Performance of Calcium Silicate-Based Carbonated Concretes vs. Hydrated Concretes under Freeze-Thaw Environments

First of a Three-Part Series Exploring the Durability of Solidia Concrete™ in a Variety of Testing Environments

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Abstract: Solidia Technologies® has developed a calcium silicate-based cement (CSC), which emits less CO_2 during its production as compared to Ordinary Portland cement (OPC). This CSC is hereafter referred to as Solidia Cement™. Additionally, Solidia Concrete™ made using Solidia Cement cures and hardens through a carbonation process and consumes CO_2 during this process. The total CO_2 footprint associated with cement manufacturing and use can be reduced by up to 70% when Solidia Cement replaces OPC. Although Solidia Concrete can achieve compressive strengths over 10,000 psi, the effect of freezing and thawing exposure and surface scaling in presence of salts need to evaluated. This paper presents results for two types of Solidia Concrete and a reference concrete tested as per ASTM C666 procedure A. The scaling resistance is evaluated as per ASTM C672 for air entrained Solidia Concrete and reference concrete. RDME values for both Solidia Concrete and reference concrete after 300 FT cycles were about 88%. After extended FT exposure (~800 cycles) concrete specimens performed well as RDME values did not reduce further. The scaling resistance after 50 FT cycles was very good for Solidia Concrete and reference concrete.

The Cement Sustainability Initiative of the World Business Council for Sustainable Development set 2050 CO_2 reduction targets for the global cement industry. To accomplish this goal, the use of alternative cementitious materials is becoming more prevalent. Solidia Technologies® has addressed this challenge through the development of a calcium silicate-based cement (CSC), which emits less CO_2 during its production as compared to Ordinary Portland cement (OPC). This CSC is hereafter referred to as Solidia Cement™. Additionally, Solidia Concrete™ made using Solidia Cement cures and hardens through a carbonation process and consumes CO_2 during this process. The total CO_2 footprint associated with cement manufacturing and use can be reduced by up to 70% when Solidia Cement replaces OPC.

Before Solidia Concrete can be used in widespread applications, its durability must be demonstrated. The durability of concrete is generally defined as the ability to withstand damaging effects of the environment without deterioration for a designed duration. The durability of concrete involves resistance to freeze-thaw, corrosion, carbonation, alkali-silica reaction (ASR) and chemical attack from sulfates and chlorides, and many more.

Concrete has the potential to be damaged if it is subjected to freeze-thaw cycles as water expands during freezing. Frost damage-a progressive deterioration process that starts with the surface separation (or scaling) and ends up with extensive cracking of the interior of the element-is a major concern when concrete is used in cold regions. The deterioration proceeds as freezing and thawing cycles are repeated, and the material gradually loses its stiffness and strength. In addition, increasingly irreversible expansion is induced.

Air-entraining agents are recommended for most of the concretes, principally to improve resistance to freeze-thaw cycles when exposed to water and deicing chemicals in cold regions. This paper reports RDME and mass change for Solidia Concrete and reference concretes after 300, and 800 freeze-thaw cycles. Scaling resistance is reported after 50 and 100 cycles for Solidia Concrete.

Materials, Mixture Proportions and Experiments

Materials, Mixture Proportions and Mixing Procedure

Solidia Cement, sand, coarse aggregates and water were weighed as per proportions in Table 1. Coarse aggregates were loaded to the mixer, followed by Solidia Cement and then by sand. The aggregates, cement and sand were mixed for 1 minute to produce a dry mix. While the mixer was rotating, 50% of the water was added to the dry mix to produce a wet mix. All water-reducing admixture was then added to the wet mix, followed by the remaining amount of water. Finally, all air-entraining admixture (AEA) was added to the wet mix. This wet mix was mixed for an additional 3 minutes. The mixer was stopped for one minute to settle the wet mix. Wet mix was mixed for an additional 30 seconds and unloaded into a pan.

The air content and the unit weight of fresh concrete were measured as per ASTM C231. Rectangular concrete beams with the dimensions of 3 in x 4 in x16 in (75 mm x 100 mm x 405 mm) and 4 in x 8 in (100 mm diameter x 200 mm length) cylinders

Ingredients	Solidia Concrete	Reference Concrete
Ordinary Portland Cement		325
Solidia Cement	400	-
Fly ash	0	81
Sand	689	765
Coarse Aggregates	1134	1032
Water	116	170
AEA, ml/kg of cement	3	3
Water reducer, ml/kg of cement	10	0
Water to cementing materials	0.30	0.42
Air Content, %	5.5	6.7

Table 1: The proportions of raw materials for concrete mixtures, kg per cubic meter

were cast in metallic molds in three layers. For the scaling test, concrete slabs of 12 in x 6 in x 3 in (302 mm x 151 mm x 76 mm) were cast in mold in two layers. With the addition of each layer, the mold was vibrated on a table vibrator for 30 seconds. All Solidia Concrete specimens were demolded after 4-5 hours. The demolded concrete specimens were placed in a chamber for CO_2-curing. The CO_2-curing parameters to achieve the designed compressive strength were 60°C and 98% inlet CO_2 concentration for 65 hours.

Comparable reference-concrete specimens were demolded after 24 hours and submerged in lime-saturated water for 28 days to achieve designed compressive strength. Reference concrete contained 20% fly ash as cement replacement which is typical in pavement concretes in Indiana. The water to cement ratio for Solidia Concrete was selected such as to obtain similar porosity and compressive strength as that of reference concrete.

Solidia Concrete specimens were produced at Solidia Technologies' facility in Piscataway, N.J. The freeze thaw resistance test as per ASTM C666 (Procedure A – freezing and thawing in water) and scaling resistance test as per ASTM C672 were performed at Purdue University, West Layette, Ind. The results of these tests were already presented at ACI Fall Convention in 2014.

Procedure to Determine Freeze-Thaw Resistance

Prior to initiating the FT testing both, the CO_2-cured Solidia Concrete specimens and water-cured reference concrete specimens were preconditioned by saturating them in tap water for 48 hours. Initial mass and fundamental transverse frequencies were measured for saturated specimens. Each specimen was then placed in a container with water surrounding it. The containers were placed in the freeze-thaw apparatus. Each freeze-thaw cycle consisted of lowering the specimen temperature from 40° to 0°F and then increasing the temperature back to 40°F over a time duration of about 5 hours. The mass and resonant frequencies were measured for each specimen after the completion of each 30 freeze-thaw cycles. Measurements were done immediately after the completion of the thawing cycle.

Freeze-thaw durability was calculated using the following equations:

$$\text{RDME} = \frac{\text{(Dynamic Modulus of elasticity at Cycles)}}{\text{(Dynamic Modulus of elasticity at 0 Cycles)}} \quad (1)$$

According to ASTM C666, the freeze-thaw test process is continued to 300 cycles or to the point when Durability Factor (DF) drops below 60%, whichever occurs first. As the number of FT cycles are 300, RDME is equal to DF. As Solidia Concrete is a novel concrete, no long-term durability information under FT exposure is available. Therefore, while ASTM C666 requires testing up to 300 cycles, this testing was continued beyond 700 cycles to evaluate the freeze-thaw potential of Solidia Concrete.

Procedure to Determine Scaling Resistance

CO_2-cured Solidia Concrete and water-cured reference concrete specimens were preconditioned in air (23°C, 50% RH) for 14 days. A dam was installed using a waterproof material around the perimeter of the specimens. This dam was designed to expose the top surface area of the specimen to a salt solution consisting of 4% calcium chloride.

For the scaling resistance measurement, the specimen was at freezing temperature (-18°C) for 11 hours and at thawing temperature (23°C) for 11 hours. Water was added to the surface if the solution level dropped below 6 mm during the experiment. After every freeze-thaw cycle, the solution was changed. During measurements, the calcium chloride solution was removed. The surface was flushed with water, pictures were taken to record changes due to exposure to FT action in presence of salt solution, and the mass of each specimen was recorded.

Results and Discussions

Freeze-Thaw Test Results

Mass Changes: The changes in mass and resonant frequencies of all specimens were measured after every 30FT cycles. Figure1 shows the normalized mass (with respect to the initial mass) for Solidia concrete specimens (both air entrained and non-air entrained) and for the reference concrete (air entrained). Values higher than 1.00 indicate mass gain whereas values lower that 1.00 indicate mass loss. For Solidia Concrete, the increase in mass is attributed to water absorption during the test as most of the mixing water is removed from Solidia Concrete during CO_2 curing. Although specimens were soaked in water for 48 hours before the commencement of test, the smaller size pores present in Solidia concrete are not expected to saturate within this time period. It can also be observed from Figure 1 that the mass gain is greater for air-entrained specimen due to the presence of large air voids. For the reference concrete, continuous mass loss is observed after exposure to FT cycles. After 500 cycles, air entrained Solidia Concrete has about 1% mass gain, while air entrained reference concrete lost about 6% of its mass. Non-air entrained Solidia Concrete lost less than 0.1% mass. This behavior can also be seen in Figure 2, which shows photographs of Solidia concrete and reference concrete specimens after exposure to 300 FT cycles. In Figure 2, aggregates are clearly visible on the surface of reference concrete specimens. This is the result of scaling of the OPCbased concrete surface. In contrast, the Solidia Concrete specimens do not show that much scaling and thus do not experience significant mass loss.

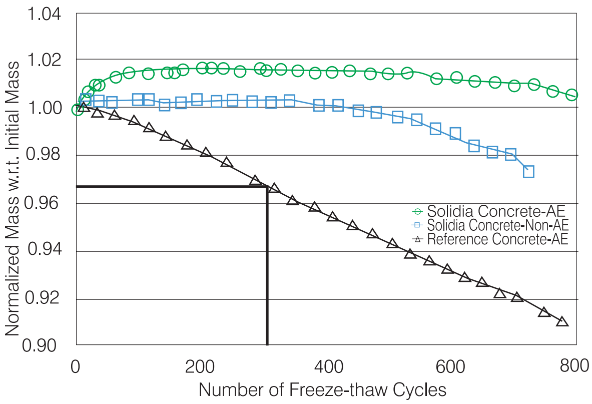
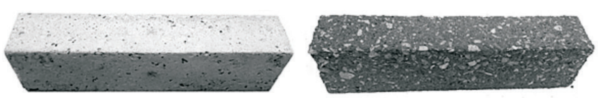


Figure 1: Mass change for reference concrete and Solidia Concrete over the duration freeze thaw test up to 800 FT cycles



(A) Solidia Concrete (B) Reference Concrete

Figure 2: Concrete specimens made with (A) Solidia Cement and (B) OPC cement after completion of the FT test (350 cycles)

Relative Dynamic Modulus of Elasticity: The relative dynamic modulus of elasticity was calculated for all concrete specimens exposed to freezing and thawing after every 30 cycles using Equation 1. Figure 3 shows RDME values for Solidia Concrete (air entrained and non-air entrained) and reference concrete (air entrained) after exposure to different number of FT cycles.

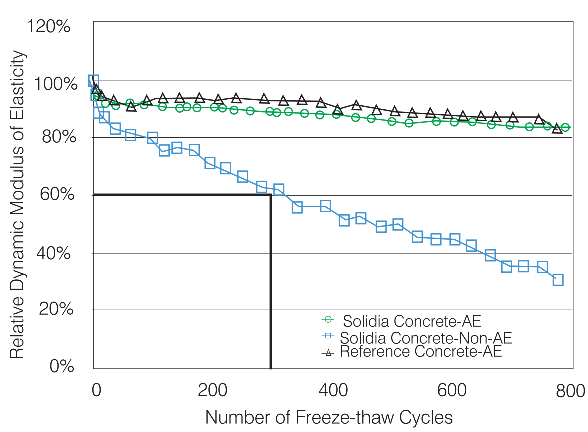
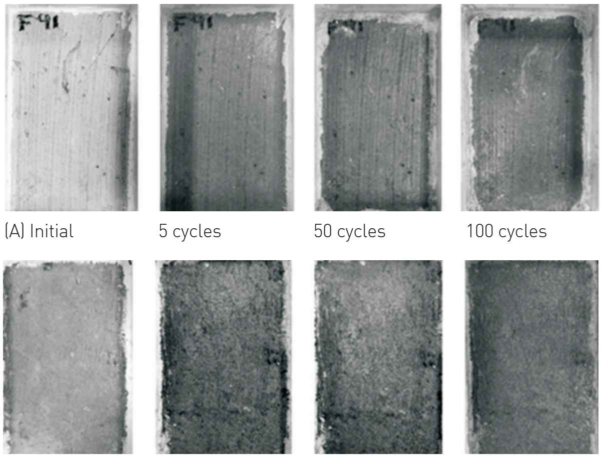


Figure 3: Relative Dynamic Modulus of Elasticity for Reference and Solidia Concretes over the duration freeze thaw test up to 800 FT cycles

The RDME values decrease due to the formation of micro-cracks in concrete specimens when exposed to FT action. After first measurement, the RDME values were reduced for all specimens and remained at about 90% or more after 300 cycles for both air entrained Solidia Concrete and reference concrete. For non-air entrained Solidia Concrete, RDME values decreased to about 80% after 100 FT cycles and to about 58% after 300 FT cycles. These values of RDME indicate very good FT resistance for air entrained Solidia Concrete but marginal performance for non-air entrained concrete.



(A) Initial 5 cycles 50 cycles 100 cycles

(B) Initial 10 cycles 15 cycles 50 cycles

Figure 4: Solidia Concrete (A) and reference concrete (B) scaling specimens before and after completion of different number of FT exposure cycles

Scaling resistance test results

Specimens from the Solidia Concrete and reference concrete scaling tests are shown in Figure 4A and 4B respectively before and after different number of FT cycles. The tests were performed as per ASTM C672, which is described in section 2.3.

Figure 4A shows no surface scaling for Solidia Concrete after 50 FT cycles in the presence of calcium chloride solution, which indicates good scaling resistance. The test was continued further to evaluate long-term performance for Solidia Concrete, which shows no visible scaling after completion of 100 cycles.

Figure 4B shows reference concrete specimens after different numbers of FT cycles. After exposure for 50 cycles, the reference concrete specimen does not show any visible scaling and, thus can also be considered having good scaling performance.

Conclusions

In this study, the freeze-thaw and scaling resistance was evaluated as per ASTM procedures for Solidia Concrete and reference concretes. The following can be concluded form the data collected from the study:

1) Air entrained Solidia Concrete performed extremely satisfactorily in freeze-thaw testing, retaining 88% of the original dynamic modulus even after 500 FT cycles and displaying essentially no mass loss. Even without air entraining, the performance of Solidia Concrete was judged to be reasonably satisfactorily-essentially zero mass loss being recorded even after 500 cycles, while the relative dynamic modulus at 300 cycles (the standard duration of testing) was reduced to about 60% of the original value.
2) Air entrained Solidia Concrete showed no visible deterioration in the standard scaling test, even after 100 cycles, i.e. twice the number of cycles usually administered in this form of testing.
3) Despite the relatively satisfactory performance displayed by non-air entrained Solidia Concrete, normal air entraining is recommended where freezing and thawing are anticipated.