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► **To cite this version:**

Quoc Hung Vu, Jean-Michel Pereira, Anh Minh Tang. Effect of fines content on soil freezing characteristic curve of sandy soils. *Acta Geotechnica*, 2022, 17 (11), pp.4921-4933. 10.1007/s11440-022-01672-9 . hal-03905344

HAL Id: hal-03905344

<https://enpc.hal.science/hal-03905344v1>

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1 Effect of fines content on soil freezing characteristic curve of
2 sandy soils

3 Quoc Hung VU, Jean-Michel PEREIRA, Anh Minh TANG

4 Laboratoire Navier, Ecole des Ponts, Univ Gustave Eiffel, CNRS, Marne-la-Vallée, France

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7

8

9 Corresponding author:

10 Dr. Anh Minh TANG

11 Research Director

12 Ecole des Ponts ParisTech

13 6-8 avenue Blaise Pascal

14 77455 Marne-la-Vallée

15 France

16 Email: anh-minh.tang@enpc.fr

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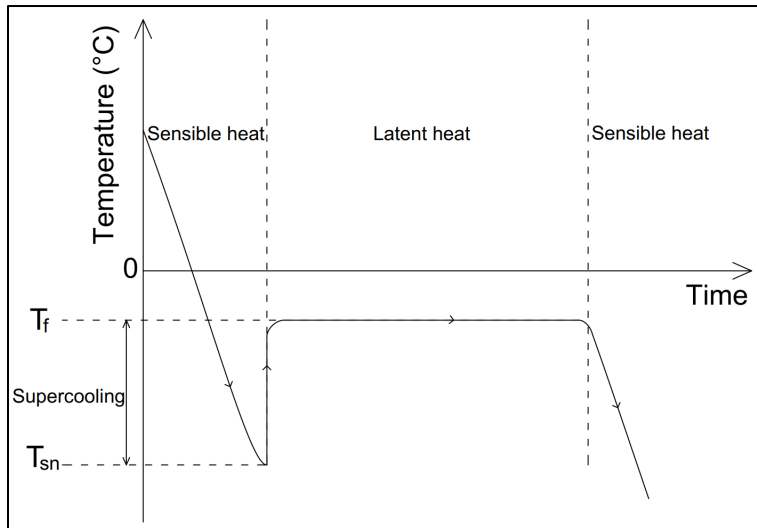
19 **Abstract:** Soil freezing characteristic curve (SFCC) represents the relationship between soil
20 temperature and unfrozen water content of soil during freezing and thawing processes. In this
21 study, SFCC of sandy soils was determined in laboratory. Pure sand was mixed with clay at
22 various contents (0, 5, 10, 15, and 20% of the total dry mass) and the mixtures were compacted
23 to their respective maximum dry density. Compacted specimens were then placed in a close
24 and rigid cell and the soil's temperature was decreased step-by-step to freeze the soil water and
25 then increased back to thaw it. During this thermal cycle, soil's temperature and volumetric
26 water content were monitored in order to determine the SFCC. The results show that SFCC
27 was strongly dependent on the fines content: at higher fines content, the temperature of
28 spontaneous nucleation was lower and the residual unfrozen volumetric water content was
29 higher.

30 **Keywords:** temperature of spontaneous nucleation, hysteresis, soil freezing characteristic
31 curve, residual water content.

32 1. Introduction

33 Frozen soil consists of mineral particles, liquid water, ice and gas. It is formed from unfrozen
34 soil during freezing, when a fraction of liquid water solidifies into ice at temperatures
35 sufficiently low below 0 °C [1]. This phase change causes significant modifications of physical-
36 hydraulic-mechanical properties of soils [2]. The freezing-thawing process is encountered in
37 cold regions, seasonal cold regions as well as construction works using artificial ground
38 freezing technique. Two main consequences of this process that need to be mentioned are frost
39 heave and thaw settlement. These phenomena can induce damages to infrastructure [3–6].

40 The freezing-thawing process in porous media has been investigated not only in civil
41 engineering and geosciences but also in physics [7–11]. While bulk water melts at 0 °C, water
42 in porous media melts at temperatures below 0 °C because of physical interactions between
43 water and solid particles [12–14]. Freezing process of a soil sample (where heat is extracted
44 from the sample with a constant rate) can be divided into three steps (as shown in Fig. 1): (i)
45 supercooling with release of sensible heat; (ii) first water freezing with release of latent heat;
46 (iii) further water freezing with release of sensible heat. In the first step, during cooling
47 (extraction of heat from soil), soil temperature decreases to reach a certain value from that it
48 cannot decrease anymore. This value is called temperature of spontaneous nucleation T_{sn} where
49 the first ice embryo nucleus forms because it attains the critical size [15, 16]. Formation of ice
50 crystals releases latent heat and thus increases soil temperature. From T_{sn} , soil temperature
51 increases to reach another value which is called freezing temperature T_f , where it remains on a
52 plateau for a while. During this second step, soil water is gradually frozen along with releasing
53 latent heat. After that, within the third step, soil temperature decreases with further water
54 freezing. Freezing temperature T_f , also considered to be equal to thawing temperature T_t at
55 which soil state changes from frozen to unfrozen, is usually used as a boundary value index to
56 distinguish between frozen soil and unfrozen soil [17–19]. These characteristic temperatures
57 (T_{sn} and T_f) were investigated in several studies [15, 18, 20].



58

59 **Fig. 1** Freezing process of soil-water system.

60 Soil freezing characteristic curve (SFCC) represents the relationship between the temperature
 61 and the quantity of liquid water in soil. It is one of the most essential data in studying the
 62 freezing-thawing process in soils. On the one hand, several SFCC models were empirically
 63 developed. From SFCC obtained experimentally, empirical models were proposed using power,
 64 piecewise or exponential functions [21–28]. On the other hand, SFCC can be derived from soil
 65 water characteristic curve (SWCC). This approach is based on the theory of similarity between
 66 freezing-thawing and drying-wetting processes that is illustrated by Clapeyron equation [29–
 67 37]. More generally, various physical models were developed based on theory of capillarity,
 68 sorption or that of interface pre-melting [38–40]. Most of the existing SFCC models consider
 69 the effect of fines content but this effect is considered in different ways. For instance, some
 70 empirical models used specific surface or liquid limit as input data while physics-based models
 71 consider absorption parameters of soil. Due to the diversity of SFCC models, there is no unified
 72 standard for choosing SFCC in numerical simulations [41]. In addition, except few models (e.g.,
 73 [35]), most of the existing ones consider a unique relationship between unfrozen water content
 74 and temperature. However, this relation obtained on the freezing path can differ from that of
 75 that of the thawing path; at a given temperature, water content of the freezing path can be higher
 76 than at of the thawing path. This hysteresis is usually ignored in the models.

77 To determine SFCC in the laboratory, a soil specimen is usually subjected to a freeze-thaw
 78 cycle, while unfrozen water content is measured. Although controlling specimen's temperature
 79 is technically feasible, measuring unfrozen water content is much more challenging. Several
 80 methods and techniques have been developed to evaluate the unfrozen water content at negative
 81 temperature, including dilatometry [42, 43], gas dilatometry [44], adiabatic calorimetry [45,
 82 46], isothermal calorimetry [28], differential scanning calorimetry [10, 47, 48], X-ray
 83 diffraction [49, 50], time/frequency domain reflectometry (TDR/FDR) [51–53] and pulsed
 84 nuclear magnetic resonance (P-NMR) [38, 54, 55]. Among these methods, TDR and P-NMR
 85 are the two most common ones. P-NMR is widely acknowledged as a highly accurate and non-
 86 destructive technique. However, the equipment required for this technique is generally
 87 expensive [56]. Compared to P-NMR, TDR/FDR can be used in the laboratory as well as in the
 88 field and it is cheaper, quicker, and more portable. With TDR, unfrozen water content is inferred
 89 from the measurement of apparent dielectric constant of soil using an empirical equation [57,
 90 58] or dielectric mixing models [51, 59, 60]. It is noted that several factors such as temperature
 91 or bound water can affect its accuracy.

92 Several studies have determined SFCC in the laboratory in both freezing and thawing processes
93 [18, 40, 42, 55, 61–64]. These studies recognized that hysteresis exists in SFCC in which the
94 unfrozen water content is different in thawing and freezing processes at the same temperature.
95 Hysteresis in freezing-thawing process was believed to be similar to that of wetting-drying
96 process. However, the mechanism inducing hysteresis in SFCC is complex and it may be
97 influenced by several effects such as supercooling, pore blocking, capillarity, free energy
98 barriers, contact angles and electrolytes [55, 62]. It is also noted that hysteresis is significant at
99 temperatures between $-2\text{ }^{\circ}\text{C}$ and $0\text{ }^{\circ}\text{C}$ [42, 65, 66] and that it should not be ignored due to
100 impacts on unfrozen water content on frost heaving [67, 68], creep behaviour of frozen soils
101 [69, 70] as well as thermal regime of frozen ground [71].

102 Beside hysteresis effect, it is found that the shape of SFCC depends also on several factors,
103 including liquid limit [28], stress condition [72], salt content and solute types [38, 73], initial
104 water content or degree of saturation [74–76], types of soil [18, 62, 65], pore-size distribution
105 [55], and fines content [18, 28, 55, 63]. Among these factors, fines content can influence others
106 (liquid limit, pore-size distribution and types of soil). As far as fines content is concerned, by
107 determining unfrozen water content of several clays, a silt and a gravel, Tice et al. [28] observed
108 significantly different unfrozen water contents at the same temperature below $0\text{ }^{\circ}\text{C}$. Tian et al.
109 [63] carried out tests on three soils corresponding to three clay contents and found that unfrozen
110 water degree of saturation also changed in different ways in both freezing and thawing
111 processes. For soils containing higher clay fraction, unfrozen water degree of saturation was
112 higher at any given temperature below freezing point and the hysteresis loop was smaller. The
113 same findings concerning SFCC were obtained in the study of Zhang et al. [18] on silty clay,
114 and silt and in the study of Li et al. [55] on silty clay, fine sand, and medium sand. Some other
115 authors also investigated different soils but the effect of fines content was out of their focus [22,
116 30, 54, 62].

117 The present study aims at systematically investigating the effect of fines content on the SFCC
118 of sandy soils. Clean sand was mixed with clay at dry state firstly and water afterward to obtain
119 sandy soils with clay content of 0, 5, 10, 15, and 20% prior to compaction at the Proctor
120 maximum dry density followed by a saturation phase. The specimen's temperature was then
121 decreased progressively to freeze the soil specimen in undrained conditions prior to applying
122 the thawing process. During this freezing-thawing cycle, soil's temperature and unfrozen water
123 content were measured. After the introduction, the second section of this paper presents the
124 materials and experimental methods. Experimental results are presented in the third section,
125 before being discussed in the fourth section.

126 **2. Materials and experimental methods**

127 **2.1. Experimental setup**

128 The experimental setup is shown in Fig. 2 and the details of the sensors used are presented in
129 Table 1. Soil specimen was contained in a rigid metallic cylindrical cell (150 mm in height and
130 150 mm in diameter). The cell was immersed in a temperature-controlled bath (F38-EH
131 JULABO with $\pm 0.03\text{ }^{\circ}\text{C}$ accuracy). Soil temperature was measured with a PT100 sensor, soil
132 volumetric water content was measured with a ML2x Thetaprobe sensor, and soil suction was
133 measured with a tensiometer. As Thetaprobe sensor measures soil apparent dielectric constant
134 (K_a) which is the ratio of the dielectric permittivity of a substance to free space, soil unfrozen
135 volumetric water content (θ_u) was estimated from measured K_a by using empirical equations
136 of Smith and Tice [58] (1) and Topp et al. [57] (2) for frozen and unfrozen states of soil,
137 respectively. Equation (2) was used only for the initial state (before the occurrence of freezing)

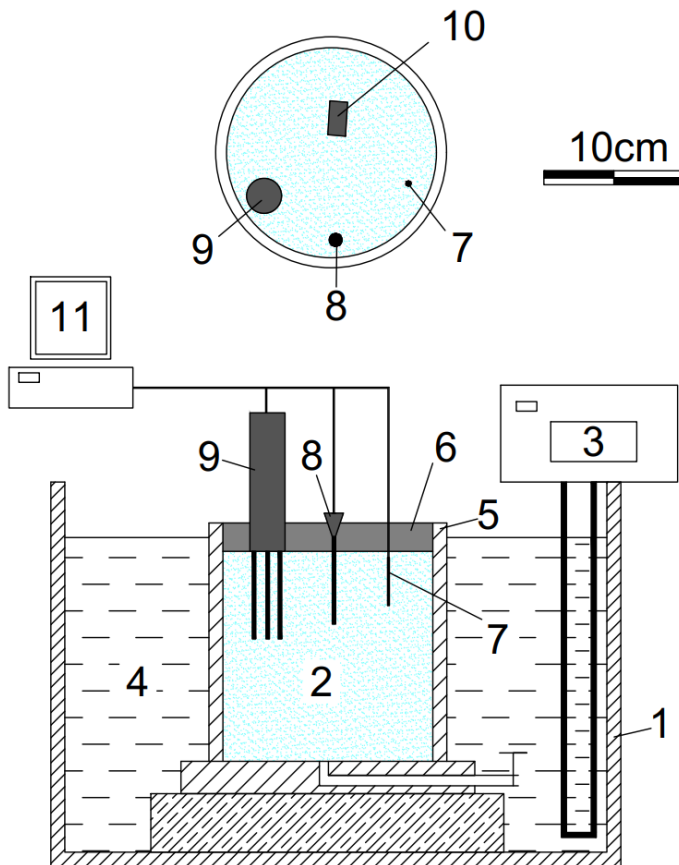
138 and for the final state where thawing is complete. Equation (1) is used where ice is expected to
 139 exist in soil (i.e. after the occurrence of freezing and before the completion of thawing).

140 $\theta_u = -0.1458 + 3.868 \times 10^{-2} \times K_a - 8.502 \times 10^{-4} \times K_a^2 + 9.92 \times 10^{-6} \times K_a^3$ (1)

141 $\theta_u = -5.3 \times 10^{-2} + 2.92 \times 10^{-2} \times K_a - 5.5 \times 10^{-4} \times K_a^2 + 4.3 \times 10^{-6} \times K_a^3$ (2)

142 Table 1: Properties of sensors using in freezing-thawing tests.

Measured parameters	Principle	Type	Accuracy	Range
Temperature	Resistance temperature detector	PT100	±0.03 °C	-200 to 400 °C
Volumetric unfrozen water content	Time domain reflectometry (dielectric constant)	ThetaProbe ML2x (4 rods)	0.01 m ³ /m ³	0.01 to 1 m ³ /m ³
Tensiometer	Piezoelectric transducer	T5x	±0.5 kPa	-160 to 100 kPa
Thermal conductivity	Transient line heat source	KD2-Prob (RK-1)	10%	0.1 to 4 W/(m.K)



143

144 **Fig. 2** Schematic view of the experimental setup. (1) Temperature-controlled bath; (2) Soil
 145 specimen; (3) Temperature controlling system; (4) Temperature-controlled liquid (30%
 146 ethylene glycol + 70% water); (5) Metallic cylindrical cell; (6) Insulating cover; (7)

147 Temperature sensor; (8) Tensiometer; (9) Soil water sensor; (10) Thermal conductivity probe
148 (results are not presented in this study); (11) Data logger system.

149 2.2. Material

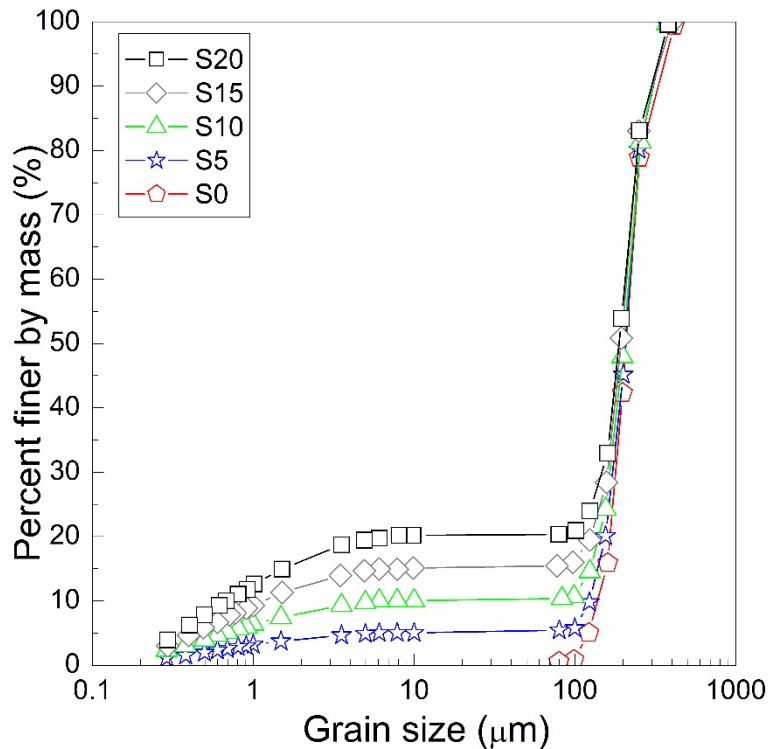
150 Fontainebleau sand was carefully mixed with Speswhite kaolin clay at dry state using an
151 automatic mortar mixer in order to obtain sandy soils with fines content (dry mass of clay
152 divided by dry mass of soil) of 0, 5, 10, 15, and 20%. The physical properties of sand and clay
153 are shown in Table 2 and Table 3, respectively. Fig. 3 presents the grain size distribution of
154 these soils. In this study, the name of each soil corresponds to its clay content (for instance, S10
155 corresponds to a soil having 10% of clay in dry mass). Prior to the preparation of the soil
156 specimens, each soil was carefully mixed with distilled water using the mortar mixer to obtain
157 optimum water content (determined from the Normal Proctor compaction curves obtained on
158 the same soils [77]). Afterward, wet soil was packed in a plastic bag for at least 24 h to ensure
159 the homogenisation of water content, prior to compaction in the cylindrical cell to reach its
160 maximum dry density.

161 Table 2. Physical properties of sand.

Property	Value
Median grain size, D_{50} (mm)	0.21
Uniformity coefficient, C_U	1.52
Minimum void ratio, e_{min}	0.54
Maximum void ratio, e_{max}	0.94
Particle density, ρ_s (Mg/m ³)	2.65
Minimum dry density, $\rho_{d,min}$ (Mg/m ³)	1.37
Maximum dry density, $\rho_{d,max}$ (Mg/m ³)	1.72

162 Table 3: Physical properties of clay.

Property	Value
Liquid limit, LL (%)	55
Plastic limit, PL (%)	30
Plasticity index, PI	25
Specific surface area (m ² /g)	0.94
Particle density, ρ_s (Mg/m ³)	2.65
Particle diameter < 0.002 mm (%)	79
Particle diameter > 0.01 mm (%)	0.5
Maximum dry density, $\rho_{d,max}$ (Mg/m ³)	1.45



163

164 **Fig. 3** Grain size distribution curves.

165 **2.3. Experimental procedure**

166 After soil compaction in the cell, sensors were installed as shown in Fig. 2 and an insulating
 167 cover made of expanded polystyrene was placed in order to avoid heat exchange between soil
 168 specimen and ambient air. The whole system was then transferred inside the temperature-
 169 controlled bath. Prior to the freezing-thawing test, soil specimen was saturated by injecting
 170 water from the bottom of the specimen during 0.5 to 2 days depending on fines content. After
 171 the saturation (when a layer of water of 10 mm was visible on the top of the specimen), the
 172 temperature of the bath was first set at a temperature between 0 °C and -1 °C (slightly higher
 173 than the expected T_{sn}). Each test started with the cooling path. The bath temperature was
 174 decreased in steps of 0.1 °C to freeze the soil pore water. Once the freezing was triggered, the
 175 temperature continued to be decreased in steps of 0.2 °C until -2 °C or -3 °C to observe the
 176 change of liquid water content during further cooling. Afterward, during the heating path, the
 177 bath temperature was increased in steps of 0.2 °C until 0 °C to thaw the frozen soil. During both
 178 cooling and heating paths, the bath temperature was changed to the subsequent step only when
 179 soil temperature and volumetric unfrozen water content (measured by the sensors) had reached
 180 their equilibrium state. The equilibrium state was considered reached when these two quantities
 181 did not change (< 0.05 °C for temperature and $< 1\%$ for water content) during at least 2 h.

182 The test program is shown in Table 4. The test number shows the soil tested (S0 to S20)
 183 followed by the number of replicate test (T1 to T4). At least two tests were performed for each
 184 soil. Tests T1 were performed following the procedure described above to obtain the complete
 185 SFCC curves. For the other tests (T2, T3, T4), only the freezing path of the same procedure was
 186 performed in order to replicate the characteristic temperatures.

187 Table 4: Physical properties of soils.

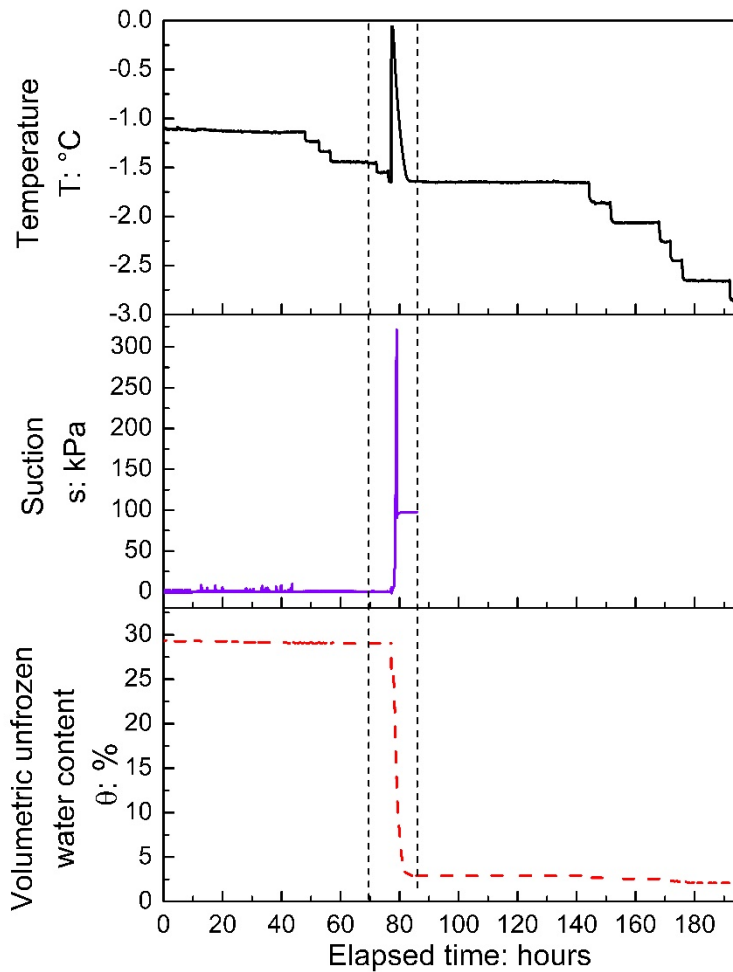
Test No.	Fines content (%)	Dry density (Mg/m ³)	Porosity (-)	Test duration (h)
S20-T1	20	1.98	0.25	754
S20-T2	20	1.96	0.26	26
S15-T1	15	1.99	0.25	712
S15-T2	15	2.00	0.25	64
S10-T1	10	1.91	0.28	590
S10-T2	10	1.90	0.28	153
S5-T1	5	1.78	0.33	817
S5-T2	5	1.78	0.33	143
S5-T3	5	1.78	0.33	190
S0-T1	0	1.67	0.37	756
S0-T2	0	1.67	0.37	286
S0-T3	0	1.67	0.37	75
S0-T4	0	1.68	0.37	187

188 **3. Experimental results**

189 **3.1. Typical test (S10-T1)**

190 As an example, the results of test S10-T1 are shown in Fig. 4 where soil temperature, suction,
191 and volumetric unfrozen water content are plotted versus elapsed time for the cooling path.

192 From -1.2 °C, soil temperature was decreased in steps of 0.1 °C down to -1.6 °C. During this
193 period, soil temperature was controlled through the bath's temperature, suction remained equal
194 to zero and volumetric water content remained constant. When soil temperature reached -1.6
195 °C, soil freezing started inducing abrupt changes in the three measured quantities. Results
196 obtained during this stage (elapsed time of 70 – 86 h) are shown in Figure 5 for a better view.

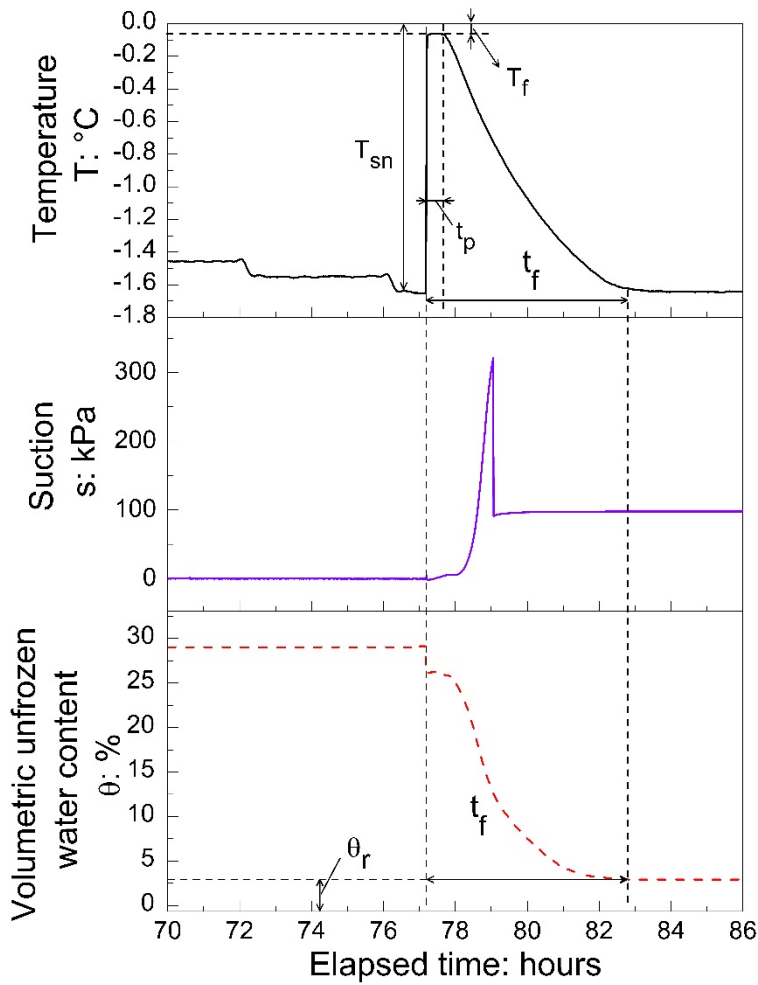


197

198 **Fig. 4** Soil temperature, volumetric unfrozen water content and suction versus elapsed time
 199 during the cooling path of test S10-T1.

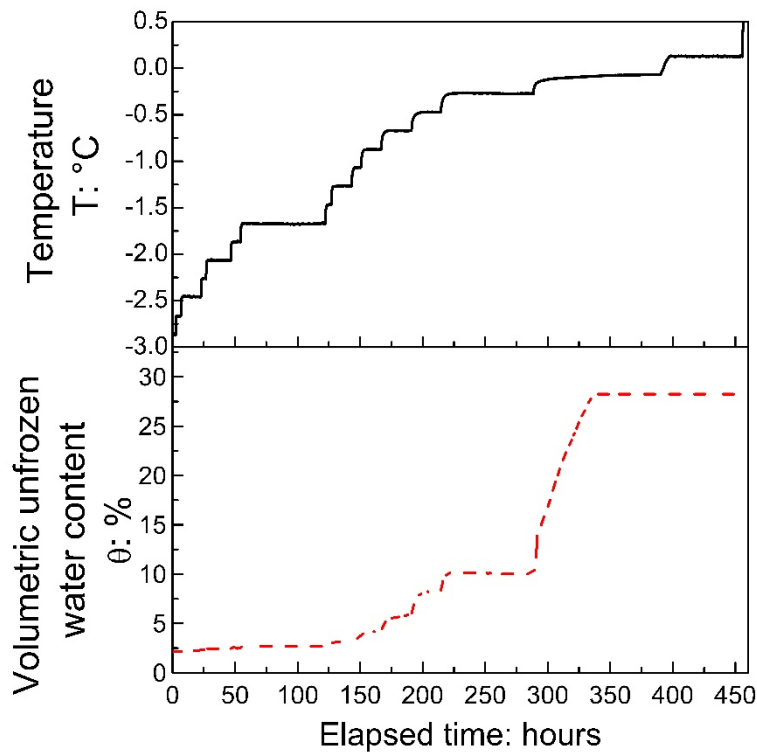
200 As shown in Fig. 5, when the bath temperature was changed from -1.5 °C to -1.6 °C (at 76 h),
 201 soil temperature changed to -1.6 °C after a few minutes. At 77 h, while the bath temperature
 202 was still maintained at -1.6 °C, soil temperature increased abruptly to -0.1 °C prior to a
 203 progressive decrease and reached the imposed temperature (-1.6 °C) again at 83 h. Soil suction
 204 started to increase at 78 h and reached a maximum value of 300 kPa prior to fall down to 100
 205 kPa. At 77 h, soil water content decreased abruptly from 28% to 26% prior to decrease
 206 progressively to 3 % at 82 h. These results are representative of a freezing process in soil (Fig.
 207 1) where the phase before 77 h corresponds to the supercooling step. At 77 h, soil water started
 208 to freeze: soil temperature increased abruptly because of latent heat release prior to decrease
 209 because of heat diffusion toward the liquid surrounding the cell; soil suction increased quickly
 210 because of the cryogenic suction induced by ice formation in the pore space (the sudden
 211 decrease of suction from 300 kPa to 100 kPa corresponded to the cavitation of the tensiometer,
 212 after this moment, the sensor did not provide anymore the real soil suction); volumetric water
 213 content decreased because of ice formation. From these typical results, the following parameters
 214 were defined to characterise the freezing process (see Figure 5): (i) temperature of spontaneous
 215 nucleation, T_{sn} ; (ii) freezing point, T_f ; (iii) residual volumetric unfrozen water content, θ_r (the
 216 value recorded at temperature equal to T_{sn}); (iv) duration of the temperature plateau, t_p ; (v)
 217 duration of the freezing process, t_f .

218 After the freezing process (from 83 h), decrease of temperature induced slight decrease of
 219 volumetric unfrozen water content (see Figure 4) while soil suction measurement was no longer
 220 available because of the cavitation of the tensiometer.



221
 222 **Fig. 5** Soil temperature, volumetric unfrozen water content and suction versus elapsed time
 223 during the freezing process of test S10-T1 (detailed view from 70h to 86 h).

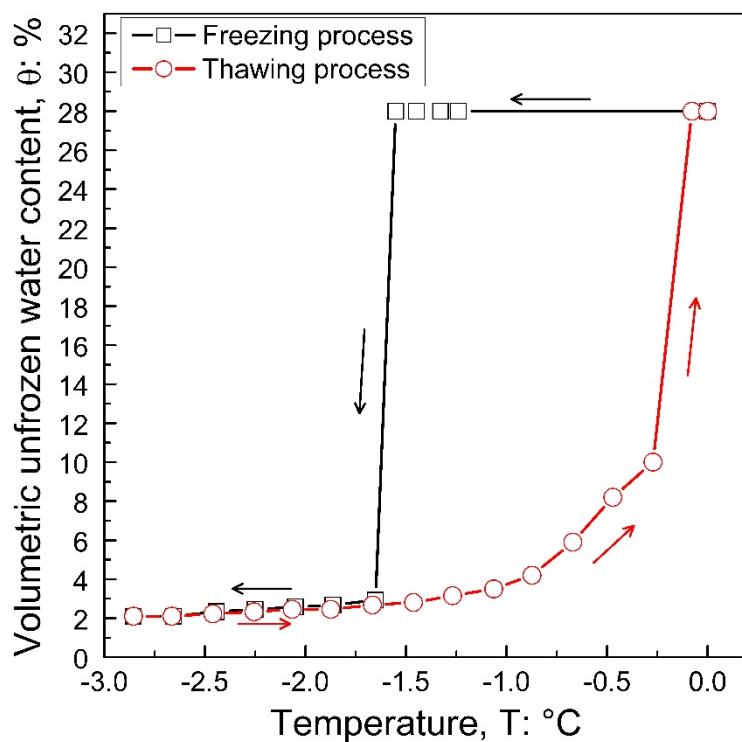
224 Fig. 6 shows the results of test S10-T1 during the heating path. During this path, temperature
 225 was increased by steps of 0.2 °C from -2.8 °C to 0 °C. It induced thawing of frozen water
 226 (corresponding to a gradual increase of unfrozen water content).



227

228 **Fig. 6** Soil temperature and volumetric unfrozen water content versus elapsed time during the
 229 heating path of test S10-T1.

230 From the results shown in Fig. 4, Fig. 5 and Fig. 6, volumetric unfrozen water content obtained
 231 at the end of each step is plotted versus the corresponding soil temperature for test S10-T1 in
 232 Fig. 7. These results correspond to the SFCC of soil S10 obtained from test S10-T1, which
 233 include both freezing and thawing paths.



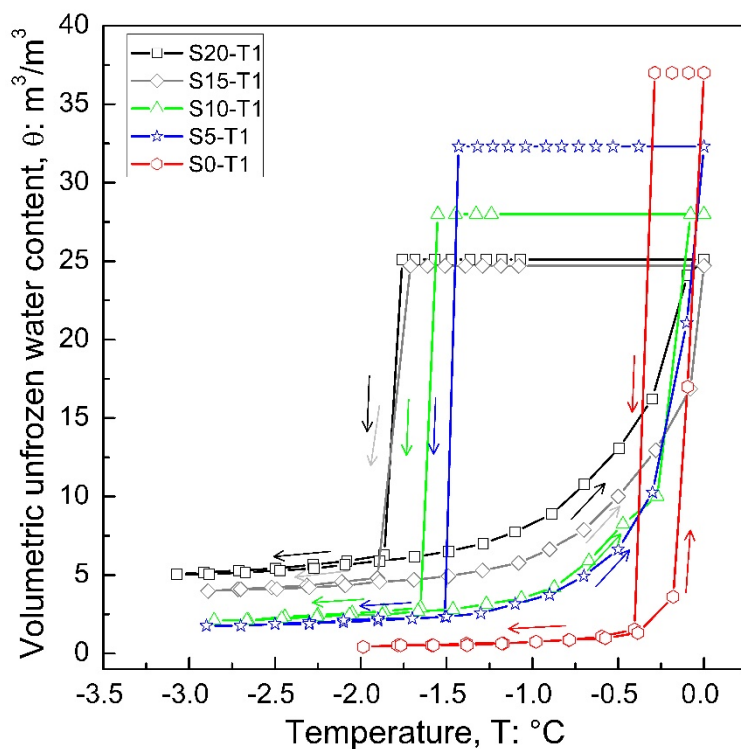
234

235 **Fig. 7** Soil freezing characteristic curve determined from test S10-T1.

236 **3.2. Effects of fines content**

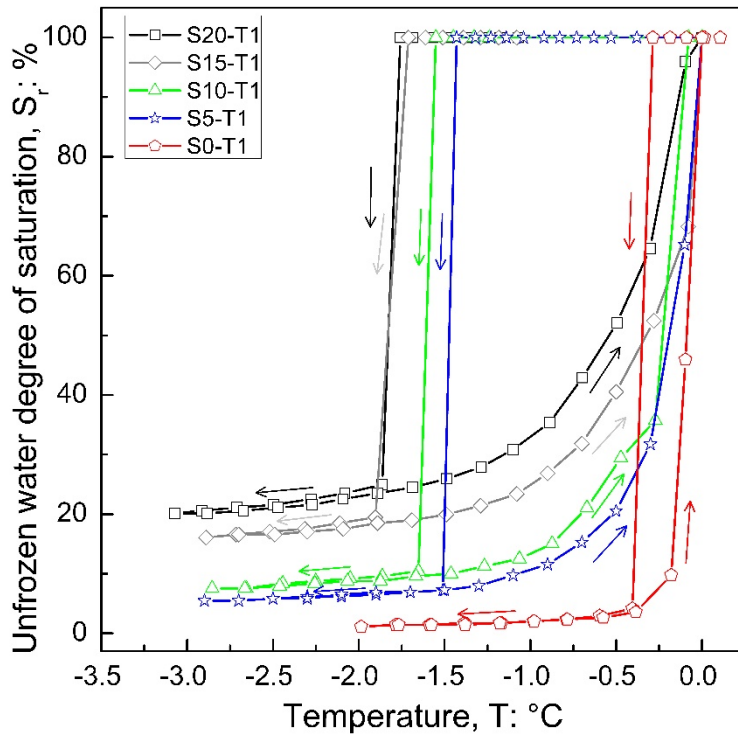
237 SFCC of all soils are shown in Fig. 8 where volumetric unfrozen water content was plotted
238 versus temperature. As the initial volumetric water content (which depends on soil dry density)
239 was different from one soil to the others, it is thus difficult to analyse the effect of fines content
240 from these results. For this reason, volumetric unfrozen water content was used to calculate
241 unfrozen water degree of saturation ($S_r = \theta/\theta_{sat}$; where θ_{sat} is the volumetric unfrozen water
242 content at saturate state). Fig. 9 shows SFCC of all soils where unfrozen degree of saturation
243 was plotted versus temperature. For each soil, from the initial saturated state, when soil
244 temperature decreased from 0 °C, soil remained saturated with unfrozen water. When
245 temperature reached the temperature of spontaneous nucleation, freezing was triggered
246 inducing significant decrease of unfrozen water degree of saturation. After this step, cooling
247 induced only slight decrease of unfrozen water degree of saturation. During the heating path,
248 unfrozen water degree of saturation increased gradually with temperature and the relationship
249 between these two quantities was significantly different from the cooling path for all soils.

250



251

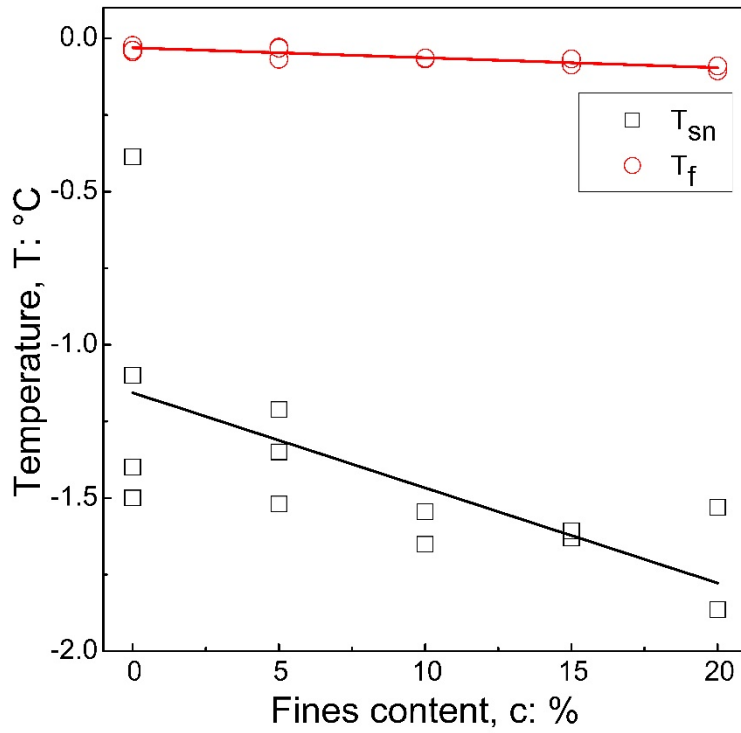
252 **Fig. 8** Soil freezing characteristic curve (volumetric unfrozen water content versus temperature)
253 for all soils.



254

255 **Fig. 9** Soil freezing characteristic curve (unfrozen water degree of saturation versus
 256 temperature) for all soils.

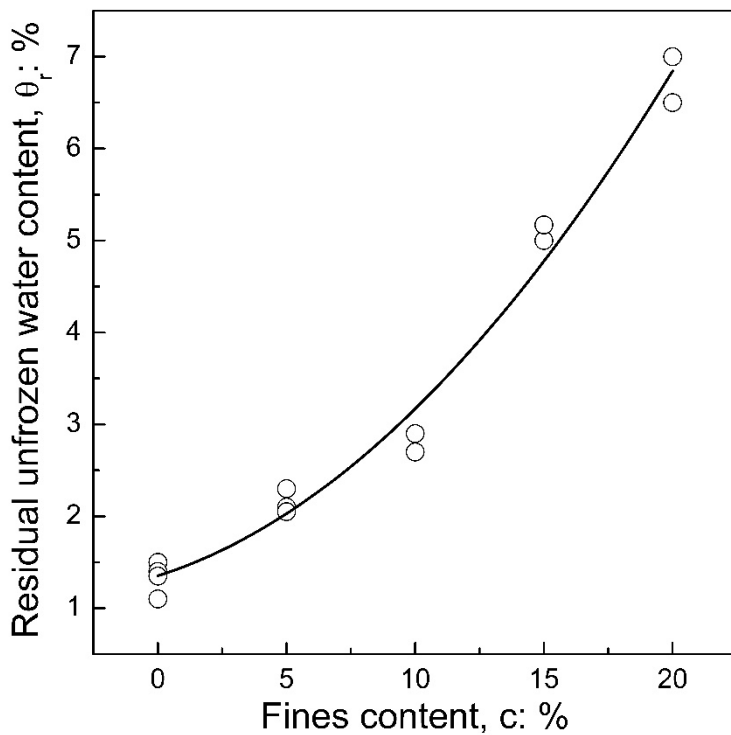
257 In order to quantitatively assess the effects of fines content, temperatures of spontaneous
 258 nucleation T_{sn} and freezing point T_f were plotted versus fines content (Fig. 10). The results
 259 show that the temperature of freezing point was close to 0 °C for all soils. The results were
 260 quite repeatable (with variation less than 0.1 °C) and only a slight trend of decrease of T_f when
 261 fines content increase could be observed. For T_{sn} , results showed a higher scattering (up to 0.5
 262 °C, except for test at 0% of clay content where this value varied from -0.4 °C to -1.5 °C). In
 263 general, T_{sn} is lower at a higher clay content.



264

265 **Fig. 10** Temperatures of spontaneous nucleation and freezing point versus fines content

266 Fig. 11 shows the residual unfrozen water content θ_f (the value determined at a temperature
 267 equal to T_{sn} , see Fig. 5) versus fines content. A good repeatability (with a scattering of 0.5 %)
 268 could be observed. The results show that residual unfrozen water content was higher at a higher
 269 fines content.



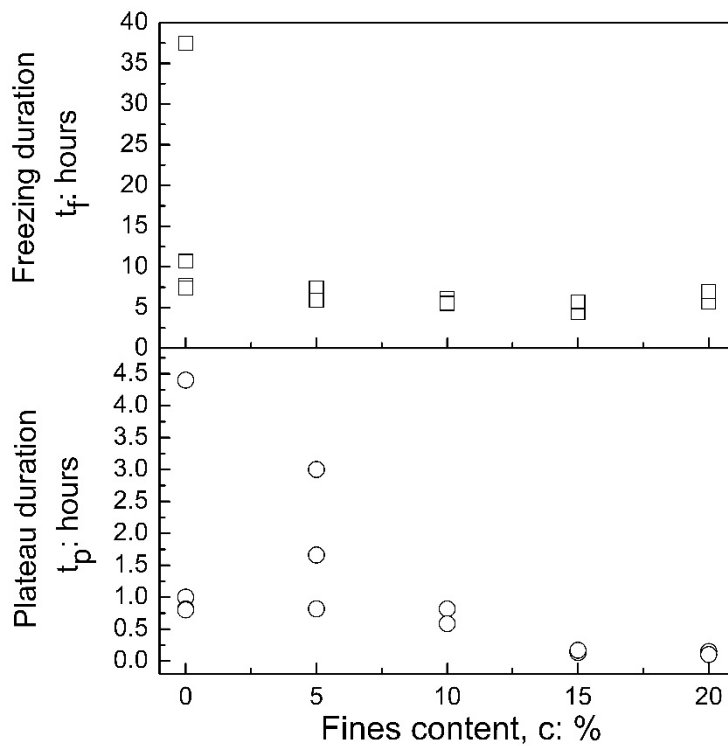
270

271 **Fig. 11** Residual unfrozen water content versus fines content.

272 Fig. 12 presents the duration of the temperature plateau t_p and the duration of the freezing
 273 process t_f (see the definition on Fig. 5) versus fines content. Results of t_p were quite scattering
 274 for 0 and 5% of fines content, varying from 0.80 to 4.40 h. They were more repeatable at higher
 275 fines contents. A general decrease of this duration when the fines contents increased could be
 276 observed. Results of t_f varied between 5 and 10 h (except one test, S0-T1 where it was very
 277 long, 37.50 h). These results did not show any clear trend.

278 Table 5 shows the obtained characteristic parameters of all tests for better comparison.

279



280

281 **Fig. 12** Duration of the temperature plateau and duration of the freezing process versus fines
 282 content.

283 Table 5: Summary of characteristic parameters in freezing of tests

Test No.	Fines content (%)	T_{sn} (°C)	T_f (°C)	θ_r (-)	t_p (h)	t_f (h)
S20-T1	20	-1.86	-0.11	6.5	0.15	5.65
S20-T2	20	-1.53	-0.09	7.0	0.10	6.95
S15-T1	15	-1.61	-0.07	5.2	0.17	5.70
S15-T2	15	-1.63	-0.09	5.0	0.13	4.00
S10-T1	10	-1.65	-0.07	2.9	0.58	5.50
S10-T2	10	-1.55	-0.06	2.7	0.80	6.10

S5-T1	5	-1.52	-0.03	2.3	0.82	5.90
S5-T2	5	-1.21	-0.07	2.1	3.10	7.35
S5-T3	5	-1.35	-0.03	2.1	1.70	7.40
S0-T1	0	-0.39	-0.04	1.5	4.40	37.50
S0-T2	0	-1.51	-0.02	1.10	1.00	7.70
S0-T3	0	-1.10	-0.04	1.40	0.80	10.70
S0-T4	0	-1.40	-0.02	1.35	0.80	7.40

284

285 **4. Discussion**

286 In this study, in order to determine the relationship between unfrozen water content and
 287 temperature during a freezing-thawing cycle, large soil specimens (150 mm in height and 150
 288 mm in diameter) were prepared in order to embed several sensors within the soil mass. To
 289 minimise thermal and any other gradients, soil temperature was changed by small steps and
 290 equilibrium was checked at the end of each step prior the subsequent step. At equilibrium, the
 291 soil temperature and unfrozen water content were thus supposed to be homogeneous within the
 292 specimen. Similar large soil specimens were equally used in previous studies investigating
 293 SFCC with TDR method for measurement of unfrozen water content [18, 53, 75, 78]. Smaller
 294 specimens were used when measurements were performed by pulsed-NMR method [30, 63,
 295 79]. In several previous works, specimens were immersed in a cooling bath with constant
 296 cooling rate or at low temperature (between -15 °C and -30 °C) and kept for several hours [17,
 297 73, 80, 81]. For determining SFCC, unfrozen water content was measured at various controlled
 298 temperatures [18, 53, 75, 76, 78, 79]. The difference between two successive controlled
 299 temperatures in these studies varies between 0.3 °C and 5 °C. In the present work, temperature
 300 steps of 0.1 °C and 0.2 °C were chosen before the occurrence of freezing phenomenon and
 301 afterward, respectively, in order to determine more accurately the freezing point, the
 302 temperature of spontaneous nucleation and the SFCC.

303 The measurement of unfrozen water content in the present study was converted from the
 304 measurement of apparent dielectric constant. In this study, under the influence of temperature,
 305 dielectric constant of each phase in soils changes, particularly those of water and ice [82, 83].
 306 Several models exist to estimate moisture content from unfrozen soil apparent dielectric
 307 constant [51, 53, 57–60, 84–86]. The most used is Topp's empirical model [57] but it is not
 308 compatible with frozen soils [44, 58, 87]. Otherwise, Smith and Tice [58] proposed a model
 309 based on comparison of unfrozen water content measured from NMR and TDR methods for 25
 310 soils covering a wide range of specific surface areas. For this reason, in the present work, the
 311 model of Smith and Tice [58], which provides an accuracy of $\pm 3\%$ compared to measurements
 312 from NMR method, was used for frozen soils.

313 Hysteresis of SFCC (difference between the freezing and the thawing curves) is usually
 314 attributed to the same factors inducing hysteresis in SWCC, such as the effect of electrolytes,
 315 pore geometry, pore blocking, effect of contact angle and change in pore structure [78].
 316 Actually, in a freezing process, increasing solute concentration by forming ice from water
 317 increases the effect of electrolyte. Otherwise, forming ice also changes soil skeleton that affects
 318 matric potentials of soils. In addition, the hysteric behaviour is also mainly attributed to
 319 supercooling of pore water [18, 54, 62, 63]. Instead of freezing at 0 °C, pore water is necessarily
 320 supercooled at lower temperature. In the present work, an insignificant hysteresis of θ_u was

321 observed for all soils below T_{sn} (at frozen state). First, the effect of electrolytes can be ignored.
322 Second, temperature below T_{sn} of -1 °C to -2 °C corresponds to a suction of 1 MPa to 2.5 MPa
323 following the Clapeyron equation. This high range of suction corresponds mainly to water in
324 micropore (intra-aggregates) in the clay matrix where SWCC is also reversible. As a result,
325 hysteresis of SFCC observed in the present work could be contributed mainly to supercooling.
326 After the triggering of freezing, SFCC obtained at temperature lower than T_{sn} were generally
327 reversible (see Fig. 8 & Fig. 9).

328 Results shown in Fig. 9 demonstrate significant effect of fines content on the thawing path of
329 SFCC; at a given temperature, a higher unfrozen water degree of saturation was obtained at a
330 higher fines content. These results are consistent with the findings of previous works [28, 55,
331 63, 70]. Following these studies, Gibbs-Thompson equation can be used to relate the pore size
332 distribution and the thawing path of SFCC; a lower temperature corresponds to a smaller pore.
333 In the present work, soil having higher fines content would have a larger volume of micropores
334 (inter-aggregates and intra-aggregates pores) a lower volume of macropores (space between
335 sand particles).

336 T_{sn} determined in this study can be associated to supercooling. Fig. 10 shows that this parameter
337 generally decreased with an increase of fines content and it was measured with a relatively high
338 scattering. For bulk water, T_{sn} depend on numerous factors such as sample volume, cooling
339 velocity, the presence and concentration of solutes, the presence of solid impurities, effects of
340 external fields (impulse waves, electromagnetic radiation, etc.) [9, 89, 90]. In the case of soils,
341 additional factors can be soil components and their fractions. Many studies determined T_{sn} of
342 various soils and found that increasing clay content in soils decreases temperature of
343 spontaneous nucleation to lower range [15, 18]. These studies focused on clays or clay and silt
344 and these results agree with sandy soils in the present study. It is noted that the supercooling is
345 considered as a necessary phase to activate nucleation process and it appears in both cases,
346 either in free pure water or within the porous volume of soils. Because of the high value of
347 released latent heat, about 334 J/g, which appears during nucleation process, water needs to be
348 supercooled at T_{sn} for equilibrating energy before crystallization. According to Yershov [91],
349 T_{sn} is remarked as the temperature at which embryo nuclei form and grow to the critical sizes,
350 about 472 H₂O corresponding to 10⁻²⁶ m³. The relatively high scattering of results obtained in
351 the present work can be thus explained by the random behaviour of the crystallization process.
352 The slight effect of fines content on T_{sn} can be explained by the effect of soil pore size
353 distribution on the supercooling: soil having a higher fines content would have higher volume
354 of micropores, and T_{sn} is generally lower in a smaller pore.

355 Numerous studies investigated T_f and showed that T_f depends on many factors such as salt
356 content [20, 73, 80, 92, 93], salt types [17, 81], initial water content [15], soil types [10, 18, 30,
357 55, 94, 95], etc. In the present study, T_f was found close to 0 °C for all soils. This result can be
358 explained by two main reasons: soils were studied at saturated state and fines content is
359 sufficiently low. Bing and Ma [81] obtained similar results with saturated sandy soil containing
360 less than 7.5% of clay. Furthermore, freezing point remains constant also above a certain value
361 of water content for all soils [9, 81, 96]. Actually, for the soils considered in the present study,
362 with relatively low contents of low plasticity kaolin clay, the amount of bound water should be
363 negligible and T_f should be similar to that of bulk pure water, i.e. close to 0 °C.

364 Residual unfrozen content was found higher at a higher fines content (Fig. 11). It is believed
365 that residual unfrozen relates almost directly to the amount of specific surface of soils.
366 According to several studies [18, 28, 30, 55, 63], unfrozen water content remaining in soils at

367 the same temperature decreased in the following order: clay, silts, sands and gravel. Following
368 Bing and Ma [81], only free water was frozen when freezing is triggered. Unfrozen water should
369 then correspond to bound water. According to Tian et al. [63], the amount of bound water in
370 soils is proportional to the thickness of the electric double layer and specific surface area. In the
371 present study, a higher fines content corresponds to a higher specific surface area and then a
372 higher amount of bound water.

373 The duration of temperature plateau, t_p , would depend then on the amount of latent heat released
374 when freezing is triggered. This amount mainly depends on T_{sn} , as shown in Table 5. As the
375 results of T_{sn} show significant scattering and a general slight increase when fines content
376 increased (Fig. 10), similar trends were observed with t_p (Fig. 12). The duration of the freezing
377 process, t_f , which is much longer than t_p , correspond to the thermal diffusion of latent heat
378 released during the whole freezing process. This duration would depend thus mainly on the
379 thermal diffusivity of the frozen soil (which at the same time evolves during freezing).

380

381 5. Conclusions

382 The results obtained in this study show that fines content in sandy soils significantly influenced
383 the soil behaviour under a freezing-thawing cycle. Based on the investigation of five levels of
384 fines content (varying from 0 to 20 %), the following conclusions can be addressed:

- 385 - When the temperature decreased from 0°C, freezing was triggered at T_{sn} inducing a
386 sudden decrease of θ_u from the saturated state to the residual state. Afterward, θ_u
387 continued to decrease but with a lower rate. The subsequent heating induced an increase
388 of θ_u (which represents a progressive melting of frozen water).
- 389 - The thawing path of SFCC was strongly dependent on the fines content; at a given
390 temperature, a higher θ_u was observed for a higher fines content.
- 391 - T_{sn} was higher at a higher fines content and varied between -1.0 °C and -2.0 °C.
- 392 - T_f varied between 0°C and -0.2 °C, only a slight decrease of T_f with an increase of fines
393 content was observed.
- 394 - θ_r (varied from 1 % to 7 %) was higher at a higher fines content.
- 395 - t_p was found scattering and slightly decreased when fines content increased.
- 396 - t_f was found independent of fines content.

397 The findings of the present study would be helpful to predict the soil behaviour under freezing-
398 thawing process. That would imply several applications in cold regions and also in geotechnical
399 engineering ground improvement by artificial ground freezing.

400 Data availability

401 The datasets generated during and/or analysed during the current study are available from the
402 corresponding author on reasonable request.

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