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Criteria for Freeze-Thaw Resistant Concrete Mixtures

Reference

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ABSTRACT

Concrete, especially for improved durability, is typically specified with prescriptive provisions. More recently there has been increasing interest in evolving towards performance-based specifications, both within state highway agencies and industry (FHWA, 2014, "Guide to Developing Performance-Related Specifications, FHWA-RD-98-155, FHWA-RD-98-156, FHWA-RD-98-171, Vol. III, Appendix C," http://www.fhwa.dot.gov/publications/research/ infrastructure/pavements/pccp/pavespec/, last accessed July 28, 2014; ACI Committee 329, Report on Performance-Based Requirements for Concrete, American Concrete Institute, Farmington Hills, 2010; The P2P Initiative, 2014, "National Ready Mixed Concrete Association, Silver Spring," http:// www.nrmca.org/p2p/, last accessed July 28, 2014). One of the challenges in successfully implementing performance-based specifications is the existence and use of reliable test methods and specification criteria that can measure the potential durability of concrete mixtures and provide the expected service life. A state pooled fund research project (TPF-5 (179), 2014, "Evaluation of Test Methods for Permeability (Transport) and Development of Performance Guidelines for Durability," http://www.pooledfund.org/Details/Study/406, last accessed July 28, 2014) was developed with an objective to propose performance criteria for concrete that will be resistant to penetration of chlorides, cycles of freezing and thawing, and sulfate attack. This paper summarized results pertaining to freeze-thaw resistance. Concrete freeze-thaw (F-T) performance was evaluated by ASTM C666/C666M-15 (AASHTO T161-08

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(Standard Method of Test for Resistance of Concrete to Rapid Freezing and Thawing, Standard Specifications for Transportation Materials and Methods of Sampling and Testing, Part 2A: Tests, AASHTO, Washington DC, 2013)) and deicer salt scaling resistance was evaluated by ASTM C672/C672M-12. It was examined whether F-T performance of concrete correlated with results of rapid index tests for fluid transport characteristics of concrete. These tests included the rapid chloride permeability, absorption, and initial and secondary sorptivity. The impact of degree of saturation on the F-T resistance of concrete was also explored. Criteria for F-T resistant concrete mixtures depending on type of exposure were suggested.

Keywords

freeze-thaw resistance, degree of saturation, performance-based specifications

Nomenclature

DOS_i = Degree of saturation of concrete at a given instant

- $DOS_{mc} = Degree$ of saturation of concrete at the end of moist curing
- $\mbox{DOS}_{\mbox{cr}} = \mbox{Critical}$ degree of saturation of concrete beyond which freeze-thaw damage can occur
 - M_m = Absorption capacity of a concrete specimen expressed as its dry mass. It is the measured absorption of water by the specimen subjected to a vacuum and is assumed to represent 100 % degree of saturation
 - RDM = Relative dynamic modulus of elasticity of concrete measured in accordance with ASTM C215-14 [31] used in ASTM C666 [10]

Introduction

Exposure to cycles of freezing and thawing (F-T) is a major cause of concrete deterioration when the in-place concrete does not have the required characteristics for this service condition. Water expands by about 9 % as it cools to become ice. Based on this, it is generally accepted that when the water in a capillary pore exceeds 91.7 % of the volume of the pore, hydraulic pressure results as freezing progresses [1], and this can generate tensile stresses that exceed the tensile strength and cause cracking. Deterioration due to exposure to freezing and thawing cycles on exposed concrete surfaces is called scaling. Scaling manifests itself as loss of paste and in severe cases can result in raveling of the underlying coarse aggregates. Scaling is exacerbated in the presence of deicing salts as this tends to increase water absorption and retention [2]. In this report, the degree of saturation is defined as the ratio of the actual volume of absorbed water to the total volume of water that can be absorbed by the concrete (also referred as absorption capacity). It has been suggested that when the saturation level of concrete exceeds a critical degree of saturation, DOS_{cr} , freeze-thaw damage can occur [3–6]. Fagerlund [7] proposed that, with time, the air voids fill with water during environmental exposure, and when the saturation of concrete exceeds DOS_{cr} , a single freezing cycle results in tensile stresses that exceed concrete tensile strength causing damage.

Resistance to damage from cycles of freezing and thawing is provided by entrained air. The presence of multiple well-distributed and small air voids relieve pressure generated as water expands and freezes to form ice. The expanding water enters the air voids to prevent pressure build up. In addition to air entrainment a minimum compressive strength of 500 psi is required before concrete is subjected to its first freezing event, and a minimum compressive strength of 3500 psi is required before concrete is subjected to cycles of freezing and thawing [8]. A maximum w/cm is also typically specified.

For buildings, the ACI 318-11 [9] Building Code for Structural Concrete establishes exposure classes for concrete members based on anticipated exposure and has requirements as shown in **Table 1**. Guidance is provided in the commentary to the building code whereby exterior walls, beams, girders, and slabs not in direct contact with soil, can be assigned an exposure class F1 as those members are not subject to snow and ice accumulation; exterior elevated slabs, foundation, or basement walls extending above grade that have snow and ice buildup against them can be assigned an Exposure Class F2 as those members will be subject to snow and ice accumulation; and horizontal members exposed to deicing chemicals such as in parking structures and most transportation structures can be assigned to Exposure Class F3. Concrete members assigned to exposure classes F2 and F3 are more likely to achieve a critical degree of saturation when exposed to F-T.

The objective of this research project was to suggest performance criteria as an alternative to the maximum w/cm requirement in ACI 318-11 and other project specifications. The maximum w/cm limit is invoked as a prescriptive requirement to reduce water penetration and the potential to attain a DOS_{cr} in a portion of the member that results in F-T damage. However, the composition of the cementitious

TABLE 1

ACI 318-11 requirements for concrete exposed to freezing and thawing.

Exposure Class	Max. w/cm	Minimum f'_c , psi (MPa) (psi)	Air Content,%	Limits on SCM
F0 – Concrete not exposed to F-T cycles	N/A	2500 (17)	N/A	N/A
F1 - Concrete exposed to F-T cycles and	0.45	4500 (31)	4.5 ^a	N/A
occasional exposure to moisture				
F2 - Concrete exposed to F-T cycles and	0.45	4500 (31)	6.0 ^a	N/A
in continuous contact with moisture				
F3 - F2+exposed to deicing chemicals	0.45	4500 (31)	6.0 ^a	Table 4.4.2 ^b

^aThese air contents are for ASTM C33 [17] No. 57 1 in. (25 mm) nominal maximum size) aggregate. For different size aggregates the air contents are provided in Table 4.4.1 in ACI 318-11. ^bTable 4.4.2 in ACI 318-11. materials that impacts water penetration is not taken into consideration by the singular w/cm requirement in the specifications.

The study was conducted in 2 phases:

In Phase A, air-entrained concrete mixtures were made at varying w/cm, supplementary cementitious material (SCM) type, and dosages. Concrete F-T performance was measured by ASTM C666/C666M-15 [10] (Procedure A) and scaling resistance by ASTM C672/C672M-12 [11]. Rapid index tests that provide an indication of fluid transport characteristics of concrete were performed, and correlatation of results of these tests to the F-T performance of the concrete mixtures, and thereby develop performance criteria for F-T resistant concrete mixtures, was attempted.

In Phase B, by varying the degree of saturation (DOS) of the specimens, quantifying the critical degree of saturation, DOS_{cr} , that caused F-T failure in ASTM C666 [10] was attempted. It is postulated that mixtures that take longer to reach DOS_{cr} are likely to perform better when exposed to cycles of freezing and thawing. The time to attain DOS_{cr} for concrete continuously exposed to moisture (ACI 318 exposure classes F2 and F3) depends on the concrete's DOS at any instant (DOS_i), mixture sorptivity (ASTM C1585-13 [12]), and absorption capacity (M_m). On this basis, performance criteria for F-T resistant concrete could be developed.

Materials and Mixture Proportions (Phase A)

The following materials were used for the concrete mixtures:

- ASTM C150/C150M-16 [13] Type II portland cement (PC) with $C_3A = 8$ %;
- ASTM C618-15 [14] Class F fly ash (FA);
- ASTM C989/C989M-14 [15] slag cement (SL);
- ASTM C1240-15 [16] silica fume (SF);
- ASTM C33/C33M-16 [17] No. Fifty-seven crushed coarse aggregate;
- ASTM C33 [17] natural sand with an FM = 2.88;
- ASTM C260/C260M-10a [18] Air-Entraining admixture, blend of saponified rosin and organic salts;
- ASTM C494/C494M-15a [19] Type F, polycarboxylate based.

Concrete mixtures were proportioned to obtain an air content from 5 to 8 % and the Type F high range water reducing admixture dosage was varied to attain a target slump from 5 to 7 in. (125 to 175 mm). The mixture proportions and test results are provided in **Table 2**. Of the eight mixtures evaluated in Phase A, six mixtures had a w/cm that is higher than that required for F-T exposure in **Table 1**. This was intentional because it was expected that air-entrained mixtures with a w/cm ≤ 0.45 would not experience F-T failure by ASTM C666 [10]. Mixture designations were assigned by the w/cm followed by the SCM type and dosage. For example, 0.49SL25 refers to mixture with a w/cm of 0.49 and 25 % slag cement. Mixtures without SCM use the designation "PC."

Procedures (Phase A)

Concrete mixtures were mixed in a revolving drum laboratory mixer in accordance with ASTM C192/C192M-16 [20]. Fresh concrete was tested for slump (ASTM C143/C143M-15a [21]), temperature (ASTM C1064/C1604M-12 [22]), air content

TABLE 2

Yield adjusted mixture composition and test results (phase A).

	Mixture Designation	0.57 PC	0.50 PC	0.50 FA20	0.50 SL30	0.50 SL25SF5	0.60 SL25SF5	0.45 PC	0.45 SL30
Type Informent, blyd²506539442385385383592414Slag, blyd²16137137137177Fly ab, hlyd²7257225SCM, %070.02030300.030w/m0.770.500.050.000.050.050.050.07STM C640 [18] AEA, or/ord0.790.844.261.170.861.390.741.87STM C145 [21], Slump, in766556.556.5STM C145 [21], Slump, in766556.55.77.6ASTM C18 [24], Carawiert ic M, %7.77.71.48.11.47.71.47.31.47.31.46.5ASTM C18 [24], Carawiert ic M, %7.77.77.87.77.87.	Calculated Batch Quantities								
Sile, Jord163137126177Fy ah. Ibyd ³ 11112525SCM, whyd ³ 00020303030030w(m070.4200.500.500.500.500.500.510.74STM C60 [18] AEA, oz(ev)0.70.540.400.582.590.511.570.74ASTM C64 [19] Typ F, oz(ev)-0.560.400.582.590.511.577.6Freat Concrete Properties0.50.610.576.65.75.77.6STM C64 [13] May, fin67.266.26.56.27.07.07.07.0STM C163 [24], Compensive Call, Terr, fin, Fin, Fin, Fin, Fin, Fin, Fin, Fin, F	Type I/II cement, lb/yd ³	506	539	442	385	385	353	592	414
Fly abs. Itypy ¹ 111Stite Turn, Itypy ² 00020300.00	Slag, lb/yd ³				165	137	126		177
Silica Func, InvolvedUU	Fly ash, lb/yd ³			111					
SCM, %00020303030000w/m0.570.500.500.500.600.600.400.45ASTM C69 [18] Type F, oz/ovt-0.560.400.582.590.511.371.16Freis0.566.00.55.06.27.0 <td< td=""><td>Silica Fume, lb/yd³</td><td></td><td></td><td></td><td></td><td>27</td><td>25</td><td></td><td></td></td<>	Silica Fume, lb/yd ³					27	25		
w/m0.570.500.500.500.500.600.640.64ASTM C260 [18] AEA, or/evt0.790.740.750.740.75ASTM C494 [19] Type F, or/evt-0.500.400.580.590.510.74Fresh Concrete PropertiesASTM C494 [21], Slump, in.76656.56.277.6ASTM C138 [24], Gravineritic Air, %5.77.85.56.06.13.77.8ASTM C138 [24], Gravineritic Air, %5.77.87.85.56.06.13.77.8ASTM C138 [24], Gravineritic Air, %5.77.87.87.07.27.07.07.07.0Hardened Concrete PropertiesASTM C166 [22], Temperature, "F7.57.57.37.07.27.0 <td>SCM, %</td> <td>0</td> <td>0</td> <td>20</td> <td>30</td> <td>30</td> <td>30</td> <td>0</td> <td>30</td>	SCM, %	0	0	20	30	30	30	0	30
ASTM C260 [18] AEA, oz/cvdt0.790.844.261.170.861.390.741.87ASTM C364 [19] Type F, oz/cvdt-0.560.610.582.590.511.371.16Fresh Concrete PropertiesASTM C133 [21], Simm, in.766556.52.277.6ASTM C138 [24], Gravimetric Air, %5.77.85.56.06.15.27.27.4ASTM C138 [24], Chensity, Ib/n ²¹ 148.1145.7147.7148.1147.71.43146.5ASTM C138 [24], Chensity, Ib/n ²¹ 148.1145.717.87.07.07.07.07.0Hardened Concrete PropertiesASTM C139 [25], Compressive Strength, psi28 day4.9184.8954.1015.3766.2494.8445.4275.1629 day4.9184.8951.811.471.241.561.611.2056 dancmal cure1.851.651.811.361.441.741.461.741.3320 dacelerated cure1.853.032.0411.073.225.163.511.14121 dacelerated cure1.853.032.0411.073.235.163.511.14121 dacelerated cure1.85.0141.0471.241.561.611.5222 da celerated cure1.85.0141.0471.241.561.611.521.524 dacelerated cure (In	w/cm	0.57	0.50	0.50	0.50	0.50	0.60	0.45	0.45
ASTM C494 [19] Type F, oz/oví-0.560.400.582.590.511.371.16Fresh Concrete PropertieASTM C431 [21], Shump, in.766556.55.226.0ASTM C331 [23], Air., %67.266.26.56.27.4ASTM C331 [24], Carsyinetric Air., %5.77.85.56.06.15.227.27.4ASTM C136 [24], Cremyenture, *F757.37.07.27.07.07.07.0ASTM C106 [22], Temperature, *F757.37.36.2494.8445.4275.18.1ASTM C106 [22], Compressive Strength, psi28 day4.9184.015.3766.2494.8445.42724 dacelerated cure2.281.811.411.471.241.56<	ASTM C260 [18] AEA, oz/cwt	0.79	0.84	4.26	1.17	0.86	1.39	0.74	1.87
Presh Concrete Properties ASTM C143 [21], Shump, in. 7 6 6 5 5.5 5.25 6 ASTM C123 [23], Air, % 6 7.2 6 6.2 6.5 6.2 7.2 7.4 ASTM C138 [24], Gravimetric Air, % 5.7 7.8 5.5 6.0 6.1 5.2 7.4 ASTM C138 [24], Density, Ib/ft ³ 148.1 145.7 147.7 148.1 147.7 147.3 147.3 147.3 146.5 ASTM C138 [24], Compressive Strength, pi ASTM C139 [25], Compressive Strength, pi 5.37 7.3 7.0 7.2 7.0 7.0 7.0 Stad ors (25) [25], Compressive Strength, pi - 5.37 6.249 4.844 5.427 5.182 Water Absorption Test (drying at 122 °F), we chance in masce control or stad	ASTM C494 [19] Type F, oz/cwt	-	0.56	0.40	0.58	2.59	0.51	1.37	1.16
ASTM C143 [21], Slump, in. 7 6 6 5 5 6.5 5.25 6.6 ASTM C138 [24], Caravimetric Air, % 5.7 7.8 5.5 6.0 6.1 5.2 7.2 7.4 ASTM C138 [24], Density, Ibft ¹¹ 148.7 147.7 147.3 147.3 146.5 ASTM C164 [22], Temperature, "F 75 75 73 70 72 70 70 70 Hardened Concrete Properties 5.75 6.249 4.844 5.427 5.182 Star C139 [25], Compressive Strength, pt 4.918 4.895 4.101 5.37 6.249 4.844 5.427 5.182 Vater Absorption Test (drying at 122 "F), we change in masses 1.41 1.47 1.24 1.56 1.61 1.20 56d normal cure 1.85 1.65 1.81 1.36 1.44 1.74 1.76 1.39 ASTM C1202 [27], Rapid Chloride Permea-billty, Coulture 1.65 1.61 1.20 56d normal cure 6.015 0.33 0.63 0.63 0.63 0.63 0.63 0	Fresh Concrete Properties								
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ASTM C138 [24], Gravimetric Air, %5.77.85.56.06.15.27.27.4ASTM C138 [24], Gravimetric, Nir, Nir, Nir, Nir, Nir, Nir, Nir, Nir	ASTM C231 [23], Air, %	6	7.2	6	6.2	6.5	6.2	7	7.6
ASTM C138 [24], Density, Ib/ft ³ 148.1 145.7 147.7 147.3	ASTM C138 [24], Gravimetric Air, %	5.7	7.8	5.5	6.0	6.1	5.2	7.2	7.4
ASTM C1064 [22], Temperature, °F757573707270707070Hardened Concrete PropertiesASTM C39 [25], Compressive Strength, psi28 days4,9184,8954,1015,3766,2494,8445,4275,182Water Absorption Test (drying at 122 °F), c'haure in mass28 da ceclerated cure2.281.811.411.471.241.561.611.2056d normal cure1.851.651.811.361.441.741.761.39ASTM C1202 [27], Rapid Chloride Permetrik, Coultrie48763634287155446984829571143 ⁻¹¹ 56d normal cure48760.0390.04110.07733251626.00851 ⁻¹⁻¹ 56d normal cure48760.0390.0410.0490.0630.0630.0360.0360.039180 days0.0450.0390.0410.0490.0650.0770.080.0580.0530.0630.0630.0630.0560.0710.580.590.590.510.511.51.	ASTM C138 [24], Density, lb/ft ³	148.1	145.7	147.7	148.1	147.7	147.3	147.3	146.5
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ASTM C39 [25], Compressive Strength, pi 4,918 4,958 4,101 5,376 6,249 4,844 5,427 5,182 Water Absorption Test (drying at 122° F), & charge :: 1,81 1,41 1,47 1,24 1,56 1,61 1,20 56d normal cure 1,85 1,61 1,61 1,61 1,60 1,61 1,60 SGTM C1202 [27], Rapid Chloride Perma-Witter 5015 578 2014 1,077 332 516 2630 851 56d normal cure 4876 503 4287 1,554 469 848 2957 1143 ASTM C157 [26], Length Change, W V 0,37 0,63 0,063 0,036 0,039 0,017 0,053 0,063 0,036 0,039 180 days 0,076 0,057 0,063 0,063 0,076 0,053 0,063 0,039 0,014 0,49 0,053 0,063 0,054 0,059 0,053 0,053 0,053 0,053 0,053 0,053 0,053 0,053 0,053 0,053 0,053 0,053 0,053 0,0	Hardened Concrete Properties								
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28d accelerated cure 2.28 1.81 1.41 1.47 1.24 1.56 1.61 1.20 56d normal cure 1.85 1.65 1.81 1.36 1.44 1.74 1.76 1.39 ASTM C1202 [27], Rapid Chloride Permeability, Coulome 5015 3578 2014 1077 332 516 2630 851 5d accelerated cure 5015 363 4287 1554 469 848 2957 1143 ASTM C157 [26], Length Change, W 0.045 0.039 0.041 0.049 0.053 0.063 0.036 0.039 180 days 0.076 0.059 0.057 0.063 0.065 0.077 0.058 0.058 ASTM C1585 [12], Rate of Water Absorptor (Strptivity, x10 ⁻⁴ mm/s ^{1/2} 6.0/ 3.2 6.2/ 3.5 - 5.0/ 3.1 ASTM C666 [10], Freezing and Thawing Restance - Verture Verture 1.61 .00 1.00 0.01 0.01 0.01 0.02 6.0/ 3.2 9.7 </td <td>Water Absorption Test (drying at 122 ° F).</td> <td>, % change ir</td> <td>n mass</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	Water Absorption Test (drying at 122 ° F).	, % change ir	n mass						
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28d accelerated cure (Initial/Secondary) 17.5/6.7 10.7/4.7 8.7/3.0 - 5.4/1.9 7.1/3.3 5.9/4.1 - 56d normal cure (Initial/Secondary) 13.7/3.6 8.1/3.4 14.1/9.8 - 6.0/3.2 6.2/3.5 - 5.0/3.1 ASTM C666 [10], Freezing and Thawing Resistance - Relative Dynamic Modulus, % and Mass Loss, % RDM @ 300 c 99 100 99 97 100 100 RDM @ 300 c 99 100 99 100 99 97 100 100 RDM @ 300 c 97 98 95 72 96 96 98 97 RDM @ 3,200 c ⁸ 86 91 82 46 93 91 90 97 Mass Loss @ 300 c 0.0 0.1 0.1 0.0 0.5 1.1 0.0 0.1 Mass Loss @ 3,200 c ⁸ 8.2 5.6 9.2 7.8 6.4 8.8 6.2 5.8 ASTM C672 [11], Salt Scaling Resistance - 0 - no scaling: 5 - severe 5.9 3.0 3.0 3.0 2.3 3.8 2.0 0.5 Visual R	ASTM C1585 [12], Rate of Water Absorp	tion (Sorptiv	ity), x10 ⁻⁴ n	nm/s ^{1/2}					
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ASTM C666 [10], Freezing and Thawing Resistance - Relative Dynamic Wodulus, % and Mass Loss, % Mass Loss, % RDM @ 300 c 99 100 99 100 99 97 100 100 RDM @ 300 c 97 98 95 72 96 96 98 97 RDM @ 3,200 c ⁸ 86 91 82 46 93 91 90 97 Mass Loss @ 300 c 0.0 0.1 0.1 0.0 0.5 1.1 0.0 0.1 Mass Loss @ 3,200 c ⁸ 5.0 2.8 5.9 5.9 4.3 6.8 3.7 5.4 Mass Loss @ 3,200 c ⁸ 8.2 5.6 9.2 7.8 6.4 8.8 6.2 5.8 ASTM C672 [11], Salt Scaling Resistance - 0 - no scaling: 5 - severe 3.0 3.0 2.3 3.8 2.0 0.5 Visual Rating (0 - 5) @ 50 cyc 5 3.0 3.0 3.6 4.3 2.8 1.5	56d normal cure (Initial/Secondary)	13.7/3.6	8.1/ 3.4	14.1/9.8	-	6.0/ 3.2	6.2/ 3.5	-	5.0/ 3.1
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RDM @ 3,200 c ³ 86 91 82 46 93 91 90 97 Mass Loss @ 300 c 0.0 0.1 0.1 0.0 0.5 1.1 0.0 0.1 Mass Loss @ 3,200 c ³ 5.0 2.8 5.9 5.9 4.3 6.8 3.7 5.4 Mass Loss @ 3,200 c ³ 8.2 5.6 9.2 7.8 6.4 8.8 6.2 5.8 ASTM C672 [11], Salt Scaling Resistance - 0 - no scaling; 5 - severe Visual Rating (0 - 5) @ 50 cyc 5 3.0 3.0 2.3 3.8 2.0 0.5 Visual Rating (0 - 5) @ 180 cyc 5 ^b 3.5 4.5 3.0 3.6 4.3 2.8 1.5	RDM @ 2,500 c	97	98	95	72	96	96	98	97
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Mass Loss @ 2,500 c 5.0 2.8 5.9 5.9 4.3 6.8 3.7 5.4 Mass Loss @ 3,200 c ^a 8.2 5.6 9.2 7.8 6.4 8.8 6.2 5.8 ASTM C672 [11], Salt Scaling Resistance - 0 - no scaling; 5 - severe - 5.8 ASTM C672 [11], Salt Scaling Resistance - 0 - no scaling; 5 - severe -	Mass Loss @ 300 c	0.0	0.1	0.1	0.0	0.5	1.1	0.0	0.1
Mass Loss @ 3,200 c ^a 8.2 5.6 9.2 7.8 6.4 8.8 6.2 5.8 ASTM C672 [11], Salt Scaling Resistance - 0 - no scaling; 5 - severe -	Mass Loss @ 2,500 c	5.0	2.8	5.9	5.9	4.3	6.8	3.7	5.4
ASTM C672 [11], Salt Scaling Resistance - 0 - no scaling; 5 - severe Visual Rating (0 - 5) @ 50 cyc 5 3.0 3.0 2.3 3.8 2.0 0.5 Visual Rating (0 - 5) @ 180 cyc 5 ^b 3.5 4.5 3.0 3.6 4.3 2.8 1.5	Mass Loss @ 3,200 c ^a	8.2	5.6	9.2	7.8	6.4	8.8	6.2	5.8
Visual Rating (0 - 5) @ 50 cyc 5 3.0 3.0 3.0 2.3 3.8 2.0 0.5 Visual Rating (0 - 5) @ 180 cyc 5 ^b 3.5 4.5 3.0 3.6 4.3 2.8 1.5	ASTM C672 [11], Salt Scaling Resistance	- 0 – no scali	ng; 5 - severe	2					
Visual Rating (0 – 5) @ 180 cyc 5 ^b 3.5 4.5 3.0 3.6 4.3 2.8 1.5	Visual Rating (0 – 5) @ 50 cyc	5	3.0	3.0	3.0	2.3	3.8	2.0	0.5
	Visual Rating (0 – 5) @ 180 cyc	5 ^b	3.5	4.5	3.0	3.6	4.3	2.8	1.5

++ Terminated at 75 cycles

*** All F-T specimens were taken out of the F-T chamber at about 3120 F-T cycles and kept in the moist room (to enhance saturation and thereby accelerate potential for F-T damage) for 90 days prior to the final F-T exposures for 80 cycles on one specimen only. Inch-lb to Metric Conversion:

 $1 \text{ lb/yd}^3 = 0.5933 \text{ kg/m}^3$

1 oz/cwt. = 65.21 ml/100 kg 1 in. = 25.4 mm

 $1 \text{ lb/ft}^3 = 16.0185 \text{ kg/m}^3$

1 psi = 0.00689 MPa

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by the pressure method (ASTM C231/C231M-14 [23]), and density (ASTM C138/C138M-16 [24]). The gravimetric air content was also calculated in accordance with ASTM C138.

Two types of curing of the specimens followed:

- Standard curing when specimens were stored in a moist room at 73.5 ± 3.5°F (23 ± 2°C) immediately after casting the specimens for the duration prior to testing.
- Accelerated curing when specimens were subjected to seven days of standard curing followed by 21 days of curing in water at 100 ± 3.5°F (38 ± 2°C). This curing was used for specimens of some of the rapid index tests as indicated.

Tests on hardened concrete included compressive strength (ASTM C39/C39M-16 [25]) measured on two 4 by 8 in. (100 by 200 mm) cylindrical specimens standard cured to an age of 28 days and length change (ASTM C157/C157M-08(2014)e1 [26]) on three 3 by 3 by 11 (1/4 in.; 75 by 75 by 285 mm) prisms, with seven days standard curing followed by up to 180 days of air drying in a $73 \pm 3^{\circ}$ F ($23 \pm 2^{\circ}$ C), 50 % relative humidity environment. This is a variation of ASTM C157 [26] that requires 28 days of moist curing prior to drying.

Rapid index tests to measure the transport characteristics of concretes included the rapid chloride permeability test (RCPT) (ASTM C1202-12 [27]/AASHTO T277-07 [28]), absorption, initial and secondary sorptivity (ASTM C1585 [12]), and were conducted at the following ages: after 28 day accelerated curing and after 56 days of standard curing. All the rapid index tests involved casting two 4 by 8 in. (100 by 200 mm) cylindrical specimens. Before the test, the specimens were cut and only the top 2 in. (50 mm) from the finished surface was tested.

ASTM has recently standardized a bulk absorption test, ASTM C1757-13 [29]. However, the absorption test used in this study was based on the British Standard BS 1881-122-11 [30]. The specimen was placed in an oven at 122°F (50°C) for 72 ± 2 h followed by cooling for 24 ± 0.5 h in a dry airtight vessel maintained at the laboratory temperature. The specimen was immersed in water for 30 ± 0.5 min and the quantity of water absorbed was determined. Absorption is the % increase in mass from the dry condition.

For the ASTM C666 [10] F-T test, two replicate beam specimens (3 by 4 by 15.5 in.; 75 by 100 by 395 mm) were prepared. Procedure A that involves freezing and thawing in water was utilized. Specimens were standard cured for 28 days followed by air drying in an environment at $73 \pm 3^{\circ}$ F ($23 \pm 2^{\circ}$ C) and 50 % relative humidity for 28 days. The standard ASTM C666 test requires 14 days of moist curing unless otherwise specified. In this research program, a longer curing period was used to allow for the mixtures containing SCMs to achieve their potential durability properties. This is consistent with longer curing adopted in ASTM C1202 [27] rapid indication of chloride ion penetrability tests. A drying period prior to the F-T exposure was incorporated to simulate the Exposure Class F1. Deterioration of concrete was measured by the relative dynamic modulus of elasticity (RDM) (ASTM C215-14 [31]) and mass change measurements after different number of F-T cycles

For the ASTM C672 [11] deicer salt scaling test, two replicate slab specimens (14 by 8 by 3 in; 350 by 200 by 75 mm) were prepared. Specimens were standard cured for 28 days followed by air drying in a $73 \pm 3^{\circ}$ F ($23 \pm 2^{\circ}$ C), 50 % relative

humidity environment for 28 days. The surface of the specimens was ponded with a 4 % calcium chloride solution and subjected to F-T cycles by placing the slabs in a freezer for 17 h and in laboratory air at 73°F for 7 h. The surface of the specimens was visually rated from 0 to 5 after every 5 cycles: 0 = no scaling and 5 = severe scaling. Ratings were made by four individuals and the average ratings are provided in **Table 2** after 50 and 180 cycles.

Note that ASTM C666 [10] is typically terminated after 300 cycles and ASTM C672 [11] after 50 cycles.

Discussions of the F-T Test Results (Phase A)

Concrete mixtures with RDM greater than 80 % after 300 cycles are considered to have excellent performance to F-T exposure. Even after 3200 F-T cycles, with the exception of Mixture 0.50SL30, all mixtures indicated excellent freeze-thaw performance as indicated by RDM that exceeded 80 %. The test data are consistent between specimens from a given mixture. It is unclear why Mixture 0.50SL30 showed reduced performance, as a very similar mixture (0.45SL30) did not show deterioration with 97 % RDM after 3200 F-T cycles.

Mass loss of specimens tested by ASTM C666 [10] due to surface scaling is plotted as a function of F-T cycles in Fig. 1. After 3200 F-T cycles all mixtures had substantial scaling and the mass loss varied between 5.6 and 9.2 %. After 300 F-T cycles, the mass loss of all mixtures was less than 0.5 % except for the 0.60SL25SF5 mixture; however, there are no limits for mass loss to classify mixtures based on their F-T durability, like there is based on RDM.

The average visual rating of specimens due to deicer salt scaling tested by ASTM C672 [11] varied widely between 0.5 and 5.0 after 50 cycles. Fig. 2 shows a comparison of specimen deterioration of various mixtures and the corresponding visual ratings after 180 cycles.

The 28-day compressive strength of the concrete mixtures varied between 4100 (28) and 6250 (43) psi (MPa) with most of mixtures between 4100 (28) and 4900 (34) psi (MPa). The air contents varied between 6 and 7.6 %. These results suggest that mixtures with acceptable RDM can be made even with w/cm as high as 0.60, as long the air content is above 6 % and specified compressive strength is greater than 3500 psi (24 MPa). It is important to remember that the specimens were introduced





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FIG. 2 Specimens subjected to ASTM C672 [11] salt scaling after 180 F-T cycles.

in the F-T chamber following 28 days of air drying. It is surmised that the air drying process lowered the degree of saturation of the specimens considerably thereby improving the performance of the concrete specimens in ASTM C666 [10].

Correlating the F-T Test Results and Rapid Index Test Results

There was no correlation observed between the rapid index test results and the RDM and mass loss measured in accordance with ASTM C666 [10] after 3200 F-T cycles. Even w/cm, or strength, did not correlate well with the RDM and mass loss. This is due to the considerably good F-T performance of most of the mixtures.

Correlations between visual ratings after 50 cycles tested by ASTM C672 [11] are plotted against rapid index test results, w/cm, and strength in Fig. 3. The rapid index test results, strength, air content or w/cm do not correlate well with the visual rating from ASTM C672 tests even though sorptivity was found to correlate with scaling resistance [32]. A minimum measured 28 day compressive strength of 5100 psi (35 MPa), separates the better performing mixtures with a scaling rating after 50 cycles varying between 0.5 and 3.0 from the mixtures that had greater scaling. These mixtures are 0.45PC, 0.45SL30, 0.50SL30, and 0.50SL25SF5. All mixtures contained at least 6 % air. Increasing the strength requirement to more than 5100 psi (35 MPa) does not better categorize mixtures with an even lower scaling rating as Mixture 0.50SL25SF5 had the highest strength, 6250 psi (43.1 MPa), but had slight to moderate scaling (rating of 2.3). Average 28 day compressive strength of 5100 psi (35 MPa) approximately corresponds to specified 28 day compressive strength of about 4500 psi (31 MPa). If the strength requirement is combined with a 0.45 maximum w/cm requirement, or even if just the 0.45 maximum w/cm requirement was used, then the mixtures that remain have a scaling rating after 50 cycles that vary between 0.5 and 2.0, a narrower range. The w/cm requirement could not be replaced with a suitable rapid index test requirement.

It is generally recognized that ASTM C672 [11] is a severe test and does not correlate well to scaling resistance in field concrete. Surface scaling in field concrete is significantly influenced by the finishing and curing procedures used [33].

FIG. 3 (a)-(f): Correlation between the visual ratings after 50 cycles and fresh air content, w/cm, compressive strength and various rapid index tests.



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Phase A Summary

Concrete members assigned to exposure class F1 are exposed to cycles of freezing and thawing with limited exposure to moisture. These members are not subjected to snow and ice accumulation and are not exposed to deicing salts; hereby, it is anticipated that these members will not be critically saturated to result in deterioration due to F-T. Based on the excellent ASTM C666 [10] F-T performance (acceptable RDM) of Phase A mixtures in this study, even those with w/cm as high as 0.60, it is suggested that the w/cm ratio requirement for concrete assigned to an ACI 318 exposure class of F1 can be as high as 0.60. Based on results in this study, a minimum specified strength requirement of 3500 psi (24 MPa) is considered to be adequate. Concrete mixtures have to be air-entrained.

Members assigned to exposure class F3 are exposed to cycles of freezing and thawing and are frequently exposed to moisture and deicing salts. In this study, ASTM C672 [11] results indicate that a maximum w/cm of 0.45, specified compressive strength of 4500 psi (31 MPa) and air content of 6 % is required for resistance to deicer scaling. However, specifiers should also consider the impact of finishing and curing practices on scaling.

Phase B

The main objective of this phase was to quantify the DOS_{cr} that caused F-T failure in ASTM C666 [10]. If DOS_{cr} is established for a concrete mixture, it follows that mixtures where it takes a longer time to reach DOS_{cr} are likely to perform better when exposed to F-T cycles.

Relation Between DOS, Sorptivity, M_m and F-T Resistance of Concrete

It is suggested that concrete mixtures at low degree of saturation, DOS (drier), low sorptivity and a high absorption capacity, M_m , will require a longer time to attain DOS_{cr} and will therefore perform better when exposed to cycles of freezing and thawing. This is explained as follows based on presumed specimen characteristics in **Table 3**.

Specimen 1 with a certain mass (m_i) and known mass in dry (m_d) and saturated (m_s) conditions (**Table 3**) is exposed to water. With time, the specimen absorbs water and its mass and DOS increases. With continued water absorption, the DOS of the specimen reaches DOS_{cr} (assumed 90 %). For specimen 1 the increase in mass necessary for the specimen to change its saturation level from DOS_i (corresponding to m_i) to DOS_{cr} (corresponding to m_{cr}) can be calculated as 1.5 g.

ASTM C1585 [12] determines the rate of absorption (sorptivity) of water by concrete by measuring the increase in the mass of a specimen as a function of time when only one surface of the specimen is exposed to water. From ASTM C1585 it can be stated that

$$\Delta m = a \times d \times (S \times \sqrt{t} + b) \tag{1}$$

Example to illustrate the relation between DOS, sorptivity, M_m and time to DOS _{cr} .									
Specimen #	m _d , g	<i>m</i> _{<i>i</i>} , <i>g</i>	<i>m_s</i> , <i>g</i>	<i>M_m</i> , %	DOS _i , %	DOS _{cr} , %	m_{cr}, g	$(m_{cr} - m_i), g$	
1	100	103	105	5	60	90	104.5	1.5	
2	100	103	110	10	30	90	109	6	
3	100	106	110	10	60	90	109	3	
4	100	108	110	10	80	90	109	1	

TABLE 3

Evample to illustrate	the relation betwee	n DAS corntivity	M and time to DOS
LATING LU IIIUSLIALE		11DOS, 301DUVILY,	

 $m_d = \text{mass of dry specimen (0 \% saturation)}$

 $m_i =$ mass of specimen at given instant

 $m_s =$ mass of saturated specimen (100 % saturation)

 M_m = Absorption capacity of specimen, i.e., at 100 % saturation, $M_m = (m_s - m_d)/m_d$

 M_i = Moisture content at DOS_i, percent of dry specimen mass

 $DOS_i = Degree \text{ of saturation at a given instant, } DOS_i = (M_i/M_m) = (m_i - m_d)/(m_s - m_d)$

 $DOS_{cr} = Critical degree of saturation$

 $m_{cr} = \text{mass of specimen at DOS}_{cr}, m_{cr} = m_d + (m_s - m_d) \times DOS_{cr}$

where:

 $\Delta m =$ calculated mass increase for the specimen over time,

S = sorptivity of concrete,

t = time,

a = exposed area of the specimen,

d = and density of water, and

b = constant

ASTM C1585 [12] actually defines an initial and secondary sorptivity; however, in this section for simplicity the term sorptivity denotes both initial and secondary sorptivity.

Concrete with a higher sorptivity absorbs water at a faster rate. Thereby, it would require a shorter duration for the specimen's DOS to reach DOS_{cr}.

Specimen 2 is identical to specimen 1 except that it has twice the absorption capacity (M_m) . As a result, the specimen's DOS_i can be calculated as half that of specimen 1 and the increase in mass necessary for the DOS_i to reach DOS_{cr} is now calculated as 6 g, i.e., four times the increase in mass of that of specimen 1. Assuming that specimens 1 and 2 have the same sorptivity, it will require a much longer time for specimen 2 to attain DOS_{cr}. If specimen 2 were to have the same DOS_i as specimen 1 (such as specimen 3 in Table 3) on account of conditioning, it would still need twice the increase in mass necessary for the Specimen DOS_i to reach DOS_{cr} as specimen 1.

However, a specimen with a higher M_m does not always attain a better freezethaw resistance. If specimen 2 were to be at a higher DOS_i than specimen 1 (such as specimen 4 in Table 3), on account of conditioning, it would need only two thirds the increase in mass necessary for the Specimen DOS_i to reach DOS_{cr} as specimen 1! Results from this study discussed later show that mixtures with lower w/cm and SCMs reduce M_m , but also lower the DOS.

The increase in mass necessary for the Specimen DOS_i to reach DOS_{cr} can also be expressed as:

$$m_{cr} - m_i = m_d \times M_m \times (DOS_{cr} - DOS_i)$$
⁽²⁾

Generally, a structure is cured, and at the end of the curing period, its DOS can be assumed to be DOS_{mc} . Since a structure could be exposed to moisture immediately after the end of the curing period $DOS_i = DOS_{mc}$. Clearly, DOS_{mc} should be less than DOS_{cr} or else the structure will fail if it is exposed to F-T cycling immediately after the end of the moist curing period. In general, structures are not immediately exposed to F-T cycles. They undergo wetting and drying, and correspondingly, their DOS will increase and decrease, respectively, depending on their sorptivity and drying rate. DOS at any instant is also inversely proportional to the absorption capacity of the concrete mixture. Cracked specimens will experience a rapid increase in DOS.

It has been reported that exposed concrete in the field can have relative humidity in excess of 80 % at a depth greater than 2 in. from the surface of the concrete [34,35]. However, relative humidity is not a direct measure of DOS [36]. Service life models have been developed that calculate the service life as the time taken for the DOS of the concrete to attain DOS_{cr} when the concrete is exposed to moisture [4,7,37]. DOS measurements in the field can help validate these service life models.

Materials and Mixture Proportions (Phase B)

The same materials used in Phase A were used, except for a different source of coarse aggregate. Four mixtures were selected for this phase of the study – three with portland cement and one containing slag cement. Variables include w/cm (0.65 and 0.45) and air content. Marginal air content was chosen so that the specimens would have a greater potential to fail during the F-T test. The mixture proportions and test results are provided in **Table 4**. The four mixtures were selected to evaluate the effect that changes in intrinsic porosity (w/cm, and presence of slag cement) and air content will have on DOS_{cr} .

Procedures (Phase B)

Concrete mixtures were mixed in a revolving drum laboratory mixer in accordance with ASTM C192/C192M-16 [20]. An ASTM C494 [19] Type F water reducing admixture was used at varying dosage to attain a slump in the range from 4 to 7 in. An ASTM C260 [18] air-entraining agent was used to attain the target air content. Fresh concrete was tested for slump (ASTM C143), temperature (ASTM C1064), air content (ASTM C231), and density (ASTM C138). A total of nine 4 by 8 in. (100 by 200 mm) cylinders and twelve 3 by 3 by 11.25 in. (75 by 75 by 285 mm) beams for the F-T test were made for each mixture. Specimens were moist cured till testing age.

Tests on hardened concrete included compressive strength (ASTM C39 [25]) at an age of 28 and 56 days; measured on two 4 by 8 in. (100 by 200 mm) cylindrical specimens at each age.

Rapid index tests to measure the transport characteristics of concretes included the rapid chloride permeability test (RCPT) (ASTM C1202 [27]), initial and secondary sorptivity (ASTM C1585 [12]) after 56 days. All the rapid index tests involved casting two 4 by 8 in. (100 by 200 mm) cylindrical specimens. Before the test, the specimens were cut and only the top 2 in. (50 mm) from the finished surface was subjected to the test. One of the cylindrical specimens was reserved for hardened air void analysis (ASTM C457/C457M-12 [38]).

TABLE 4

Yield adjusted mixture proportions and test results (phase B).

Mixture Designation	0.65PC-4.0	0.65SL35-4.0	0.45PC-4.0	0.45PC-6.5
Calculated Batch Quantities				
Type I/II cement, lb/yd ³	424	279	596	596
Slag cement, lb/yd ³		150		
SCM, %	0	35	0	0
Coarse Agg. (No.57), lb/yd ³	2019	2046	2038	2036
Fine Aggregate, lb/yd ³	1303	1311	1202	1092
Mixing Water, lb/yd ³	276	279	268	268
w/cm	0.65	0.65	0.45	0.45
ASTM C494 [19] AEA, oz/cwt	0.25	0.25	0.25	0.70
ASTM C494 Type F, oz/cwt	-	1.27	1.91	1.30
Fresh Concrete Properties				
ASTM C143 [21], Slump, in.	5.00	6.25	4.00	6.75
ASTM C231 [23], Pressure Air, %	4.0	4.0	4.1	6.4
ASTM C138 [24], Gravimetric Air, %	5.0	3.7	4.1	6.7
ASTM C138, Density, lb/ft ³	150.1	151.7	153.1	148.9
ASTM C1064 [22], Temperature, °F	78	73	72	75
Hardened Concrete Properties				
ASTM C39 [25], Compressive Strength, psi				
28 days	3,940	3,570	5,740	5,040
56 days	_	3,920	6,280	5,650
77 days	4,430			
ASTM C1202 [27], Rapid Chloride Permeab	ility, Coulombs			
56 days	-	985	3451	3716
77 days	5570			
ASTM C1585 [12], Rate of Water Absorption	n (Sorptivity), x10 ⁻⁴ mr	n/s ^{1/2}		
56d normal cure (Initial/Secondary)	29.4 / 17.9	19.4 / 7.8	14.1 / 7.0	20.8 / 9.9
ASTM C457 [38], Air Void System in Harde	ned Concrete			
Air Content, %	2.5	3.1	3.6	5.6
Spacing Factor, in.	0.0143	0.0129	0.0086	0.0074
Specific Surface, in. ² /in. ³	423	426	613	585
Moisture Condition of ASTM C666 [10] Spe	cimens			
M _m , % of dry mass	4.9	3.9	3.9	5.1
DOS _{mc} , %	93	89	86	77

Inch-lb to Metric Conversion: 1 lb/yd³ = 0.5933 kg/m³ 1 oz/cwt. = 65.21 ml/100 kg 1 in. = 25.4 mm 1 lb/ft³ = 16.0185 kg/m³ 1 psi = 0.00689 MPa 1 in.²/in.³ = 0.03937 mm²/mm³

CONDITIONING OF THE F-T TEST (ASTM C666) SPECIMENS

The ASTM C666 [10] test was conducted on specimens that had been conditioned and maintained at four different target degree of saturation (DOS) levels – 100, 92, 86, and 82 % (two specimens at each DOS) for each of the four concrete

mixtures. In addition, standard cured control specimens that were not controlled to a target DOS were tested. The DOS of the standard-cured ASTM C666 specimens was expected to vary during the test as the specimens were not sealed.

Following 28 days of moist curing, the six prisms were wiped dry to a saturated surface dry condition and the mass was measured. The specimens were dried at 140°F (60°C) for 7 days following which their dry mass (m_d) was measured. The specimens were vacuum saturated (pressure of 0 mm Hg) in accordance with the procedure in ASTM C1202 [27]. The SSD mass of the vacuum saturated specimens was measured after the 18 h water immersion period (m_s). The vacuum saturation process was repeated until the gain in mass due to water absorption by the specimens was less than 2 g (0.05 % of specimen mass). The total mass gain from the dry condition to the vacuum saturated condition is referred to as M_m , representing the absorption of water by the specimens are assumed to have achieved a DOS of 100 %.

In preliminary tests, it was found that drying to 220° F (100° C), instead of 140° F (60° C), prior to vacuum saturation gave similar absorption capacity values of the specimen (M_m). Moreover, it was felt that drying specimens at 220° F (60° C) could cause internal cracking which could impact F-T performance. For these reasons, specimens in this study were conditioned by drying for seven days at 140° F (60° C) followed by vacuum saturation to constant mass.

It is assumed that the vacuum applied to the specimens extracted air from the entrained air void system of the concrete and the subsequent soaking allowed for some or all of the entrained air voids to be filled with water. This DOS, therefore, is considered a higher level of saturation than water filling the capillary porosity of concrete. The saturation of the air void system, however, cannot be isolated from that of the capillary porosity. It is also assumed that during the subsequent drying to a target DOS, the water filled air voids emptied to some extent to allow for the typical functionality of the entrained air void system.

Following the final vacuum saturation phase, four out of the six specimens were removed while the other two specimens were left submerged in water. The four specimens were dried at $73 \pm 3^{\circ}$ F ($23 \pm 2^{\circ}$ C) and 50 % relative humidity. The mass of the specimens was measured every 30 min until the specimens reached an estimated DOS of 86 and 92 %.

When the target DOS was attained, the specimens were wrapped with shrink wrap. The wrapped specimen was double layered with a vacuum bag and sealed using vacuum sealers used for food storage. For 100 % DOS condition, two specimens were sealed in the vacuum saturated condition. Two specimens, representing control specimens, were tested without additional conditioning from the moist cured condition. The degree of saturation of the moist cured specimens (DOS_{mc}) was determined as the average value from the six other specimens for that mixture. For the 82 % DOS condition, two specimens were removed from the moist room. If their DOS_{mc} exceeded 82 %, the specimens were dried at 73 ± 3°F [(23 ± 2°C) and 50 % relative humidity to reach DOS of 82 % and sealed. If their DOS_{mc} was less than 82 %, the specimens were immediately sealed.

In summary, the following were the specimens used:

- Vacuum saturated specimens (two each) at 100, 92, and 86 % DOS
- Moist cured specimens (two each) conditioned to 82 % DOS or below and control specimens at ${\rm DOS}_{\rm mc}$

The specimens conditioned to a target DOS were sealed to ensure that the DOS did not change during the F-T testing. ASTM C666 [10] testing was commenced three days after the specimens were sealed to allow for some moisture redistribution within the specimens. The specimen masses were measured at the end of the three-day period and the DOS value was corrected as in some case there was a slight moisture loss. The control specimens, representing the typical specimen moisture condition used in C666 tests, were not sealed.

Specimens were placed in the F-T machine at the same time at an age of about 56 days. After about every 25 F-T cycles, the specimen seals were removed and the RDM and mass were measured. Changes in mass of sealed specimens were used to estimate the DOS. The specimens were then resealed as before and placed in the F-T machine. Specimens were considered to reach failure when the RDM dropped below 60 % before 300 F-T cycles.

Discussions of the F-T Test Results (Phase B)

The compressive strengths were in line with expectations with the w/cm of the mixtures. The total air contents of the fresh concrete, measured by ASTM C231, were within the tolerance of the target air content. The measured hardened air void content was reasonably consistent with the measured air content of fresh concrete. With the exception of mixture 0.45PC-6.5, all mixtures have an inadequate air void system considering the spacing factor and specific surface of air voids. The air void system is considered to be adequate when the air void spacing factor is equal to or less than 0.008 in. (0.2 mm) and the specific surface is greater than 600 in²/in³ (24 mm²/mm³) [1].

The following observations can be made from **Table 4** results:

- 1. For high w/cm (0.65) and low air content (4 %) mixtures, a 35 % slag cement replacement decreased the M_m from 4.9 to 3.9 %, decreased the DOS_{mc} from 93 to 89 %, and decreased the sorptivity. The RCPT value indicates an expected reduction with the use of slag cement.
- For a PC mixture reducing the w/cm from 0.65 to 0.45 at the same low air content (4 %) decreased the M_m from 4.9 to 3.9 %, decreased the DOS_{mc} from 93 to 86 %, and decreased the sorptivity. The RCPT value indicates an expected reduction for the reduction in w/cm.
- 3. For a PC mixture at a low w/cm of 0.45 increasing the air content from 4.1 to 6.4 % increased the M_m from 3.9 to 5.1 %, decreased the DOS_{mc} from 86 to 77 %, and increased the sorptivity. The RCPT value is similar for these mixtures as expected from the same w/cm.

The results suggest that concrete mixtures with a lower w/cm or use of 35 % slag cement at the same air content of 4 % results in lower absorption capacity, M_m , lower degree of saturation of moist cured specimens, DOS_{mc} , and lower measured sorptivity. It can be surmised that these mixtures have a finer pore structure and this is validated by RCPT test results. It is surmised that the finer pore structure of these

mixtures contributed to the lower observed values for sorptivity, whereas a reduced porosity contributed to the lower absorption capacity, M_m . It is also observed for continuously moist cured mixtures with a lower w/cm that the capillary porosity becomes discontinuous at an earlier age [39]. It is surmised that this contributed to a lower degree of saturation at the end of moist curing (DOS_{mc}) for mixtures at a lower w/cm compared to those at a higher w/cm. Increased entrained air content at the same w/cm of 0.45 increased the porosity, which contributed to a higher absorption capacity, M_m . The addition of discontinuous entrained air bubbles in 0.45 w/cm concrete is unlikely to have an effect on the age at which capillaries become discontinuous for continuously moist-cured concrete. However, the air-entrained concrete had a lower DOS_{mc} as a result of its higher M_m .

ASTM C666 RESULTS AND DISCUSSIONS

Ideally, a sealed specimen should not experience a change in mass (or DOS) during F-T testing. An increase in mass is likely if the specimen seal is compromised during the F-T test, thereby permitting water to be absorbed, and increasing the DOS relative to the target value to which the specimen was conditioned to. When measurements were performed on the specimens, removal of the seals could have resulted in some drying that would reduce the DOS from the target value. Each specimen had a mass of about 4000 g and a change in DOS of 1 % corresponds to mass change of about 2 g. The F-T test results (RDM) are plotted as a function of the number of F-T cycles in **Figs. 4a–7a**. It was noted during the experiment that in some of the test specimens, it was not possible to maintain a constant DOS as the F-T cycles increased. So for those test specimens, the F-T test results (RDM) and the DOS are plotted as a function of the number of F-T cycles in **Figs. 4b–7b**. For all 4 mixtures, the unsealed control specimens had severe scaling on the surface whereas none was observed for the sealed specimens.

Mixture 0.65PC-4.0

Specimens with DOS of 100, 92, and 87 % failed before 300 F-T cycles. The specimens with a lower DOS withstood a greater number of cycles prior to failure. Observing the 87 % DOS specimen (Fig. 4b) shows that the seal appears to have been compromised between 160 and 212 cycles, resulting in an increase in the DOS from 87 to 96 %. Between 212 and 257 cycles, the DOS dropped back to 91 %. RDM





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began to decrease after 160 F-T cycles, with a significant rate of decrease noted after 212 cycles. The specimens at DOS of 78 % showed no reductions in RDM. However, in one of those specimens, the seal had been compromised between 0 and 51 cycles and the DOS increased from 78 to 88 % and stayed around 87 % throughout the test. No reduction in RDM was noted throughout the test. From these data, it is surmised that for the 0.65PC-4.0 mixture DOS_{cr} is between 88 and 91 %. Control specimens failed at around 444 cycles. The DOS of the control specimens was 93 % at the start of F-T test. These specimens were not sealed during the F-T test.

Mixture 0.65SL35-4.0

Specimens with DOS of 100, 92, and 85 % failed before 300 F-T cycles. The specimens with a lower DOS withstood a greater number of cycles. Observing the 85 % DOS specimens (Fig. 5b), it is seen that the DOS increased from 85 to 88 % between 138 and 193 cycles-and stayed above 90 % after that. An initial reduction in RDM was observed at 193 F-T cycles, which progressed to decrease thereafter. The specimens at DOS of 78 % showed no reductions in RDM. From these data it is surmised that for the 0.65SL35-4.0 mixture, DOS_{cr} is about 88 %. The DOS of the control specimens was 89 % at the start of F-T test. The control specimens failed rapidly

0.45PC-4.0 mixture

Specimens with DOS of 99 % failed before 60 F-T cycles. Specimens at DOS of 92, 78, and 77 % did not fail. The acceptable performance of the 92 % DOS specimens



FIG. 6 Mixture 0.45PC-4.0 ASTM C666 [10] results versus number of cycles (a) RDM and (b) RDM, DOS of one of the specimens.

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FIG. 5



was unusual. Observing the 92 % DOS specimens (**Fig. 6b**) shows that even though the DOS started at 92 %, it had decreased to 89 % by 180 cycles. DOS decreased to 85 % by 391 cycles and leveled off. Correspondingly, RDM decreased until about 180 cycles and steadied off suggesting no further F-T deterioration. If RDM had continued to decrease at the same rate, then it should have indicated a failure by 250 cycles. From these data, it is surmised that for the 0.45PC-4.0 mixture, DOS_{cr} is around 90 %. The DOS of the control specimens was 86 % at the start of F-T test and did not show reduction of RDM at 300 F-T cycles. The excellent F-T performance of a marginal air content mixture is not unusual [40].

0.45PC-6.5 mixture

Specimens with DOS of 99 %, 92 % failed before 300 F-T cycles. Observing the 92 % DOS specimens (**Fig. 7b**) shows a reduction of the DOS to 90 % by 98 cycles, remaining constant thereafter. A rapid decrease in RDM is observed through 98 cycles and a much more gradual decrease thereafter. By 147 cycles, the RDM had reached the failure point. It is surmised that for the 0.45PC-6.5 mixture, DOS_{cr} is around 90 %. Specimens at DOS of 86 or 80 % did not show any reduction in RDM. The DOS of the control specimens was 77 % at the start of F-T test and did not show reduction of RDM at 300 F-T cycles.

The RDM for the specimens at a DOS of 92 % after 98 cycles was about 63 %. In comparison, for the 0.45PC-4.0 mixture, the RDM for the specimens at a DOS of 92 % after about 100 cycles can be estimated to be about 75 %. The DOS at that stage was comparable at 90 % for both mixtures. This suggests that at a similar DOS greater than DOS_{cr} , the higher air content of the 0.45PC-6.5 mixture does not really help improve F-T resistance over that of the 0.45PC-4.0 mixture.

Phase B Summary

CRITICAL DOS

When the ASTM C666 [10] test results are analyzed, it appears that there is a critical degree of saturation, DOS_{cr} for failure due to exposure to cycles of freezing and thawing to occur. Failure is defined as RDM < 60 % within 300 F-T cycles. For any mixture, as expected, the lower the specimen's DOS the better the F-T performance. The critical degree of saturation, DOS_{cr} , for F-T failure is around 88 %. This represents the total available porosity in the concrete, including entrained air

FIG. 7 Mixture 0.45PC-6.5 ASTM C666 [10] results versus number of cycles (a) RDM and (b) RDM, DOS of one of the specimens.

voids, and is more than the saturation level of the capillary porosity. This observation appears to be independent of the air content, SCM type/content, and w/cm evaluated in this study. The value of DOS_{cr} is similar to that reported in the literature [6,7].

When the DOS was greater than DOS_{cr} , even air-entrained concrete with a low w/cm resulted in failure due to cycles of freezing and thawing in ASTM C666, as indicated by the RDM. Conversely, when the DOS was lower than DOS_{cr} even concrete with a w/cm of 0.65, a low air content, and low compressive strength did not fail in the freeze-thaw tests. The latter observation is consistent with the observation in Phase A that a maximum w/cm as high as 0.60 may be acceptable for ACI 318-11 exposure class F1.

DEVELOPING F-T RESISTANT CONCRETE MIXTURES

The control specimens of both mixtures with a 0.65 w/cm had significant reductions in RDM and failed in the F-T test. These specimens had $\text{DOS}_{mc} \ge 89$ %. The control specimens of the 0.45PC-4.0 mixture had adequate F-T resistance as measured by ASTM C666 RDM, even though they had a marginal air content and air void spacing factor. These specimens had a DOS_{mc} of 86 %. The ASTM C666 test of 300 F-T cycles takes about 50 days, and for only about 20 % of that time (10 days), the specimens thaw in water (they are frozen for the remainder of the time). Since it is very unlikely for a moist cured specimen to undergo an increase in DOS during the 10 day exposure to below 40°F (5°C) water, it can be argued that as long as $\text{DOS}_{mc} < \text{DOS}_{cr}$, the specimen should not suffer a reduction in RDM during the 300 F-T cycles of the ASTM C666 test, even if the DOS is not controlled by sealing during the F-T cycles.

From **Table 4**, it is clear that increased air content leads to a higher M_m and lowers the DOS at any point (as evidenced by the lower DOS_{mc}), which can extend the time to attain DOS_{cr} . The F-T resistance of the higher entrained air content mixture can be improved further if its sorptivity is also reduced. From **Table 4**, it can be observed that concrete mixtures with a lower w/cm or SCMs have lower sorptivity. These mixtures also reduce the M_m but are expected to have a lower DOS (as evidenced by the lower DOS_{mc}) and therefore the net result is expected to be an increase in the time to attain DOS_{cr} . Another option would be to seal the concrete, which would reduce the sorptivity without affecting M_m . Sealers could be a good choice, particularly for those areas that have a high tendency to become critically saturated. Based on the excellent ASTM C666 performance of the 0.45 w/cm mixtures, a maximum w/cm criterion of 0.45 is suggested. More experimental validation is required to suggest alternative sorptivity requirements. The corresponding 28 day specified strength requirement would be 4500 psi (31 MPa).

For all the mixtures, the specimens at 100 % DOS (that were sealed) showed significant reductions in RDM and failed in the F-T test. There was no surface scaling for these specimens. If scaling occurs primarily due to freezing and thawing of water inside the concrete at the surface, then the sealed specimens should have scaled as there was adequate moisture. It appears that the physical formation of ice on the concrete surface is necessary for surface scaling. A possible explanation is that the freezing and thawing of ice on the concrete surface may be subjecting the concrete surface to restrained tensile forces, which causes scaling. Since the presence of the

TABLE 5

w/cn
0.60
0.45
0.45

Recommended mixture selection criteria for F-T resistance.

^aThese air contents are for ASTM C33 [17] No. 57 (1 in.) aggregate. For different size aggregates the air contents provided in Table 4.4.1 in ACI 318-11 can be used. The air content tolerances are ± 1.5 %.

shrink wrap and double layered vacuum bag prevented that from happening (the ice formed on the bag and not on the concrete surface), scaling did not occur. From a practical point of view, if similar results are demonstrated with a sealer or membrane it could help achieve scale-resistant concrete driveways and sidewalks even with a high w/cm of 0.65 and low compressive strength. However, it is possible that shrink wrap and double layered vacuum bag provides a physical barrier of greater magnitude than any sealer or membrane.

Conclusions

The critical degree of saturation, DOS_{cr} , for F-T failure was found to be around 88 %. When the DOS was greater than DOS_{cr} , even air-entrained concrete with a low w/cm had poor F-T performance.

Concrete mixtures at low DOS (drier), low sorptivity and high absorption capacity require a longer time to attain DOS_{cr} , and will therefore perform better when exposed to cycles of freezing and thawing. Air-entrainment improve the F-T performance by increasing the absorption capacity, and as a result, help attain a lower DOS of the concrete at any given external exposure. The use of low w/cm, and slag cement improved the F-T performance by reducing the sorptivity, and as a result, help attain a lower DOS of the concrete.

Based on the Phase A and B summary, the recommended mixture selction criteria for F-T durability are summarized in **Table 5**.

The effect of higher amounts of SCM on scaling was not studied in this project. Thus, no recommendations are made as to these limits in ACI 318 for concrete with application of deicing chemicals (Exposure Class F3). Alternative criteria for the prescriptive w/cm requirement are $DOS_{mc} < DOS_{cr}$. More experimental validation is required to recommend alternative sorptivity criteria, though. Sealers are recommended particularly for those applications that have a high tendency to become critically saturated.

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