SOIL Discussions

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Comment on soil-2021-86

Anonymous Referee #2

Referee 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-RC2, 2021

The work presented by the authors is very important. It states the effect that freezing has on the structure of the material, and shows how such technique is not suitable for the circumstances in which the microstructure needs to be investigated. Therefore, some statistical analysis on at least three subvolumes needs to be presented on subvolumes having the same size and same initial properties (e.g., density, depth of extraction, water content). Additionally, The authors should provide some perspectives on how the presented results can be confirmed, and above all, how the other scientist could avoid freezing the subsamples. The methodology presented lack in many details that the authors need to provide before accepting this paper for publication.

Major comments:

- Lines 25-27: I am not sure that the pores detected post-freezing were not detected pre-freezing. Perhaps, they have been created in the freezing process.
- Subsection 2.1: The readers would be interested in more details about the sampling. For instance, why these types of peats, why extracted in 25-40 cm depth, and finally, why stored at 3-4 degrees? Please spend few words on arguing more the field sampling methodology.
- Subsection 2.2 - Line 111: can you please specify the correct size of the undisturbed volume? A few centimeters can be a couple or a dozen.
- Why is the physico(physical)-chemical analysis relevant?
- Line 122: how is the sample hermetically sealed?
- Line 127: In the authors' opinion, this water loss of 0.1g is relevant or can be considered as negligible? And why?
- Line 122 and 124: I believe that 'submitted' is not the correct verb to use. I suggest finding a more appropriate term. Suggestion: "The sample was first imaged/observed/analysed through x-ray and then frozen.."
- Line 136: why the authors decided to convert the 16 bit image in 8bit? This choice implied a huge loss of data passing from a greyscale range of 65536 values to only

256. Such reduction could have definitely improved the measurements presented in the following sections.

- Line 140: How the authors preserved the freezing status of the samples during the 4 hours scanning time? What were the scanning conditions (e.g., temperature and humidity)?
- Line 141: Until this point, I thought that the sample was scanned before the freezing status and after freezing, ergo, frozen. But as I understand in this section the sample was scanned the second time after being defrosted, am I right? If this is the case, I strongly suggest the authors to rephrase the previous sections and clearly express that the 'after freezing' correspond to a status post-unfreezing.
- Lines 143-146: For the consistency of the measurements and the comparison of the two samples, the authors should have extracted the same volume for both. Why your two samples have two different volume size?
- Line 150: What does 'well inside the sample' mean? Please indicate the exact distance from the edges and the reason behind this choice.
- Line 155: How was measured the signal to noise ratio?
- Moving average filter: how is this filter defined? How does it work? Why was this specific filter chosen, respect to other ones (e.g. anisotropic or bilateral filters?). In figure 1, you could add the limits of the threshold used for the segmentation. Such info would help the reader following the procedure.
- Lines 158-159: Here I am having some trouble understanding. The 3D volumes of the sample should have been normalized, in order to have greyscale values coinciding for each phase within the scanned object, and to have an automatic threshold choice. A threshold is defined as a range. Is the range going from 0 to this value or from this value to 256, or else? This section is very important for all of your results are based on this technique. Please implement the information and clarify all the choices.
- Line 161: How are the pores identified? By 'scanning the image' is not an appropriate terminology. Is the identification done manually? Or otherwise?
- Line 178-181: What does this conclusion imply?
- Line 202-203: I would define the difference in grey scale values as darker grey (pores are not black, otherwise they would correspond to a unique value) for the pores and lighter grey for the organic material
- Line 204-207: Please define better in the Figure and in the text this differentiation. Such statement is confusing and not well described.
- Line 220-225: As pointed out before, how is defined the quantification of the pores? This is very unclear.
- Line 237: Why have the authors chosen two different volume sizes for the two samples. How can you make a comparison between the two cases, if you are using a different volume size?
- Line 239: air-filled ovoid pores, why did you use the term 'ovoid'?
- Line 265: How is this volume of water - equal to 8.7% - quantified? This seems to be an important step in your methodology and the description is missing.
- These results need to be implemented. I suggest the authors to run the same analysis on other sub-volumes and present a statistical comparison.
- Line 290-293: unclear statement.
- Eq7 and Eq 8: what does 1.087 represent exactly?
- Line 302: As asked in previous comments, why the authors state that small pores were detected only after freezing, but perhaps these pores was produced by the freezing process. Why is this hypothesis not taken into account?
- Line 307: pores calculated theoretically, how?
- Conclusions: The work presented by the authors is very important. It states the effect that freezing has on the structure of the material, and shows how such technique is not suitable for the circumstances in which the microstructure needs to be investigated. Therefore, some statistical analysis on at least three subvolumes needs to be presented on subvolumes having the same size and same initial properties (e.g., density, depth of extraction, water content). Additionally, The authors should provide some perspectives

on how the presented results can be confirmed, and above all, how the other scientist could avoid freezing the subsamples.

Minor comments:

- Figure 1: It would be easier for the readers to understand the difference in the grey value distribution if the before and after freezing curves are in the same plot, perhaps plotted with different color or line format.
- Figure 2: The arrows are not very clear. The authors should highlight in a different way the pores that appear before/after freezing. Maybe with a circle or shades of two different colors.
- Figure 3: The volumes are not aligned in the row direction. Scale is missing in the figure
- Figure 4: Same comment as Figure 3
- Figure 7: The authors should modify this figure, showing the reader where this subvolume is located in the global volume. Why is this size of subvolume chosen?
- Figure 8: Same comment as Figure 7
- Table 2: How is this porosity measured in the images? Based on the segmented image? After reading the manuscript, this info is still unclear.
- Title: "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". I am not sure about the second part of the title, 'when used prior to x-ray micro tomography'. I'd suggest the authors to clarify it.
- Abstract: The specification of the tomography (lines 20-23) are irrelevant in the abstract.
- The Introduction can use some improvement in the English format.
- Line 129: the authors already said that the sample depth corresponded to 30-37 cm in subsection 2.3, hence, no reason to repeat it here.