Freeze-thaw impacts on macropore structure of fiber-reinforced clay by industrial computed tomography scanning

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ABSTRACT

In cold climates, freeze-thaw cycling is an important issue in engineering. In freeze cycles, translocation of water and ice can be caused by thermodynamic conditions at temperatures just below 0 °C, impacting the mechanical properties of soils. Major efforts across geotechnical studies have attempted to develop reinforcement methods to decrease the adverse effect of these cycles. Recent studies have shown that Polypropylene fibers can provide an effective method to increase soil strength. However, there are few macrostructural investigations available to demonstrate the underlying physical basis for this effectiveness. In this study, Computed tomography (CT) images were taken from clayey reinforced samples with the addition of 2% Polypropylene fibers that were subjected to a maximum of 10 closed-system freeze-thaw cycles (FTCs). The results show that the significant effects of FTCs on soil structure are the reduction of macropores and augmentation of mesopores, while the addition of Polypropylene fibers changes the reduction amount of macropores from 28% to 18% following 10 FTCs.

RÉSUMÉ

Dans les climats froids, le cycle gel-dégel est un problème important en ingénierie. Dans les cycles de gel, la translocation de l'eau et de la glace peut être causée par des conditions thermodynamiques à des températures juste en dessous de 0 °C, ce qui a un impact sur les propriétés mécaniques des sols. Des efforts majeurs dans les études géotechniques ont tenté de développer des méthodes de renforcement afin de diminuer l'effet négatif de ces cycles. Des études récentes ont montré que les fibres de polypropylène peuvent fournir une méthode efficace pour augmenter la stabilité du sol, cependant, il existe peu d'études macrostructurales disponibles pour démontrer la base physique sous-jacente de cette efficacité. Dans cette étude, des images de tomodensitométrie (CT) ont été prises à partir d'échantillons renforcés d'argile avec l'ajout de 2 % de fibres de polypropylène qui ont été soumis à un maximum de 10 cycles de congélation-décongélation (FTC) en système fermé. Les résultats montrent que les principaux effets significatifs des FTC sur la structure du sol sont la réduction des macropores et l'augmentation des mésopores, tandis que l'ajout de fibres de polypropylène diminue la quantité de réduction des macropores de 28% à 18% après 10 FTC.

1 INTRODUCTION

Soils, especially at shallow depths, are exposed to freeze-thaw cycles (FTCs) every year in cold climates. It has been recognized that these cycles considerably change the structure of soils and thus have a significant influence on engineering properties of soils, such as physical features including hydraulic permeability, densification, unfrozen water content, and mechanical features involving strength, compressibility, and bearing capacity. During seasonal soil freezing, ice lenses, which form in free spaces between soil aggregates, force them apart and alter characteristic structures in micro and macro scales (Roustaei et al., 2015). In Canada, it has been found that the embankment constructed on soils that had never experienced FTCs, was damaged in just one year resulting in the loss of bearing capacity (Leroueil et al., 1991).

Equally, newly constructed highway embankments that are left unpaved for a few years may be subjected to damage by freeze-thaw cycles (Eigenbrod, 1996). This strong weathering phenomenon affects engineering activities significantly. Recent studies have shown that FTCs can change the structure of soils, considerably reducing the strength and resilient modulus of the soils and increase subgrade settlement, cracking, and deformation (Ghazavi & Roustaei, 2013; Ghazavi & Roustaie, 2010; Qi et al., 2006).

A primary reason for these physical and mechanical changes is the change in soil porosity during freezing. These microstructural changes have been investigated through SEM (scanning electron microscope), and it was found that a significant increase in the permeability of clayey soil was observed after freezing and thawing (Hohmann-Porebska, 2002), and also the soil becomes looser as the equivalent diameter decreases (Cui et al., 2014; Roustaei et al., 2015). SEM images of the soil samples before and after FTCs show coagulation of soil particles as a result of dehydration which occurs due to the migration of free water from the unfrozen part of the soil to the freezing front during freezing (Roustaei et al., 2015).

X-ray computer tomography (CT) technology has been recently added to the FTCs experiments (Nguyen et al., 2019; Wang et al., 2018). In the field, one of the main factors causing engineering quality degradation is the soil porosity variations related to frost heave in the cold season and later thawing of these ice lenses.

Considering the influence of freeze-thaw cycles on soils, researchers have focused on using additives that can control the effects of these cycles (Asl et al., 2019; Hazirbaba & Gullu, 2010; Roustaei et al., 2015; Zaimoglu, 2010). These studies have shown that some additives, such as the addition of polypropylene fibers, have a significant effect in reducing the impact of FTCs. Still, to our knowledge, there are very few macrostructural studies that demonstrate the underlying physical basis for this effectiveness.

The present study was conducted to investigate the effect of FTCs on macrostructural changes of unreinforced and reinforced clayey samples with 2% of polypropylene fibers after 3, 6, and 10 FTCs.

2 MATERIALS

2.1 Soil

This study used an EPK Kaolin manufactured by Edgar Minerals Inc. and classified as MH in the Unified Soil Classification System. Noticeably, the effects of freeze-thaw cycles are stronger in fine-grain soils in comparison with sand or gravel (Qi et al., 2006)

The soil properties are presented in Table 1. Standard Proctor Compaction tests were performed on the soil, and a maximum dry mass density of approximately 1.52 g/cm^3 at the optimum moisture content (OMC) of approximately 28 % was obtained. The specimens are reinforced using 2% of polypropylene fiber contents of the weight of dry soil.

Table 1. Characteristics of tested soil (Palat et al., 2019)

Liquid Limit (%)	58
Plastic Limit (%)	41.57
Plasticity Index (%)	16.43
Optimum Moisture Content (OMC) (%)	28
Maximum Dry Density (kN/m^3)	15.2

2.2 Fibers

The specimens were reinforced by adding 2% polypropylene fibers relative to the weight of dry soil. The fibers were supplied by MiniFIBERS. Inc. and their properties are presented in Table 2.

Table 2. Properties of the polypropylene fibers (Palat et al., 2019)

Length (mm)	4-5
Thickness (mm)	0.035
Specific gravity (g/cc)	0.91

3 EXPERIMENTAL PROCEDURE

3.1 Sample preparation

All cylindrical samples were prepared with 15 mm diameter and height with the maximum dry unit weight and optimum water content. To prepare unreinforced samples, firstly, the necessary OMC was determined and mixed with the soil and stored in plastic bags for 24 hours. Preparation of polypropylene reinforced samples was more challenging, the first half of the dry clay and half of the water were mixed prior to adding fibers. The fibers were not added to the clay–water mixture at this stage, since the fibers tend to adhere together if they are added at this point. The remaining half of the soil, water, and all fibers were then added to the clay–water mixture prepared in the previous step. The mixture of clay–water–fibers is then mixed gently. This procedure is found to be the effective at creating homogeneous samples (Ghazavi & Roustaie, 2010). Finally, compaction of soil mixtures was done using a mold and arbor press with a constant number of blows.

It should be noted that prior to sample compaction, the inside of the mold is coated with a lubricant in order to minimize the friction between the mold and sample. This step minimized fracture of the sample following removal from the mold. Following removal, the sample is immediately covered with a plastic layer to protect it from water evaporation.

3.2 Freeze-thaw cycles (FTCs)

To prepare the samples for the closed system freezing and thawing cycles, specimens were placed in a digital refrigerator at −20 °C for 12 h and then at +20 °C for the thawing phase for 12 h. These temperatures have been used previously (Ghazavi & Roustaei, 2013; Kalhor et al., 2019; Qi et al., 2006; Roustaei et al., 2015). Twelve hours is a proportional period after which the alteration of specimens' height would become constant. This means that the height increase in the freeze phase and the height decrease in the thawing phase are negligible. The cycles were continued up to 10 cycles since at this point most soil strength reduction will have occurred in primary cycles, and after 5–10 cycles a new equilibrium condition becomes predominant (Ghazavi & Roustaie, 2010).

3.3 Scanning and image processing

Micro Computerized tomography (µ CT) was used to examine the 3D internal mesostructure of the compacted clay/PP fibers mixtures. This method is a non-destructive technique that has been useful in the investigation of geological porous media (Ketcham & Carlson, 2001; Kozaki et al., 2001; van Geet et al., 2005). It consists firstly in recording a set of two-dimensional X-ray radiographs of an object at multiple angles (typically 180 or 360 degrees), and secondly reconstructing the 2D slices from the radiographs using a mathematical algorithm. The final 3D image of the internal structure is obtained by stacking the slices. The final measurement is the attenuation coefficient to X-rays which depends on the mass density and the atomic number of the object (Ketcham & Carlson, 2001; Saba et al., 2014; van Geet et al., 2005).

The scans presented here were carried out with a Nikon XTH 225 ST, and the soil samples were scanned at 185 Kv and rotated 360° with an exposure time of 250 ms, resulting in a voxel (3D volume element representing pixel resolution and slice thickness) of 10 µm. The images were reconstructed into three-dimensional gray-scale volumes using CT pro 3D software and finally analyzed using Dragonfly 2022 image processing software.

4 RESULTS

Figure 1a presents a typical reconstructed 3D CT image of a clay specimen. The image was cropped with a circular mask to eliminate the plastic wrap around the sample. Figure 1b and c provide representative images for void space and fibers, respectively. These figures show that the effective voxel size of scans (10 µm) was sufficient to clearly show the void spaces as well as fiber orientations.

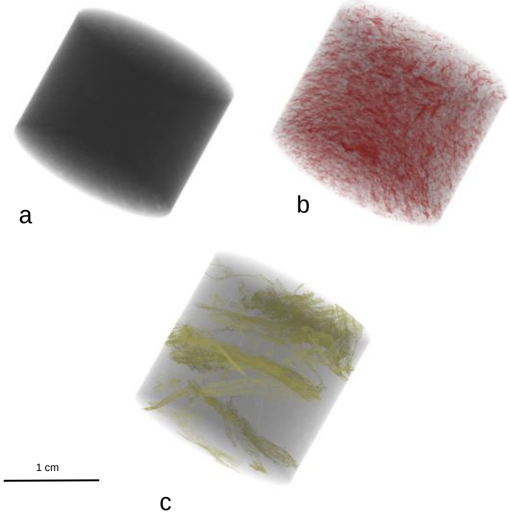

Figure 1. CT image of a) clayey sample b) void spaces c) fibers

To evaluate the effect of FTCs on macrostructural changes of unreinforced and reinforced samples, a histogram was created after each scan by plotting the linear attenuation coefficient (µ) versus the voxel populations. The linear attenuation coefficient (µ) characterizes how easily a volume of material can be penetrated by a beam of X-rays while the voxel population is the population of volume pixels, the smallest distinguishable element of a 3D image. By creating a histogram of values from each sample, distributions of density values are presented (Figure 2). Curves are named NC to show the N freeze-thaw cycles that were applied to the samples.

The shape of the histogram changes with the proportion of the component materials. In fact, a histogram is a representation of the volumetric content in a sample (Calmels et al., 2010). Figure 2 shows that FTCs significantly affect the first peak which corresponds to the pore spaces of the sample. This increase in the voxel population is an indication of larger pores and porosity changes after the cycle which is in agreement with the results of a previous study showing the augment of larger pores after the cycles through SEM imaging (Ghazavi & Roustaie, 2010). This survey also showed that using propylene fibers in the clayey soil affects the changes in physical and mechanical soil characteristics caused by freeze-thaw cycles since they act as a tensile element between the soil particles during the frost heave of the freezing period. This could be the reason for fewer changes in histograms of fiber-reinforced samples in comparison to unreinforced ones in this study (Figure 2).

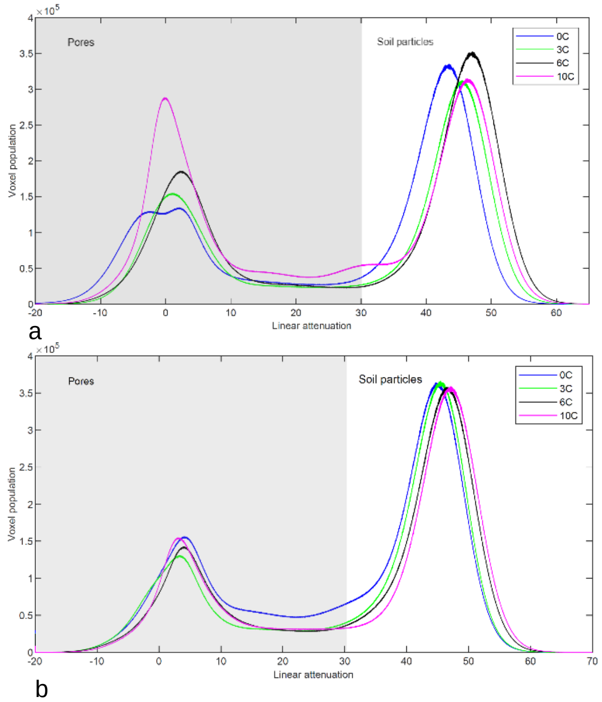

Figure 2. Histograms of scanned a) unreinforced clayey sample, and b) fiber-reinforced clayey samples after FTCs

In order to identify the mesostructural changes of soil samples after FTCs, and quantitatively characterize the effect of adding PP fibers, the following image processing steps were adopted in this study. First, a cylindrical region of interest (ROI) with 2 mm diameter and 8 mm height was selected from the same location of each scan (figure 3). This step saves processing time and improves visualization of void spaces. Then, a multi-ROI analysis was applied to the selected ROI to separate the void spaces based on their volumes (Figure 3).

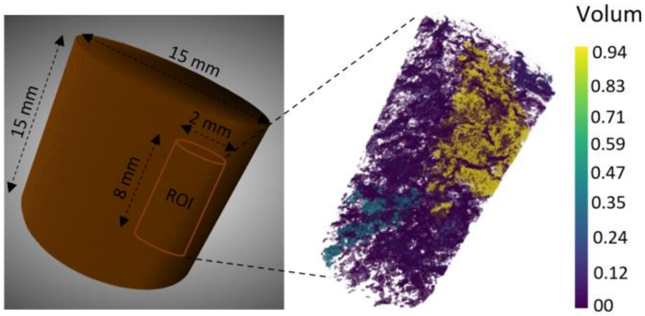

Figure 3. Visualization of ROI and the multi-ROI analysis results

A multi-ROI analysis automatically groups labeled voxels into components based on connectivity and then calculates properties such as volume, surface area, and mean/max/min Feret diameter. The Feret diameter is the distance between the two parallel planes restricting the object perpendicular to that direction.

To implement a porosity analysis on the selected ROI from the samples, the pores were separated into three main categories; fissures, macropores, and mesopores based on their mean Feret diameters.

Greenland 1977 defined soil fissures as macropores with a diameter larger than 0.5 mm, which was adopted in this study. Macropores refer to voids comprising at least two connected voxels since single-voxel voids could be artifacts from imaging and its processing (Fan et al., 2021; Hatano et al., 1992; VandenBygaart & Protz, 1999).

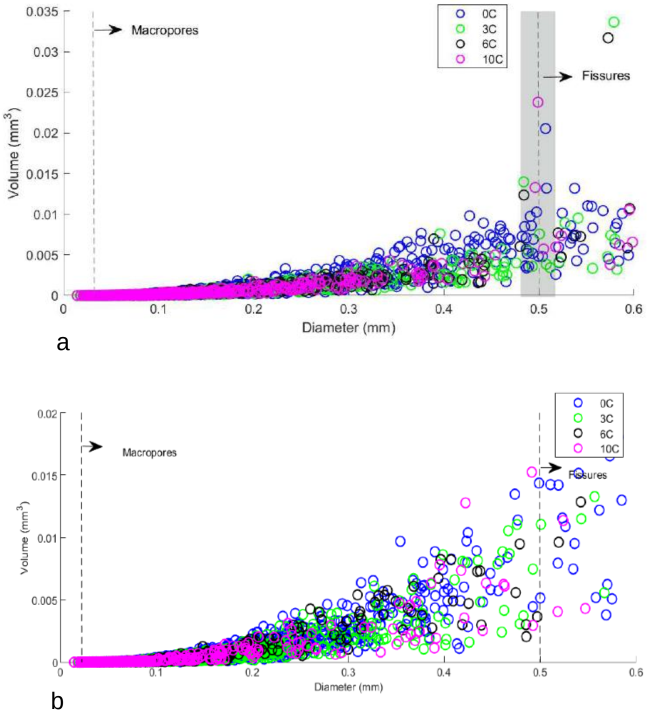

Figure 4. Mean Feret diameters of pores vs their volumes in a) unreinforced, and b) fiber-reinforced clayey samples after FTCs

The macropores were assumed to have a diameter between 0.5-0.02 mm while the mesopores were smaller than 0.02 mm. Figure 4 shows the Feret diameters of pores vs their volumes in unreinforced and fiber-reinforced samples after FTCs. It is obvious from this figure that, based on the pixel size of the scans, the main changes are happening in macropores and small fissures, and augmentation of bigger pores after the cycles is clear (gray area). This change is less in fiber-reinforced samples by comparison with unreinforced ones.

Figure 5 visualizes the variations of fissures, macropores, and mesopores within the ROI to highlight the effect of FTCs on unreinforced and fiber-reinforced samples. This figure shows that as the number of freeze-thaw cycles increases, the pore structure of the soil changes, and the main effect of FTCs on soil structure is the reduction of macropores and augmentation of mesopores (showed in blue dots). This result is in agreement with a previous study using particle-size distribution and shows that as the FTCs progress, soil permeability becomes stronger as the pore size distribution curve shifts to the smaller pores (Xu & Wang, 2022).

For a more precise comparison of these changes and to find a better understanding of the effect of fibers in reducing the effect of FTCs, the area of fissures, macropores, and mesopores in different slices of the ROI were plotted in figure 6. This figure indicates that FTCs significantly reduce the area of fissures and macropores mainly in the first 3 cycles and the changes become negligible after. Previous studies also showed that the effect of FTCs is more notable in the first 5-6 cycles after which a new equilibrium condition becomes predominant (Roustaei et al., 2015).

In addition, similar to Figure 5, the area of mesopores within the top half of the unreinforced ROI stayed constant or slightly increased after the cycles and this increase is more significant in the fiber-reinforced sample. So it means that the fibers are more effective in reducing the effect of FTCs on the fissures and macropores in comparison to the mesopores.

The pore size distribution of samples can also be evaluated after the cycles. Figure 7 shows the distribution in pie charts before and after FTCs for unreinforced and fiber-reinforced samples. These charts show that in both samples FTCs result in reducing the macropores and increasing the mesopores, hence the portion of fissures increases significantly after the cycles which can affect the strength, permeability, and settlement of soil mixtures. These results show a 28% reduction in the portion of macropores (32% to 23%) after the cycles for the unreinforced sample, while this decrease is 18% for the fiber-reinforced one. So, the reduction of macropores in the fiber-reinforced sample is less than in the unreinforced sample. This change could be due to the tensile behavior between the soil particles during freezing and less frost heave and porosity changes in macropores.

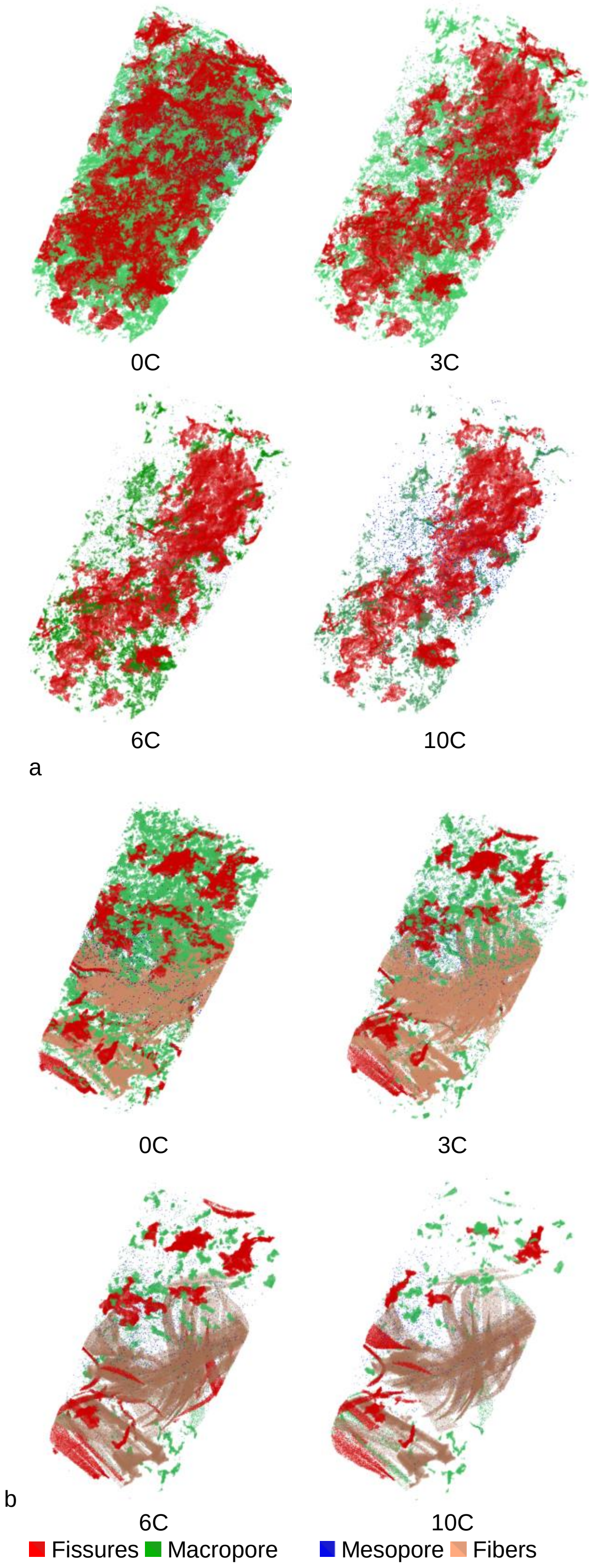

Figure 5. Visualization of fissures, macropores and mesopores in a) unreinforced and b) fiber-reinforced clayey samples

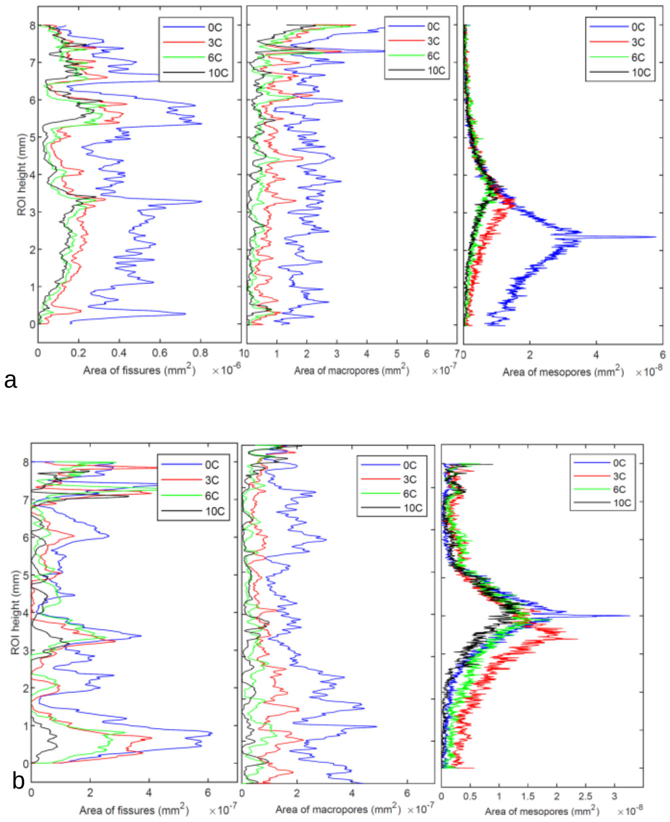

Figure 6. Area of fissures, macropores, and mesopores in a) unmodified and b) fiber-reinforced clayey samples within the adopted ROI

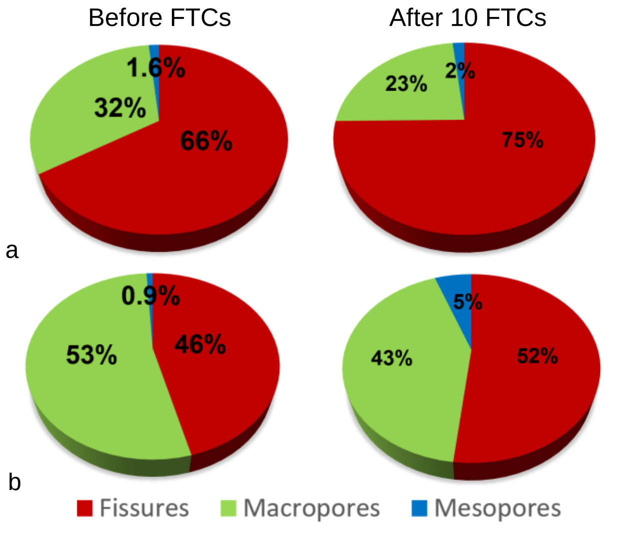

Figure 7. Porosity distribution of a) unreinforced and b) fiber-reinforced clayey samples before and after FTCs.

5 CONCLUSION

FTCs are one of the most common phenomena in cold regions, resulting in structural damage and deterioration of soils. This study investigated the mesostructural changes of unreinforced and fiber-reinforced clayey soil through micro-computed tomography as a non-destructive method. Image processing and porosity analysis were effective at showing the variation of soil samples following FTCs. Based on the results the following conclusions can be drawn:

- Histogram changes of scanned samples and porosity analysis after FTCs show augmentation of larger pores and small fissures as well as porosity changes.
- The most significant effect of FTCs on soil structure is a reduction of macropores and augmentation of mesopores.
- The reduction of macropores in the fiber-reinforced sample is less than in the unmodified sample, i.e., 28% reduction in the portion of macropores after the cycles for the unreinforced sample decreases to 18% for the fiber-reinforced one.
- Polypropylene fibers and their tensile effect between the soil particles during frost heave effectively reduce the effects of FTCs on macropores variation.

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