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Concrete Testing for MTC: Oxocrete™ Surface Treatment

By Haley Mims

PURPOSE: This technical note provides an in-depth review of results for the concrete testing performed on a Materials Testing Center (MTC) project. At the request of the sponsor, Mr. Allan Shantz, this document specifically focuses on the difference in the physical and chemical properties between treated and untreated concrete cores and beams.

INTRODUCTION: Control (untreated) and treated specimens were externally sampled and delivered to the US Army Engineer Research and Development Center (ERDC). Treated samples were impregnated with a proprietary silicate blend using the Oxocrete™ application method to achieve penetration and impregnation of silicate into the matrix. The treatment is expected to produce a difference in strength and chemical analysis compared to the untreated samples. Specimens' sizes were sampled according to the appropriate minimum size required by the test being performed. For the testing of cylinders, two sizes of cylindrical cores for strength and abrasion were sampled, 2 in.* diameter × 4 in. high and 6 in. diameter × 2 in. high. Once the cores arrived at the MTC, their ends were cut and ground as appropriate for each specification. The two types of concrete beams were sampled, 3.5 in. × 4.5 in. × 12 in. and 4 in. × 4 in. × 15 in. Three replicates of treated and untreated specimens were used, totaling six specimens for each test method. Due to perceived inconsistences in pH and compressive strength measurements, these standards were repeated with larger 4 in.-diameter specimens for comparison.

TEST METHODS: The test methods performed were in accordance with the American Society for Testing and Materials (ASTM) procedures, except for two tests that do not have an ASTM procedure. For concrete strength and durability testing, the four following ASTM procedures were performed:

- ASTM C39 / C39M-21, *Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens* (ASTM International 2021)
- ASTM C78-02, *Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)* (ASTM International 2002)
- ASTM C666 / C666M-15, *Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing* (ASTM International 2015)

* For a full list of the spelled-out forms of the units of measure used in this document, please refer to *US Government Publishing Office Style Manual*, 31st ed. (Washington, DC: US Government Publishing Office, 2016), 248–52, https://www.govinfo.gov/content/pkg/GPO-STYLEMANUAL-2016/pdf/GPO-STYLEMANUAL-2016.pdf.

- ASTM C944 / C944M-19, *Standard Test Method for Abrasion Resistance of Concrete or Mortar Surfaces by the Rotating-Cutter Method* (ASTM International 2019)

For chemical properties analysis, two ASTM methods were performed:

- ASTM D4262-05(2018), *Standard Test Method for pH Of Chemically Cleaned or Etched Concrete Surfaces* (ASTM International 2018)
- ASTM C267-20, *Standard Test Methods for Chemical Resistance of Mortars, Grouts, and Monolithic Surfacings and Polymer Concretes* (ASTM International 2020)

For chemical properties analysis, two non-ASTM procedures were performed:

- Permeability (sorptivity)
- Depth of Carbonation (phenolphthalein test)

RESULTS: Images of the specimens were taken upon arrival and throughout testing. Figure 1 shows images of three treated and three untreated cores and beams taken after they arrived. These images are representative of the samples as a whole. There was limited visual indication of treatment on some specimens.

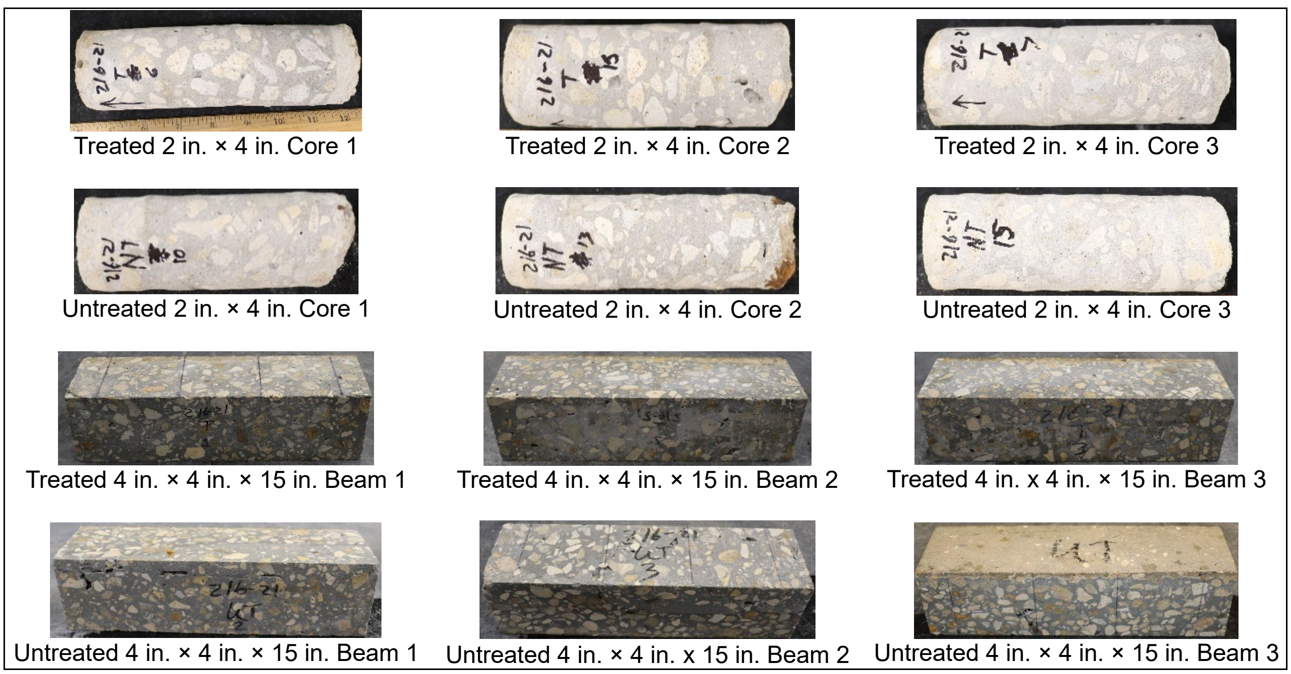


Figure 1. As-received samples.

For the concrete compressive strength of the 2 in. × 4 in. cores and flexural strength of the 4 in. × 4 in. × 15 in. beams, tests were conducted at two ages: Day 0 and Day 90. In this case, Day 0 refers to the original test date of as received samples and Day 90 refers to 90 days after the Day 0 test. Three replicates were tested at each age. The compressive strength at Day 0 for the untreated cores averaged 2,300 psi, while the treated cores averaged 2,500 psi. At Day 90, the untreated cores had an average compressive strength of 1,870 psi, and the treated cores had an average of 1,700 psi. In both 0- and 90-day compressive strength tests, the differences between treated and untreated cores were about 10% of the total compressive strength; we consider this difference negligible considering normal concrete compressive strength variability. We consider it noteworthy, however, that the 90-day testing produced meaningfully lower strengths than the 0-day testing. This may indicate dehydration of the samples that occurred in the time since coring.

Due to perceived inconsistencies in the data, compressive strength was tested again using 4 in. diameter cored specimens based on the hypothesis that the larger specimens should provide greater certainty in results. Like the 2 in.-diameter core tests, three treated and three untreated cores were tested. The core samples were not long enough for the standard 2:1 length:diameter ratios as prescribed in ASTM C39, so correction factors were necessary to compare the results to standard values. ASTM C42, *Standard Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete* (ASTM International 2020b) was used to apply correction factors, and based on specimen length, factors ranging from 0.87 to 0.93 were used. Treated cores had an average compressive strength of 3,770 psi, and untreated cores had an average of 3,500 psi. These results appear to be more consistent with expectations than the 2 in. diameter specimens.

The untreated 4 in. × 4 in. × 15 in. beams had an average flexural strength at Day 0 of 465 psi, while the treated had an average of 490 psi. There were an insufficient number of samples provided for 90-day treated flexural tests.

Abrasion testing resulted in an average loss of 26 g for both the treated and untreated 6 in.-diameter × 2 in. tall cores with an average depth of wear of 2.5 mm for both core types. ASTM C944 calls for three 2 min periods in three separate areas of the representative surface; however, to prevent overlapping, only one period of abrasion was performed over a 2 min time frequency using a double load of 44 lbf due to the limited surface area of the specimens. Figure 2 shows images of the cores after abrasion.

Untreated samples after abrasion | Treated samples after abrasion

Figure 2. Abrasion samples after testing.

Freezing-and-thaw testing was conducted on three treated and three untreated 4 in. × 4 in. × 12 in. beams in accordance with ASTM C666 as summarized in Table 1. The beams were cured in their shipping boxes for 14 days and then placed in the cure room at 72°F for 48 hr to reach a moist condition prior to testing. After the curing period, the specimens were placed in the thawing water at 40°F for 24 hr to begin the thawing phase. Then, the initial weights and frequencies were recorded, the freezing-and-thawing cycles began at 40°F and dropping to 0°F ± 3°. The specimens were removed from the apparatus in a thawed condition at intervals not exceeding 36 cycles of freezing-and-thawing for data sampling, which included determinations of the fundamental transverse frequency and the mass of the specimens. The specimens were returned to the apparatus after each data sampling; data sampling was conducted three times per week until failure occurred, or 300 freezing-and-thawing cycles were completed. The treated specimens completed 300 cycles, and the untreated failed after 166 cycles. Table 1 represents the results of averages for weight change, transverse frequency, durability factor, and relative dynamic elastic modulus.

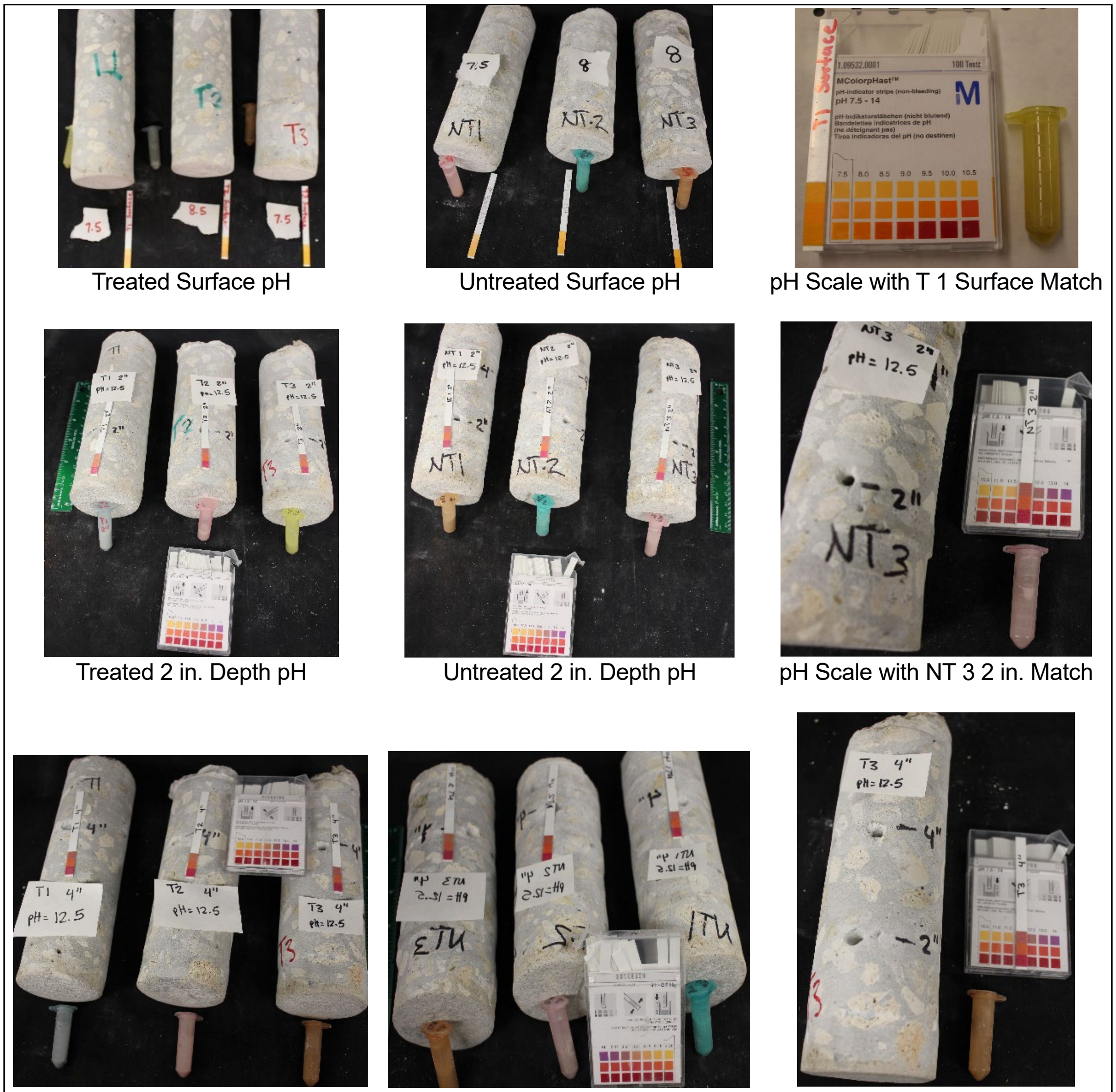
Test Results	Treated 1	Treated 2	Treated 3	Untreated 1	Untreated 2	Untreated 3
Weight Change (lb)	−0.07	−0.01	−0.38	0.03	−0.05	0.37
Frequency (Hz)	2.26	2.26	2.13	2.05	2.30	2.29
Relative Dynamic Elastic Modulus (%)	76	75	71	62	66	68
Durability Factor	42	41	39	19	21	22

Table 1. Freeze / thaw results.

Chemical properties analysis was completed on the 2 in. × 4 in. cylindrical cores for pH readings utilizing pH paper at three depths: the surface, 2 in. from the surface, and 4 in. from the surface. For the surface reading, 60 grit sandpaper was used to sand the surface to obtain approximately 1 g of the specimen that was placed in a centrifuge tube with 1 mL of water for a 1:1 ratio. A drill and concrete bit were used to drill holes 2 in. and 4 in. down from the surface, and the inner material of the cores was collected to repeat the process. Sanding was not required for these two depth readings, because the drill provided the amount of material needed. After sample collection and mixing was completed, pH paper was placed in the mixture for at least 2 sec and matched to the pH scale for the most accurate reading. Table 2 tabulates the pH readings for each sample. The surface pH for all cores was between 7.5 and 8.5, while a pH of 12.5 was recorded for all readings at 2 in and 4 in. depths for treated and untreated specimens. Figure 3 shows images at each reading depth for all specimens.

Testing for pH was evaluated a second time using an alternative test method on a new set of 4 in.-diameter cores at the sponsor’s request. Two cores were tested—one treated and one untreated. Each core was sliced at depths of 1, 2, 3, 4, and 6 in. below the surface. The downward facing exposed side of each cut was then milled to collect a 0.5 g sample of fine material. There was no sample taken directly from the surface of the cores. Each 0.5 g sample was added to 1.0 ml distilled, pH-neutral water in a centrifuge tube and agitated to combine the constituents. The tube was allowed to rest for 30 min before testing. Results of these tests are shown in Table 3. For the untreated core, results were near constant and essentially the same as the initial round of testing.

Results for the treated core specimen were variable from 10.0 to 12.0. Given the lower pH expected to be found on the concrete surface and lower results at the 1 in. depth for both 4 in. specimens, it may be more accurate to conclude that the pH varied from 10.5 to 12.0, because the 1 in. depth may have been altered by proximity to the surface. In either case, the pH was found to be lower in the treated core using this method.



Treated Surface pH | Untreated Surface pH | pH Scale with T 1 Surface Match

Treated 2 in. Depth pH | Untreated 2 in. Depth pH | pH Scale with NT 3 2 in. Match

Treated 4 in. Depth pH | Untreated 4 in. Depth pH | pH Scale with T 3 4 in. Match

Figure 3. pH testing.

Depth of Testing	Measured pH					
	Treated 1	**Treated 2**	**Treated 3**	**Untreated 1**	**Untreated 2**	**Untreated 3**
Surface	7.5	8.5	7.5	7.5	8	8
2 in. Depth	12.5	12.5	12.5	12.5	12.5	12.5
4 in. Depth	12.5	12.5	12.5	12.5	12.5	12.5

Table 2. pH results.

Table 3. pH results (alternate method).

Depth of Testing	Measured pH	
	Treated 4 in. Diameter Core	Untreated 4 in. Diameter Core
1in. Depth	10.0	12.0
2 in. Depth	12.0	12.5
3 in. Depth	11.0	12.5
4 in. Depth	10.5	12.5
6 in. Depth	10.5	12.5

Depth of carbonation testing was conducted on 2 in. × 4 in. cores that were split in half using a press, creating a break throughout the middle of the core to test carbonation on both halves. The inner sections of the cores were cleaned with water to remove any dust and debris, rinsed with phenolphthalein, and allowed to dry. All treated and untreated cores were carbonated throughout the depth, starting from 1 to 1.5 in. from the surface. Images were taken before and after carbonation of all specimens as shown in Figure 4.

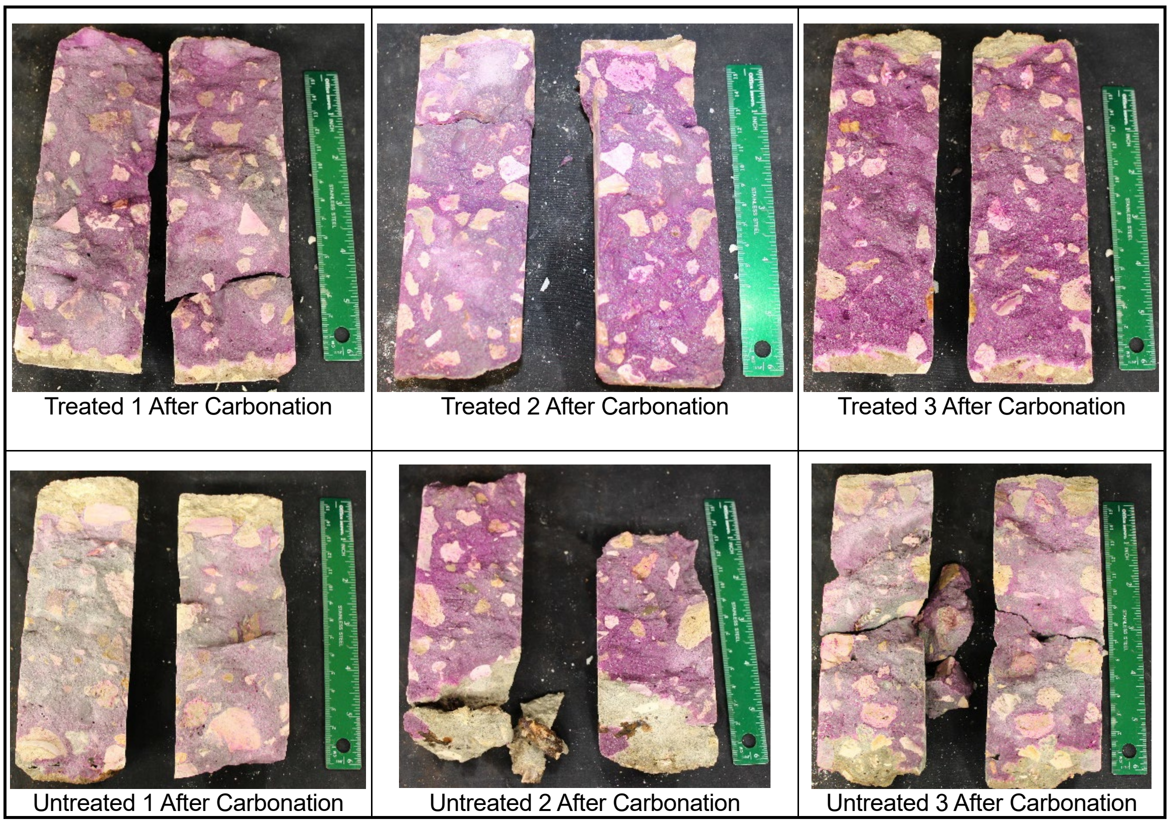


Figure 4. Depth of carbonation results.

Chemical resistance was performed on three treated and three untreated 2 in. × 4 in. cores. The specimens were weighed initially, and visual observations were documented. A 10% sulfuric acid solution was used for the test. The solution was made using 700 mL of sulfuric acid and 6,300 mL of de-ionized water yielding 7 L of solution allowing the cores to be completely to submerged in the solution. The specimens were placed in a clean bin on their sides without touching, 7 L of 10% sulfuric acid solution was added to cover all six specimens, and the bin was covered. After 24 hr, the specimens were quickly rinsed three times with cold tap water and blotted dry by with a paper towel between each rinse. The specimens were then air dried for ½ h while resting on their curved surface before weighing. The weight after immersion and a posttest visual inspection were recorded for each specimen and the solution. Mass change percentage was calculated using the initial and posttest masses. Prior to testing, all specimens were gray in color with pitted aggregates, had air voids visible throughout the cores, and there were no cracks or softening. Untreated 1 and untreated 3 had visible oxidation at the bottom of both cores. After testing, discoloration occurred for all six specimens; all specimens turned white, and their aggregates became more discolored. All specimens still contained pitted aggregates and air voids visible throughout. No cracking occurred in any specimen, but softening occurred for all, causing pieces of the concrete to remain in the solution and crumble easily. The oxidation on both untreated 1 and untreated 3 cores was easily removed after chemical testing. Finally, the mass change percentage was slightly greater for the untreated specimens, which had an average mass change of 1.7%, while the treated specimens averaged 1.5%. These findings imply that the treated specimens were more resistant to the chemical soaking. Figure 5 shows pretest and posttest images of each specimen.

Permeability testing was performed by creating a barrier at the surface of three treated and three untreated 2 in. × 4 in. cores using impermeable tape and a 1:1 ratio of epoxy along the surface. The barrier was tested by ponding water on the surface of each core overnight to make sure leaks did not occur. Once all specimens were prepared with epoxy and tape, masses were recorded to the nearest 0.1 g prior to testing. Water was placed on the surface of each core for 15 min, 30 min, 45 min, 1 hr, 2 hr, 4 hr, 6 hr, and 24 hr. After each time frame was completed, the surfaces were patted dry with a paper towel to a saturated surface dry condition, and masses were recorded. Water was re-introduced to each surface for the next time interval. After roughly 2 hr, the treated specimens reached their sorption limit, and mass gain ceased. The untreated specimens continued to absorb water until there was no change from 6 to 24 hr, indicating the treatment reduced the sorption capacity after a certain period. Once the specimens reached their full sorption capacity, their mass was maintained and did not increase. Table 4 provides the masses recorded initially and at each time interval for each specimen.

Figure 5. Chemical resistance.

Time Period	Measured Weight (g)					
	Treated 1	Treated 2	Treated 3	Untreated 1	Untreated 2	Untreated 3
Initial	774.6	718.3	794.5	731.9	782.8	785.4
15 min	775.4	721.1	797.7	733.6	785.3	789
30 min	779	722.8	799	733.9	785.6	789.7
45 min	783.2	725	801.5	734.2	786	790
1 h	785.5	725.3	802.9	734.2	786.1	790.2
2 h	785.9	725.3	804.1	734.3	786.2	790.3
4 h	786	725.3	804.1	736.7	787.9	791
6 h	786	725.3	804.1	738.9	788.7	791.2
24 h	786	725.3	804.1	739	788.7	791.2
Total Weight Change (g)	11.4	7	9.6	7.1	5.9	5.8

Table 4. Sorptivity results.

CONCLUSIONS: Control (untreated) and treated concrete specimens were evaluated for compressive strength, flexural strength, abrasion resistance, freeze-thaw resistance, pH, depth of carbonation, chemical resistance, and sorptivity by ponding water. In compressive strength, flexural strength, abrasion resistance, and depth of carbonation, both the control and treated samples performed similar or nearly the same. Chemical resistance to sulfate was moderately better in treated samples (1.5% mass loss compared to 1.7%). Sorptivity by ponding testing exhibited more water ingress into the sample in treated specimens verses untreated. This may be due to pre-treatment techniques and was generally opposite to expected results. pH testing was conducted by two methods—this laboratory's typical method and an alternative method proposed by the sponsor. The alternative method showed a lower pH in the treated core specimens compared to the untreated by approximately 1 to 2 logarithmic units. The first method showed equivalent pH in both treated and untreated samples. Concrete pH testing can be difficult to accurately assess, and differences of these magnitudes may not be indicative of true differences between specimen treatments. Additionally, in this case in which the samples were cored from existing concrete and allowed to dry, any change in the original concrete structure, sample location or drying time may influence results. The largest and perhaps the most significant difference in performance was observed in freeze-thaw testing. The treated specimens had an average durability factor of 41 after 300 cycles while the untreated specimens failed after 166 cycles with roughly half the durability factor compared to the treated specimens. This may indicate significantly better freeze–thaw performance in the field compared to the control specimens.

POINT OF CONTACT: For additional information, contact Haley Mims (haley.m.mims@usace.army.mil). This technical note should be cited as follows:

> Mims, Haley. 2024. *Concrete Testing for MTC: Oxocrete™ Surface Treatment*. ERDC/GSL TN-24-1. Vicksburg, MS: US Army Engineer Research and Development Center, Geotechnical and Structure Laboratory.

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