

1 **Evaluation of frost heave and moisture/chemical migration**
2 **mechanisms in highway subsoil using a laboratory simulation**
3 **method**

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7 ABSTRACT: Seasonal processes in cold countries significantly affect the engineering
8 characteristics of highway subsoil over time. Cyclical freeze-thaw leads to changes in thermal
9 and moisture conditions. As a result, road bearing capacity can progressively change from the
10 initial design. In this work, a modified laboratory method was developed, with cyclical freeze-
11 thaw of soil samples and simultaneous supply of deionised water and a de-icing agent (sodium
12 chloride) to the base. The benefits of the test procedure included slow freezing, simulating the
13 conditions that can be experienced by highway soils in cold environments, extended soil
14 column heights and a larger number of identical soil samples, which allowed experimental
15 variability to be assessed. The method included the monitoring of moisture and chemical mass
16 transfer in the soils. Samples supplied with deionised water experienced ice segregation in their
17 upper parts, and significant heave. While soils supplied with NaCl solution behaved in a similar
18 fashion during their first freeze-thaw cycle, the second cycle saw a reduction in the rate of
19 migration of the freezing front within the soils and also less ice segregation and less heave due
20 to increased salinity. Salt was preferentially transferred upwards in the soil columns as a result
21 of the thermal gradient, including negative pressure associated with cryosuction, and osmotic

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22 pressure. The new method provides a more realistic laboratory approach to assessing potential
23 freeze thaw impacts, and the effects of de-icing agents on soils beneath roads, and in different
24 settings.

25 *Keywords:* Freeze-thaw, highway subsoil, de-icing chemicals, laboratory test, sandy clay.

26 **1. Introduction**

27 Highway subsoils are subject to significant variability in terms of their moisture-temperature
28 regime, especially in countries with a severe cold climate. As highway pavements have higher
29 densities and thermal conductivities, the near surface layers freeze before, and at a faster rate
30 than roadside soils (Vasilenko, 2011; Vasilenko, 2011; Han Chunpeng *et al.*, 2010; Simonsen,
31 Janoo and Isacsson, 1997). The temperature gradient in the highway sub layers may also induce
32 a significant upwards and lateral migration of moisture supplied from the ground water table
33 (Konrad and Samson, 2000).

34 In a highways context, this groundwater may be modified by deicing agents, with a resultant
35 depression of the freezing point easily down to -25 and with more difficulty up to -41 °C,
36 depends on the chemical content (Wan, Lai and Wang, 2015). Wan et al. present a modified
37 methodology to examine this depression and the degree to which it enhances migration of soil
38 moisture towards the freezing front under the pavement structure. It also explores whether the
39 de-icing chemical migration responds to the thermal gradient in the highway subsoils. When
40 the temperature of a soil falls sufficiently, the interstitial water starts to segregate into ice
41 crystals. The remaining, unfrozen, water becomes progressively enriched with dissolved salts
42 and has a depressed eutectic temperature (Bing and He, 2011; Torrance and Schellekens, 2006).

43 Previous studies have provided contradictory evidence for the movement of de-icing agents
44 within soil: Vidyapin and Cheverev, (2008) report de-icing chemicals moving towards the

45 freezing surface, while Brouchkov, (2000) detected no obvious salt migration during the
46 freezing period. As a result, little certainty can be derived from the published literature about
47 the potential nature of frost heave in highway subsoils.

48 The nature of change in mechanical characteristics during the spring thaw is also unclear.
49 Thawing occurs both from the pavement and base of the frozen soils. However, some parts of
50 the saturated sub soils can remain frozen for some time and block the drainage of moisture
51 downwards. Localised ice lenses and associated deformation of subsoils may lead the
52 significant strains in the pavement structures due to the rapid oversaturation and the weakening
53 of sub base soils (Miller, 1972). A thorough review based on the frost heave thermodynamics
54 has been prepared by Henry (2000).

55 Numerous studies have attempted to explain the soil properties during the freeze and thaw
56 period since early 1900s (Miller, 1972; Hoekstra, 1969; Hoekstra, 1966; Taber, 1930). Most of
57 the studies have taken place under laboratory conditions to provide experimental control and
58 improved accuracy in the results (Nagare *et al.*, 2012; Bronfenbrener and Bronfenbrener,
59 2010).

60 Progressive approaches have been performed in a triaxial cell within a negative temperature
61 (Cui and Zhang, 2015; Zhang *et al.*, 2013; Hazirbaba, Zhang and Leroy Hulsey, 2011; Sinitsyn
62 and Løset, 2011; Ishikawa *et al.*, 2010; Qi, Vermeer and Cheng, 2006; Brouchkov, 2002).
63 Triaxial cell tests enable contemporaneous mechanical loading but only one sample can
64 normally be tested at a time. The size of the tested sample is also restricted by the size of the
65 test cell.

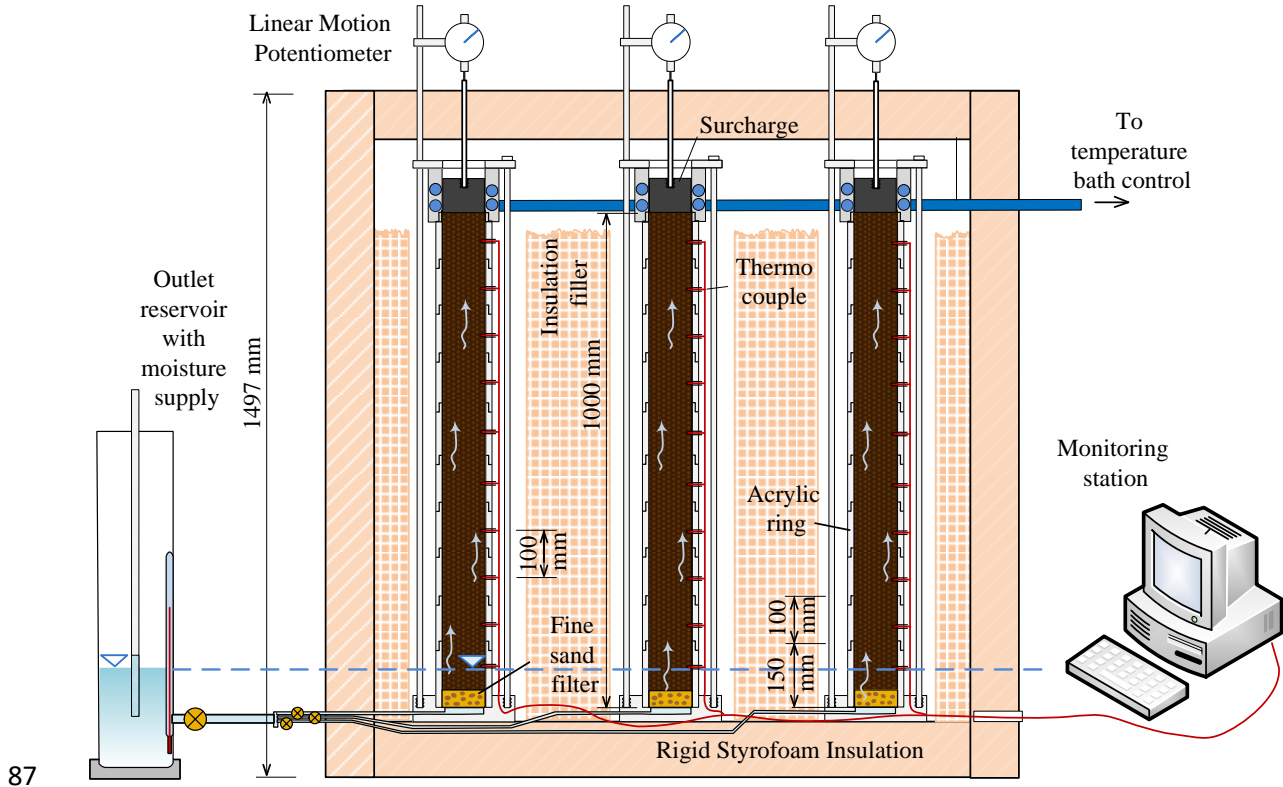
66 There are several standard methods which are used for freeze-thaw cycles of soils in different
67 countries: BS 812-124:2009, ASTM D 5918-06, GOST 28622-2012 and BS EN 1997-2:2007,

68 section 5, 5.5.10. These typically involve testing 15 cm high soil samples over a limited and
69 controlled temperature range, usually +3 and -3 °C. The number of simultaneously tested
70 samples is normally limited to four. The standard methods provide uniformity and
71 predictability of the obtained results and are suitable for classification tests. However, the
72 sample height and limited range of variables do not allow for a detailed examination of the role
73 of thermal gradient, including a descending freezing front, migration of pore water, or the
74 effects of changing soil water chemistry to be examined. Therefore, in order to better
75 understand the complex processes in the highway sub soils, a laboratory method for freeze-
76 thaw cycles with simultaneous supply of water at the base of the sample was developed.

77 **2. Materials and methods**

78 A new laboratory method with freeze-thaw cycles has been developed from the ASTM D 5918-
79 06 Standard. This allows a more realistic simulation of freeze-thaw with depth.

80 The height of the soil column was increased up to 1.00 m, including 5 cm of saturated soil at
81 the base. Water was supplied to the base through a 5 cm fine sand filter (Fig. 1). The samples
82 were made from non-saline soil. This enabled the observation of salt mass transfer from a
83 solution supplied at the base of each soil column. Application of the non-saline soils and
84 feeding the base 5 cm layer with sodium chloride solution in the new method facilitates the
85 observation of the chemical mass transfer and its possible secondary salinization in
86 consequence of the freeze-thaw of the soil mass.



87

88 **Fig. 1.** Environmental chamber for freeze-thaw cycles with 9 soil samples capacity

89 *2.1 Experimental soil characteristics*

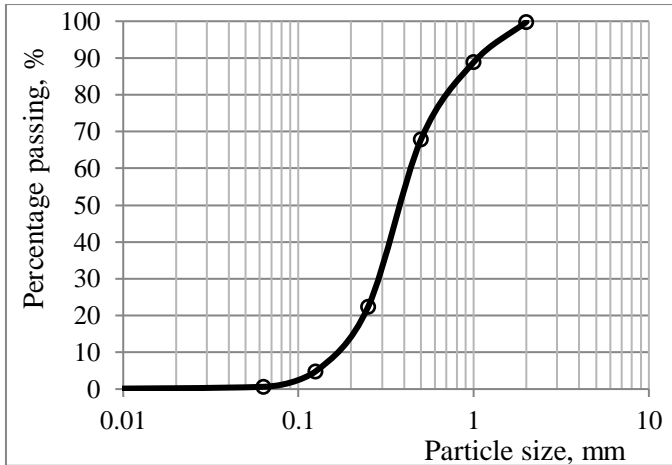
90 The soils used in this experiment were remolded on geotechnical data from Astana, Kazakhstan
 91 (Karaganda GIIZ, personal communication), where the winter air temperature can drop to
 92 below -35 °C. Kazakh Steppe soils typically consist of ancient sedimentary rocks that have
 93 been transformed by chemical and physical weathering to residual layers, together with alluvial
 94 soils of irregular thickness according to KazGIIZ geological report. To ensure comparability
 95 in laboratory tests a remolded sandy clay, reflecting frequently occurring soils 1 metre below
 96 the surface, was manufactured from 50% sand with angular shape of the grains (cross-sectional
 97 dimension less than 2 mm) and 50% kaolinite clay. A particle size distribution of the sand part
 98 in the soil sample is presented in Fig. 2. Average plastic and liquid limits were 23.77 % and
 99 37.05%, respectively. Initial classification properties of the remolded soils are presented in the
 100 Table 1.

101 Table 1 – Initial soil characteristics of soils. Soil tests followed the procedures in BS1377-
 102 1:1990. * Note Angle of internal friction is high as a result of the drained, consolidated shear
 103 test used on the soil

Characteristic	Symbol	Unit	Value	Annotation
Initial moisture content	W	%	17.2	See Fig. 5 – according to 95% max. dry density – moisture content relationship
Angle of internal friction	ϕ	°	24.1°*	CD direct shear test, moisture content $W=17.2\%$
Cohesion	C	kN/m ²	10	
Particle density of sandy clay	ρ_s	g/cm ³	2.615	Soil mixture by mass: 50% of sand and 50% of kaolinite
Average dry density before freezing cycle	ρ_{dry}	g/cm ³	1.814 ± 0.012	BS Light compaction test operating with 2.5 kg rammer. The mechanical energy applied to soil is 596 kJ/m ³
Initially bulk density at the beginning of the test	P	g/cm ³	2.128 ± 0.015	
Uniformity coefficient	C_u	-	2.4	Uniformly-graded sand
Coefficient of curvature	C_c	-	3.65	
Activity of Clays	A	-	0.25	Inactive clays
Liquid limit	w_L	%	37.05	CI – Medium plasticity Cone penetrometer test used
Plastic Limit	w_P	%	23.77	
Average linear shrinkage	L_S	%	4.8	
Plasticity Index	PI	%	13	

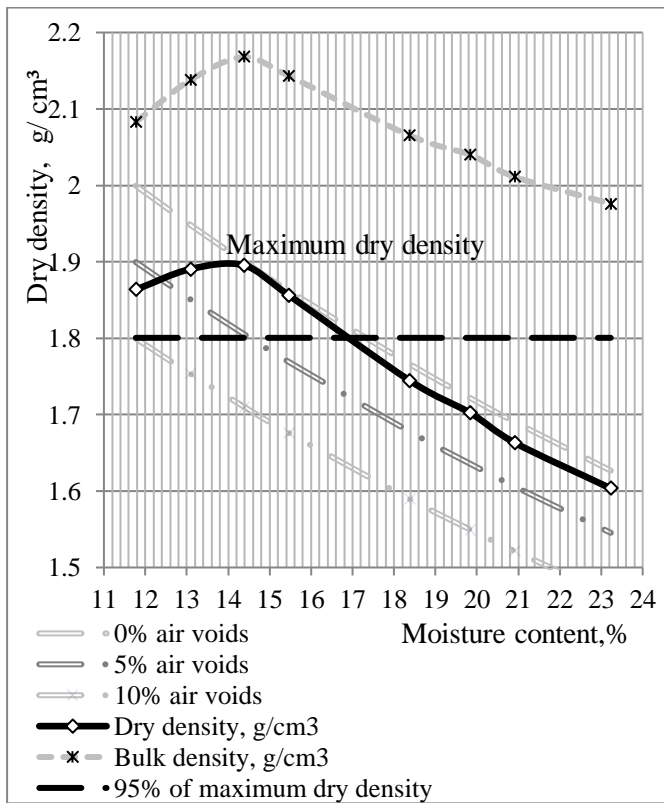
104 Table 2 - CBR results and corresponding dry density values for the manufactured soils with
 105 different moisture contents

W, %	CBR, %	Dry density, Mg/m ³
4.00	100.41	1.587
15.06	48.83	1.543
16.50	17.19	1.495
17.65	10.69	1.469
19.50	5.50	1.443
20.61	3.90	1.406
22.24	2.75	1.370



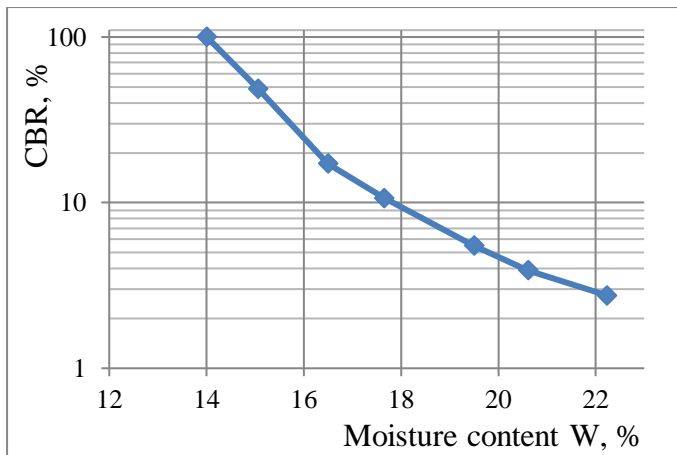
106

107 **Fig. 2.** Particle size analysis of the sand part in the remolded soils, determined by dry
 108 sieving. The graph presents only the sand part of the soil which is 50 %, the rest 50 % is a
 109 kaolinite clay.



110

111 **Fig. 3.** Dry density – moisture content curves for the remolded sandy clay. Compaction was
 112 achieved using a 2.5 kg hammer, following BS 1377-4:1990.



113

114 **Fig. 4.** California Bearing Ratio results for the remolded sandy clay.

115 The variability in dry bulk density with moisture content of the remolded soil is shown in Fig.
 116 3. The 95% value of maximum dry density is 1.8 Mg/m³, which corresponds to the w=17.2%
 117 moisture content, with the compacted soil being almost completely saturated.

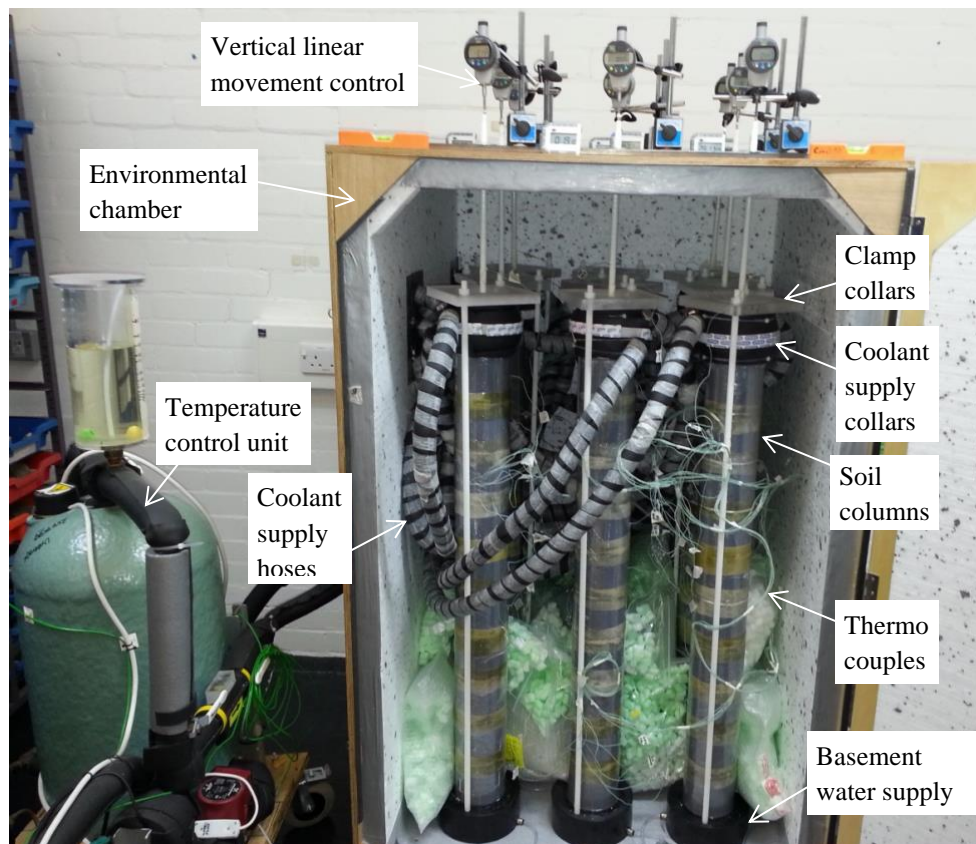
118 California bearing ratio was derived according to BS 1377-4:1990. The percentage of standard
 119 forces for penetrations of 2.5 and 5 mm were calculated. A sequence of CBR tests was
 120 conducted for a range of moisture content values (Fig. 4). The corresponding dry density and
 121 CBR values at different moisture contents are given in Table 2.

122 2.2 Sample preparation

123 The dry soil mass was mixed with 17.2% of deionised water by mass and stored for 24 hours
 124 to let the moisture distribute uniformly. Previous studies have used salinized soil from the start
 125 (e.g. Nguyen *et al.*, 2010; Arenson *et al.*, 2005). The moistened manufactured soil was then
 126 compacted within heavy duty plastic 10 cm x 10 cm cylinders using a 2.5 kg hammer to produce
 127 a dry bulk density of 1.8 Mg/m³. This was done by alternately adding 5 cm of soil and then
 128 compacting using 31-32 hammer blows, to provide the same compactive effort as in BS1377-
 129 4:1990. After filling and compaction, each stack weighed 16.784 ± 0.1 kg. The cylinder at the

130 bottom included a basal, 5 cm thick layer of fine sand to act as a filter layer. Water was supplied
131 to the base via a pipe to produce saturated conditions in the bottom 5 cm of the manufactured
132 soil. Air taps were left open for 24 hours to allow void excess air pressure to dissipate before
133 the experiment began.

134 The friction between the soil sample and the mold was managed with the polished coating
135 inside the plastic tubes. Such mode couldn't exclude the friction completely, however avoided
136 the mechanical disruption during the consisted compaction of the 1 meter height soil samples
137 and chemical interference during the freeze-thaw cycles. To provide the moisture insulation
138 the plastic collars connections were covered with silica gel and rubber sleeves, and the whole
139 soil columns were wrapped with clean film thoroughly (Fig. 5). Nine soil columns were
140 prepared for each of the two tests reported here.

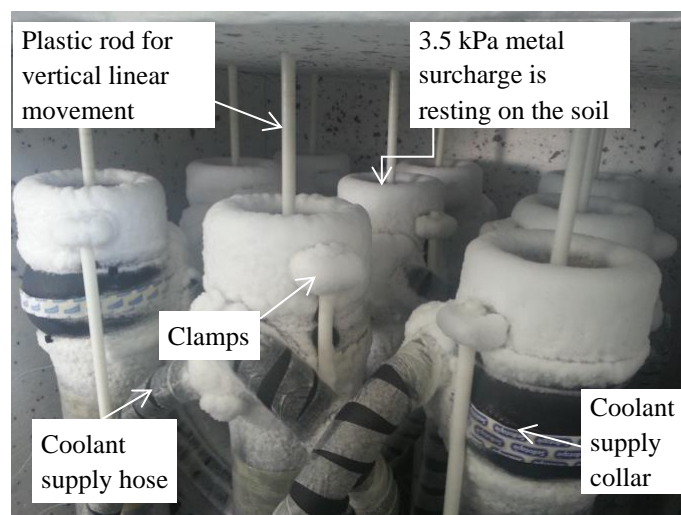


142 **Fig. 5.** Environmental chamber for freeze-thaw cycles with a capacity of nine soil columns

143 During the first test, the soil columns were supplied with deionised water throughout the freeze-
144 thaw cycles. In the second test, the soils were supplied with a sodium chloride solution, where
145 the salt content made up 11 g of NaCl per litre, based on the previous studies of salt distribution
146 in the roadside area (Lundmark and Olofsson, 2007; Pedersen, Randrup and Ingerslev, 2000).
147 Samples for both tests ran with the same freezing regime and equal initial soil properties.

148 *2.3 Environmental chamber design*

149 An insulated environmental chamber with a capacity of nine 1 metre soil columns was designed
150 for the freeze-thaw tests (Fig. 5). The top ring of the columns was supplied with refrigerant
151 liquid circulated to a thermos controlled bath. Circular metal surcharges of 3.5 kPa were placed
152 on the top of the soil samples to provide a load similar to that imposed by pavement layers (Fig.
153 6). These metal surcharges also acted to equally distribute the temperature reduction created
154 by the cooling collars. To prevent the formation of condensation inside the chilling collars they
155 were covered with clean film.



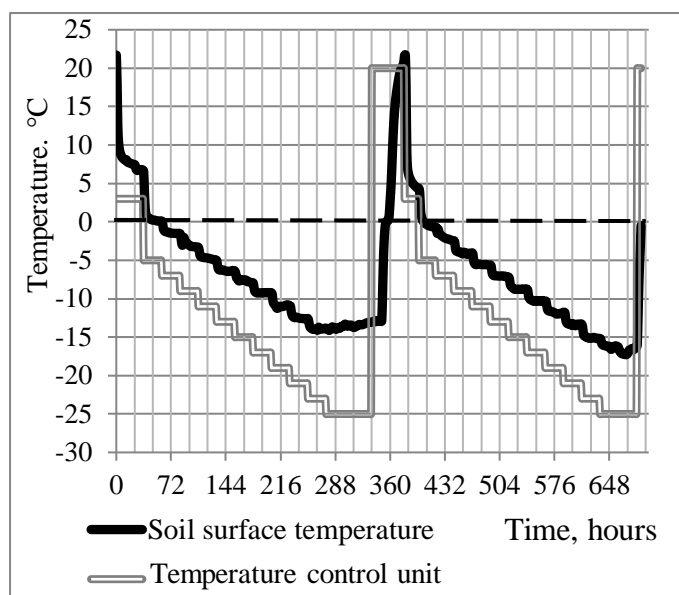
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157 **Fig. 6.** The surcharges inside the cooling collars evenly cool the soil surface and facilitate the
158 vertical linear movement control.

159 The base of the soil columns was supplied from a Mariotte bottle with deionised water or 11000
160 mg/litre sodium chloride solution located in a separate refrigerator.

161 2.4 Freeze-thaw regime

162 The freezing rate was 2 °C per day, simulating the natural conditions of roadside soils. The
163 temperature timeline for the freeze-thaw cycles is presented in Fig. 7. The temperature
164 distribution in the top layers depended on the moisture accumulation and the heat capacity of
165 the soils. Daily fluctuation of the environmental temperature also brought the test close to the
166 natural conditions.



167

168 **Fig. 7.** Relationship between the set for the temperature control unit and the actual
169 temperature of the soil surface.

170 In the first test the soil columns were supplied from the base with deionised water. The first
171 three columns were removed from the chamber immediately after the first freezing cycle and
172 processed for data analysis. Three other were removed after the first thawing period. The last
173 three columns were removed at the end of the second freezing cycle.

174 In the second test, the soil columns were fed with 11000 mg/litre sodium chloride solution
175 and all nine columns were kept in the same conditions until the end of the test.

176 *2.5 Temperature monitoring*

177 The temperature in each 10 cm section of the entire 1 m soil column was recorded with K
178 type thermocouples and PicaLog technology LTD logger at hourly intervals throughout the
179 test. Supplementary thermocouples were measuring the temperature of air in the
180 environmental chamber, supplied water or solution and the chilling collar temperature at the
181 top of the sample.

182 *2.6 Monitoring of frost heave and consolidation rates*

183 Vertical linear movements were recorded every 12 hours from the digital gauges mounted
184 outside the environmental chamber. These were connected through the chamber's top to the
185 metal surcharges via low thermal conductivity plastic rods. This allowed observation from
186 outside the chamber and so minimised heat loss.

187 *2.7 Post freeze-thaw sampling and testing*

188 After removal from the environmental chamber, each column was weighed and placed in a
189 horizontal position. The soil in the columns was sliced at 10 cm sections along the joins of the
190 mould rings. Each section was weighed once more and sampled from the top and bottom side
191 of each section for the moisture and chemical content determination.

192 *2.7.1 Determining the moisture content*

193 At the end of the freeze-thaw cycles the soil columns were immediately sliced without waiting
194 for the thaw and sampled to determine the total moisture content, including ice and unfrozen

195 water content. The total moisture mass was defined with the weight difference after drying at
196 105 °C for 24 hours. The moisture content of the top 10 cm section was determined at a 1 cm
197 interval, while the lower sections were sampled every 10 cm.

198 *2.7.2 Obtaining the sodium chloride content*

199 A multi-range conductivity meter HI 9033 was used to determine the chemical mass content in
200 the soils after the freeze-thaw test. To produce a calibration standard chart for electrical
201 conductivity, a 50 g of oven dried sample of the unused soil was mixed in 500 ml of deionized
202 water and measured for the electrical conductivity. This established a relative “zero” sodium
203 chloride content at the beginning of the test. A calibration standard was then established by
204 progressively adding known amounts of sodium chloride to this sample and mixing thoroughly.
205 At the end of the test run with a sodium chloride solution supply, the soil samples were taken
206 from the columns, labelled and oven dried, and then mixed in deionised water in the same
207 proportion to obtain the chemical content of the solution. The determined salt content was
208 considered in mg per dried soil mass in grams or mg/litre respectively to the moisture content
209 of the sample after the test.

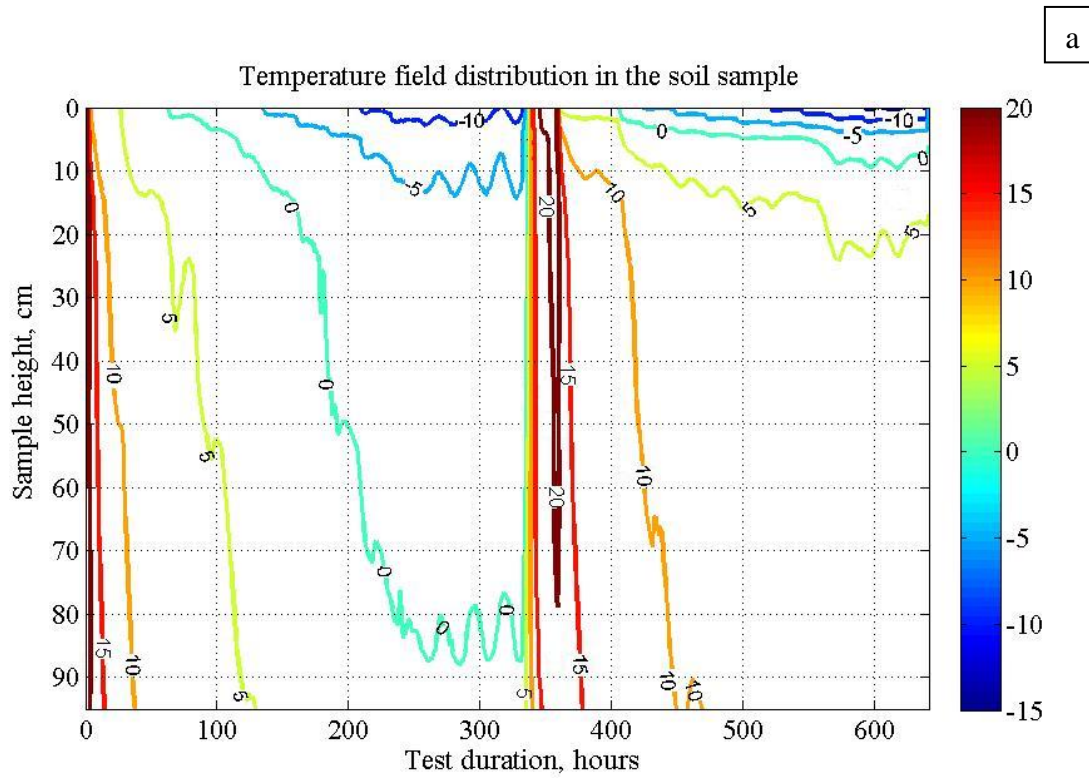
210 *2.7.3 Further tests*

211 The remaining undisturbed soil was also tested with ultrasound for elasticity modulus value
212 using a Pundit Plus ultrasonic equipment (Model PC1006), direct shear test, oedometer
213 consolidation and CBR.

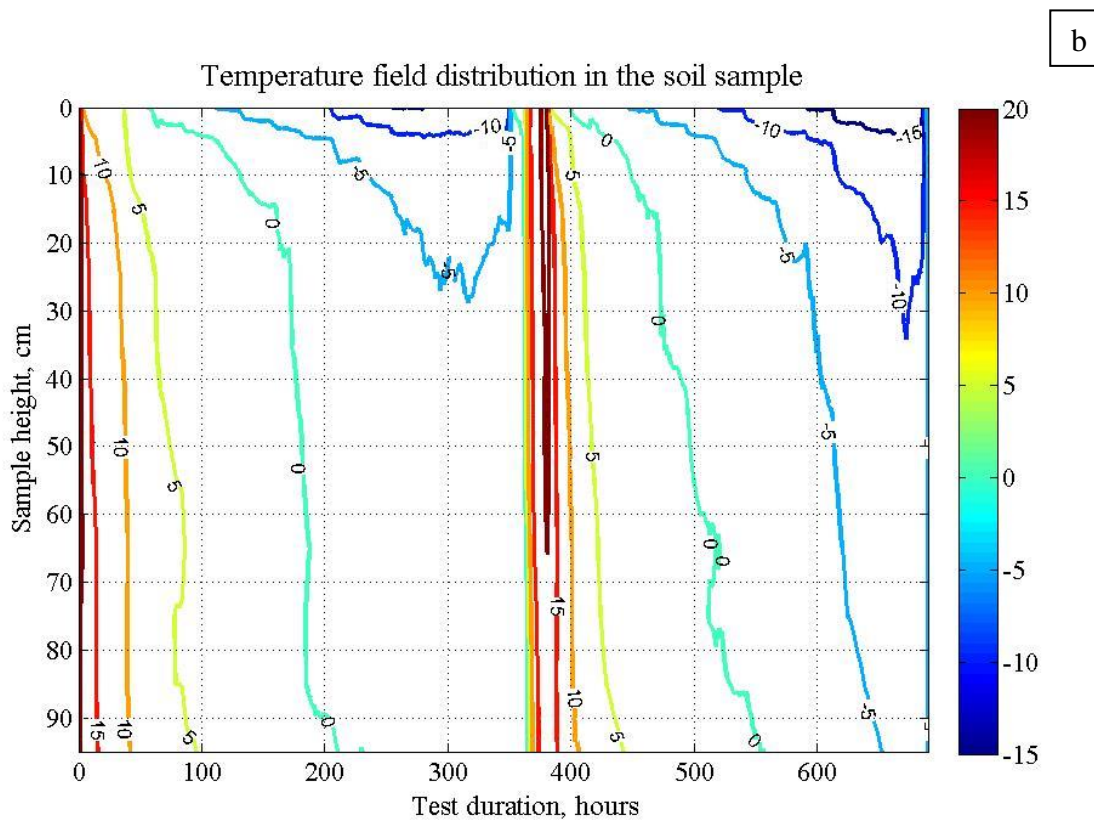
214 **3. Results**

215 *3.1 Temperature field distribution*

216 The evolution of temperature fields in the columns developed in a similar way in each test.
217 Some variations within the time line are due to minor variations in the structure of the soil
218 particles and some variation in the thermal conductivities.



219



220

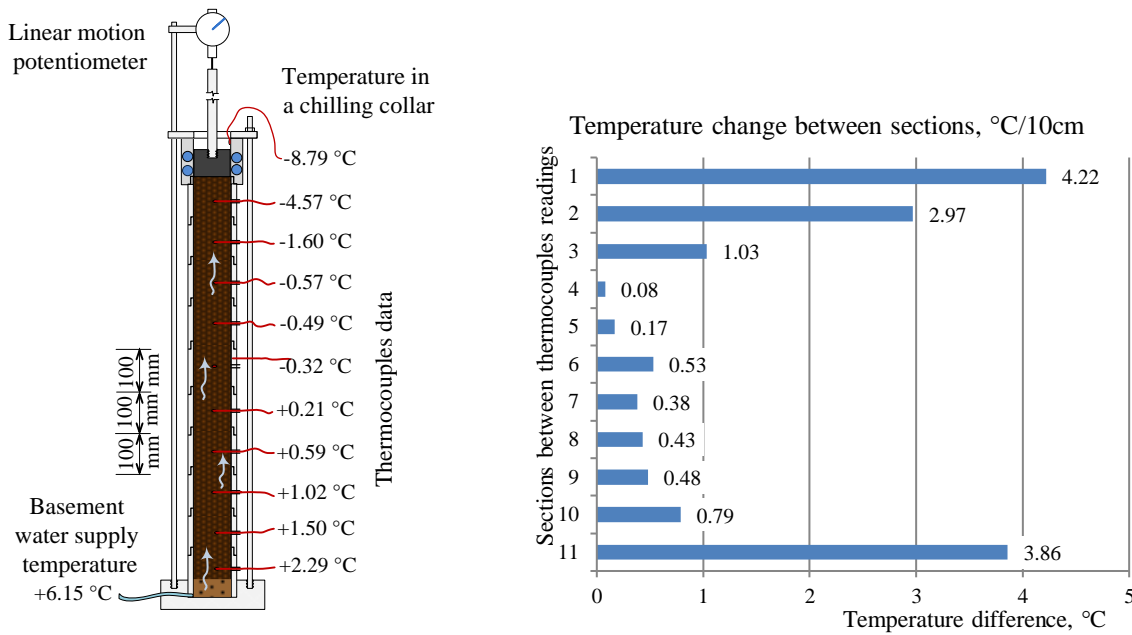
221 **Fig. 8.** Example of temperature development in column #2 during the freeze-thaw test, cm: a
222 - supplied from the basement with deionised water; b - supplied from the basement with
223 11000 mg/litre sodium chloride solution

224 As shown in Fig. 8 a and b, the first cycle of both tests followed a similar temperature range,
225 while the second cycle differed significantly. The heat capacity of soils supplied by the clean
226 water increased because of greater moisture collection at the top of the soil columns. In the
227 second freezing cycle the heat conductivity and the temperature gradient between sections
228 decreased because of extensive moisture collection. Thus the enlarged heat capacity of the
229 moistened soils required intensified heat loss to chill down in the second freezing cycle (Fig.
230 8a).

231 In Fig. 8b, the soil was steadily salinized by chemical solution from the base supply. However,
232 along with increased ion content, it provided better heat conductivity and accelerated the soil
233 cooling. This explains the divergence in the second freezing cycle. The temperature range in
234 the second cycle significantly decreased, in contradiction to the soils supplied with deionised
235 water.

236 The change in temperature by the height of the soil column is not uniform. In the soil column
237 illustrated in Fig. 9 the temperature gradients varied from 0.22-0.30 °C/cm in the upper frozen
238 section, 0.02-0.05 °C/cm in the middle section and 0.08-0.10 °C/cm in the lowest section.

239 The significant drop in temperature was associated with the crystallization of the pore water,
240 which is explained by the enthalpy in transition zones. As the cooling process was
241 implemented gradually for a metre depth, the temperature jumps were recorded in depth
242 sequentially in time.



243

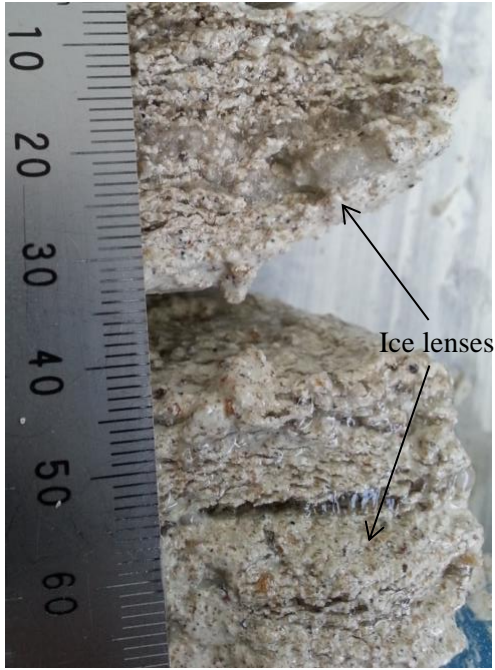
244 **Fig. 9.** The temperature field distribution on example of column #2 during the 201st hour of
 245 the freeze-thaw test.

246 *3.2 Change in moisture distribution*

247 Moisture content raise in the top soils was up to 82.5% in the deionised water supplied test.

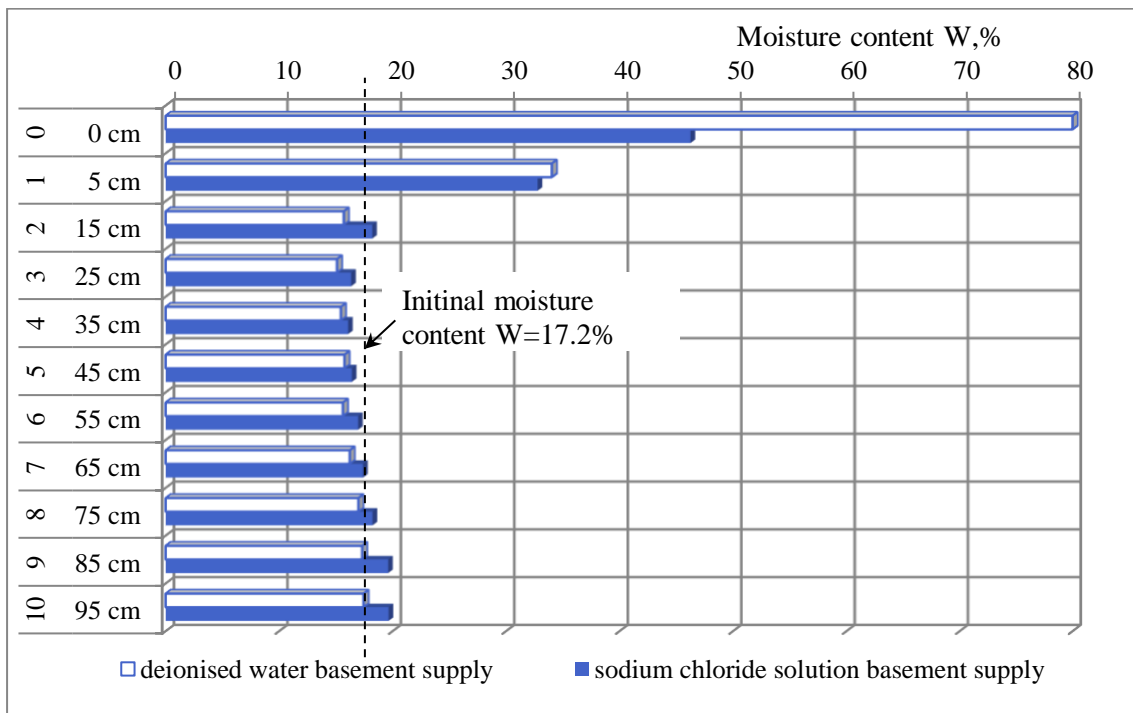
248 The heterogeneous dispersion of moisture in the top 10 cm of soil mass is explained by the
 249 growth of segregated ice lens formation that produced a layered fabric (Fig. 10; cf. van
 250 Everdingen 2005, Fig. 8c).

251 The migration of moisture toward the freezing front led to desiccation in the middle height
 252 zone from 15 cm to 55 cm, with a moisture content decrease of around 2% (Fig. 11). Negative
 253 pressure in top zone forced further feeding from the base water and composed 17.4% in the
 254 base zone. Each column had absorbed 170 g on average from base deionised water supply by
 255 the end of the test.



256

257 **Fig. 10.** Horizontal ice lenses present in the top soil with thickness up to 2 mm at the end of
 258 the freeze-thaw test, supplied with deionised water.



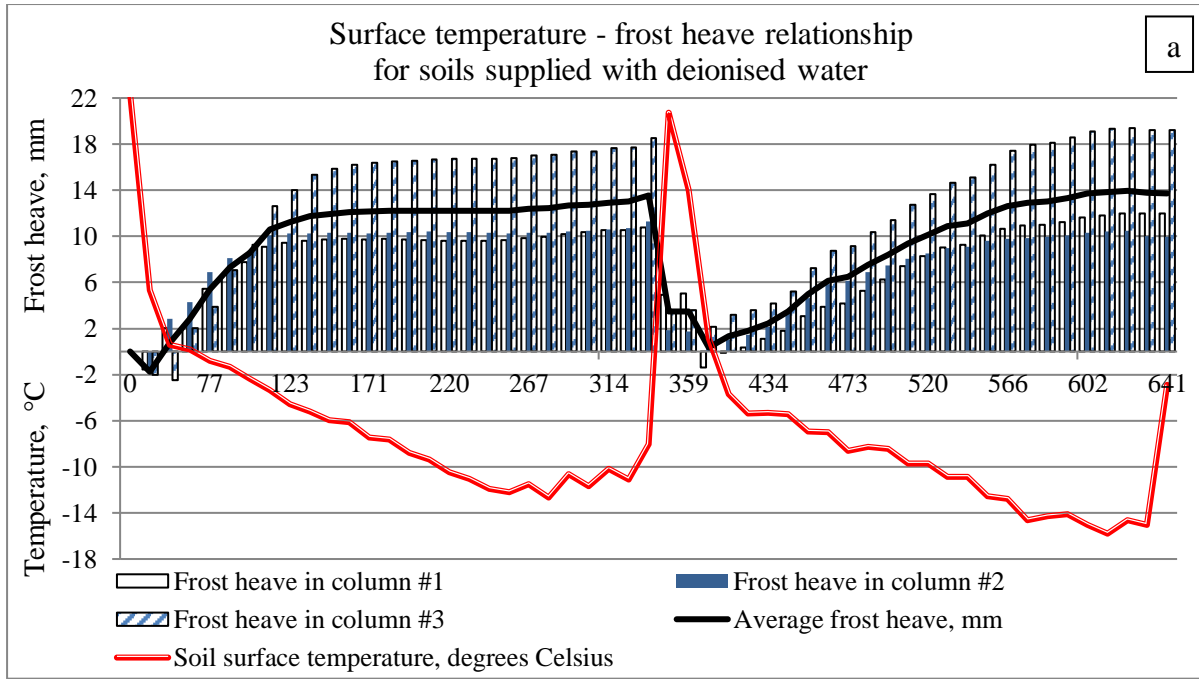
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260 **Fig. 11.** Average moisture content distribution after 2 freeze-thaw cycles.

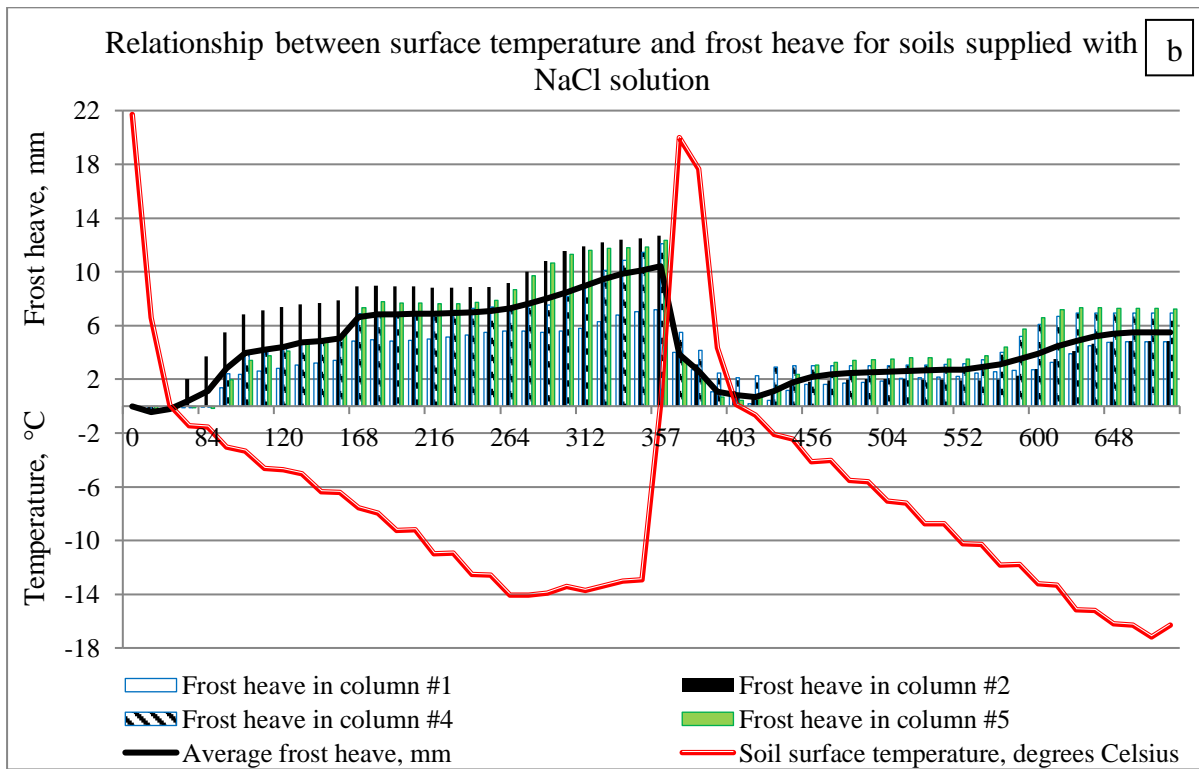
261 In comparison with the distilled water test, the solution supplied test resulted in a smoother
262 moisture redistribution and less hydraulic conductivity. The top soils increased by 46.2% of
263 moisture content at the end of two freeze-thaw cycles. Moisture content in the middle zone was
264 insignificantly desiccated to 16.3% during the test and base soils were moisten by 19.6% of
265 salt-water solution. The average solution intake during the test drew up 130 g per column.

266 *3.3 Frost heave during the freeze-thaw cycles*

267 Frost heave obtained in the deionised water supply test between 13-14 mm on average at the
268 end of the first cycle, which is around 1.4 % from the initial height (Fig. 12a). The frost heave
269 extension during the first 3 days of the first freezing cycle, 46-122 hours, was within the range
270 of 2.6-5.2 cm/day. The freezing speed by the sample length at that period was 2.0 cm/day.
271 While the next day (122-146 hours in Fig. 12) the frost heave extension decreased to 1.0-1.3
272 cm/day, while the freezing speed increased to 5.5 cm/day. During the next six days (146 to
273 290 hours) the frost heave extension was 0.1-0.5 cm/day. The freezing speed rose dramatically
274 at the 15 -75 cm depth of the sample achieving the freezing rate of 20.0-35.0 cm per day. At
275 the end of the first freezing cycle the freezing rate had slow down to 5.2 cm/day. In the second
276 freezing cycle the similar heave level was achieved in shorter period. Some individual columns
277 had up to 19 mm of frost heave. The frost heave extension in the first three days of the second
278 freezing cycle (410-482 hours by the timeline), was in a range of 1.4-3.2 cm/day. The further
279 7 days period, 482-626 hours, the heave extension varied between 0.2-2.0 cm/day. However
280 the freezing speed was only 0.63 cm/day and by the end of the test just the top 5 cm was frozen.



281



282

283 **Fig. 12.** Relationship between surface temperature and frost heave with time, h: a - deionised
 284 water supplied test; b - soils supplied from the base with 11 g/litre sodium chloride solution.

285 In the test supplied by chemical solution, the final frost heave had decreased to 10 mm on
286 average (Fig. 12b). The freezing speed in the first cycle had a similar pattern to the test with
287 deionised water. The frost extension during the first freezing cycle varied between 0.1-3.4
288 cm/day, with less heave extension at the surface temperature below -7°C . During the second
289 freezing cycle, the vertical linear heave extension of soils did not exceed 1.1 cm/day and by
290 the end of the test was less than 6 mm. This might be explained by chemical mass migration
291 towards the cooling zone and a consequent reduction of the freezing point. The segregation of
292 ice crystals requires its separation from the solution part, which leads the remained salt solution
293 to become even more concentrated. Therefore, the salinization of ground water leads to the
294 reduction in frost heave. The freezing speed in the second freezing cycle was 2.85 cm/day in
295 the 403-447 hours' time interval, and 9.6 cm/day and more afterwards.

296 *3.4 Chemical Conductivity*

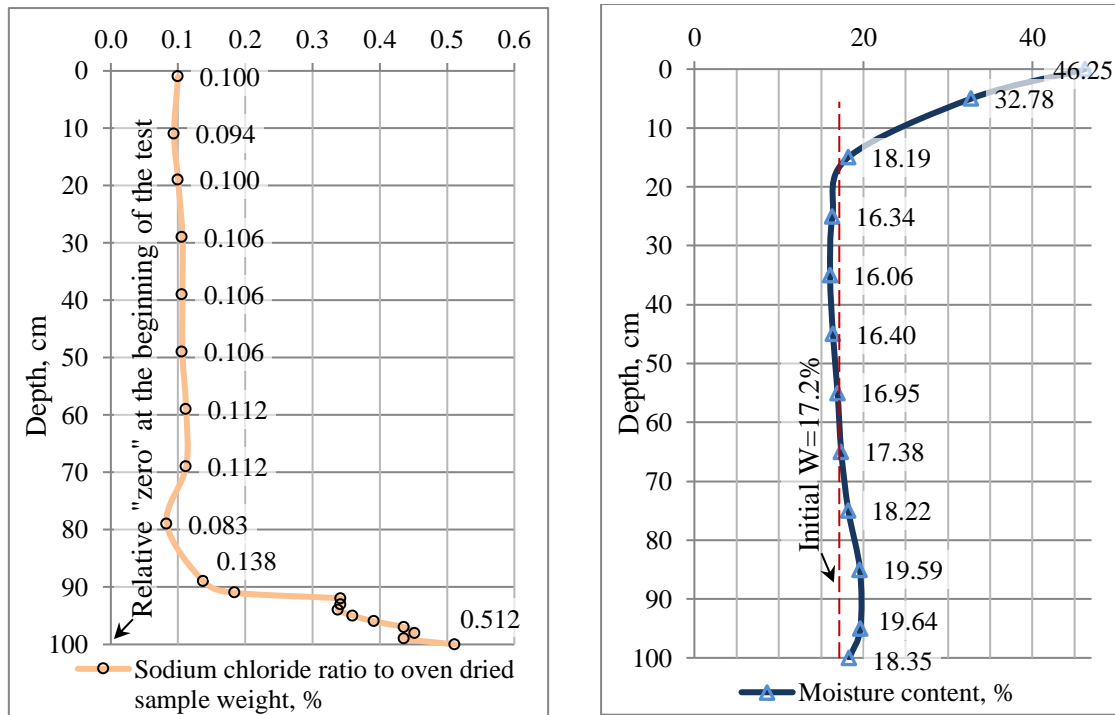
297 The measurement of sodium chloride content after freeze-thaw cycles was performed over
298 the entire height of the soils columns with basement chemical solution supply (Table 3).

299 The average mass transfer of the sodium chloride and moisture content distribution at the end
300 of freeze-thaw cycles is plotted in Fig. 13. Here the relatively zero value represents the initial
301 chemical content measured for the soil sample before the test. In the range of about 20,000-
302 30,000 mg/litre sodium chloride concentration in pore water was obtained after the test,
303 throughout the soils in the middle height zone between 0.1-0.9 m depths. This corresponds to
304 the chemical mass transfer of 0.10-0.14% to the dry soil's weight. Such dimensions basically
305 exceeded the fresh water concentration limits and drastically changed the freezing point of soil
306 samples.

307 Table 3 – Calculation of sodium chloride content after freeze-thaw cycles with basement
 308 solution supply of the soil columns.

Soil sample depth, cm	Electrical conductivity average for 9 columns, μ S	NaCl, obtained by electrical conductivity g/250ml	Moisture content average for 9 columns, %	Mass of water dried out from the sample, g	Mass of NaCl per litre of pore water, g	Ratio of the weight of NaCl to the weight of dried soil, %
1	0.27	0.0250	46.3	11.563	8.65	0.100
11	0.26	0.0235	18.9	4.728	19.88	0.094
19	0.27	0.0250	16.9	4.225	23.67	0.100
29	0.29	0.0266	16.3	4.075	26.11	0.106
39	0.29	0.0266	16.3	4.075	26.11	0.106
49	0.29	0.0266	16.7	4.175	25.49	0.106
59	0.30	0.0280	17.2	4.300	26.05	0.112
69	0.30	0.0280	17.8	4.450	25.17	0.112
79	0.24	0.0208	18.9	4.725	17.61	0.083
89	0.35	0.0344	19.6	4.900	28.08	0.138
91	0.45	0.0460	19.6	4.900	37.55	0.184
92	0.78	0.0855	19.6	4.900	69.80	0.342
93	0.78	0.0855	19.6	4.900	69.80	0.342
94	0.77	0.0845	19.6	4.900	68.98	0.338
95	0.81	0.090	19.6	4.900	73.47	0.360
96	0.89	0.098	19.6	4.900	80.00	0.392
97	0.98	0.109	19.6	4.900	88.98	0.436
98	1.01	0.113	19.4	4.850	93.20	0.452
99	0.98	0.109	19.1	4.775	91.31	0.436
100	1.14	0.128	18.9	4.725	108.36	0.512

309 The electrical conductivity results after freeze-thaw cycles with de-icing chemical solution
 310 basement supply have confirmed that sodium chloride did migrate along with moisture toward
 311 the freezing top through the soils.



312

313 **Fig. 13.** The chemical mass transfer and corresponding moisture distribution with the column
 314 height after two freeze-thaw cycles.

315 **4. Discussion**

316 The applied method allowed clear differences in the results from distilled water and the sodium
 317 chloride solution supply to be observed.

318 The temperature distribution in the samples with the deionised water supply test registered the
 319 obvious change in the second freezing cycle, which was explained by severe moisture
 320 redistribution of moisture within the soil sample during the slow rate freezing.

321 On the contrary in the samples supplied with 11000 mg/litre sodium chloride solution the lower
 322 temperature was achieved throughout the columns height and less frost heave noted. A similar
 323 reduction in the frost heave rate in saline soil samples was observed by Wan, Lai and Wang,
 324 (2015). The vertical linear movement gauges displayed the increased soil volume after the first
 325 freeze-thaw cycle (Fig. 12), while above zero degree temperatures confirmed the thawing of

326 the entire column length (Fig. 8). This can be explained with the moisture uptake to the soil
327 samples which were at the maximum dry density achievable during sample preparation, and
328 close to zero void content.

329 The inverse relationship of the frost heave extension and the freezing speed by the soil sample
330 length was admitted. The maximum frost heave extension was observed at the beginning stage
331 of the first freezing cycle when the rate of freezing through the sample was 2.0 cm/day. Iushkov
332 and Sergeev (2015) also found that the maximum frost heave in clay soils is obtained at the
333 slow freezing rate in a range 2-3 cm/day. The second freezing cycle showed the reduced
334 freezing speed in the deionised water supply test and slightly accelerated freezing rate in the
335 solution supplied one.

336 The results from the sodium chloride content measurements support the version of chemical
337 mass transfer induced by the gradient in temperature. The presence of sodium chloride in the
338 ground water influenced the significant cooling in the second freezing cycle and led to less
339 moisture collection in the top 20 cm of the soil columns, with higher moisture content in the
340 soils below this depth. The supply of sodium chloride solution also reduced the frost heave in
341 the second freeze cycle despite the increase of moisture in the cooling zone. Furthermore, there
342 was a clear migration of NaCl upwards to the freezing zone, which was more than expected
343 given the observed water mass transfer.

344 The phenomenon of chemical mass transfer during the freeze-thaw cycles was induced by the
345 diffusion of NaCl along a vertical concentration gradient, in excess of the redistribution that
346 would be expected by the cryosuction-induced migration of the pore water.

347 Similar previous studies on freezing have produced somewhat contradictory results. Significant
348 salt redistribution in a downward freezing test was observed by Baker and Osterkamp (1989),

349 where the chemical mass was rejected from the frozen zone to produce an enriched brine in the
350 unfrozen part of the soil sample. However no salt movement was observed in the upward
351 freezing. Significantly, no additional moisture supply was provided in the Baker and
352 Osterkamp (1989) test.

353 In contrast, Brouchkov (2000) reported no significant salt migration in a horizontally
354 positioned sample experiencing a long term temperature gradient.

355 Bing's experiments with red clay samples showed an accumulation of chemical ions in the
356 lower unfrozen zone and no change in the content in the upper frozen part of the sample (Bing
357 and He, 2008). Here the samples were supplied with 5% sodium sulphate solute from the base.
358 The amount of chemical mass transfer was sensitive to the temperature drop, enhancing the ion
359 accumulation in the unfrozen part as temperature decreased.

360 It is worth noting that these previous studies for chemical mass transfer during freeze-thaw
361 cycles were performed with soils that were already saline.

362 It is important to note that further work on chemical mass transfer is required as the degree of
363 salt rejection during freezing, and the associated transfer of salt to the remaining soil water
364 solution, is likely to be affected by freezing rate (Iushkov and Sergeev, 2015).

365 The current experimental design does not include monitoring of pore water pressure. The
366 method also does not allow direct observation of water migration or ice segregation. Clearly,
367 while the method is a more realistic representation of road subsoils than existing standards, it
368 does not directly simulate the more complex soil conditions likely to be encountered on site.

369 **5. Conclusions**

370 From the experimental results using the new method, the following observations can be made:

- 371 1. The negative temperature distribution in the salt solution-fed soils was faster and more
372 intense than for the deionised water supplied soils, particularly in the second freeze
373 cycle of the test.
- 374 2. Moisture input from the base water table was 30% greater in the deionised water test
375 than with 11000mg/litre sodium chloride solution supply.
- 376 3. The frost heave value in the deionised water test was greater than during the sodium
377 chloride solution test, especially in the second freeze cycle. There is an inverse
378 relationship between the frost heave extension rate and the freezing speed in the clay
379 soils. The maximum frost heave rate was obtained when the freezing speed was 2
380 cm/day.
- 381 4. The chemical migration that occurred over the entire height of the columns confirmed
382 the salt migration together with water redistribution towards the freezing top of soils.
383 The chemical mass transfer obtained at the end of two freeze-thaw cycles was within
384 0.10-0.14%, using the ratio of sodium chloride mass to the dry soil mass. This
385 corresponds to a 20,000-30,000 mg/litre concentration of sodium chloride in pore
386 water.
- 387 5. The phenomenon of chemical mass transfer during the freeze-thaw cycles appears to
388 be induced by both the thermal gradient and osmotic pressure caused by equilibration
389 of the chemical potential throughout the soil column.
- 390 6. The migration of de-icing chemicals through the subsoil under a pavement area is likely
391 to progressively change the chemical content in the ground water of soils and hence
392 freezing regime under the road over time. Therefore the potential effect of de-icing
393 chemicals and subsequent change of engineering properties of highway subsoils need
394 to be considered at the design period.

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