SOIL Discussions

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Reply on RC1

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Author comment on "Effect of freezing on the microstructure of a highly decomposed peat material close to water saturation when used prior to X-ray micro computed tomography" by Hassan Al Majou et al., SOIL Discuss., https://doi.org/10.5194/soil-2021-86-AC1, 2021

Reply to Reviewer #1

We thank Reviewer #1 for his comments which were very helpful in improving the manuscript submitted for publication.

Reviewer #1: This study analyzed the structure of a peat material before and after freezing by investigating looking at the air-filled pore using X-ray µ-CT. Many scholars have studied the effects of freeze-thaw cycles on soil structure, and this study only focused on before and after freezing. Although the thesis of the study can attract our insights to read the paper, lots of questions still have not resolved clearly. Some suggestions should be useful to improve this manuscript.

Reviewer #1:

- Author seems to use only two samples for the experiment. Is the sample number enough to support the conclusion of the study?

The results were indeed recorded for two samples of a highly decomposed sphagnum and Molinia peat material exhibiting some differences of composition (Table 1). We first performed similar work using several other highly decomposed peats materials but using a X-Ray µ-CT Bruker SkyScan 1278 with a smaller resolution than used in that paper. The results were similar to those included in our paper but some questions remained because of the limited resolution. Thus, on the basis of preliminary results, we used a X-Ray µ-CT device Nanotom 180NF (see sections methods) with a better resolution making possible to have a voxel size of 60x60x60 μm^3 with the characteristic of the samples studied (size and chemical composition) when it was 100x100x100 μm^3 with the first X-Ray µ-CT used. With a voxel size of 60x60x60 μm^3, it was possible to study ovoid pores of the organic matrix and very small tubular pores, both air-filled.

For us, there is no doubt about the generic character of the results recorded for highly decomposed peat materials. In the conclusion, we restrict the validity of our results to peat materials with similar characteristics to those of the samples we studied. If Reviewer #1 thinks that we are going too far with "Thus, the use of freezing prior to any study of the structure of peat material similar to the one studied here and close to water saturation should be avoided", we suggest to replace the last sentence of the conclusion by:

"Thus, the possible consequences of freezing prior to any study of the structure of peat materials should be investigated, particularly for highly decomposed peat materials."

Reviewer #1:

- Second, some methodological details are missing. Essential information about methods and materials is crucial for readers to evaluate the results. The description of the experimental process is not clear enough. What is the basis of freezing and thawing temperature selection?

We agree on the major importance of the information about the methods used.

The values of -10°C for freezing and 20°C for room temperature during the experiment were chosen to reproduce the conditions used by several authors who studied poorly decomposed peat materials and already used freezing to study peat structure (Quinton al al. 2009; Rezanezhad et al., 2009 and 2010; Ramirez et al., 2016) (see Line 182). Our objective was to discuss the validity the method they developed when applied to highly decomposed and water saturated peat materials.

Now, if we discuss the possible effect of freezing on the structure of a peat material, others conditions should be sought to increase freezing speed and thus to produce amorphous ice which dramatically reduce the volume change produced when water turns from liquid to solid state. Several studies were published during the 80s about the effect of freezing on the soil structure according to the freezing conditions. Among these studies, there is a paper published by one of the co-authors in the Journal of Soil Science (Bruand and Prost, 1987, Effect of water content on the fabric of a soil material: an experimental approach, 38, 461-472) who studied the consequences of on the soil structure of the method used to remove water from a sample prior its study in scanning electron microscopy and mercury porosimetry. However, for our samples of very large in size compared to the sample size required to produce amorphous ice using liquid nitrogen as often used, production of amorphous would require significant material investment to perform experiments with a very uncertain result.

To summarize, the conditions used are intentionally those which were used by the authors who earlier worked on the structure of peat by using freezing to prepare the samples.Our objective was to study the consequences of their protocol for highly decomposed peat materials.

Reviewer #1:

What container is the sample placed in when scanning□(line 121: a transparent plastic tube; line 137: were mounted and waxed on a glass rod)

The text is not clear as noted by reviewer #1. We suggest to replace the sentence "The samples were mounted and waxed on a glass rod" (Lines 136 and 137) by:

"Each transparent PVC tube with its sample of peat material inside was mounted and waxed on the glass rod of the chamber which rotates by 360 degrees during acquisition."

The word "PVC" will be used at the place of "plastic" in the whole text to be more specific.

Reviewer #1:

- Another major issue, the experimental procedure was not described in detail. This study only paid attention to air-filled pores, how to determine that the changes in soil structure before and after freezing are not the result of water change or migration.

This is indeed a major issue and we paid lot attention to that point during the experiment (Lines 180 and 181). It is probably not clear enough in the text. We suggest to add information to the two sentences Lines 184 and 186 as follows:

"Each sealed transparent PVC tube with its peat material inside was weighted at the different steps of the process, i.e. prior and after each analysis using X-ray X-Ray µ-CT, to check the absence of water loss during the acquisition of the successive 2D images. Measurements showed that the weight variation between two successive steps and between the first and last step was <0.1 g for the samples studied. Water condensation on the walls of the transparent tube was not observed during the experiment."

Reviewer #1:

- What is the purpose of sub-images of the 3D images. This information is present in Figs. 3 and 4, which only slightly enlarged in the sub-images.

Yes, the information present in Figs. 3 and 4 was slightly enlarged in the sub-images but also clarified by removing part of the initial information contained in Figs. 3 and 4. Information in Figs. 7 and 8 was indeed reduced by decreasing the lengths of the x, y and z axis, thus leading to fewer pores present in the 3D volume shown and consequently more readable information on the 3D porosity, especially for the very small discontinuous tubular pores and the ovoid pores of the organic matrix, both air-filled. This needs to be explained in text.

We suggest to modify the text "The selected … 8b and 8e)" (Lines 238 to 240) as follows:

"The selected sub-images show fewer pores, making it possible to analyze more precisely the morphology of the pores occupied by air, in particular the pores <500 voxels before and after freezing. Thus, Figures 7b and e and Figures 8b and e show that the pores <500 voxels corresponded to ovoid pores of several voxels to several dozen voxels, and to discontinuous fine tubular pores, both air-filled."

Reviewer #1:

- As for the results, we found that the upper and lower characteristic pores of the sample were relatively corresponding before and after freezing. Why did the middle part change so much.

We are not sure we fully understood the reviewer's comment. We are wondering if there is not some confusion in the reading of Figure 5. When we compare the pore size distribution recorded for sample A before freezing (Fig. 5a, 5a') and after freezing (Fig. 5b, 5b'), there is a dramatic change for all sizes of pores. Similar results were recorded for sample B as shown by Fig. 5c and 5c' (before freezing) and Fig. 5d and 5d' (after freezing).

Reviewer #1:

- The manuscript needs a major editorial revision to improve the writing quality. I can see some grammar mistakes, improper words, though I'm not a native speaker.

The English of the manuscript was edited by a native English speaker whom it is the professional activity. We did some very small changes throughout the manuscript after her editing work and may introduce some grammar mistakes and other type of mistake. The English of the version integrating modifications on the basis of reviewers' comments will be edited again by a native English speaker before final approving.

Reviewer #1:

For example lines 30-34 "Theoretical calculation of the consequences of the increase in the specific volume of water by 8.7 % when it turns from liquid to solid because of freezing led to the creation of a pore volume in the organic matrix which remains saturated by water when returning to room temperature and consequently to the desaturation of the largest pores of the organic matrix as well as the finest tubular pores which were water-filled before freezing."

At the place of "…and consequently to the desaturation of the largest pores of the organic matrix as well as the finest tubular pores which were water-filled before freezing.", we suggest:

"… and consequently to the desaturation of the finest tubular pores as well as largest ovoid pores of the organic matrix which were both water-filled before freezing." to make the sentence clearer.

Reviewer #1:

Lines 129-130 "X-ray µ-CT was performed for the sub-samples 4x4x7 cm in volume cut between 30 and 37 cm depth using a micro X-ray µ-CT device Nanotom 180NF (GE Phoenixx-ray, Wunstorf, Germany)." Suggest reorganizing the language.

We forgot to remove "cut between 30 and 37 cm depth" during the last correction step prior submission. The sentence was indeed confused.

We suggest:

"X-ray µ-CT was performed for the sub-samples 4x4x7 cm^3 in volume using a micro X-ray µCT device Nanotom 180NF (GE Phoenixx-ray, Wunstorf, Germany)."