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1	A laboratory-scale physical model for freeze-thaw studies in soil columns under
2	simulated climate change conditions
3	By
4	Madhumita Sahoo, Paul Bentley, Andrew Smith, Paul Blackbourn, Kieren Howarth, Domenico
5	Bau and Steven Thornton
6	School of Mechanical, Aerospace and Civil Engineering, University of Sheffield, Sheffield
7	United Kingdom
8	
9	Corresponding Author:
10	Madhumita Sahoo
11	MSCA Postdoctoral Researcher,
12	Groundwater Protection and Restoration Group (GPRG),
13	School of Mechanical, Aerospace and Civil Engineering, University of Sheffield, Sheffield
14	United Kingdom
15	Email: m.sahoo@sheffield.ac.uk; sahoomadhu1989@gmail.com
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## Abstract

Responses of soils to climate warming during winter can be studied by monitoring soil 30 temperature variations in the vadose zone. Freeze-thaw cycles during winter can have a 31 significant impact on soil biogeochemical and physical processes. Observing soil 32 temperature at different depths offers important insights into heat availability, which 33 influences biogeochemical activity and solute movement driven by temperature 34 35 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 36 37 interface. This paper describes the development and experimental verification of a laboratory-scale physical model to assess the effect of freeze-thaw cycles on 38 biogeochemical processes in unsaturated soil. The results show that the experimental 39 40 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 41 below the freezing interface for pore water sampling. The rate of freezing is suitable for 42 quick freeze-thaw cycles (0.19°C/minute). This setup can be further developed to observe 43 solute transport and attenuation in variably-saturated soil and soil moisture status for 44 different freeze-thaw regimes under simulated climate change scenarios. 45

- 46
- *Keywords* Freeze-thaw cycle, freezing jacket, soil column, soil temperature, unsaturated soil
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1.

## INTRODUCTION

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

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

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

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

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

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# 2. MATERIALS AND METHODS

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#### 2.1 Conceptual design of physical model

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

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



# column setup, showing temperature gradients (a) soil column; (b) Coolant circulation mechanism through the freezing jacket.

At this stage, only the freezing mechanism and freezing efficiency of the jacket was tested without adding any other arrangements to observe solute movement. Soil temperature sensors (thermocouples [Fig. 1]) were placed within the depths corresponding to the layers of freezing jacket. One thermocouple was installed at a deeper depth of soil column awayfrom the jacketed layer to test the soil temperature in the unfrozen layer.

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# 2.2 Soil column

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



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

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

a 100 $\mu$ m stainless steel mesh to retain soil particles, while allowing samples of free draining water to be collected from the base of the column. The soil total porosity was maintained between 55 – 57%, with a corresponding bulk density of 1.18 – 1.2 g/cm<sup>3</sup>.

182 **2.3. Freezing jacket** 

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



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

Each layer was further machined to obtain two sub-layers (each 2 cm in width) to achieve freezing temperatures within the desired depth [Fig. 3(b)]. The internal plates were fitted with a groove to form a circuit for coolant circulation [Fig. 3(c)]. A chiller circulator [Fig. 2(b)] was used to circulate chilling fluid (i.e. glycol in the present study) through the freezing jacket. The detachable layers can be added or removed depending on the required freezing depth. One or more 4 cm-layers (or two sub-layers together) was either added or removed to test the performance of the jacket across different simulated freezing depths.
The unjacketed part of the soil column should always remain above freezing temperature
during the experiment.

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# 2.4. Soil and ancillary materials

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

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

#### **3. RESULTS AND DISCUSSION**

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





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

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

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

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









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





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

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

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

305 4. CONCLUSIONS

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

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

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# 500 Author contributions

- 501 *Conceptualization, writing, reviewing, editing, data analysis, and visualization:* M.
- 502 Sahoo, D. Baú, and S. Thornton; *Resources, methodology, software:* P. Bentley, A. Smith, P.
- 503 Blackbourn, and K. Howarth; *Supervision:* D. Baú and S. Thornton.
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517 Data will be made available upon request.