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Abstract

Responses of soils to climate warming during winter can be studied by monitoring soil temperature variations in the vadose zone. Freeze-thaw cycles during winter can have a significant impact on soil biogeochemical and physical processes. Observing soil temperature at different depths offers important insights into heat availability, which influences biogeochemical activity and solute movement driven by temperature gradients. However, it is difficult to replicate the relevant processes in soil columns without maintaining freezing temperatures with an unfrozen soil layer below the freezing interface. This paper describes the development and experimental verification of a laboratory-scale physical model to assess the effect of freeze-thaw cycles on biogeochemical processes in unsaturated soil. The results show that the experimental design can (i) induce cyclical freezing of soil down to desirable depths, (ii) maintain vertical temperature gradients and (iii) ensure the rest of the column remains unfrozen below the freezing interface for pore water sampling. The rate of freezing is suitable for quick freeze-thaw cycles (0.19°C/minute). This setup can be further developed to observe solute transport and attenuation in variably-saturated soil and soil moisture status for different freeze-thaw regimes under simulated climate change scenarios.

Keywords – Freeze-thaw cycle, freezing jacket, soil column, soil temperature, unsaturated soil

54 1. INTRODUCTION

55 Freeze-thaw cycles (FTCs) are increasingly recognized as a critical process
56 influencing soil systems in temperate and high-latitude regions. Warming winters and an
57 increase in snow-free days, driven by climate change, have resulted in frequent, intense, and
58 rapid FTCs these regions (Kreyling et al., 2008; Schmitt et al., 2008; Sang et al., 2021). Soil
59 FTCs can affect the movement of solutes (Man et al., 2022; Xu, 2022; Zong et al., 2023),
60 pore and soil structure (Leuther and Schlüter, 2021; Miranda-Velez et al., 2023; Zhang et
61 al., 2021), and plant and microbial activity (Koponen et al., 2006; Kreyling et al., 2010;
62 Sorensen et al., 2018). FTCs have been found to increase nutrient fluxes from soil, which
63 can be lost via leaching or as upward accumulation on soil surface (Yanai et al., 2004;
64 Hentschel et al., 2008; Matzner and Borken 2008; Gao et al., 2018; Sun et al., 2021; Liu et
65 al., 2023). Understanding factors which affect the development of FTCs in variably-
66 saturated soil and simulating future FTC patterns is therefore important in managing
67 potential impacts of nutrient loads and pollutant export from to soil and water resources
68 under these conditions.

69 Observation of FTCs in the field is difficult, due to uncertainties associated with the
70 occurrence and frequency of these phenomena. Long-term seasonal monitoring over several
71 years provides important observations at the catchment scale (Zhang et al., 2022; Liu et al.,
72 2023), but it is unrealistic to use these observations for accurate simulation and prediction.
73 Laboratory-based studies have been undertaken to overcome the uncertainties related to
74 field studies, by collecting soil samples from a field site and inducing FTCs in a controlled-
75 temperature environment (Sharma et al., 2006; Vestgarden and Austnes, 2009; Stres et al.,
76 2010; Li et al., 2024). FTCs in soil can be induced by keeping the samples (e.g. undisturbed
77 soil cores) in a temperature-controlled cold room (Rooney et al., 2022), incubators
78 (Koponen and Martikainen, 2004; Wu et al., 2014), or laboratory freezers (Sharma et al.,

79 2006). Soil cores were packed in PVC cylinders or glass jars and kept in controlled-
80 temperature environments. Arrangements for different chemical sampling were made
81 destructively, i.e., air-drying and sieving, water extraction for chemical analyses (Wu et al.,
82 2014; Wu et al., 2021; Rooney et al., 2022) or non-destructively for microbial sampling
83 (Sharma et al., 2006), at the end of the freezing/thawing. This procedure, however, cannot
84 be used for instantaneous measurements of soil temperature and moisture movement. Soil
85 column experiments which can be used for unsaturated freezing-thawing experiments are
86 rare. To overcome these limitations, soil column experiments have been conducted to
87 experimentally simulate field conditions (Fitzhugh et al., 2001; Hermansson, 2004; Evirgen
88 and Tuncan, 2019; Xu et al., 2019; Libby et al., 2020). Fitzhugh et al. (2001) and Libby et
89 al., (2020) attempted to simulate field conditions using soil columns in outdoor and indoor
90 sites, respectively. The experiment by Hermansson (2004) experiment involved freezing a
91 soil column from the top and observing the frost penetration. However, this experiment, is
92 not reliable due to poor reproducibility and may result in inadequate freezing depths. The
93 experiment by Xu et al. (2019) involved circulating warm and cold fluids at the top and
94 bottom of a soil column to develop FTCs. Evirgen and Tuncan's (2019) experiment
95 circulated coolant fluid through pipes which were buried within soil to freeze it. The
96 objectives for each experiment were different from the objectives of the present study. The
97 main objective of the present study was to develop a mechanism for freezing/thawing
98 experiments which can be useful in studying unsaturated soil behaviour (both chemically
99 and physically) during quick and frequent FTCs. Previous experimental designs used to
100 investigate FTCs in unsaturated soil have important limitations, in that (i) they are unsuitable
101 to create rapid FTCs (as is observed in the field), (ii) conducted at unrealistically low (< -
102 10°C), and (iii) they cannot create a warmer soil mass beneath the frozen soil zone, as occurs
103 naturally. Topsoil temperatures <-10°C are not observed during quick and frequent FTCs in

104 temperate regions. Existing studies using laboratory freezers typically use a minimum
105 temperature of -20°C, thus killing soil organisms responsible for microbial activity during
106 FTCs at deeper levels (Henry, 2007). As not all soil water freezes during winter, active solute
107 transport may still occur through the soil profile, driven by the developed temperature
108 gradients. Therefore, maintaining a warm soil zone beneath the frozen layer is important to
109 observe instantaneous changes occurring during FTCs in soil.

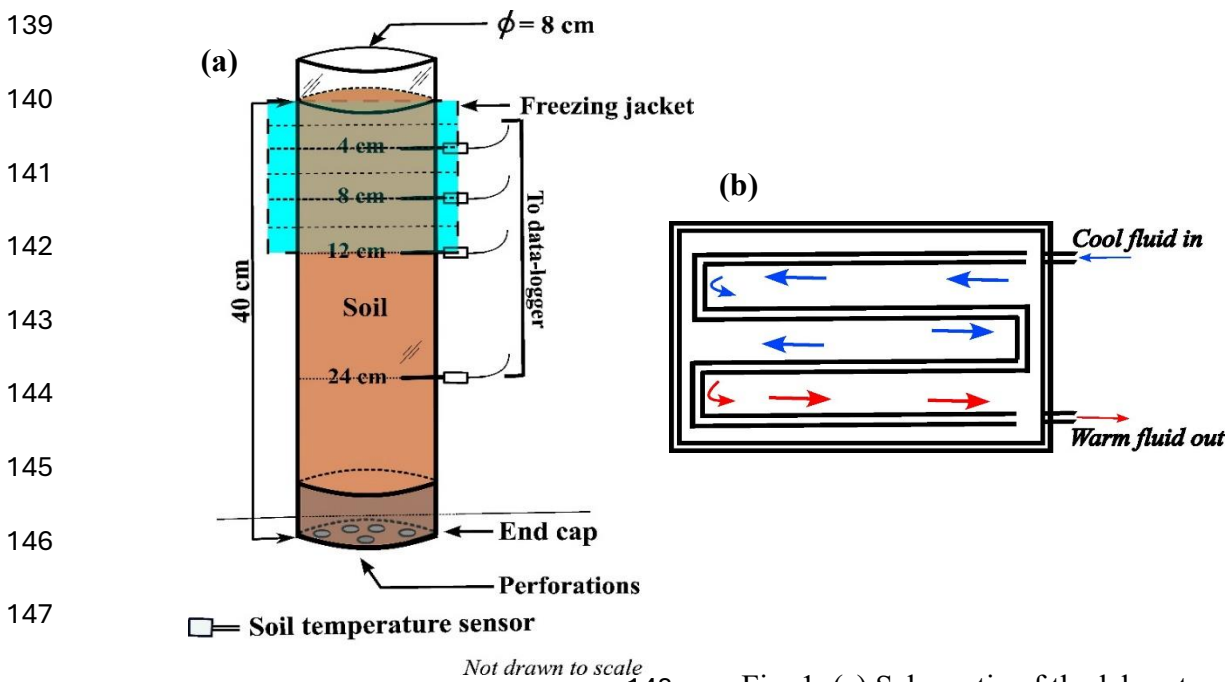
110 The present study describes the development of a novel laboratory-scale physical
111 model to experimentally determine the effect of FTCs on biogeochemical and solute
112 transport processes in variably-saturated soil under simulated climate change conditions.
113 The specific objectives of the research are to: (i) develop the experimental model to maintain
114 freezing temperatures and unfrozen soil simultaneously in a soil column, and (ii) verify the
115 performance of the physical model in replicating FTC propagation and temperature
116 gradients in soil columns.

117 2. MATERIALS AND METHODS

118 2.1 Conceptual design of physical model

119 Soil acts as a heat reservoir and can retain heat during winter to support microbial activity
120 at deeper depths (Li et al., 2020; Sahoo, 2022). The effective plant and microbial activity
121 can be found within the top 30 cm of soil (Hao et al., 2021). Soils across the temperate zone
122 and high latitudes experience multiple and quick freeze-thaw cycles during winter
123 (Kreyling, 2010). Frequent FTCs can have a significant impact on solute movement in soil,
124 which may result in leaching of soil nutrients to groundwater (Matzner and Borken, 2008),
125 upward accumulation of solutes on soil surface (Liu et al., 2022), and seasonal elevation of
126 nutrient content in stream water during early spring (Creed et al., 1996; Chen et al., 2020).
127 Observing the movement of solutes in unsaturated soil due to imposed temperature gradients
128 can be challenging at the field-scale due to uncertainties related to replication. A simplified

129 laboratory-scale physical model was conceptualized to replicate FTCs in a soil column and
 130 changes in critical soil physical-chemical properties resulting from them. Assuming an
 131 average root-zone soil depth of 100 cm and active depth of soil microbial activity as 30 cm,
 132 the occurrence FTCs was hypothesized within the top 30 cm soil depth. An efficient freezing
 133 mechanism which can freeze only the top 30 cm and create precise temperature gradients
 134 within the soil profile, while ensuring the remaining depth remains unfrozen was necessary.
 135 A jacket-like mechanism was proposed, which remains wrapped around a soil column
 136 extracting the heat from the soil down to the desirable depth. With the aim of testing the
 137 efficiency of a freezing jacket to freeze the soil to a desirable depth, a 1/4th scaled-down
 138 version of laboratory set up was constructed (Fig. 1).



148 Fig. 1. (a) Schematic of the laboratory soil
 149 column setup, showing temperature gradients (a) soil column; (b) Coolant circulation
 150 mechanism through the freezing jacket.

151 At this stage, only the freezing mechanism and freezing efficiency of the jacket was
 152 tested without adding any other arrangements to observe solute movement. Soil temperature
 153 sensors (thermocouples [Fig. 1]) were placed within the depths corresponding to the layers

154 of freezing jacket. One thermocouple was installed at a deeper depth of soil column away
155 from the jacketed layer to test the soil temperature in the unfrozen layer.

156 2.2 Soil column

157 An unplasticized polyvinyl chloride (UPVC) tube (8 cm internal diameter and 48 cm
158 height) was used to construct a soil column (Fig. 2). UPVC has a thermal conductivity of
159 the same order of magnitude as a natural soil and therefore a weak temperature gradient will
160 exist between soil and the tube. Lateral soil moisture movement towards the tube is likely

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171 Fig. 2. (a) UPVC column used for the FT experiments, and (b) laboratory set up for FT
172 studies.

173 to be negligible (Gray and Granger, 1986), thus inducing a one-dimensional flow within the
174 column. The side wall of the tube was roughened to restrict any side wall flow (Lewis and
175 Sjöstrom, 2010). Air-dried soil was packed in the tube by regular tamping to obtain a
176 uniform packing throughout the column. The soil column was maintained at a height of 40
177 cm, leaving the upper 8 cm for the accumulation of any water/snow during the FT
178 experiments. The tube was fitted with a hollow end cap [Fig. 2(a)]. The end cap contained

179 a 100 μ m stainless steel mesh to retain soil particles, while allowing samples of free draining
180 water to be collected from the base of the column. The soil total porosity was maintained
181 between 55 – 57%, with a corresponding bulk density of 1.18 – 1.2 g/cm³.

182 2.3. Freezing jacket

183 A controlled depth of freezing in a soil column can be achieved by wrapping the
184 column with a freezing jacket down to the required depth. Ideally, the freezing jacket should
185 remain tightly held to the column to remove the heat from the soil at any given depth. In the
186 present study, the freezing jacket was constructed from aluminium alloy for its good heat
187 conductance, even distribution of heat, and resistance to corrosion [Fig. 3(a)] and a coolant
188 (ethylene glycol) circulated through it to achieve the desired freezing temperature in soil.
189 The freezing jacket consists of three detachable layers (each 4 cm in width) for controlling
190 freezing depth up to 12 cm depth.



191 Fig. 3. (a) Freezing jacket, (b) front-view of freezing jacket with detachable layers, and (c)
192 internal circuit for coolant circulation.

193 Each layer was further machined to obtain two sub-layers (each 2 cm in width) to
194 achieve freezing temperatures within the desired depth [Fig. 3(b)]. The internal plates were
195 fitted with a groove to form a circuit for coolant circulation [Fig. 3(c)]. A chiller circulator
196 [Fig. 2(b)] was used to circulate chilling fluid (i.e. glycol in the present study) through the
197 freezing jacket. The detachable layers can be added or removed depending on the required
198 freezing depth. One or more 4 cm-layers (or two sub-layers together) was either added or

199 removed to test the performance of the jacket across different simulated freezing depths.
200 The unjacketed part of the soil column should always remain above freezing temperature
201 during the experiment.

202 **2.4. Soil and ancillary materials**

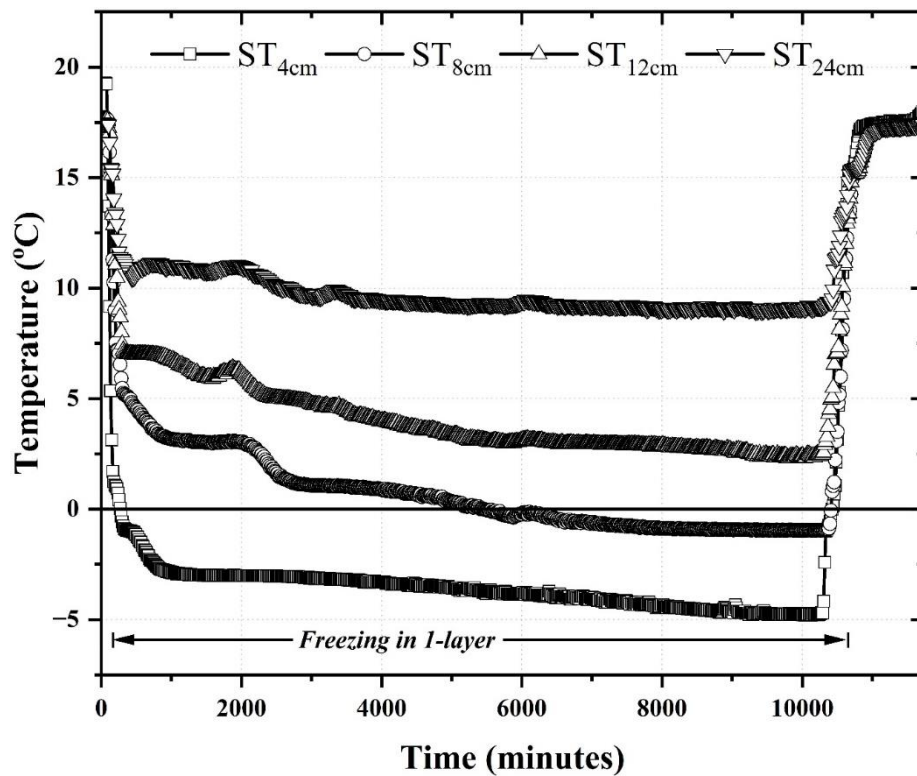
203 Soil samples were collected from an agricultural farm in Wakefield, United Kingdom.
204 The soil was predominantly weathered sandstone with impermeable layer starting
205 approximately at 15 cm depth. Large stones, debris or litter material were removed before
206 the experiment by sieving the soil sample. Screened and homogenized soil from this
207 preparation was used to create a soil column for good method reproducibility. The sample
208 has sandy loam texture (71% sand, 18% silt, and 11% clay) and the column contained soil
209 with particle sizes 3.35 mm or finer in grain size. The soil drains rapidly and has an organic
210 matter content of 7.2 wt %. The initial soil saturation was completed from the base upwards,
211 allowing the moisture to wick upwards by capillary action (Lewis and Sjöstrom, 2010).
212 Subsequent saturation with water was done from the top. Freezing of the soil was started
213 once the soil reached a soil moisture content of 35 – 40% (field capacity of the soil used). A
214 constant head permeability test determined the hydraulic conductivity of the soil as 26.36
215 cm/day.

216 Thermocouples were installed at 4 cm, 8 cm, and 12 cm from the soil surface (three
217 depths within the full jacketed zone) and 24 cm (unjacketed zone) of the soil column to
218 observe temperature changes within the soil profile during FT experiments. The
219 thermocouples monitored and recorded temperature changes within the centre of the column
220 at 15-minute intervals. The duration of the experiments varied between 1 – 9 days, to
221 examine the performance of the overall set up for representative and realistic durations of
222 FTCs. The laboratory set up was operated at a room temperature of 22°C.

223

224 **3. RESULTS AND DISCUSSION**

225 The freezing jacket decreased the soil temperature from 20.4°C to 0.08°C (near
226 freezing temperature) in 105 minutes (1 hour 45 minutes) at a rate of 0.19°C/minute. The
227 freezing temperature can be maintained until the chiller circulator begins operating. Once
228 the chiller circulator was switched off, the soil started thawing at a rate of 0.05°C/minute.
229 The coolant was circulated through the freezing jacket at -10°C. The chiller circulator could
230 operate continuously for long durations (up to 7.06 days in the present study), maintaining
231 freezing temperatures within the jacketed region of the soil column (Fig. 4). Freezing
232 temperatures beyond the unjacketed region started appearing after 4 days, reaching a
233 minimum temperature of -0.95°C. The minimum temperature within the jacketed region

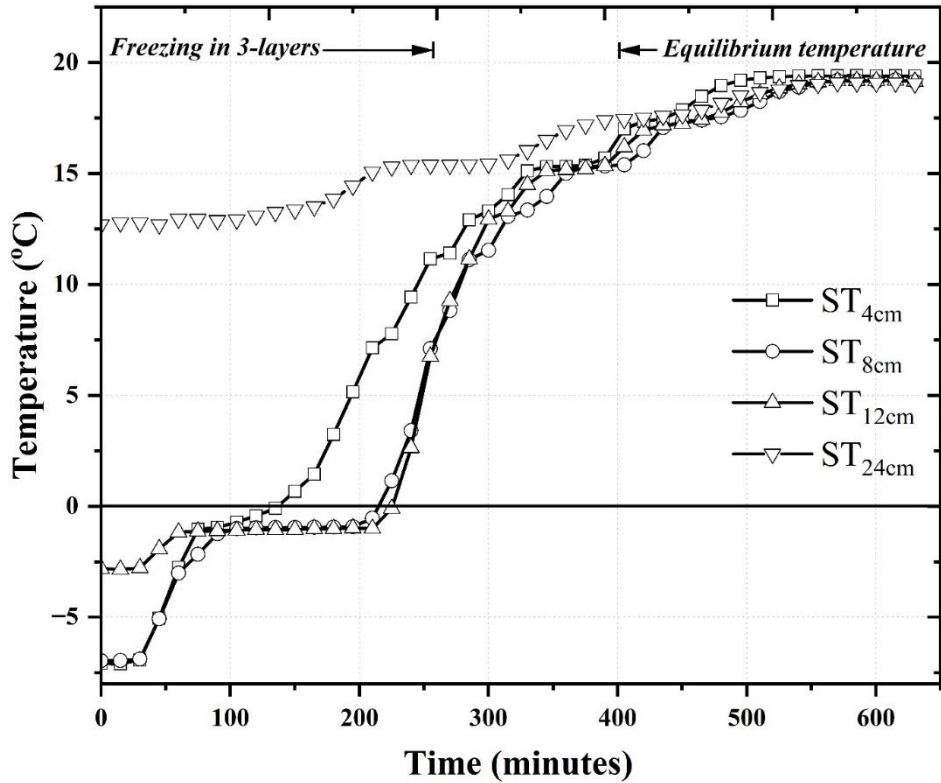


243 Fig. 4. Freezing soil column with 1-layer of jacket (4 cm depth).

244 varied between -7.10°C and -4.79°C. The temperature at 24 cm depth always remained
245 above +10°C (Fig. 4-6).

246 Temperature gradients occur at different depths in soil at the field-scale (Contosta et
247 al., 2016; Sahoo, 2022). The freezing jacket was able to maintain a consistent temperature

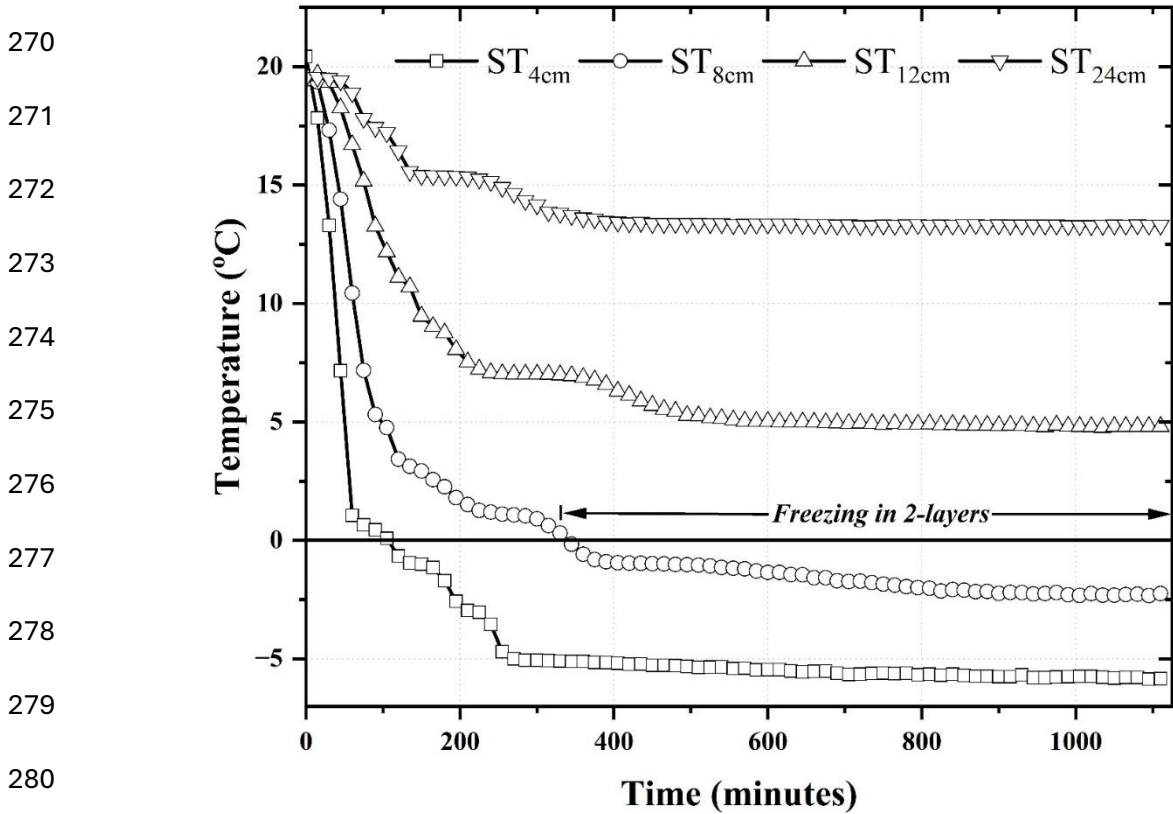
248 difference within the jacketed region for significant time periods (Fig. 4-6). Soil at deeper
249 depths was warmer than at the soil surface. After thawing, soil temperatures within the entire
250 column reach an equilibrium temperature (Fig. 5).



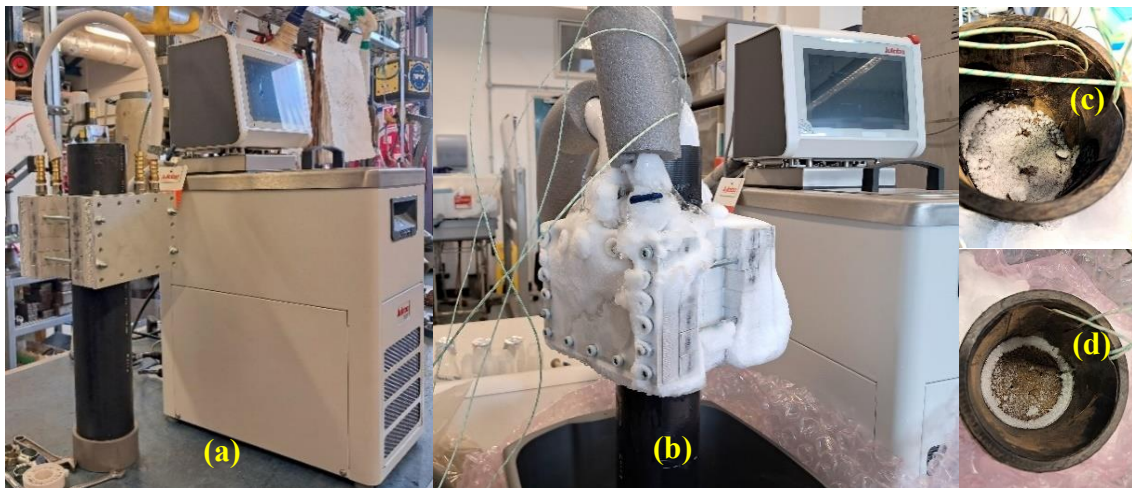
262 Fig. 5. Freezing soil column with 3-layers of jacket (12 cm depth).

263 The efficiency of the jacket in restricting the freezing depth was precise and could be
264 achieved down to 4 cm depth with each aluminium layer (Fig. 6). The addition or removal
265 of layers suggests that the freezing jacket is effective in cooling the soil down to the desired
266 depth and duration and can maintain soil temperature differences within the frozen soil
267 depth. With changes in the number of layers in the jacket, the amount of accumulated ice on

268 the soil surface is significantly reduced (Fig. 7). This change suggests that the freezing jacket
 269 layers were able to replicate mild and severe winter scenarios at the laboratory-scale.



281 Fig. 6. Freezing soil column with 2-layers of jacket (8 cm depth).



282 Fig. 7. (a) Column with the freezing jacket, (b) frozen column with 3-layers of
 283 jacket, and (c) and (d) accumulation of ice on frozen soil surface, with 3-layers and 1-layer
 284 of jacket, respectively.

285 The unjacketed portion of the soil column was not affected by the environmental
286 temperature changes. UPVC is a poor conductor and heat does not affect the temperature of
287 soil within the column. The temperature changes within the unjacketed column were due to
288 the movement of the freezing front from above the soil column, the heat retained during
289 packing the column, and the movement of soil water during the saturation stage.

290 Existing methods of chilling soil samples for studies of solute biogeochemistry require
291 freezing the soil at $<-10^{\circ}\text{C}$ inside a laboratory refrigerator or chilling incubator (Bochove et
292 al., 2000; Henry, 2007; Patel et al., 2021; Edwards et al., 2024). However, these low
293 temperatures are deleterious to soil microorganisms and are not usually encountered in
294 temperate regions (Henry, 2007). The present setup was able to achieve and maintain
295 freezing temperatures $> -10^{\circ}\text{C}$, that can support biogeochemical reactions within the soil
296 profile. Soil temperature can be regulated in the chiller circulator to the desired temperature
297 and maintained consistently over extended periods.

298 Hence the laboratory-scale physical model developed in this study can be used to
299 reproduce field-scale scenarios of climate change-induced FTCs to understand the effect of
300 this phenomena on the status and movement of solutes within the soil profile. In particular,
301 the methodology can enable solute fluxes to surface water and groundwater resources to be
302 determined for specific climate-change scenarios. This is important to predict and manage
303 the impact of nutrients and other potential contaminants to these receiving waters from
304 different sources in catchments.

305 **4. CONCLUSIONS**

306 This study has introduced a novel laboratory-scale physical model designed to
307 investigate the effects of FTCs on the physico-chemical processes occurring in variably-
308 saturated soils. The model was used to create temperature gradients arising from FTCs
309 within a required depth at the laboratory-scale for extended periods. This setup can be used

310 for experiments which consider the influence of climate change on FTCs and which require
311 the creation of precise *in situ* soil temperature variations with depth. Modelling of
312 unsaturated zone processes are essentially considered as one-dimensional unless stated
313 otherwise. This setup was tested successfully for unsaturated soil processes and can be
314 further used to examine soil moisture distribution and solute transport induced by FTCs (or
315 winter warming) and the effect of this phenomena on solute (e.g. nutrient and potential
316 pollutant) fluxes to surface water and groundwater. In contrast to soil freezing, the jacket
317 can be used for soil heating experiments (or to increase soil temperature within specific
318 depths) by circulating warm fluid in a similar way. This jacket-column arrangement can be
319 used for one column at a time, therefore, studies which require simultaneous testing of
320 multiple samples may require multiple jackets for each soil column. Further studies will be
321 undertaken with this methodology, using a scaled-up version of the column and freezing
322 jacket, with sensor arrangements to observe solute transport and other biogeochemical
323 processes induced by FTCs under simulate climate-change scenarios.

324

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499

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516 **Data Availability**

517 Data will be made available upon request.