

# **THE EFFECT OF FREEZE/THAW CYCLES ON THE PERFORMANCE AND MICROSTRUCTURE OF CEMENT-TREATED SOILS**

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## **Abstract**

In this paper, the performance and structural changes in cement-treated soils under influence of freeze/thaw (f/t) exposure are investigated. Specimens from plastic and compacted soil-cement mix designs were exposed to different f/t scenarios to study the influence of f/t dimensionality (i.e., one-dimensional vs three-dimensional exposure) and specimens' age at the time of f/t exposure on changes in their performance. Changes in hydraulic conductivity, unconfined compressive strength, and longitudinal resonant frequency of the specimens were studied under each exposure scenario. An examination of the microstructure of the f/t exposed and control specimens using transmitted light optical microscopy was also performed to evaluate how the soil-cement matrix was disrupted after exposure to f/t cycling. Observations showed increases in water content of the mix design (when wet of optimum water content), as well as increased specimen age at the time of exposure may increase f/t susceptibility. Contrary, comparison of the performance of the specimens exposed to one-dimensional and three-dimensional f/t exposure did not show any significant variation. Microstructural analysis of petrographic thin section samples from control and f/t exposed specimens showed that while optical microscopy can detect matrix disintegration for highly damaged specimens, it is not able to identify structural degradation at early stages of damage development.

1    **INTRODUCTION**

2    The treatment of soil with Portland cement is usually carried out to improve geotechnical  
3    performance in applications such as for use in the base layer for pavements, slope  
4    protection, and water retention systems. Treatment in this way allows a soil to meet  
5    performance specifications such as strength and hydraulic conductivity (ACI, 1990).  
6    Portland cement is also used in cement-based solidification/stabilization (S/S), a source-  
7    control remediation technique, where it is mixed with contaminated materials to mitigate  
8    the release of the contaminants to the surrounding environment (ITRC, 2011). As soils and  
9    contaminants are very heterogeneous and potential engineering application numerous, a  
10   wide variety of mix designs incorporating cement can be formulated.

11   Engineered cement-treated S/S systems are expected to maintain their structural integrity  
12   for decades (PASSiFy, 2010). However, it is suggested that under environmental exposure,  
13   similar factors that influence the long term performance of concrete (e.g. chemical attack,  
14   wet/dry (w/d) cycles, and freeze/thaw (f/t) exposure) may initiate these same degradation  
15   processes in S/S systems (Klich et al., 1999). During the design stage of cement-treated  
16   materials, w/d and f/t exposure tests have been routinely used to examine their durability  
17   (e.g., ASTM-D559 (1996) and ASTM-D560 (2003) (both withdrawn in 2012)). However,  
18   comparing the results of the laboratory investigations for cement-treated materials exposed  
19   to standard w/d and f/t cycles in the literature (e.g. Felt, 1955; Al-Tabbaa & Evans, 1998;  
20   Shihata & Baghdadi, 2001) suggest that f/t exposure may generally be a dominant factor  
21   controlling the durability of soil-cement materials.

22 The influence of f/t exposure upon the integrity and mechanical properties of compacted  
23 and plastic soil-cement has been widely studied in the literature (e.g. Felt, 1955; Dempsey  
24 & Thompson, 1973; Kettle, 1986; Pamukcu et al., 1994; Shihata & Baghdadi, 2001).  
25 Jamshidi and Lake (2015) and Jamshidi et al. (2015a) conducted extensive laboratory  
26 investigations focusing on changes in the hydraulic performance of cement-treated soils  
27 after f/t exposure. Increases of up to three orders of magnitude as well as minor decreases  
28 in the hydraulic conductivity values were observed after 12 cycles of f/t exposure. Both  
29 reductions and increases in UCS values were also reported in these studies. However, UCS  
30 changes did not correspond to the variations of hydraulic conductivity values obtained  
31 within each mix design. Jamshidi et al. (2015b) suggested that the impact resonance (IR)  
32 method may be a reliable non-destructive tool in predicting the potential trends in hydraulic  
33 conductivity changes of cement-treated soils subjected to cycles of f/t.

34 The majority of laboratory studies (e.g. ASTM-D560, 2003) evaluating f/t resistance  
35 subject the soil-cement to three-dimensional freezing, as opposed to a more realistic one-  
36 dimensional exposure scenario expected in the service environment. The influence of one-  
37 dimensional freezing on mechanical properties of soil-cement has been previously  
38 investigated (e.g. Dempsey & Thompson, 1973). However, possible correlation of the  
39 results between one- and three-dimensional exposure scenarios, do not appear to exist.  
40 Also, variations in the hydraulic performance of soil-cement under one-dimensional  
41 freezing conditions have not been addressed in the literature. In addition, a systematic  
42 examination of the micro/macro-structural changes in soil-cement materials undergoing  
43 freeze-thaw in this regard is lacking.

44 The current study examines the influence of f/t cycles on two mix designs employing  
45 cement to treat silty sand; representing compacted and plastic soil-cement conditions.  
46 Although it has been shown by Jamshidi et al. (2015a) and Jamshidi et al. (2015b) that  
47 damage may progress as the number of f/t cycles increases, a significant portion of the  
48 damage was shown to occur in the initial cycles of f/t. Given that the mechanism of the  
49 initiation of this damage is of interest, three cycles of f/t were performed for this study.  
50 Three-dimensional f/t exposure was performed upon immature samples (aged 16 days) and  
51 mature samples (aged over 110 days). Mature specimens from each mix design were also  
52 exposed to a one-dimensional freezing process. Hydraulic conductivity, unconfined  
53 compressive strength, and longitudinal resonant frequency (RF) measurements were  
54 performed on control and f/t exposed specimens for each of the exposure scenarios  
55 described above. Transmitted light microscopy was also conducted on petrographic thin  
56 sections obtained from specimens exposed to the various exposure and freezing scenarios,  
57 to examine potential mechanisms of degradation.

## 58 **MATERIALS AND METHODS**

### 59 **Soil Materials Used For Soil-Cement Mixes**

60 Selected size fractions from two soils (known as soil A and soil B) were blended to create  
61 the soil used in this study (i.e., referred to as soil SIII in table 1). Soil A consisted of a  
62 glacially-derived silty-sand (ASTM-D2487, 2011) and Soil B consisted of the fine-grained  
63 by-product of a quarry operation. Both soils were collected in Nova Scotia, Canada. An  
64 analysis performed on soil A and B indicate silica, aluminum, potassium, sodium, and iron  
65 as the major oxides present (Table 2). Quartz and feldspars were also identified as the main

66 crystalline phases in both soils based on an X-ray diffraction analysis shown in Figure 1.  
67 Standard proctor tests (ASTM-D558, 2011) performed on the blended soils mixed with  
68 10% by weight Portland-limestone blended cement (CSA type GUL) showed an optimum  
69 water content (OWC) of approximately 11 percent and a maximum dry density of  
70 approximately 1975 kg/m<sup>3</sup>.

### 71 **Soil-Cement Specimen Preparation**

72 The two mix designs used in the study were chosen to represent compacted soil-cement  
73 prepared at near OWC conditions (i.e. SIII(1.2) specimens (soil SIII stabilized at a  
74 water/cement (w/c) ratio of 1.2) in Table 1), and plastic soil-cement prepared at slightly  
75 over eight percent above OWC conditions (i.e. SIII(2.1) specimens (soil SIII stabilized at  
76 a w/c ratio of 2.1) in Table 1). To prepare the specimens from each mix design, the soil,  
77 cement and water were mixed using a drill-mounted paddle at the proportions presented in  
78 Table 1. SIII(1.2) specimens were compacted in standard proctor molds in three layers in  
79 accordance with ASTM-D558 (2011). SIII(2.1) specimens were placed into cylindrical  
80 plastic molds (101 mm in diameter and 118 mm in height) in three layers, each layer being  
81 tamped with 20 blows of a standard concrete slump testing rod to provide the required  
82 consolidation. After placement, the molds were placed in sealed plastic bags for five days,  
83 when specimens were extruded and kept in a 100% humidity moist room for further curing.

### 84 **F/T Conditioning of Specimens**

#### 85 *Immature- vs. Mature-Exposure*

86 Jamshidi and Lake (2015) showed that mature specimens exposed to 12 f/t cycles exhibit  
87 higher amounts of damage compared to immature conditions. Non-destructive  
88 examinations performed by Jamshidi et al. (2015a) and Jamshidi et al. (2015b) also showed  
89 that a significant portion of the degradation during f/t exposure occurred in the initial f/t  
90 cycles. Considering these observations, in the current study, immature and mature  
91 specimens from each mix design (i.e., specimens cured for 16 days and over 110 days,  
92 respectively) were exposed to three f/t cycles. The time period of 16 days was chosen based  
93 on the work of Jamshidi et al (2015a) while the time period of 110 days was chosen to  
94 reflect a curing time in which the majority of cement hydration would be complete,  
95 minimizing further structural changes in soil-cement structure. Each f/t cycle consisted of  
96 24 hours of three-dimensional freezing at  $-10\pm 1^{\circ}\text{C}$  followed by thawing in a 100%  
97 humidity room at a temperature of  $22\pm 1^{\circ}\text{C}$ . All specimens were saturated in a set-up  
98 presented in method A of ASTM-D5084 (2010) under a minimum back-pressure of 524  
99 kPa and a confining pressure of over 558 kPa for a duration of at least seven days before  
100 f/t exposure. Previous work by Goreham et al. (2012) showed how for similar cement  
101 contents,  $\beta$  -value measurements were greater than 95% for the saturation conditions  
102 employed in this work.

### 103 ***One- vs. Three-Dimensional Specimen Freezing***

104 Ground freezing occurs due to boundary conditions created by sub-zero air temperatures.  
105 Heat flux occurs one-dimensionally, resulting in progressive downward freezing of the soil.  
106 In the current study, mature specimens from both mix (i.e., SIII(1.2) and SIII(2.1)) designs  
107 were exposed to three cycles of one-dimensional freezing followed by thawing in the 100%

108 humidity room. The changes in the performance of these specimens were then compared  
109 to similar specimens exposed to three-dimensional f/t.

110 A test set-up shown in Figure 2 was used to one-dimensionally freeze duplicate specimens  
111 from each mix design. Specimens were placed in a plexiglass tube with a wall thickness of  
112 1.5 cm covered with approximately 1.2 cm of fibreglass insulation. One-dimensional flow  
113 of temperature during the freezing process was confirmed by Mcknight-Whitford (2013)  
114 in a similar test setup. To provide intimate contact between specimens and the plexiglass  
115 tube walls (i.e. the interior surface of plexiglass tubes), layers of plastic wrap were placed  
116 around each specimen and the final surface was coated with vacuum grease prior to the  
117 placement in the tubes. A dummy sample, having a similar mix design to specimens being  
118 tested, with a height of approximately 5 cm was placed underneath each specimen to  
119 facilitate achieving sub-zero temperatures at the bottom of the specimens during the  
120 freezing process. A mixture of ice and water was used to maintain the temperature at the  
121 base of the dummy sample at 0°C. A compact refrigerated circulator (i.e. HAAKE DC30  
122 liquid chiller with a K10 bath) was used to circulate a mix of water and antifreeze at a  
123 temperature of  $-10\pm 1^{\circ}\text{C}$  through the stainless steel caps placed on top of each of the  
124 duplicate specimens. The whole set-up was placed in a room with an ambient temperature  
125 of  $3\pm 1^{\circ}\text{C}$  to provide the required working conditions for the compact refrigerated  
126 circulator. Thermocouples were placed between the specimens and the dummy samples to  
127 monitor the temperature changes at the base of the specimens and to ensure complete  
128 freezing of the specimens in order to provide proper comparison to the three-dimensional  
129 freezing conditions. Each freezing phase lasted for three days, after which specimens were



130 extruded and placed in a moist room for approximately 24 hours for complete thawing.  
131 This process was repeated for a total of three f/t cycles.

## 132 **Specimen Testing**

133 Hydraulic conductivity, unconfined compressive strength (UCS), and longitudinal  
134 resonance frequency (RF) measurements were performed on the control and exposed  
135 specimens to monitor the changes in the performance of each mix design under different  
136 exposure scenarios and freezing conditions. Transmitted light microscopy was also  
137 performed on petrographic thin sections obtained from control and f/t exposed specimens  
138 in order to provide information regarding the microstructural changes and damage  
139 propagation mechanisms under various exposure conditions. A summary of the testing  
140 procedures used in the study follows.

### 141 ***Hydraulic Conductivity***

142 Duplicate specimens were tested for hydraulic conductivity values before (i.e. control) and  
143 after three cycles of f/t exposure. General guidelines suggested in method A of ASTM-  
144 D5084 (2010) (i.e. constant head flexible-wall method) were followed during each test.  
145 Test duration ranged from 3 days to 2 weeks depending on the hydraulic conductivity of  
146 the specimens. Permeation was performed under a hydraulic gradient ranging from 29 to  
147 60. The test was continued until a steady hydraulic conductivity value was achieved as  
148 described in the standard.

149

150 ***Impact Resonance (IR)***

151 Vibration-based non-destructive techniques have been used for structural health  
152 monitoring purposes in various applications (e.g. Nagy, 1997; Sansalone, 1997; Shah et  
153 al., 2000; Jin & Li, 2001; Gheorghiu et al., 2005). Since resonant frequency (RF) of a  
154 material measured using these techniques is related to its physical properties including  
155 density, shape, and the dynamic modulus of elasticity (Malhotra, 2011), it can be used as a  
156 reliable indicator for monitoring the changes in the material due to external stresses.  
157 Jamshidi et al. (2015b) showed that the IR method, as an example of vibration-based non-  
158 destructive techniques, can be used for early detection of damage in cement-treated soils  
159 during exposure to cycles of f/t.

160 In the current study, longitudinal resonance frequency (RF) of specimens was measured  
161 using the IR method, before f/t exposure and subsequently at the end of the thawing phases  
162 of cycles 1, 2, and 3. The tests followed the general procedures presented in ASTM-C215  
163 (2008). Impact load was generated using a steel ball with a diameter of 9.5 mm attached to  
164 a plastic band. An accelerometer (PCB model 352C68) and a Freedom Data PC Platform  
165 (Olson Instruments Inc.) were used for the data acquisition and signal processing. A  
166 bandwidth filter of 500-15000 Hz, sampling rate of 500 KHz, and record size of 8192  
167 samples were used during the test. Each test consisted of five trials on the specimen with  
168 the average values being reported as the RF.

169

170

171 ***Unconfined Compressive Strength (UCS)***

172 Unconfined compressive strength (UCS) measurements were performed on duplicate  
173 specimens for the control and f/t exposed conditions. Sulfur-capped specimens were  
174 subjected to a vertical deformation rate of 0.5 mm/min during the loading process.

175 ***Transmitted Light Optical Microscopy***

176 A study of the microstructural changes in the exposed specimens using petrographic  
177 methods can provide an insight on the mechanisms of damage formation during f/t  
178 exposure. Petrographic thin section samples were prepared by a specialized laboratory at  
179 the Geology and Earth Sciences Department of Dalhousie University. Thin sections were  
180 taken from horizontal and vertical planes of resin impregnated samples from the control  
181 and exposed specimens. An examination of the thin sections was performed using a Nikon  
182 Optiphot-Pol polarized light microscope equipped with a 12 megapixel digital scanning  
183 camera (Kontron ProgRes 3012).

184 **RESULTS AND DISCUSSION**

185 A summary of the hydraulic conductivity, UCS, and resonant frequency results obtained  
186 from the study is presented in Table 3. Below is a discussion of these results as well as  
187 microstructural changes observed from the thin section samples using transmitted light  
188 microscopy.

189

190

## 191 **Hydraulic Conductivity**

192 Figure 3 shows the influence of water content in the mix design on hydraulic conductivity  
193 of the treated soil under both immature and mature curing conditions (i.e. without f/t  
194 exposure). Results from measurements presented in Jamshidi and Lake (2015) on a similar  
195 soil at different water contents are also presented in Figure 3 to better demonstrate the  
196 variations with respect to OWC conditions. The results show that the lowest hydraulic  
197 conductivity can be achieved by preparing mix designs slightly wetter than OWC  
198 conditions. Hammad (2013) previously showed that for specimens cured for 28 days, the  
199 lowest hydraulic conductivity occurred at a water content ranging from 2 to 6 percent above  
200 the standard proctor OWC conditions for a similar soil. It is hypothesized that this water  
201 content range produced the lowest hydraulic conductivity because, similar to compacted  
202 clay soils (Mitchell et al., 1965; Benson and Daniel, 1990), the samples slightly wet of  
203 optimum produced a more dispersed structure and a resulting soil kneading action during  
204 compaction at this moisture level (i.e. near its plastic limit). The resulting fabric is more  
205 aligned in a direction perpendicular to flow and any clods are kneaded during the  
206 compaction process. Figure 3 shows that hydraulic conductivity values increase at a slow  
207 “rate” as water content is increased from near OWC (11%) up to a moisture content of  
208 18%, after which the hydraulic conductivity seems to be very sensitive to a one percent  
209 increase in moisture content (i.e. 19%). This is possibly due to the excessive bleeding of  
210 the water during preparation of mixtures at high water contents creating high porosity areas  
211 within the final structure.

212 Hydraulic conductivity values also seem to be sensitive to water content changes when  
213 below OWC conditions. Approximately a three to four orders of magnitude increase in  
214 hydraulic conductivity is observed as the water content during compaction of the mix  
215 design is decreased from a value of 11% (i.e., near OWC conditions) to 9% (slightly below  
216 OWC conditions). This increase is likely due to a combination of less water available for  
217 hydration and/or insufficient “lubrication” of soil and cement particles during the  
218 compaction process to assist with the “kneading” action (Mitchell et al., 1965; Benson and  
219 Daniel, 1990). Figure 3 also shows a decrease of as much as two orders of magnitudes in  
220 the hydraulic conductivity values between immature and mature measurements as a result  
221 of the progression of the hydration process in the specimens.

222 Changes in the hydraulic conductivity values for SIII(1.2) and SIII(2.1) specimens obtained  
223 under various f/t exposure conditions are presented in Figure 4. After three cycles of three-  
224 dimensional f/t exposure, a wide range of changes in hydraulic conductivity values was  
225 observed which appears to be dependent on the curing conditions and mix design. For the  
226 SIII(1.2) mix design, immature specimens showed a slight reduction in hydraulic  
227 conductivity values, while mature specimens exhibited an increase of about one order of  
228 magnitude. Also of note is that the hydraulic conductivity values after f/t exposure, even  
229 though they have undergone an increase, remain low (less than  $10^{-10}$  m/s). Under similar  
230 conditions, immature and mature specimens from SIII(2.1) showed increases of about 12  
231 and 320 fold in the hydraulic conductivity values, respectively. Increase in hydraulic  
232 conductivity values after f/t exposure is likely a result of crack initiation and subsequent  
233 structural degradation due to expansion of pore water in soil-cement during the freezing  
234 process.

235 The theory of damage development in cement-based materials during f/t exposure is  
236 discussed in previous research studies (e.g. Powers, 1945; Setzer, 2001). Conversely, the  
237 decrease in hydraulic conductivity values after f/t exposure of immature SIII(1.2) is likely  
238 due to the interaction of the continuing hydration of cement, as well as various healing  
239 mechanisms (e.g. mineral precipitation) acting in parallel with the deleterious effect of the  
240 f/t process. Hydraulic conductivity recovery of damaged soil-cement specimens after post-  
241 exposure curing was also reported in Jamshidi and Lake (2015).

242 Examination of one-dimensional freezing conditions compared to three-dimensional  
243 freezing conditions has been conducted in the literature by Othman & Benson (1993) for  
244 compacted clay soils. Othman & Benson (1993) showed that f/t dimensionality has  
245 negligible influence on the changes in the hydraulic performance and on the crack  
246 formation pattern in compacted clay specimens. Comparing the hydraulic conductivity  
247 changes in mature specimens exposed to one- and three-dimensional freezing conditions  
248 in the current study (Figure 4) also shows changes in the hydraulic conductivity values are  
249 within the same order of magnitude for both freezing scenarios.

### 250 **Longitudinal Resonant Frequency (RF)**

251 Figure 5 presents the RF values measured on specimens at control conditions (i.e. cycle 0)  
252 and subsequently after 1<sup>st</sup>, 2<sup>nd</sup>, and 3<sup>rd</sup> f/t cycles. Values obtained for control specimens  
253 show mature specimens exhibit higher RF values compared to immature specimens, which  
254 is a result of increased structural integrity due to longer curing. Also, both immature and  
255 mature SIII(1.2) specimens show higher RF values compared to SIII(2.1) specimens.  
256 Higher RF values suggest a higher stiffness of these specimens. Values are also in

257 agreement with lower hydraulic conductivity measurements presented in the previous  
258 section.

259 For SIII(1.2)-immature specimens, RF values were relatively constant (slightly over 12000  
260 Hz) through different f/t cycles, which suggests minor structural changes. This was in  
261 agreement with the slight reductions in hydraulic conductivity values of these specimens  
262 after f/t exposure. For SIII(1.2) mature specimens exposed to three-dimensional f/t, RF  
263 values showed over 1000 Hz reduction after the initial cycle and reached a value of about  
264 10800 Hz (i.e. a total decrease of approximately 2600 Hz) at the end of the third cycle.  
265 Reductions in the RF values were in agreement with the increases observed in the hydraulic  
266 conductivities of these specimens (see Figure 4). For SIII(1.2) mature specimens exposed  
267 to one-dimensional freezing conditions, RF values showed only minor variations over three  
268 f/t cycles, which is in contrast to approximately five times increase observed in their  
269 hydraulic conductivity values. However, as noted previously, the final hydraulic  
270 conductivity values are still lower than  $10^{-10}$  m/s, suggesting that the specimens are still  
271 relatively intact.

272 For SIII(2.1)-immature and SIII(2.1)-mature specimens, average RF values dropped from  
273 8300-9600 Hz to values close to 2000 Hz after three cycles of f/t. This extensive drop in  
274 RF values is consistent with several orders of magnitude change in hydraulic conductivity  
275 for this mix, both observations suggesting a significant change in structure within the soil-  
276 cement specimens.

277 For the mature specimens tested from SIII(2.1) mix design, variations of results for one-  
278 and three-dimensional freezing conditions were insignificant. Also, irrespective of

279 exposure scenario, the initial cycle had a significant effect in the degradation of the  
280 specimens, resulting in over 38 percent reduction in the RF values.

### 281 **Unconfined Compressive Strength (UCS)**

282 Trends in the UCS values for the treated soil at w/c ratios ranging from 1 (below OWC) to  
283 2.1 is shown in Figure 3. Data presented in Figure 3 include both measurements performed  
284 in this study and UCS measurements presented in Jamshidi and Lake (2015) on a similar  
285 soil treated at different w/c ratios. Maximum UCS values appear to correspond with the  
286 OWC, with values decreasing at a higher rate above OWC. Observations by Felt (1955)  
287 previously showed that for non-plastic soils the maximum UCS occurs slightly below the  
288 OWC obtained from the standard proctor test.

289 Figure 6 presents the results of UCS testing for SIII(1.2) and SIII(2.1) specimens after three  
290 cycles of f/t exposure. On average, SIII(1.2) specimens showed a general increasing trend  
291 (with a maximum increase of about six percent) in UCS values for both immature and  
292 mature exposure conditions. SIII(2.1)-immature showed an average reduction of 17 percent  
293 after f/t exposure while SIII(2.1)-mature remained relatively unchanged after three-  
294 dimensional f/t exposure. A 19 percent reduction was observed for similar specimens  
295 exposed to one-dimensional freezing. Comparing the UCS measurements to the  
296 observations on hydraulic conductivity changes for similar mix designs, it is apparent that  
297 UCS is not a suitable indicator of the changes in the hydraulic performance of cement-  
298 treated soils. For instance, it is possible that micro-crack formations during the freezing  
299 process can lead to increases in the hydraulic conductivity values, while under similar  
300 conditions the compressive forces applied during the UCS test can cause the micro-cracks



301 to contribute to the strength of the specimen as a result of friction development on the crack  
302 walls.

### 303 **Optical Microscopy**

304 An examination of the microstructure of the cement-treated soil using transmitted light  
305 optical microscopy was performed to examine how the soil-cement matrix was disrupted  
306 after exposure to f/t cycling. Comparison of the micrographs between the horizontal and  
307 vertical planes obtained from control and f/t exposed specimens did not suggest any  
308 noticeable spatial variation in the soil-cement structure and the damage formation  
309 mechanisms. Typical micrographs obtained from specimens under control and three-  
310 dimensional f/t exposed conditions are presented in Figure 7. The blue color in the  
311 micrographs presented in Figure 7 represents the pore structures that were filled with the  
312 resin used during the impregnation of the samples prior to thin section preparation.

313 The soils used in the study appear to be “young”, in the respect that they contain minerals  
314 associated with the disintegration/degradation of igneous and metamorphic rocks. These  
315 minerals (which are relatively thermodynamically unstable), form angular, coarser clasts.  
316 Minerals identified in the coarser fraction include microcline, plagioclase, ferromagnesian  
317 minerals, and micas. Some of the clasts present in the soil exhibit signs of degradation,  
318 including micro-cracking, suggesting that prior to use in the study, they were subject to  
319 mechanical or environmental loading (see images A, B, and G in Figure 7).

320 Discrete incidents of high porosity areas were observed in control samples from both  
321 SIII(1.2) and SIII(2.1) mix designs (see patches of blue color in images C and G in Figure

322 7). These features were likely induced during the placement of the specimens as a result of  
323 poor compaction and/or localized high water content areas in the matrix. It should be noted  
324 that the images presented in Figure 7 are in scales of millimeters and hence there were  
325 hundreds of potential images. Those presented in Figure 7 are considered typical based  
326 upon visual observations of the entire thin sections.

327 Considering the control conditions in Figure 7, the microstructure in samples from SIII(2.1)  
328 with higher w/c ratio, appears less dense (see images A and C in Figure 7), especially at  
329 the matrix/aggregate interface. SIII(1.2) samples prepared at lower w/c ratio have a denser  
330 packing and a less intense blue color (see images E and G in Figure 7).

331 Under the exposed conditions, in the thin sections from SIII(2.1)-immature specimens  
332 ( $K_{\text{exposed}}/K_0 \approx 12$ ), minor matrix disruptions in scattered areas throughout the paste were  
333 noticeable as is shown in micrographs presented in Figure 8. For the same mix design under  
334 mature three-dimensional f/t exposure (i.e. SIII(2.1)-mature with a  $K_{\text{exposed}}/K_0 \approx 320$ ), more  
335 evidence of matrix disruption as well as some micro-cracking along the paste-aggregate  
336 interface were observed (Figure 8). Damage in the thin sections from SIII(2.1)-mature was  
337 also evident by comparing the thin sections even at a larger scale as is shown in Figure 9  
338 (note the increased intensity of blue color in exposed sample as compared to the control  
339 sample). It can also be seen in Figure 9 that damage formation in soil-cement, at least at  
340 the high cement content used in this study, doesn't follow the mechanisms suggested for  
341 compacted clay (i.e. parallel ice lens formation), which is likely a result of higher tensile  
342 strength of these materials due to the binding capacity of cement hydration products.

343 Considering SIII(1.2)-immature samples, the damage was less evident, although some  
344 disruption of the matrix was observed (Figure 10). This was despite the reduction of the  
345 hydraulic conductivity values in this mix design after f/t exposure. This reduction is likely  
346 a result of the decreased hydraulic conductivity of the paste due to the hydration process  
347 and the isolation of the disrupted areas by unaffected materials. In SIII(1.2)-mature,  
348 although approximately ten times increase in the hydraulic conductivity values were  
349 observed after three-dimensional f/t exposure, only minor scattered degradation of the  
350 matrix were observed during the analysis (Figure 10). It should be again noted that although  
351 increases in hydraulic conductivity were observed, the final hydraulic conductivity value  
352 of the SIII(1.2) mixtures was less than  $10^{-10}$  m/s and hence tend to agree with the  
353 microscope observations. Similar to the SIII(2.1) mature mix samples, no ice lens  
354 formation was observed.

355 Microstructural analysis of one-dimensionally exposed specimens did not reveal any  
356 obvious differences in the mechanisms of f/t damage compared to three-dimensional  
357 exposure. More damage (in terms of matrix-aggregate interface cracking and matrix  
358 disruption) was observed in the micrographs obtained from SIII(2.1) specimens (i.e. one-  
359 dimensional f/t), with higher hydraulic conductivity changes. This was compared to minor  
360 structural changes in the SIII(1.2) specimens with slight increases in the hydraulic  
361 conductivity values (Figure 11).

362 Results of the microstructural analysis performed show that there are obvious limitations  
363 on sample observation using transmitted light microscopy. The degree of microstructural  
364 disturbance between samples and within individual samples is not as obvious as

365 anticipated, even though other test methods showed a reduction in structural integrity for  
366 most of the scenarios investigated. Thus, the use of transmitted light microscopy may not  
367 be able to resolve the early stages of (sub-microscopic) matrix disruption, whereas the other  
368 indirect analytical methods (i.e, hydraulic conductivity, UCS, and RF measurements)  
369 employed have this capability. However, it was established that microstructural damage  
370 was mainly in the form of matrix disruption; cracking within the matrix, and cracking at  
371 aggregate boundaries.

## 372 **CONCLUSIONS**

373 Cement-treated silty sand specimens prepared at two w/c ratios representing compacted  
374 and plastic soil-cement mix designs were exposed to three cycles of freeze/thaw (f/t).  
375 Various sample ages (i.e. immature vs. mature) and f/t dimensionality (i.e. one-dimensional  
376 vs. three-dimensional) scenarios were investigated. Changes in performance of the  
377 specimens were monitored using hydraulic conductivity, UCS, and longitudinal RF  
378 measurements. Also, microstructural degradation of the specimens were evaluated by  
379 studying petrographic thin section samples obtained from the specimens, using transmitted  
380 light optical microscopy technique. The following conclusions can be drawn from the  
381 results.

- 382 1. Performance measurements on soil-cement specimens prepared over a wide  
383 range of water contents (i.e. without f/t exposure) showed that minimum  
384 hydraulic conductivity likely occurs at water contents slightly above optimum  
385 water content. In this study, hydraulic conductivity values increased at a slow  
386 “rate” as water content is increased from near OWC (11%) up to a moisture

387 content of 18%, after which the hydraulic conductivity seemed to be very  
388 sensitive (i.e. hydraulic conductivity increased) to a one percent increase in  
389 moisture content (i.e. 19%). The maximum UCS possibly occurs at water  
390 contents near or slightly below optimum water contents. Hydraulic conductivity  
391 values seem to be more sensitive (i.e. hydraulic conductivity increased) to  
392 changes in the water contents towards dryer mix designs. Approximately a three  
393 to four orders of magnitude increase in hydraulic conductivity is observed as the  
394 water content during compaction of the mix design is decreased from a value of  
395 11% (i.e. near OWC conditions) to 9% (slightly below OWC conditions). UCS  
396 values are more sensitive (i.e. decrease in UCS values) to changes in the water  
397 contents towards wetter mix designs, with respect to optimum water content  
398 conditions.

- 399 2. Specimens exposed to three f/t cycles exhibited a wide range of performance  
400 changes including minor enhancement of the performance (possibly due to the  
401 interaction of f/t degradation mechanisms with the hydration/healing processes)  
402 as well as increases in the hydraulic conductivity values and decreases in the UCS  
403 and RF values (i.e. performance degradation). Specimens prepared at higher  
404 water content (i.e. SIII(2.1)) exhibited more damage after f/t exposure compared  
405 to the dryer mix design (i.e. SIII(1.2)). Also, it was found that mature specimens  
406 are more susceptible to f/t exposure compared to immature specimens; an  
407 observation discussed in detail in Jamshidi and Lake (2015).
- 408 3. Under the testing conditions applied in the study, no significant variation in the  
409 performance of the specimens exposed to one-dimensional and three-

410 dimensional f/t exposure was observed. This may suggest while one-dimensional  
411 f/t studies better mimic the exposure mechanisms expected in the field, laboratory  
412 investigation of soil-cement using three-dimensional f/t exposure scenarios can  
413 still provide a reliable estimation of the performance degradation.

414 4. Microstructural analysis of petrographic thin section samples from control and  
415 f/t exposed specimens showed that optical microscopy is not able to identify  
416 structural degradation at early stages of damage development, suggesting  
417 presence of possible sub-microstructural disruption mechanisms. For highly  
418 damaged specimens (i.e. in terms of hydraulic conductivity changes), however,  
419 signs of matrix disintegration as well as matrix-aggregate interface cracking were  
420 identified.

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Table 1: Summary of the mix designs utilized for SIII(1.2) and SIII(2.1).

| Mix designation | W/c ratio | Cement content, percent (dry weight of soil) | Mixing method* | Composition of the soil used (i.e. soil SIII) by dry weight, percent |              |              |              |                 | ASTM classification of the blended soil |
|-----------------|-----------|--|----------------|--|--------------|--------------|--------------|-----------------|---|
|                 |           |  |                | Soil A   |              |              |              | Soil B <0.08 mm |   |
|                 |           |  |                | 9.50-4.75 mm   | 4.75-1.20 mm | 1.20-0.30 mm | 0.30-0.08 mm |                 |   |
| SIII(1.2)       | 1.2       | 10   | C              | 9  | 30           | 21           | 10           | 30              | Silty sand                              |
| SIII(2.1)       | 2.1       | 10   | S              |  |              |              |              |                 |   |

\*C: compaction, S: self-consolidation.

Table 2. Mineralogical composition of soil A and soil B.

|        | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | K <sub>2</sub> O | Na <sub>2</sub> O | Fe <sub>2</sub> O <sub>3</sub> | LOI (1000°C)* | Other |
|--------|------------------|--------------------------------|------------------|-------------------|--------------------------------|---------------|-------|
| Soil A | 71.82            | 14.57                          | 3.27             | 3.03              | 2.66                           | 2.41          | 2.24  |
| Soil B | 65.65            | 15.31                          | 4.02             | 3.08              | 5.66                           | 1.17          | 5.11  |

\*LOI: Loss on ignition

Table 3: Summary of performance test results under control and exposed conditions.

| Test               |          | Hydraulic conductivity, m/s<br>(Relative standard deviation, %) |                                 |                                 |                                | UCS, MPa<br>(Relative standard deviation, %) |               |         |               | Longitudinal resonance frequency, kHz<br>(Relative standard deviation, %) |               |               |               |               |               |               |               |
|--------------------|----------|---|---------------------------------|---------------------------------|--------------------------------|--|---------------|---------|---------------|---|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
|                    |          | 3D  |                                 | 1D                              |                                | 3D   |               | 1D      |               | 3D  |               |               |               | 1D            |               |               |               |
| Exposure condition |          | Control   | Exposed                         | Control                         | Exposed                        | Control                                      | Exposed       | Control | Exposed       | Control   | Cycle 1       | Cycle 2       | Cycle 3       | Control       | Cycle 1       | Cycle 2       | Cycle 3       |
| SIII(1.2)          | Immature | 3.8×10 <sup>-11</sup><br>(38.7)                                 | 3.5×10 <sup>-11</sup><br>(36.2) | -                               | -                              | 10.6<br>(4.0)                                | 10.8<br>(0.5) | -       | -             | 12.4<br>(1.5)   | 12.3<br>(1.2) | 12.2<br>(1.2) | 12.4<br>(2.1) | -             | -             | -             | -             |
|                    | Mature   | 5.9×10 <sup>-12</sup><br>(10.2)                                 | 5.7×10 <sup>-11</sup><br>(68.4) | 2.0×10 <sup>-12</sup><br>(18.0) | 1.1×10 <sup>-11</sup><br>(5.3) | 11.2<br>(0.7)                                | 11.7<br>(7.6) | -       | 11.9<br>(9.1) | 13.4<br>(1.4)   | 12.0<br>(4.9) | 12.2<br>(4.8) | 10.8<br>(6.3) | 12.8<br>(0.5) | 12.6<br>(1.0) | 12.7<br>(0.4) | 12.6<br>(0.6) |
| SIII(2.1)          | Immature | 3.4×10 <sup>-9</sup><br>(11.8)                                  | 4.0×10 <sup>-8</sup><br>(19.0)  | -                               | -                              | 3.0<br>(3.3)                                 | 2.5<br>(4.0)  | -       | -             | 8.4<br>(2.2)  | 4.8<br>(7.6)  | 3.3<br>(1.9)  | 2.5<br>(2.4)  | -             | -             | -             | -             |
|                    | Mature   | 2.1×10 <sup>-10</sup><br>(2.4)                                  | 6.8×10 <sup>-8</sup><br>(48.2)  | 2.9×10 <sup>-10</sup><br>(1.8)  | 2.4×10 <sup>-7</sup><br>(60.3) | 3.7<br>(10.5)                                | 3.6<br>(6.7)  | -       | 3.0<br>(22.6) | 9.6<br>(1.6)  | 4.6<br>(3.3)  | 3.0<br>(1.0)  | 2.0<br>(1.5)  | 9.9<br>(0.3)  | 6.1<br>(1.0)  | 3.6<br>(11.1) | 2.6<br>(52.3) |

