 35 percent slag instead of 50 percent, displayed much better scaling resistance. Therefore, it is difficult to determine a cause for the deterioration besides the increase in slag content. However, Mix #7 and Mix #16, which also had 50 percent slag contents, showed good scaling resistance in the ASTM C672 and poor resistance when exposed to the modified BNQ and VDOT accelerated curing regimes.

The primary factors that reduce deicer scaling appear to be w/cm ratio, air content, and air spacing factor. Adequate air content cannot prevent scaling when slag contents of 50 percent are utilized, even at low air void spacing factors as seen in Mix #10 and Mix #13, but it helps mitigate the effects. The use of LA cement also appears to reduce the amount of deicer scaling. The increase in slag content tended to increase the amount of scaling observed.

Air Void Analysis

The fresh and hardened air content properties of each mix were determined and recorded in Table 10. A cylinder was cut longitudinally in half and then two sections 75 mm x 100 mm (3 in. x 4 in.) were polished, blackened with voids filled, and analyzed for air content and spacing factor. Table 10 shows the average air content and spacing factor of the two sections. An aggregate correction factor of 0.9 percent was applied to fresh air contents.

Table 10. Air properties and slumps of the concrete mixtures

Mixture: Cement Slag w/cm	Air-entraining Admixture (mL/m^3)	Fresh Air Content (%)	Hardened Air Content (%)	Spacing Factor (mm)	Slump (mm)
Mix #1: 100HA 0.42wc	135.7	6.0	4.8	0.247	100
Mix #2: 0.5HA 0.5SG100 0.38wc	78.6	6.2	6.0	0.196	140
Mix #3: 0.5HA 0.5SG120 0.38wc	128.6	6.4	7.0	0.178	130
Mix #4: 100LA 0.42wc	107.7	6.1	3.4	0.278	100
Mix #4 Recast: 100LA 0.42wc	105.7	9.4	10.0	0.122	100
Mix #5: 0.8LA 0.2SG100 0.42wc	85.7	7.5	7.8	0.165	130
Mix #5: 0.8LA 0.2SG100 0.42wc	75.7	6.8	5.1	0.275	120
Mix #6: 0.65LA 0.35SG100 0.42wc	75.7	7.0	6.7	0.253	135
Mix #7: 0.5LA 0.5SG100 0.42wc	92.9	6.1	5.0	0.252	140
Mix #8: 0.8HA 0.2SG100 0.42wc	107.1	6.2	4.5	0.239	120
Mix #9: 0.65HA 0.35SG100	128.6	7.0	4.9	0.283	140
Mix #9: 0.65HA 0.35SG100Recast	126.7	6.4	5.8	0.248	145
Mix #10: 0.5HA 0.5SG100 0.42wc	85.7	7.0	6.8	0.155	150
Mix #11: 0.8LA 0.2SG120 0.42wc	100	7.0	5.9	0.181	100
Mix #12: 0.65LA 0.35SG120	92.9	6.5	5.1	0.264	130
Mix #13: 0.5LA 0.5SG120 0.42wc	90	6.4	5.6	0.228	115
Mix #14: 0.8HA 0.2SG120 0.42wc	113.3	6.2	5.8	0.214	125
Mix #15: 0.65HA 0.35SG120	114.3	6.0	3.2	0.358	140
Mix #16: 0.5HA 0.5SG120 0.42wc	126.7	6.0	4.5	0.230	140
Mix #15: 0.65HA 0.35SG120 0.42wc Recast Micro Air®	106.7	7.0	7.7	0.170	140
Mix #16: 0.5HA 0.5SG120 0.42wc Recast Micro Air®	120	10.5	10.9	0.114	100

It was observed that the use of 100 percent LA cement reduced the required air entrainment dosage by about 20 percent compared to the HA cement. This is expected as an increase in alkali content tends to increase air admixture demand. Increasing Grade 100 slag contents with the LA cement progressively reduced the Vinsol resin demand, while increasing Grade 100 slag contents with HA cement progressively increased the Vinsol resin demand.

Increasing the content of Grade 120 slag when used with the LA cement progressively reduced the Vinsol admixture demand, while increasing content of Grade 120 slag used with HA cement progressively increased the Vinsol admixture demand.

In general, a higher hardened air content improved the scaling resistance regardless of the scaling procedure used. The greatest improvement was observed with the VDOT accelerated curing regime. This supports the previous hypothesis, which suggested that during curing, the slabs subjected to VDOT accelerated curing regime become more saturated, since they are submerged in $Ca(OH)_2$ solution and cured for 21 days at 38°C (100°F). This higher saturation increased scaling when freezing and thawing cycles commenced. However, with improved air content and spacing factor, the hydraulic pressures will be reduced, resulting in less scaling.

While comparing fresh air contents to hardened air contents, it was observed that in some cases the hardened air content was less than the air content obtained from the air pressure meter test even after the 0.9 percent aggregate correction factor was applied. In several cases, this discrepancy was more than 1 percent, which indicates that additional Vinsol resin admixture may need to be added to achieve adequate air content and spacing factor. Others have obtained similar reductions in hardened air properties and increases in AEA demand with an increase in slag content (Deja 2003; Giergiczny et al. 2009).

Comparing the hardened air voids with those of Mixes #15 and #16 using Micro Air® AEA admixture, it was observed that the Vinsol resin admixture produced larger air bubbles. Air-entrained samples from Mix #15, which indicate the difference in air entrainment due to Vinsol resin (Figures 5 and 6) and Micro Air® AEA admixtures (Figures 7 and 8), are presented below. As shown in Table 10, the concrete entrained with Micro Air® had smaller air bubbles, which also appeared to be more closely spaced.

With the use of Micro Air® as an air entraining agent, no increase in the AEA demand was observed, nor a reduction of the hardened air content. Therefore, it appears that Micro Air® AEA admixture performs better than the Vinsol resin when air entraining slag cements. This confirms the findings of Chatterji (2003) who clearly indicated that some air entraining agents perform better than others.

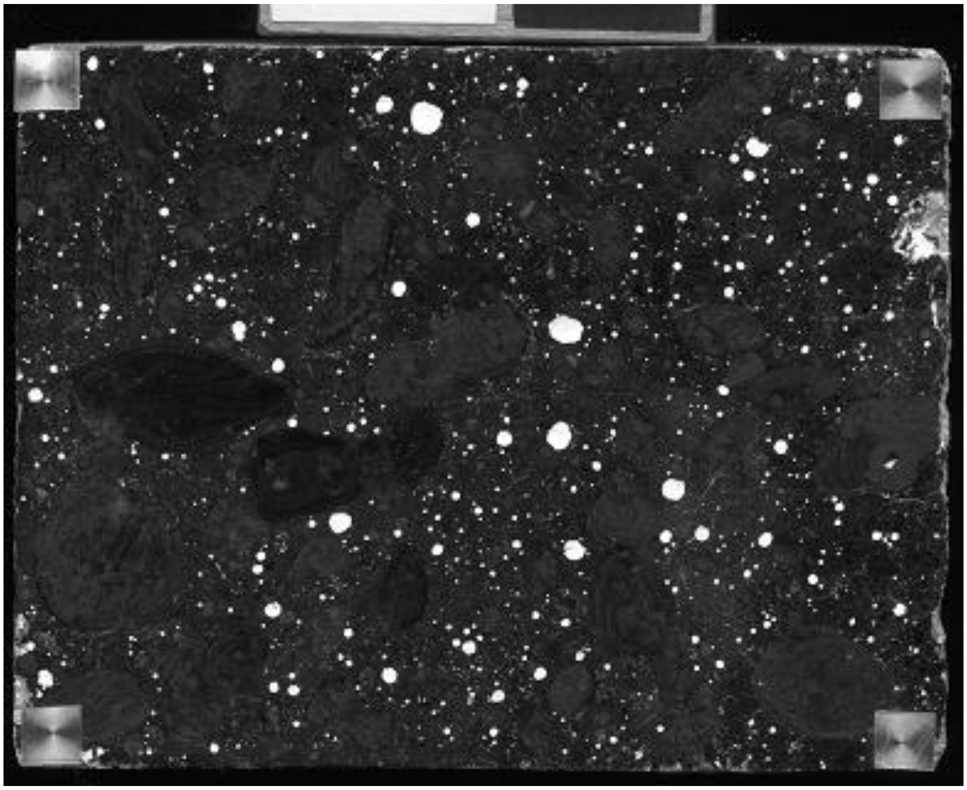

Figure 5. Scanned (top) sample of Mix #15: 0.65HA 0.35SG120 0.42wc with 3.64% air content and 0.267 mm spacing factor, using 114.3 mL/m^3 of Vinsol resin AEA

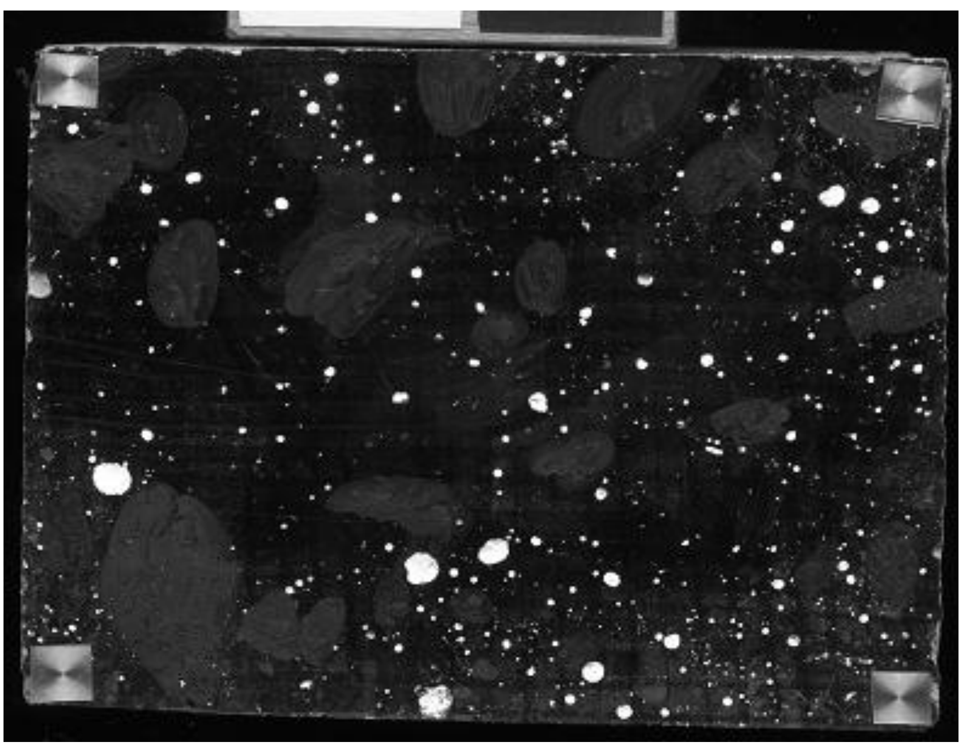

Figure 6. Scanned (bottom) sample of Mix #15: 0.65HA 0.35SG120 0.42wc with 2.79% air content and 0.449 mm spacing factor, using 114.3mL/m^3 of Vinsol resin AEA

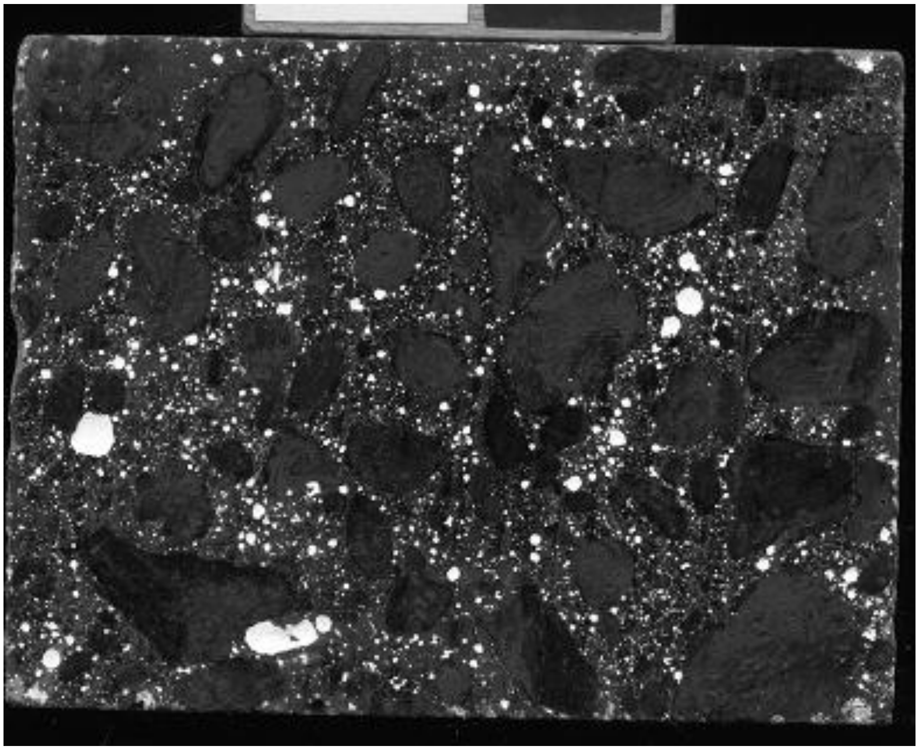

Figure 7. Scanned (top) sample of Mix #15: 0.65HA 0.35SG120 0.42wc with 8.96% air content and 0.133mm spacing factor, using 106.7 mL/m^3 of Micro Air® AEA

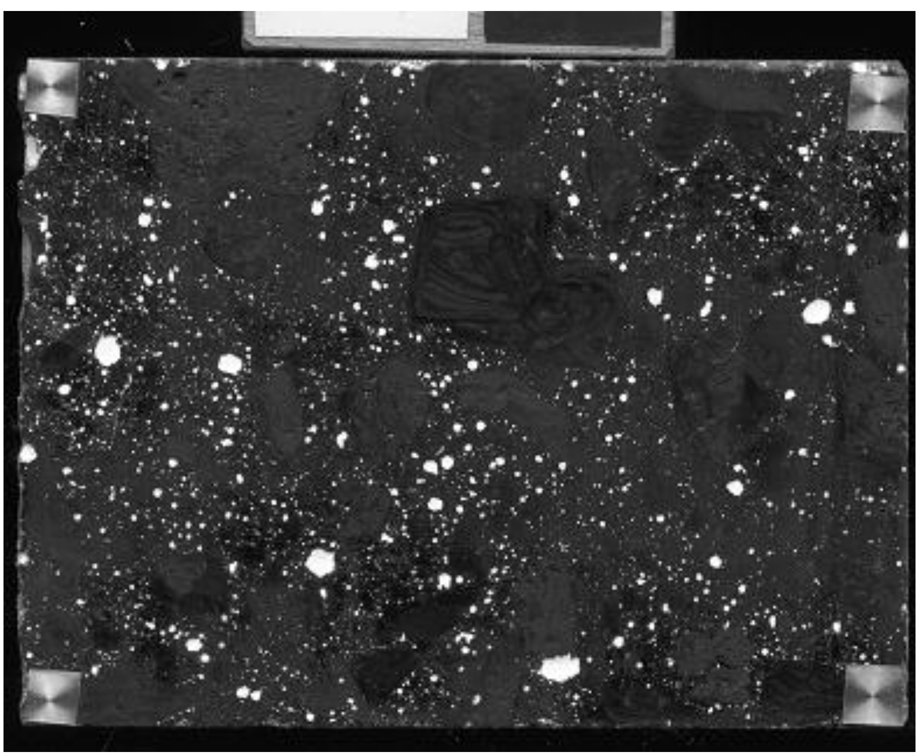

Figure 8. Scanned (bottom) sample of Mix #15: 0.65HA 0.35SG120 0.42wc with 6.35% air content and 0.207 mm spacing factor, using 106.7 mL/m^3 of Micro Air® AEA

Slab Temperatures During Freezing and Thawing Cycles

A scaling slab was instrumented with three thermocouples to measure the freeze-thaw rate: (a) just above the slab surface and submerged in NaCl solution, (b) 3 mm below the slab surface and, (c) at the slab's center of mass 42 mm below the surface. The 1st cycle was performed with the slab positioned on the top shelf of the freezer, the 2nd cycle took place positioned in the middle shelf, and during the 3rd cycle, the slab was placed at the bottom of the freezer. Readings were automatically recorded every five minutes over three 24-hour cycles, and results are displayed in Figures 9, 10, and 11.

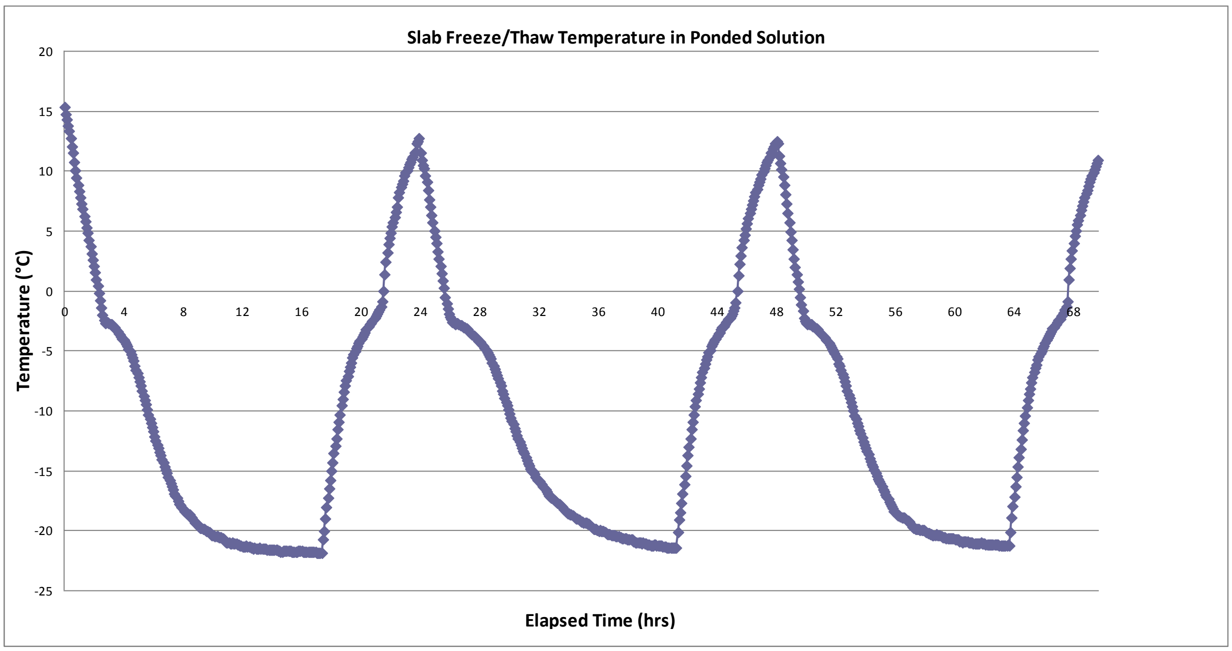

Figure 9. Temperature cycles 3 mm (1/8 in.) above the surface of instrumented slab, immersed in salt solution (3% NaCl)

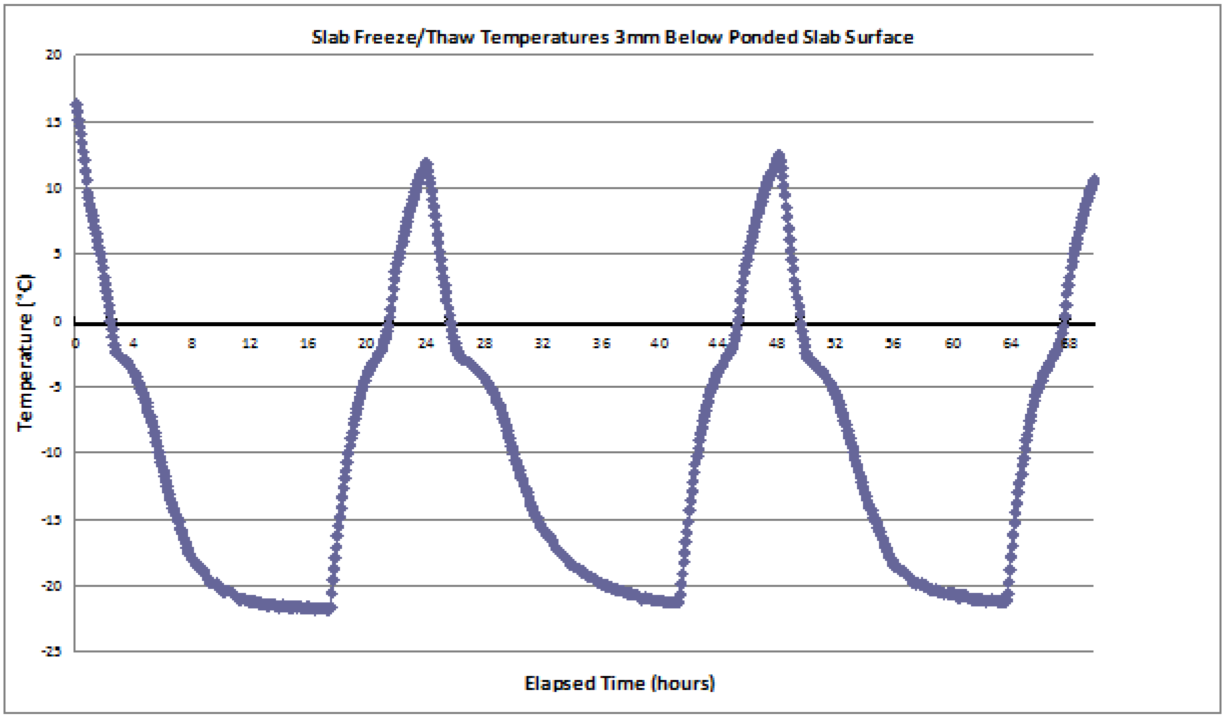

Figure 10. Temperature cycles 3 mm (1/8 in.) below the ponded surface of instrumented slab

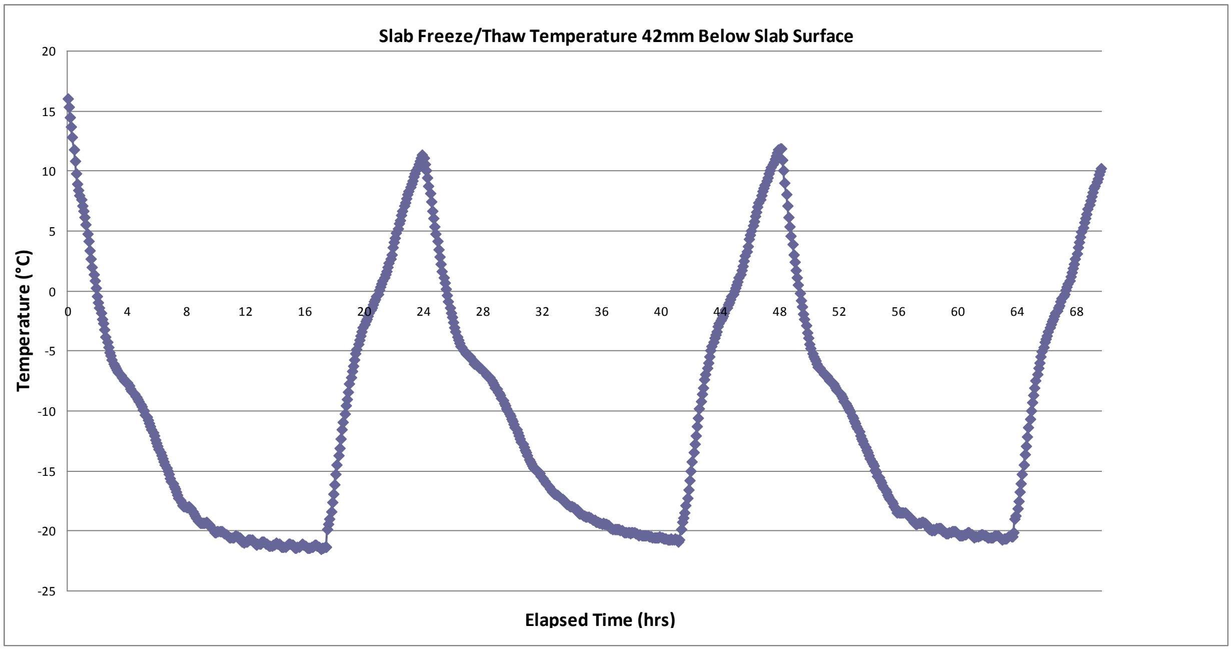

Figure 11. Temperature cycles 42 mm (1.7 in.) below the ponded surface of instrumented slab

Results were somewhat surprising as all three thermocouples appear to register almost identical temperatures during cycling. It was expected that there would be a significant delay in the rate of freezing and thawing of the slab 42 mm below the surface. This was not the case, as differences between the temperatures at different depths were quite minimal. This may be due to concrete's low R-value (thermal resistance). During this test, freezing was not one-dimensional, as all sides of the slab were also exposed to freezing; however, the thermocouples were centered in the 300 mm x 200 mm slab, so this likely did not affect results.

There was no major difference noted in the rate of freezing and thawing regardless of top, middle, or bottom location of the slab within the vertical freezer. This reduces the concern with variability in scaling results as all slabs would be subject to nearly identical freezing and thawing cycles, regardless of position in the upright freezer. From the figures, it can be observed that the initial freezing/thawing point of the NaCl solution takes place at around -3°C (26.5°F). This point is marked by the slower rate of freezing and thawing as water transitions from a solid to liquid state, indicating the change in enthalpy.

From the data, it can be observed that the thawing rate is faster than the freezing rate. Temperatures during cycling are within the ASTM C672 thawing limits and peak at about 12°C (53.5°F); however, the freezing limit (18 ±3°C) is slightly exceeded and drops to about -21.5°C (-6.5°F). This is not anticipated to have a significant impact on scaling as the limit is only exceeded by 0.5°C (2.5°F).

Carbonation and Chloride Penetration Tests

Table 11 presents results of the carbonation (phenolphthalein spray) and chloride (silver nitrate spray) penetration tests. While all depths of carbonation were small, it was observed that mixes with higher rates of carbonation also exhibited some of the highest scaling (ex: Mix #10). Carbonation depth appeared to increase with slag content, while chloride penetration depth decreased. Mixes with some of the highest levels of chloride penetration experienced almost no carbonation, such as Mix #4. These mixes also performed well in terms of scaling. Higher carbonation of slag mixtures was also shown by Copuroglu et al. (2006).

It is important to note that mixes that experienced high amounts of scaling would have a reduced (scaled off) concrete surface. This surface was probably carbonated, but not included in the total depth of carbonation. All the carbonation measurements were performed at one date (April 30, 2012) regardless of the casting date (ranging from May 26 to October 28, 2011); therefore, additional carbonation would have taken place after the termination of scaling. However, even though slabs were stored in a dry room temperature environment, they were stacked on top of each other, with the dikes acting as a partial seal and a barrier to CO_2 ingress. This is evidenced by the lack of carbonation in Mix #4, which was the first mix cast (11 months old) and stored in the manner described for over 7 months, following termination of scaling.

Table 11. Average carbonation depth and chloride ion ingress

Average Carbonation Depth and Cl ion Ingress (mm)				
Mix	Average Carbonation Depth (mm) Average Cl Ion Penetration Depth (mm)			
Test Method	ASTM C672	BNQ	VADOT	Age when Evaluated
Mix #1: 100HA 0.42wc	0.5 16	1 14	1 5.5	8 months
Mix #2: 0.5HA 0.5SG100 0.38wc	1 4	0.5 4	0.75 2.5	6 months
Mix #3: 0.5HA 0.5SG120 0.38wc	0.5 5	0.5 3.5	0.75 1.5	6 months
Mix #4: 100LA 0.42wc	0.5 15	0.75 16	0.5 14	11 months
Mix #4 Recast: 100LA 0.42wc	1 15.5	0.5 16.5	0.5 15.5	6 months
Mix #5: 0.8LA 0.2SG100 0.42wc	1.25 10	0.5 11	0.5 8	6.5 months
Mix #5 Recast: 0.8LA 0.2SG100 0.42wc	0.5 11	0.5 12.5	0.5 7.5	6.5 months
Mix #6: 0.65LA 0.35SG100 0.42wc	0.75 10	0.75 7.5	0.75 4.5	6.5 months
Mix #7: 0.5LA 0.5SG100 0.42wc	2 3.5	2.5 4	0.75 3.25	8.5 months
Mix #8: 0.8HA 0.2SG100 0.42wc	2 9	0.75 7.5	0.75 5.5	8 months
Mix #9: 0.65HA 0.35SG100	0.75 9.5	1.5 6.5	1 5	8 months
Mix #9 Recast: 0.65HA 0.35SG100	0.75 7	N/A	0.5 3.5	7 months
Mix #10: 0.5HA 0.5SG100 0.42wc	5 8	6 7	4.5 4	10 months
Mix #11: 0.8LA 0.2SG120 0.42wc	0.5 9	2 9	2.25 8.5	11 months
Mix #12: 0.65LA 0.35SG120 0.42wc	1.25 10.5	3.5 10	0.5 5.5	11 months
Mix #13: 0.5LA 0.5SG120 0.42wc	0.75 6	1.5 7	2 3	11 months

CONCLUSIONS AND RECOMMENDATIONS

Summary of Work Performed

The primary objectives of this experimental program were to evaluate three different test methods for deicer scaling resistance of concrete. The recently proposed ASTM WK 9367 (modified BNQ) test method was evaluated, with and without the VDOT accelerated curing cycle, and compared to the ASTM C672 test method using air-entrained concrete mixtures made with both low and high alkali cements, with ASTM C989 Grades 100 and 120 slag cements at replacement levels of 0, 20, 35, and 50 percent. The effects of one-dimensional freezing, the addition of a drying period after the VDOT curing cycle, and placement of geotextile at the bottom of the slab to allow for bleed water absorption were also examined. A total of 16 different concrete mixtures were cast. Two air-entraining agents, a Vinsol resin and Micro Air®, were used to study the effect of air entrainment and spacing factor. After mixing, the slump and air content were measured. Additionally, rapid chloride permeability, compressive strength, and hardened air-void analysis tests were performed. In this chapter the conclusions of the experimental research are presented and recommendations are made for further research in the assessment of deicer scaling.

Conclusions

(i) It was observed that no direct relationship exists between compressive strength and deicer scaling. Although concrete with higher compressive strength is less likely to scale when exposed to de-icing salts, the w/cm, slab curing, conditioning, slag content and air content combined with the spacing factor are far more important.

(ii) It may be concluded from the ASTM C1202 results that no direct relationship exists between charge passed through a concrete specimen and its resistance to salt scaling. In fact, the mixes with the 50 percent slag content exhibited some of the highest scaling even though they had some of the lowest coulomb values. However, when the w/cm of these mixes was reduced to 0.38 from 0.42, they not only displayed the best scaling resistance in all three scaling procedures, but they also had the lowest charges passed. In contrast, some of the mixes with the highest RCPT values actually performed very well in salt scaling. Therefore, it is concluded that the scaling resistance of slag mixes is more sensitive to a reduction in w/cm than from obtaining lower C1202 values.

(iii) After conducting deicer scaling tests according to both the ASTM C672 and the draft ASTM WK 9367 (modified BNQ) test, it was consistently observed that a direct relationship exists between an increase in slag content and scaling of the concrete surface. In most cases, the specimens exposed to the ASTM C672 testing procedure showed some of the lowest scaling levels, especially with increased slag content. In addition, the bulk of the scaling occurred during the first 15 cycles in the ASTM C672 test, at which point it tapered off. In contrast, only small amounts of scaling were exhibited by concrete slabs subjected to the ASTM WK 9367 test, with and without the VDOT accelerated curing regime, during the initial 15 cycles. However, the amount of scaling accelerated after this point was especially evident in concrete slabs with higher slag contents (except at 0.38w/cm) and/or with high air contents.

(iv) It was expected that concrete slabs exposed to the VDOT accelerated curing regime would exhibit less scaling because of the higher maturity achieved, increased curing temperature, and addition of the pre-saturation period. However, unlike previous results (Houehanou et al. (2010); Taylor et al. (2004); Panesar and Chidiac et al. (2008)),where extended curing periods were utilized, slabs exposed to the VDOT accelerated curing regime experienced come of the most severe scaling, when no drying period was included prior to pre-saturation. It is assumed that the concrete surface reached higher levels of saturation due to submerged curing in saturated lime water during the curing cycle, and therefore had lower resistance to freeze-thaw cycling.

(v) Addition of a geotextile at the bottom of the slab did not appear to have an effect on scaling performance. This is likely because little bleeding was observed in any of the mixes cast due to their low water-to-cement ratios. The benefits of adding a geotextile may only be realized at higher w/c ratios or with concretes that exhibit more bleeding.

(vi) The use of insulated slabs to obtain one-dimensional freezing drastically reduced scaling. Thawing cycles were about two hours longer under these conditions. It is likely that the insulation provides lower temperature gradients reducing crack formation in the ice and therefore reduces scaling. One-dimensional freezing could also reduce hydraulic pressures due to the slower rates of freezing, which allows more time for water to escape from the capillaries into nearby air voids.

(vii) Air entraining proved to be more difficult using the Vinsol resin air admixture compared to the Micro Air® admixture. It was observed that concretes entrained with Vinsol resin had larger diameter air bubbles with higher spacing factors, while Micro Air® produced smaller diameter air bubbles and lower spacing factors. Achieving an adequate air entrainment of 6 to 7 percent and a good spacing factor reduced deicer scaling, but did not guarantee satisfactory scaling performance.

(viii) The carbonation depth appeared to increase with slag content, while chloride penetration depth decreased. Mixes with some of the highest levels of chloride penetration experienced almost no carbonation, and these mixes also performed well in terms of scaling. It was observed that concrete mixes, which experienced more than 2 mm of carbonation, performed poorly to deicer scaling.

(ix) Finishing of the surface according to ASTM C672 by brushing appeared to have an effect on the early scaling results. Bouzoubaa et al. (2008) and Bilodeau et al. (1994) indicated that premature finishing, which traps bleed water under the surface, may damage the air void system at the concrete surface, making it less durable to salt scaling. This may have been the case in the ASTM C672 test because the concrete surfaces tended to scale during the initial 15 cycles followed by a reduction in the rate of scaling. This indicates that a weaker surface layer existed, which once removed, exposes a stronger layer of hydrated cement less susceptible to scaling.

The opposite effect was experienced with the ASTM WK 9367 and VDOT, in which case slabs were not brush finished.

The mechanisms of deicer scaling are complicated. Experimental data obtained from a variety of test methods confirms the literature indicating that scaling results are dependent primarily on w/cm ratio, curing, conditioning, degree of pore saturation, air content and spacing factor, cement type, and slag content. It has been shown through a variety of prior case studies that ASTM C672 results poorly represent field performance of concrete mixtures with SCMs, providing that correct construction practices were used to place the concretes. However, in this experimental program concrete slabs subjected to ASTM C672 exhibited some of the lowest scaling especially at slag contents greater than 35 percent. These results may be attributed to the difference in solution concentration 4wt% vs. 3wt% and lack of saturation prior to freeze-thaw cycling, unlike samples cured using the version of the VDOT accelerated curing procedure used here. Additional testing should be performed to confirm these results and several recommendations for future work are proposed in the following section.

Recommendations

(i) The effects of one-dimensional freezing should be studied to determine if longer thawing cycles reduce scaling damage. A slab could be instrumented with thermocouples to determine the freezing and thawing profile.

(ii) The effects of different air entraining admixtures must also be examined so that appropriate admixtures may be selected for slag cements. The results obtained from this research program indicate that Micro Air® provides better air entrainment capabilities than a traditional neutralized Vinsol resin. An investigation of the air void system should be performed to determine why an increase in slag content reduces the content of finer air voids. Increasing the specified air content range to 8 ±1% for slag cements may also be beneficial as long as the compressive strength is maintained and chloride penetration kept low at these air levels.

(iii) Adding a 14 day drying period, after the VDOT accelerated curing regime and before the pre-saturation period, appears to reduce scaling and needs to be further evaluated. The accelerated curing regime could also be compared to a 28 day moist curing regime where slabs are not submerged in lime water.

(iv) When casting, finishing should only be performed with a maximum of two wood screed passes in each of the longitudinal and transverse directions. Because of the low levels of bleeding typically exhibited by low w/cm concretes, final finishing is not critical and may be eliminated.

(v) Rinsing the collected scaled material with potable (tap) water needs to be evaluated to dissolve any salt crystals that may have also been collected.

(vi) It is recommended that mass loss be measured every 5 cycles and not after just 5, 10, 15, 25 and 50 cycles. It was observed that salt crystals from the saline solution begin to precipitate around the edges of the slab between the 25th and 50th cycles. At this point, the solution was removed and replaced. No such precipitation was observed when the solution was replaced every 5 cycles. Measuring the mass loss every 5 cycles will also generate a better scaling profile and improved analysis.

(vii) An extended curing regime of 28 days is also recommended, especially for mixtures containing SCMs. Pre-saturation of the slab surfaces with deicer solution is recommended in order to limit the effects of osmosis and absorption.

(viii) Standardizing the visual assessment system for scaling is also recommended so that a visual rating approximately corresponds to a mass loss range. After a thorough analysis of the scaling data, the following ratings and corresponding mass losses were determined to be appropriate, as shown in Table 12.

Table 12. Suggested equivalency chart relating visual scaling to mass loss

Equivalency Chart Relating Visual Scaling to Mass Loss		
Scaling Rating	**Mass Loss Range g/m^2**	**Characteristics of the scaling surface**
0	0 - 50	No significant scaling observed
1	51 - 210	Very slight scaling 3mm (1/8") depth, max, no coarse aggregate visible and no pop-outs present
2	211 - 500	Slight to moderate scaling and/or presence of a few pop-outs
3	501 - 1300	Moderate scaling with some exposed coarse aggregate
4	1301 - 2100	Moderate to severe scaling where the coarse aggregate is clearly exposed and there is scaling of the surface mortar
5	>2100	Severe scaling coarse aggregates are visible over the entire surface

ix) Based on these results, changes to the draft ASTM WK9367 (modified BNQ) test method have been proposed and the test method is provided in Appendix A. This test method incorporates the wood screed finishing, insulated sides of the slabs, and a 7 day pre-saturation period with 3wt% NaCl solution. It does not include use of the geotextile in the bottom of the scaling slabs. For concrete mixtures with SCMs, it also provides an option to use the VDOT accelerated curing regime.

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APPENDIX

Draft July 12, 2012
ALTERNATE TEST METHOD TO ASTM C 672: Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals
WK 9367

Rationale:

Applying a visual rating to the damage incurred in a given sample is subjective. Some authorities have adopted a system of determining the exposed surface mass loss of scaling slabs in order to make the test more objective and this system has been adopted in this method.

The current C672 method has been reported to be more severe than field experience, i.e. concretes, especially those containing pozzolans or slag cement, that fail the test have been observed to perform satisfactorily in the field. Particular concerns, which have been addressed in this replacement method, are:

- Osmotic effects are more severe in samples that have not been presoaked in the salt solution.
- Premature finishing activities have been shown to significantly influence performance.
- Insulating the sides of the scaling slabs will minimize the influence of lateral freezing.
- Additional curing or accelerated curing will allow concrete containing pozzolans or slag cements to develop their properties sufficiently prior to exposure to freezing cycles.

This method is based on a modification of the ASTM C672 test method developed by Bureau de Normalisation du Quebec (BNQ) NQ 2621-900. The accelerated curing option is based on the method adopted in ASTM C1202-12.

Standard Test Method for Quantitative Assessment of Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals Using Mass Loss[1]

1. Scope

1.1 This test method covers determination of the resistance to scaling of a horizontal concrete surface exposed to freezing-and-thawing cycles in the presence of deicing chemicals. It is intended for use in evaluating this surface resistance both qualitatively by visual examination and quantitatively using scaling mass loss.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:[2]

C125 Terminology Relating to Concrete and Concrete Aggregates
C143/C143M Test Method for Slump of Hydraulic Cement Concrete
C156 Test Method for Water Retention by Concrete Curing Materials
C173/C173M Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method
C192/C192M Practice for Making and Curing Concrete Test Specimens in the Laboratory
C231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method
C233 Test Method for Air-Entraining Admixtures for Concrete
C511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
C672/C672M Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals

2.2 Other Standards

BNQ NQ 2621-900, (2002), "Détermination de la Résistance à l'écaillage du Béton soumis à des Cycles de Gel-Dégel en contact avec des Sels Fondants" (Determination of the Scaling Resistance of Concrete Surfaces Exposed to Freezing-and-Thawing Cycles in

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[1]This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.67 on Resistance of Concrete to Its Environment.

[2]For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

the Presence of Deicing Chemicals), Bureau de Normalisation du Québec, Annexe A, pp. 19-22.

3. Terminology

3.1 Definitions—For definitions of terms used in this test method, refer to Terminology C125.
3.2 Definitions of Terms Specific to This Standard:
3.2.1 scaling – the loss of surface from a concrete element due to the expansive crystallization of salts due to evaporation and / or freezing of salt solutions in contact with the concrete surface

4. Significance and Use

4.1 This test method can be used to evaluate the effect of mixture proportioning, surface treatment, curing, or other variables on resistance to deicer scaling and is based on a modification of the BNQ NQ 2621-900 test method.
4.2 This test method is not intended to be used in determining the durability of aggregates.
4.3 No relationship has been established between the deicer scaling resistance of specimens cut from hardened concrete and specimens prepared in the laboratory.
4.4 This test method is an alternative to test method C672 for deicer scaling resistance that is thought to better relate to field performance of concretes containing supplementary cementing materials (1 ,2).

5. Apparatus

5.1 *Freezing and Thawing Apparatus*—The apparatus shall consist of a cold room or a cabinet in which the ambient air goes down to –18 ± 3°C [0 ± 5°F] during the freezing period and goes no higher than 25 ± 3°C [77 ± 5°F] during the thawing period at 23 ± 2.0°C [73.5 ± 3.5°F] and a relative humidity of 45 to 55 %

The capacity of the apparatus shall be so that the temperature at the interface of the deicing saline solution and the concrete specimen can be maintained to –18 ± 3°C [0 ± 5°F] during the last 7 hours of the freezing period (see 5.1) and be greater or equal to 5°C [40°F] at the end of the thawing period. The duration of the freezing period for which the temperature at the interface of the saline solution and the concrete specimen is at –18 ± 3°C [0 ± 5°F] shall not exceed 12 hours (Note 1). These requirements shall be met throughout the chamber.

The use of two distinct apparatus for the periods of freezing and thawing is permitted if the two conditions alternate in a continuous manner; that is there is no delay between the end of a given period and the beginning of the following period.

Note 1- On weekends or during other delays in temperature cycling, maintain the specimens in a frozen condition at –18 ± 3°C. Covering the slab surfaces with a cover plate or cling film may be useful to minimize sublimation of ponded ice.

5.2 *Oven*—An oven of sufficient size, capable of maintaining a uniform temperature of 110 ± 5°C [230 ± 9°F].

5.3 *Molds*, of the proper size for the test specimens to be used, and conforming to the requirements of Practice C 192/C 192M.

5.4 *Extruded Rigid Polystyrene Foam Insulation Sheet*, 25 mm [1in.] thick. Cut in strips 125 ± 5 mm [5 ± ¼ in.] high. Each slab requires two strips of the same length as the length of the scaling slab and two strips of 50 ± 5 mm [2 ± ¼ in.] longer than the width of the scaling slab.

5.5 Silicone Sealant---- Suitable for bonding foam insulation sheets to sides of concrete slabs and to each other.

5.6 *Tamping Rod*, conforming to the requirements of Test Method C143/C143M.

5.7 *Strik-eoff Board*— Wood strike-off and screed board approximately 40mm x 85mm [1.5 in. x 3.5 in.] in cross-section and of sufficient length to extend over the sides of the mold at all times.

5.8 *Slump Cone*, conforming to the requirements of Test Method C143/C143M.

5.9 *Air Meter*, conforming to the requirements of Test Method C173/C173M or Test Method C231.

5.10 *Scales*, conforming to the requirements of Practice C192/C192M.

5.11 *Concrete Mixer*, conforming to the requirements of Practice C192/C192M.

6. Proportioning and Mixing

6.1 *Proportioning*— The air content, cement factor, slump, water-to-cement ratio, and other characteristics of the concrete and its ingredients shall be those appropriate for the purposes for which the tests are to be made, or represent concrete intended for use in construction (Note 2).

NOTE 2- Concrete with the following characteristics has been found useful for the purposes for which this test method is generally used: (a) air content, 6 ± 1 % (Note 3) (b) cement content, 335 ± 5 kg/m^3 [565 ± 10 lb/yd^3]; (c) max w/cm of 0.45; and (d) durable aggregate (Note 4) of 25-mm [1-in.] maximum size.

NOTE 3—For additional information pertaining to air-entrained concrete proportions, see Test Method C 233.

NOTE 4—Care should be taken to use an aggregate which has a good performance record in freezing and thawing exposure.

6.2 *Mixing and Testing of Freshly Mixed Concrete*—Machine mix and test in conformance with the applicable provisions of Practice C192/C192M.

7. Specimens

7.1 Specimens shall have an exposed surface area (see 8.5) of at least 0.045 m^2 [72 $in.^2$] and be at least 75 mm [3.0 in.] in depth. At least two duplicate specimens for each combination of variables to be tested shall be made.

7.2 *Fabrication of Specimens*:

7.2.1 Coat the inside surface of the mold with a light coat of mineral oil or a suitable nonreactive release material just prior to fabrication of the specimens.

7.2.2 Fill the mold in one layer. Rod one time for each 1300 mm^2 [2 $in.^2$] of surface, leaving a slight excess of material after the final rodding. Tap the sides of the mold with a rubber mallet to close any voids and spade around the periphery with a flat trowel. Level the surface with several passes of a wood strike-off board.

7.2.3 Immediately after consolidation, strike off and level the surface using the wooden screed board (with the 40mm [1.5 in.] section laid flat on the concrete surface. Proceed in a back-and-forth (horizontal sawing) motion with a ± 50mm [2 in.] amplitude, first lengthwise, then widthwise until the surface is uniform, free of holes and free from any protruding coarse aggregate particles. Avoid excessive finishing by making no more than two passes with the screed in each of the lengthwise and widthwise directions.

7.2.4 Test specimens may also be slabs meeting the exposed test area requirements cut from hardened concrete in a structure. In this case, the specimens shall not be cut or damaged on the surface to be tested and should not be allowed to dry to a moisture condition below that of the structure from which they have been taken. This may be accomplished by wrapping the specimens in some waterproof material or by other suitable means.

8. Curing

8.1 Except where the method of curing is an element of study or is otherwise specified, cover the specimens with a polyethylene sheet immediately after finishing. Do not allow the sheet to contact the concrete surface.

8.2 Remove the specimens from the molds at an age of 24 ± 4 h after addition of water to the mix and place in moist storage as provided for in Specification C511. Do not submerge or expose the specimen to running or dripping water.

8.3 If concretes with differing rates of strength gain are to be compared, maintain the specimens in moist storage until such time as the desired strength or maturity level has been obtained as per one of the options in 8.3.1, 8.3.2, or 8.3.3.

8..3.1 Portland Cement Concretes: Remove the specimens from moist storage at the age of 14 days and store in air for 14 days at 23.0 ± 2.0°C [73.5 ± 3.5°F] and 45 to 55 % relative humidity.

8.3.2.1 Standard curing for concretes containing SCMs: Remove the specimens from moist storage at the age of 28 days and store in air for 14 days at 23.0 ± 2.0°C [73.5 ± 3.5°F] and 45 to 55 % relative humidity.

8.3.2.2 Accelerated curing for concretes containing SCMs: Remove the specimens from moist storage at the age of 7 days and place in sealed containers over water for 21 days at 38.0 ± 2.0°C [100 ± 3.5°F]. At 28 days, remove the specimens from the sealed containers and store in air for 14 days at 23.0 ± 2.0°C [73.5 ± 3.5°F] and 45 to 55 % relative humidity.

8.4 Prior to the initiation of testing and after at least 2 days of air storage to allow adequate drying of the concrete in order to allow adequate curing of the silicone sealant, place the dikes. The dikes are made from 125 mm [5 in.] high strips of 25 mm [1 in.] thick rigid, closed cell polystyrene insulation bonded to the full depth of all four sides of the slabs using silicone sealant (Notes 5 and 6) and protruding 25 mm [1 in.] above the surface of the slab. Seal intersections of the dike to the top surface of the slab with silicone sealant and seal corners of dike.

Note 5 ---- The insulation on the sides of the slabs helps ensure one-dimensional freezing cycles.

Note 6 ---- Failure to apply adequate adhesive or to allow for adequate drying time may result in leakage of the saline solution as the slab surface begins to scale during repeated freezing and thawing cycles.

8.5 After curing and air drying, cover the flat surface of the specimen with approximately 6 mm [1/4 in.] of a solution of sodium chloride and water, having a concentration such that each 100 mL of solution contains 3 g of anhydrous sodium chloride (Note 7). Cover the solution with plastic cling wrap or other suitable means of minimizing evaporation.

8.6 Keep the surface saturated with the deicer solution for seven days (Note 8).

8.7 After the seven day saturation period, initiate the freezing cycles.

NOTE 7—Other curing periods and conditions and other chemical deicers and different concentrations may be used when there is a need to evaluate their specific effect.

NOTE 8—The saturation period with deicer solution reduces osmotic potentials at the onset of freezing.

9. Protective Coatings

9.1 If protective coatings are to be evaluated, apply them in accordance with the manufacturer's recommendations regarding quantity and method of application at the age of 21 days. When a material proposed as dual-purpose curing compound/protective coating is being evaluated, apply it at the proper time of application for curing compounds, as described in Test Method C 156 (Note 7).

NOTE 7—When evaluating penetration-type coatings for application to pavement surfaces subject to traffic wear, it may be desirable to abrade the treated surface of the test specimens by sufficient wire brushing to break any films remaining on the surface after drying.

10. Procedure

10.1 Place specimens in the freezing-and-thawing apparatus. The freezing-and-thawing cycle has a duration of 24 hours divided into a freezing period of 16 ± 1 h followed by a thawing period of 8 ± 1 h. This cycle is repeated 50 times (Note 8).

NOTE 8—Generally, 50 cycles are sufficient to evaluate a surface or surface treatment. However, where comparative tests are being made, it is recommended that the tests be continued beyond the recommended minimum number of cycles if differences have not developed between treated and control specimens.

10.2 Collect and measure the mass of the scaling residue from the surface every 5 cycles, as follow: Rinse the surface of the specimen with new saline solution to remove all flaked off particles. Collect the scaling residue in a pre-weighed filter paper, then dry the filter paper and residue in the oven at 110°C [230°F] until constant mass. Measure the surface of the specimen exposed to the deicer solution to the nearest one square centimeter. Add water between each cycle as necessary to maintain the proper depth of solution.

10.3 After each measuring operation, apply new deicer solution and resume the test until 50 cycles have been completed.

10.4 Either keep specimens frozen during any interruption in the daily cycling or maintain them in a damp condition after removal of solution and flushing of surfaces.

11. Report

11.1 Report the following:

11.1.1 Cement content, water-cementing materials ratio, the kind and amount of any admixture, slump, and air content of mix,

11.1.2 Curing and drying if other than standard,

11.1.3 Type of surface treatment, time of application, and rate of application,

11.1.4 Type of deicer, whether solution or solid, concentration of the solution if used, rate of application, and time of application,

11.1.5 Any other deviations from the standard procedures

11.1.6 Mass loss in kg/m^2 [psf] after every 5 cycles and the cumulative mass loss after 50 cycles.

11.1.7 Visual rating of the surface after every 5 cycles in accordance with the following scale:

Category	Approximate Mass Loss Range g/m^2	Characteristics of the scaling surface
0	0 - 50	No significant scaling observed
1	51 - 210	Very slight scaling 3mm (1/8") depth, max, no coarse aggregate visible and no pop-outs present
2	211 - 500	Slight to moderate scaling and/or presence of a few pop-outs
3	501 - 1300	Moderate scaling with some exposed coarse aggregate
4	1301 - 2100	Moderate to severe scaling where the coarse aggregate is clearly exposed and there is scaling of the surface mortar
5	>2100	Severe scaling coarse aggregates are visible over the entire surface

11.1.8 If the test specimens are cut from hardened concrete, the size, shape, orientation of the specimens in the structure, and any other pertinent information available shall be included in the report, and

11.1.9 Photographs or a word description of the surface, or both, also should be included where possible.

12. Precision and Bias

12.1 A precision statement has still to be prepared for this method. A between-lab repeatability study of the BNQ test was conducted using seven laboratories (3) using four concretes with different cementing materials. The main differences between the inter-laboratory tests and this test are that the slabs were exposed to 56 cycles of freezing and the concrete only received 14 days of moist curing. The results are shown in Table X

Table X: Inter-laboratory test: cumulative scaling residue after 56 cycles of freezing (3)

Concrete Mixture	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Lab. 7	Averag e	Std. Dev.
Mix 1-portland cement control	0.18	0.32	0.37	0.31	0.1	0.34	0.41	0.29	0.11
Mix 2-35% fly ash	0.45	0.17	0.94	0.19	0.23	0.3	0.56	0.34	0.61
Mix 3- 35% slag cement	0.19	0.46	0.13	0.17	0.11	0.23	0.24	0.22	0.12
Mix 4- 25% fly ash	0.29	0.33	0.31	0.16	0.1	0.36	0.73	0.33	0.41

13. Keywords

13.1 concrete-weathering tests; deicing chemicals; freezing and thawing; resistance-frost; resistance-scaling

References

(1) Bouzoubaâ, N., Bilodeau, A., Fournier, B., Hooton, R. D., Gagné, R., and Jolin, M., "Deicing Salt Scaling Resistance of Concrete Incorporating Supplementary Cementing Materials: Laboratory and Field Test Data," *Canadian Journal of Civil Engineering*, Vol. 35, No. 11, 2008, pp. 1261-1275.

(2) Bouzoubaâ, N. Bilodeau, A. Fournier, B. Hooton, R.D. Gagné R. and Jolin, M. "Deicing Salt Scaling Resistance Of Concrete Incorporating Fly Ash and/or Silica Fume: Laboratory and Field Sidewalk Test Data," *Canadian Journal of Civil Engineering*, Vol. 38, 2011, pp. 373-382.

(3) Bouzoubaâ, N. Bilodeau, A. Fournier, B. *R and D Consortium on Deicing Salt Scaling Resistance of Concrete Incorporating supplementary Cementing Materials*, Natural Resources Canada, CANMET Materials Technology Laboratory Report MTL-2004-15 (TR-R) October 2004, 63pp.

Appendix

X.1 Evaluation Criteria for Scaling Mass Loss

X1.1 The Quebec Ministry of Transport uses a failure criteria of 0.5 kg/m^2 mass loss after 56 cycles of freezing.

X.1.2 The Ontario Ministry of Transportation uses a failure criteria of 0.8 kg/m^2 mass loss after 50 cycles of freezing. However, their test method (OPS LS-412 (4)) does not include the 7 day deicer saturation period prior to commencing freezing cycles.

(4) OPS LS-412, "Test Method for Scaling Resistance of Concrete surfaces Exposed to deicing Chemicals," Ontario Provincial Standard, Ministry of Transportation Ontario, Laboratory Testing Manual.