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Evaluation of Stone-Monument Repair Materials for Potential Use in Interim Repair of Frost-Damaged Concrete Navigation Structures

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ABSTRACT: Commercially available products used for conservation and restoration of stone monuments were evaluated for potential use in interim repairs of concrete navigation structures damaged by cycles of freezing and thawing. The investigation was conducted on laboratory specimens of concrete, and test method ASTM C 666 (Test Method for Resistance of Concrete to Rapid Freezing and Thawing) was used as the basis for the evaluation. It was concluded that the products probably were not suitable for practical application because of the size and dimensions of the structures, and because of the nature of the damage.

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Preface

The work reported herein was conducted at the U.S. Army Engineer Research and Center (ERDC), Vicksburg, MS, by personnel of the Concrete and Materials Branch (CMB), Engineering Systems and Materials Division (ESMD), Geotechnical and Structures Laboratory (GSL).

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The work was conceived, designed, and managed by Dr. Edward F. O'Neil, CMB. Dr. Toy S. Poole, CMB, prepared this report, along with Dr. O'Neil.

At the time of publication, Brian H. Green was Acting Chief, CMB; Dr. Larry N. Lynch was Acting Chief, ESMD; Dr. William P. Grogan was Deputy Director, GSL; and Dr. David W. Pittman was Chief, GSL.

COL Richard B. Jenkins was Commander and Executive Director of ERDC. Dr. James R. Houston was Director.

1 Introduction

Background

A line of commercial products called stone strengthening or stone consolidating materials have been marketed for a number of years for purposes of conserving or restoring strength to stone that has been structurally degraded by certain physical and chemical processes. These degradation mechanisms include salt crystallization, salt hydration, attack by salts, acid dissolution, abrasion, cycles of freezing and thawing, and microbial growth. Many U.S. Army Corps of Engineers structures built before about 1940, and a few built after this date, suffer damage from cycles of freezing and thawing. In the early 1940s, it was learned that entraining air in the cement-paste fraction of concrete would protect it from damage by cycles of freezing and thawing. The pre-1940 structures suffered damage because knowledge was inadequate at that time to prevent it. Post-1940 structures sometimes suffer because of improper application of the technology that prevents this damage.

The ultimate solution to repair of these structures is replacement of the concrete that is damaged by the cycles of freezing and thawing and replacing it with concrete that has been properly air entrained. This is expensive. In cases in which the structural integrity of a structure is not immediately threatened, it is sometimes desirable to conserve the structure, if economically feasible, in its present condition until funding for the ultimate repair or the complete replacement of the structure can be arranged.

The purpose of this work was to investigate the performance of these products for purposes of prolonging service life until the ultimate disposition of the structure can be determined. ASTM C 666, Test Method for Resistance of Concrete to Rapid Freezing and Thawing, is the test method used to evaluate the resistance of concrete to cycles of freezing and thawing, and is the basis for this evaluation.

Literature Review

There does not appear to be a great deal of recent literature on the topic of stone consolidation. Clifton (1980) reviews the technology of these materials as represented by publications through 1980, and provides a good discussion of the desirable properties and of some of the potential problems with use of these materials in stone. The following notes are taken from that source.

Clifton (1980) categorized performance requirements into two categories: primary and secondary. Primary requirements are those that all consolidants should meet. Secondary performance requirements are those that must be met only under specifically defined circumstances (i.e., optional requirements). Only primary requirements are discussed in this review. Very little in the way of standard specifications exists for these properties, although a guide for the repair and restoration of dimension stone is being developed in ASTM Committee C 18. A guide to selecting accelerated tests for determining the service life of building components and materials is also being developed in that committee. Clifton's primary requirements are contained in six categories, as follows:

- a. *Consolidating value.* This is the property of restoring cohesion among particles of deteriorated stone. Tensile strength, surface hardness, and abrasion resistance are properties sometimes used as metrics for this property. Only one recommendation for a specific limit was found in the literature review, which was that compressive strength should be at least 10% higher than the stone in the undeteriorated condition.
- b. *Durability.* Consolidated stone should generally be as durable as unweathered stone. No limits were recommended, but ASTM E 632 was recommended as a test method. This method was withdrawn in 2005, and a substitution is being developed.
- c. *Depth of penetration.* Penetration of these materials is accomplished by capillary action. The desirable condition is penetration to about 25 mm—without a sudden change in physical properties at this depth, but rather as a gradual change. Sharp physical-properties changes sometimes result in focus points for delamination.
- d. *Stone porosity.* Durability of stone is dependent partially on size of pores. Reductions in mean pore radius by use of stone consolidation treatments may have negative effects on durability to freezing and thawing. No firm guidance had been offered at the date of this report (1980).

- e. *Moisture transfer.* Total elimination of water and vapor transport can cause problems traceable to a water-saturated condition, such as cycles of freezing and thawing. Also, eliminating water impermeability but allowing water vapor permeability by use of partial pore filling sometimes results in accumulation of salts behind the barrier film, causing damage. The objective is to not change water or vapor permeability significantly. ASTM test methods C 97 and C 355 (withdrawn 1982) apply to measuring these properties.
- f. *Compatibility with stone.* Treated stone should have dimensional properties and brittleness properties similar to untreated stone.

In 1993, a collection of papers on the subject was published by RILEM (International Union of Laboratories and Experts in Construction Materials, Systems and Structures), many of which focused on desirable properties of stone consolidation materials. Presented below are some notes taken from this source.

Littmann and Sasse (1993) investigated various analytical techniques for evaluating the properties of treated stone. These included measures of depth of penetration, water absorption, and modulus. The polyurethane material that was used penetrated to 50 mm.

Chiavarini et al. (1993) reported results of tests on perfluorourethanes in durability tests. These included ultraviolet light, marine fog, wetting and drying, and thermal cycling, but not freezing and thawing.

Piacenti et al. (1993) used modulus of elasticity as a metric to evaluate effectiveness of perfluorinated protective agents.

Auras (1993) evaluated polyurethane treatments on three stone types. Penetration was to about 12 cm, but performance was irregular. In some cases there was strength loss in the first centimeter.

Sasse et al. (1993) discusses laboratory-determined properties that can be used as evaluation criteria for effectiveness of polymers for consolidating stone. The criteria focused on microstructural modifications. Material qualities such as viscosity, polarity, molecular weight, rates or reactivity, compatibility with stone, adhesion, and elastic modulus were listed. Products are not intended to repair macroscopic defects.

Objective

The objective of the work is to determine whether stone treatment products have potential to be used for interim repairs of concrete navigation structures damaged by exposure to cycles of freezing and thawing.

Approach to the Research

The objective of this work is to determine whether stone consolidation products appear to have any value in conserving concrete structures that have been deteriorating from cycles of freezing and thawing. It is not anticipated that major rehabilitation or replacement of these structures is imminent, so discovery of a technology that could conserve their remaining physical properties for a few years may have value.

Particularly notable are structures built before 1940, when technology for avoiding damage from cycles of freezing and thawing had not been developed. The Corps of Engineers owns a number of structures in the upper Mississippi and upper Ohio rivers and drainage areas that were built in the 1920s and 1930s. The damage to these structures is visually conspicuous. It is not implausible that products that conserve stone could also conserve concrete, given the similar nature of the two materials.

The approach to the work is to use ASTM C 666 as the basis for evaluating the effectiveness of stone consolidation products for conserving concrete strength. C 666 is the standard test method by which concretes are evaluated for resistance to cycles of freezing and thawing and on which specifications for new concrete are based. Also, the test method for evaluating the durability of aggregates to cycles of freezing and thawing, CRD-C 114, is also based on C 666.

Briefly, the laboratory procedure was to fabricate C 666 test specimens, and then to expose them to cycles of freezing and thawing according to the test method until significant degradation in properties was observed. Specimens were treated with stone consolidation materials and then re-exposed to additional cycles of freezing and thawing. The before- and after-treatment performance was then compared.

2 Materials, Methods, and Test Results

Materials and General Methods

Two single concrete mixture designs were used to make test specimens. The proportions (Table 1) were the same from both, except that one was air entrained and other was not air entrained.

Table 1. Description of concrete mixtures used in testing.	
Component	Mass of Each Component kg/m³
Cement	245
Fine aggregate	889
Coarse aggregate	979
Water	159
Air-entraining admixture, if used	0.086 L/m ³
Water/cement ratio	0.65
Slump	3.5" w/air, 2.25" w/o air
Air content	5.4% w/air, 2.8% w/o air
14-day Compressive strength	3243 psi w/air, 3404 psi w/o air

Both air-entrained and non-air-entrained concrete mixtures were used to generate a range of concrete deterioration conditions, with the intent of better covering the level of deterioration (due to cycles of freezing and thawing) that exists in structures. The water-cement ratio of 0.69 is also high by today's standards, but this was common in older construction. This water-cement ratio also ensured a measurable level of deterioration within a reasonable amount of laboratory time.

Specimens were cured for 14 days and soaked in water for 48 hr prior to initiation cycles of freezing and thawing, as described in C 666, Method A (freezing in water, thawing in water).

The test method allows for a range of temperature cycling rates, ranging from as few as 2 hr per cycle to as many as 5 hr per cycle. In this work, the cycling period was 2 hr. This period was not chosen for any particular reason other than it is the standard operating procedure in this laboratory and could not easily be revised.

Resonant frequency was the metric used to measure the physical integrity of the specimens. Resonant frequency is a function of modulus of elasticity, so when the resonant frequency at a particular test age is expressed as a fraction of the resonant frequency of the specimen before any cycles of freezing and thawing, the metric is then expressed as a percentage of the initial condition and called %E. Data collected from a thermocouple placed in the center of a rock core of similar size to the concrete specimens, and placed in the center of the tank, were used to monitor temperature cycling conditions.

The freezing and thawing apparatus was manufactured by Conrad/Missimer. Dynamic modulus of elasticity was calculated from resonant frequency data as described in ASTM C 215, using the impact resonance method. The accelerometer and frequency analyzer was manufactured by Pico.

Products Tested

Six products were chosen for testing, as described below. None of these products specifically claims to be adequate to repair concrete that has suffered significant damage from cycles of freezing and thawing; however, their claimed action does appear to be potentially pertinent to that process.

- a. Prosoco OH100 Consolidation Treatment. This product is based on silicic ethyl ester chemistry. It is designed for the restoration of structural integrity to stone masonry by replacing the natural cementing action that may have been degraded with a synthetic cementing action. The product does not result in large changes in thermal expansion properties. Large changes in coefficient of thermal expansion can result in stone spalling. This material is for use on all stone.
- b. Prosoco H100 Consolidation Treatment. This is also an ethyl silicate-based product with silane-based water repellent. The

product is designed to restore cementing of stone particles and is for general stone use.

- c. Prosoco HCT. The chemical basis of this product is vaguely described in the manufacturer's literature, but is referred to as a novel two-component hydroxylating material. It is specifically designed for restoration of carbonate rocks, such as limestone and marble. It claims to restore the stone grain binding that may have been removed by the action of acids.
- d. Degussa Chem-Trete BSM 40. The chemical basis of this product is isobutyltrialkoxysilane in an alcohol solvent. This product is advertised as a penetrating water repellent for concrete, masonry, and stone (i.e., not a surface sealer). It is intended to reduce the effects of corrosion, spalling, scaling, efflorescence, leaching, and staining.
- e. Degussa Dynesylan BHN. The chemical basis for this product is alkyltrialkoxysilane, which is a liquid compound. This product is a penetrating sealer specifically designed to reduce corrosion and alkali-aggregate reaction.
- f. Degussa Protectosil CIT. This product is basically a concrete sealer that is advertised to inhibit corrosion of steel and penetration of chlorides.

Specific Test Conditions and Test Results

There were three rounds of testing. Two of these involved the Prosoco products and one involved the Degussa products. Following are descriptions of the specifics of the test conditions and the test results.

Prosoco Round 1

Both air-entrained and non-air-entrained concrete was used in this round. Six test specimens were cast. Three specimens were air entrained, and three were not air entrained. Specimens were conditioned as in C 666, then exposed to cycles of freezing and thawing until the durability factor (expressed as relative dynamic modulus of elasticity, percent) fell to 60% or lower. The non-air-entrained specimens lost modulus very rapidly to about 20% after only 20 cycles. The air-entrained specimens fell to about 60% after 305 cycles.

The non-air-entrained specimens were then removed from the freezing chamber and left in water until the air-entrained specimens had reached the 60% level. All specimens were then air dried for 2 days, treated with

product, and allowed to air dry for 21 days. Then, the specimens were returned to the freezing chamber for additional cycles of freezing and thawing. Relative E was determined at various points in this treatment period.

The treatment cycle was to brush the product liberally on all surfaces, then allow the specimen dry for 30 min. This cycle was repeated two more times within a 1.5-hr period. The rinse was applied to the HCT material 30 min after the last treatment. Specimens were then left in air for the 21-day period.

Results for the non-air-entrained specimens are shown in Figures 1 and 2. Figure 1 is a plot of Relative E versus time in days. This graph emphasizes the details of the change in Relative E during the treatment period. Figure 2 is a plot of Relative E versus number of cycles of freezing and thawing. This graph emphasizes the before-and-after treatment comparison.

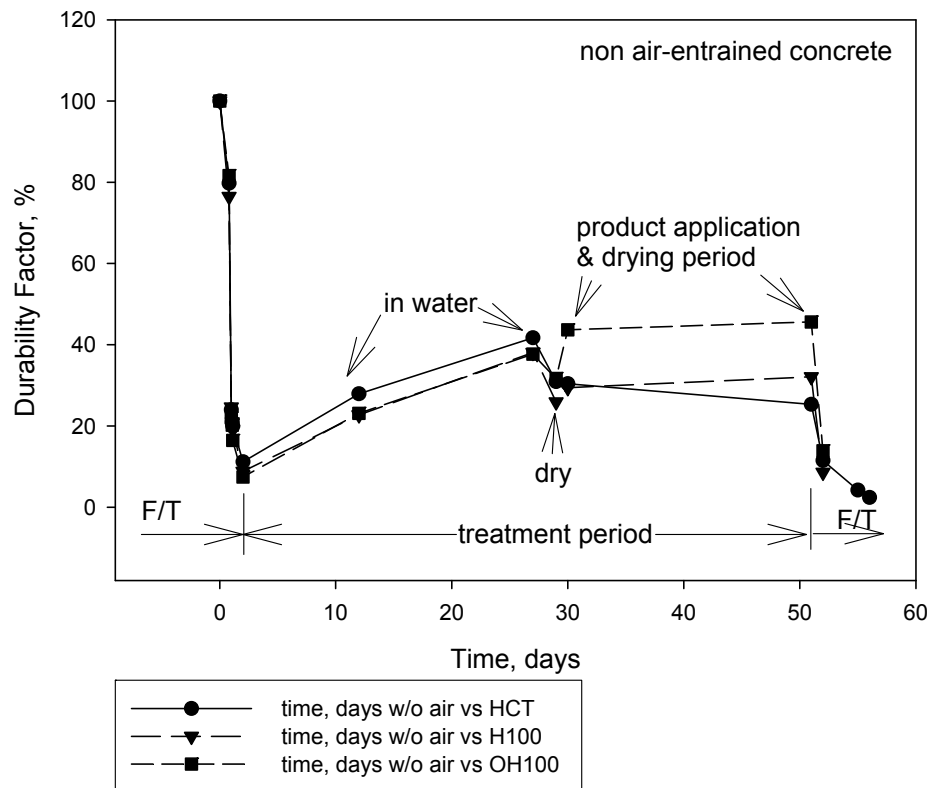


Figure 1. Durability factor (%E) versus treatment time (days) for Prosoco products used on non-air-entrained concrete samples.

As shown in Figure 1, there was some recovery of modulus following the period of initial freezing and thawing during the period of soaking in water. However, modulus decayed very rapidly upon resumption of freezing and thawing cycles.

Figure 2 puts the rate of decline in durability factor into better perspective. This presentation shows that damage accumulated with increasing cycles of freezing and thawing in a non-linear way. The shape of the decline suggests an exponential decay type function. The application of stone strengthener products seemed only to temporarily interrupt the rate of this process.

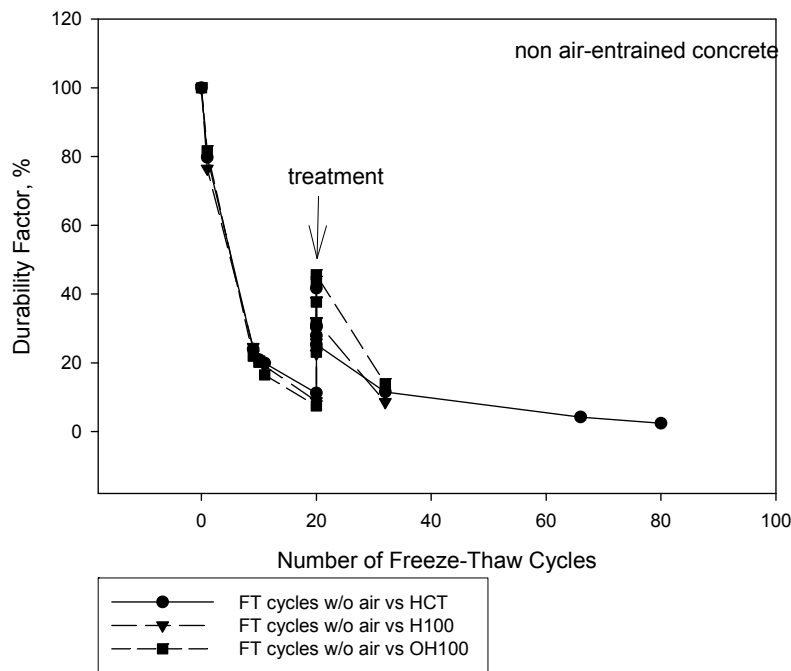


Figure 2. Durability factor (%E) versus number of cycles of freezing and thawing for Prosoco products used on non-air-entrained concrete samples.

Figures 3 and 4 show the time- and cycle-dependent change in durability factor for treatment of air-entrained specimens. As expected, the pretreatment degradation of durability factor was slower than in the case of non-air-entrained concrete. An increase in %E for product OH100 was seen during the treatment period, but none was observed in use of HCT and H100 products. Degradation of durability factor very quickly returned

to the pre-treatment levels. In the case of the air-entrained concrete, the rate of decay in durability factor appears to be approximately linear with respect to number of cycles of freezing and thawing.

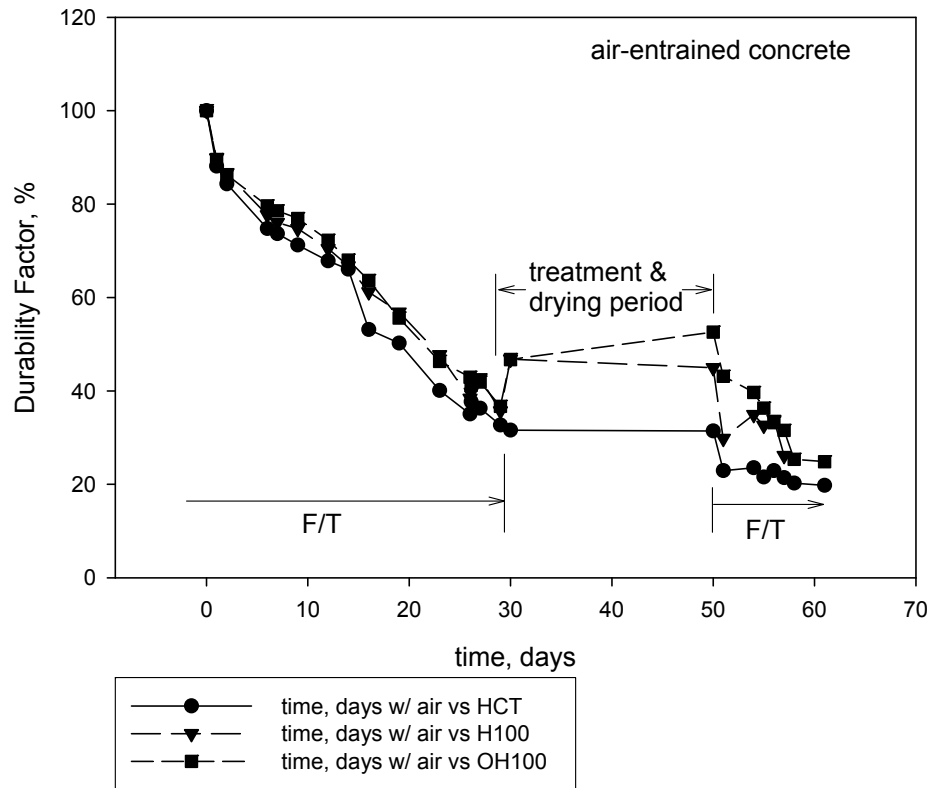


Figure 3. Durability factor (%E) versus treatment time (days) for Prosoco products used on air-entrained concrete samples.

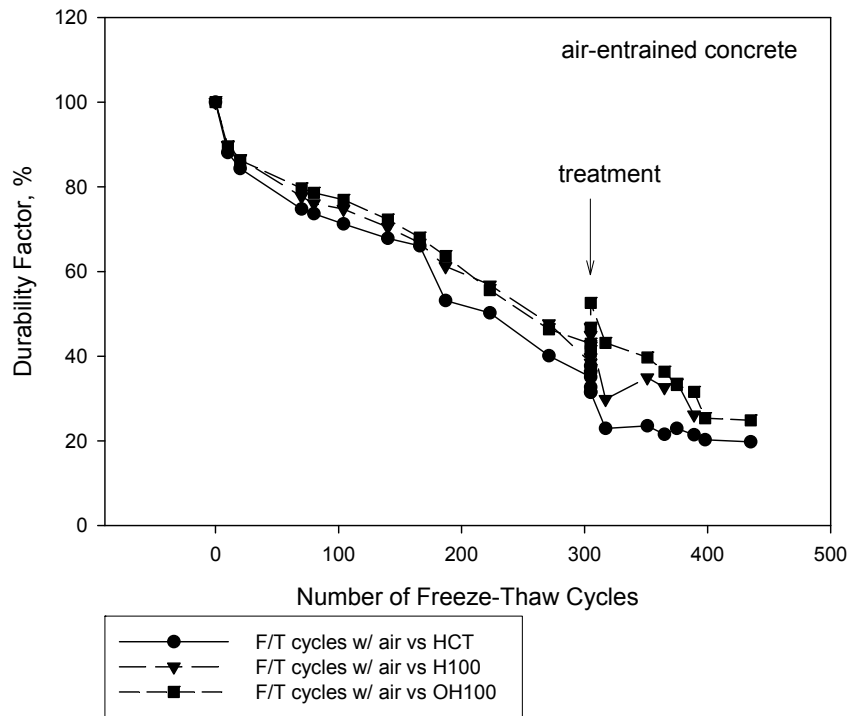


Figure 4. Durability factor (%E) versus number of cycles of freezing and thawing for Prosoco products used on air-entrained concrete samples.

Prosoco Round 2

Figure 5 shows data from the second round of testing with Prosoco products. In this case the durability factor was reduced to between 60 and 80% before treatment. No data were collected during the treatment period; so, the first post-treatment data point occurred after 11 post-treatment cycles of freezing and thawing. There was significant continued degradation of durability factor at this point. However, damage appeared to cease at this point. This also happened with the control specimen (no stone strengthener applied); therefore, this effect cannot be attributed simply to the stone strengtheners.

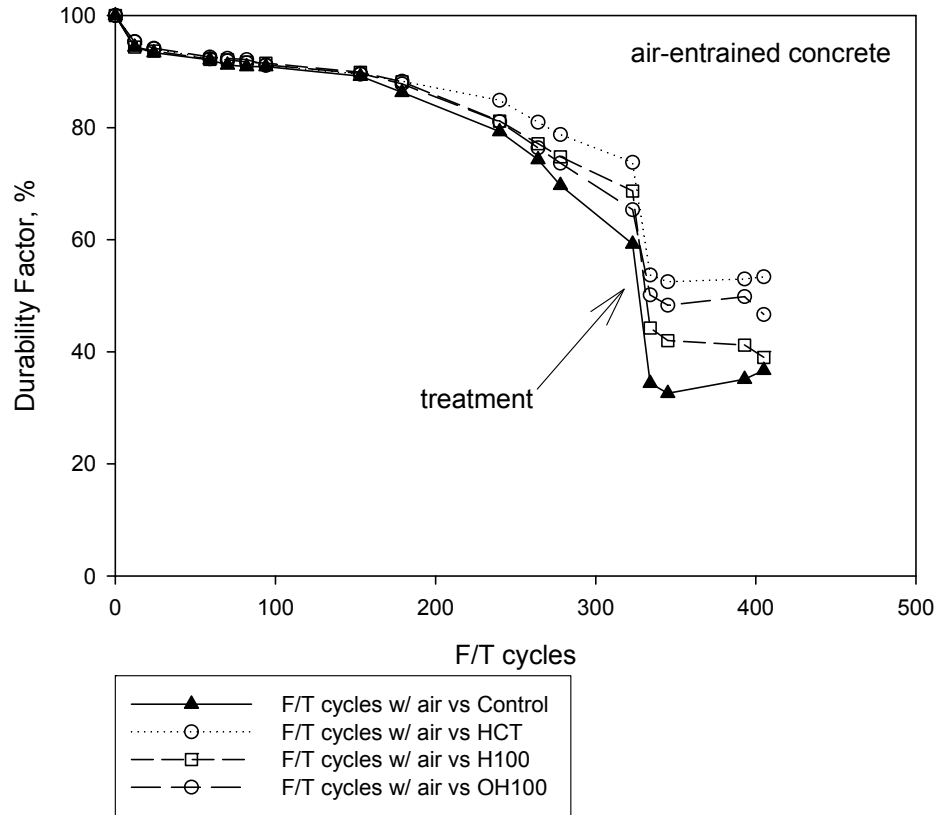


Figure 5. Durability factor (%E) versus number of cycles of freezing and thawing for Prosoco products used on air-entrained concrete samples, Testing Round 2.

Degussa

None of the Degussa products resulted in a restoration of structural integrity that persisted for even a few cycles of freezing and thawing. All of the specimens, including the control, showed a slight reduction in rate of decay of durability factor (Figure 6), which suggests that this was probably an artifact of the testing and not an attribute of the products.

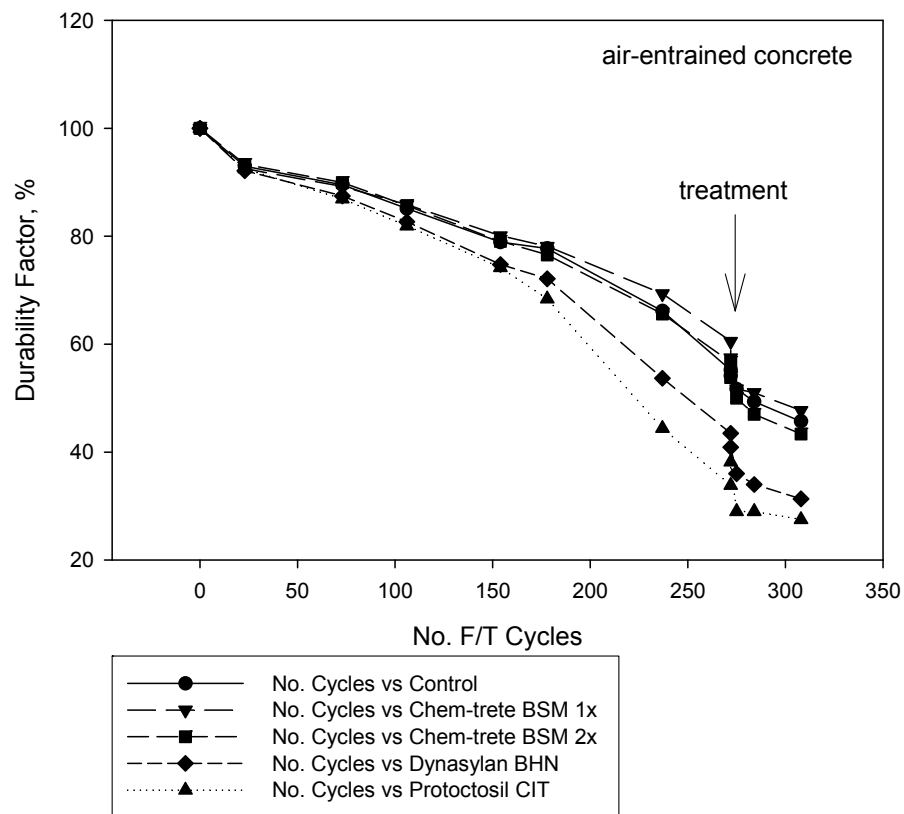


Figure 6. Durability factor (%E) versus number of cycles of freezing and thawing for Degussa products used on air-entrained concrete samples.

3 Discussion of Results

Summary of Results

The materials tested in this work are a mixture of sealers and stone consolidation products. The Degussa products were all sealers. The Prosoco products were all stone consolidation products.

Sealers are not expected to have any action in the way of restoration of strength, but they may prevent further damage by preventing development of a water-saturated condition in the concrete. There was some evidence of a change in slope of the $E\%$ versus cycle curve after the treatment. However, since the control (no treatment) specimens also showed this, it could not reasonably be concluded that the treatments were responsible. There was no significant evidence in this work that this occurred, although there is some possibility that the specimens were not adequately dried out before the application of the material. Thus, it is possible that a sufficient fraction of the concrete remained in a saturated condition after the sealers were applied, such that damage continued.

The use of sealers for protection of existing concrete from further damage by action of freezing and thawing cycles involves much uncertainty. It is commonly reported that application of sealers to pavements that have moisture access from below will actually enhance the rate of damage by trapping the concrete near the freezing zone in a saturated condition. However, sealers might have useful application on elevated structures in which saturation from below is not likely.

The stone consolidation materials are designed to restore strength by restoring the cementing among rock grains. There was no strong evidence that this occurred in frost-damaged concrete. This could be attributable to the way in which damage develops in concrete subjected to cycles of freezing and thawing. Development of macroscopic damage in the form of cracks parallel to the freezing front is common (Figure 7). Microscopic damage also develops. Stone consolidation products may have some

benefit in restoring strength loss as a result of the microscopic damage. However, it is not expected that it would repair macroscopic damage.



Figure 7. Macroscopic damage from cycles of freezing and thawing in a 1930s-era navigation structure.

It is not clear that the test methods employed would effectively differentiate among these two damage-repair scenarios. Therefore, it cannot be declared definitively that the materials had no effect. However, if the effect occurred, it was not major.

The test method used in this work, ASTM C 666, is considered to be a very aggressive test method. Any benefits derived from treatment with sealers or stone consolidation products may have been overwhelmed by the severity of this test. Although the results do not show any convincing evidence of the utility of these products in restoring concrete strength, it must be allowed that there is the possibility the products may have some temporary value under more realistic conditions.

A major problem with use of stone consolidation products—even if they could be determined to give some useful strength restoration to

frost-damaged concrete—is achieving adequate depth of penetration. Damage from freezing and thawing at Corps of Engineers' navigation structures is known to reach at least 2 to 3 ft (0.6–0.9 m), as evidenced by macroscopic damage, and may reach somewhat deeper. Application of materials as surface treatments would be expected to result in penetration to shallow depths relative to this frame of reference.

Recommendations

Concrete sealers may have some application for preservation of some structures. For example, the top surfaces of parapet walls might reasonably be treated with a sealer to prevent saturation from rainwater or from melt of accumulated snow and ice. Untreated sides would then provide breathability that would ensure that a saturated concrete microstructure did not occur as a result of capillary rise from below.

It does not appear that stone consolidation products offer great promise for large-scale application to navigation structures because there is no evidence of significant strength restoration and because of the problems with application of the materials to the required depths.

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