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Effects of Microencapsulated Phase Change Material on the Behavior of Silty Soil Subjected to Freeze–Thaw Cycles

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Abstract: Freeze–thaw (F–T) cycles are one of the most important factors affecting the performance of silty soils with high kaolin content in seasonally freezing regions. This study investigates the improvement of a high-plasticity clayey silt soil (MH) with microencapsulated phase change material (mPCM) to prevent changes in mechanical properties when subjected to freeze–thaw cycles. Unconfined compression, one-dimensional compression, and freeze and thaw tests were performed to evaluate the behavior of treated soil under different freeze/thaw cycles and with different mPCM ratios. It has been observed that the mPCM additive decreased the unconfined compression strength (UCS); however, the strength of the soil held constant during the increasing F–T cycles, and the increase in the mPCM additive content increased the strength of the soil. The inclusion of mPCM affected the compression of the soil and increased settlement (ΔH), although the settlement remained constant with increasing freeze–thaw cycles. It has been noted that the compression behavior, which is least affected by the unconfined compressive strength and freeze/thaw cycles, is achieved with the addition of 10% mPCM. As a result of the tests, it was determined that the most suitable additive mPCM ratio is 10% for the compression and strength behaviors.

Keywords: microencapsulated phase change material; unconfined compressive strength; one-dimensional compression; freeze–thaw cycle; volume change behavior; high-plasticity silt



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1. Introduction

Soils subjected to F–T cycles in Turkey are located in the Central and Eastern Anatolia Regions, where the coldest provinces are listed as Ardahan, Kars, Ağrı, Erzurum as well as Sivas. These cities are predominantly in mountainous regions, and the area has an extensive highway network. According to the statistical data available between the years of 1940 and 2023, the coldest province is Ağrı, located in the Eastern Anatolia Region [1]. The average altitude of Ağrı is 1630 m, the lowest air temperature is measured at -45°C , the highest air temperature amounts to 40°C , and the greatest snow depth is 2.25 m. Soils with a thickness of about 2 m from the ground surface are subjected to F–T more than once a year [1]. Additionally, foundations and embankments are affected by F–T cycles where the groundwater level is high.

In regions where seasonal freezing occurs, the surface temperature of the soil gradually decreases due to the decline in air temperature. The water in the soil voids freezes, leading to ice lenses forming in the frost layer when the surface temperature drops below 0°C . As the temperature falls, the frost layer can rapidly move downwards, causing the capillary and pore water to gradually move towards the frost layer [2].

In periods when the air temperature rises seasonally, the frozen soil begins to melt from the uppermost surface. F–T cycles cause significant changes in the physical and mechanical properties of the soil, deteriorating its structure. Because of an approximately 9% increase in the volume of frozen water, the voids between the soil particles enlarge, and the particles move away from each other, hence, soil becomes structurally weaker than before F–T. Ice lenses formed during freezing induce capillary cracks in the soil after thawing and boost

water seepage through the soil compared to before F-T [3–5]. Repeating this process in each F-T cycle expands the soil volume and has an adverse effect on its mechanical properties. Thus, microstructural changes lead to variations in mechanical properties on a macroscopic scale, depending on many factors, such as climatic conditions, soil type (grain distribution, mineralogical structure) in addition to properties, water content, surcharge load, and temperature [6,7]. F-T processes under varying weather conditions may cause stability problems by affecting the thermal, mechanical, and physical properties of foundation soils or embankments in cold regions [8,9].

High-plasticity silty soils in cold regions should only be used in engineering applications with improvement [10–12]. For this purpose, typical chemical stabilizers such as fly ash, lime, and cement have been used in long-term improvement studies [13–17]. However, it has been stated that F-T cycles after improvement with aforementioned materials decrease the strength of the soil [18–25]. These findings highlight the need to identify a more effective method to improve the F-T performance of the soil. Phase change materials (PCMs) are used as a new material, preventing the freezing of asphalt pavements [26] and melting ice and snow on concrete pavements [27], and allowing the temperature control of mass concrete [28] and the thermal insulation of buildings [29].

Farnam et al. [27] proposed the use of PCMs to melt ice in concrete pavements, while Chen et al. [30] and Si et al. [31] used them to reduce high and low temperatures in asphalt pavements. Pilehvar et al. [32] investigated the mechanical properties of two different mPCMs containing geopolymers concrete and Portland cement concrete subjected to F-T cycles. They concluded that the reduction in compressive strength in the F-T cycles decreased below 2.5% with the inclusion of mPCM [32]. Experimental studies on PCM including asphalt, concrete, and cement provided a theoretical basis for the improvement of the F-T performance of soils in cold regions. PCM has been used in studies [27,33–36] to enhance the F-T performance of the soil, with promising results for the behavior of engineering structures. PCM molecules are concurrently retained in the porous clay structure due to their physical interactions, such as capillarity, surface tension forces and weak intermolecular attractions [37]. Thus, PCM improves the mechanical and physical properties of soil.

Few studies have investigated the influence of the incorporation of liquid or microencapsulated PCM in the physical and mechanical properties belonging to plastic soils that are highly frost sensitive. Mahedi et al. [25], Chen [38] and Rao [3] carried out strength tests by adding liquid (PCM) and microencapsulated PCM (mPCM) to the soil, and suggested the usage of mPCM for subgrade soil under the F-T effect.

Plastic silts are susceptible to the effects of frost action. When these soils are used as embankment fill material or foundation soil, they will cause stability problems such as settlement, differential settlement, and cracking due to the F-T effect. The main objective of this study is to evaluate the changes in the physical and mechanical properties of a high-plastic clayey silt soil (MH) with high kaolin content, depending on the mPCM ratio and the number of F-T cycles, when an mPCM additive is employed for enhancement purposes. The following sections present the materials, experimental studies employed, results, and discussions.

2. Materials and Methods

2.1. Materials

2.1.1. Soil

This study used white MH with a high kaolin mineral content, known to be sensitive to frost and have high plasticity, supplied from the Kocaeli region. Sieve and hydrometer analysis (ASTM D6913 [39] and ASTM D7928 [40]), Atterberg limits (ASTM D4318 [41]), and standard compaction (ASTM D1557 [42]) tests were performed throughout the experimental program. The test results are summarized in Table 1. The soil class is determined as high-plasticity clayey silt (MH) according to the Unified Soil Classification System (USCS). The particle size distribution curves of the soil and mPCM are presented in Figure 1a,b.

The relationship between optimum water contents (OWC) and dry densities (ρ_k) was determined by standard compaction energy (Figure 2). In XRD and XRF analyses conducted in the MH, kaolin (89%), quartz (4%), feldspar (4%), and illite–montmorillonite (3%) minerals were observed in the soil (Figure 3).

Table 1. Soil properties observed in the study.

Property	Value
Silt (%)	55.00
Clay (%)	45.00
Specific gravity (Gs)	2.65
Liquid limit (LL) (%)	54.00
Plastic limit (PL) (%)	37.19
$\rho_{kmax-standard}$ (g/cm ³)	1.36
OWC _{standard} (%)	34.80

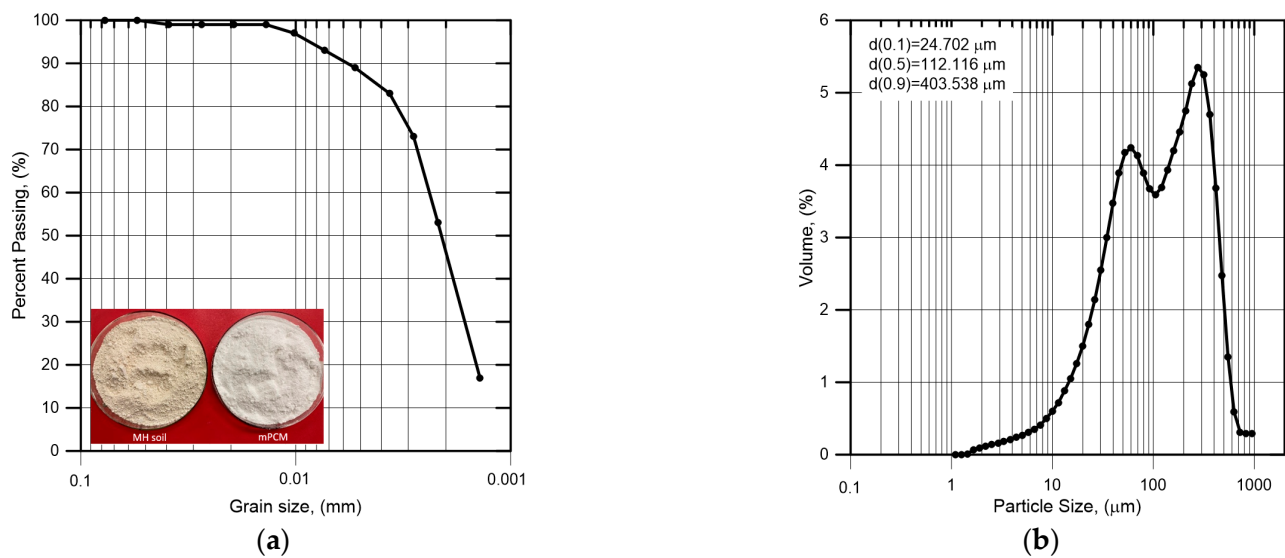


Figure 1. Particle size distribution curve: (a) MH; (b) mPCM.

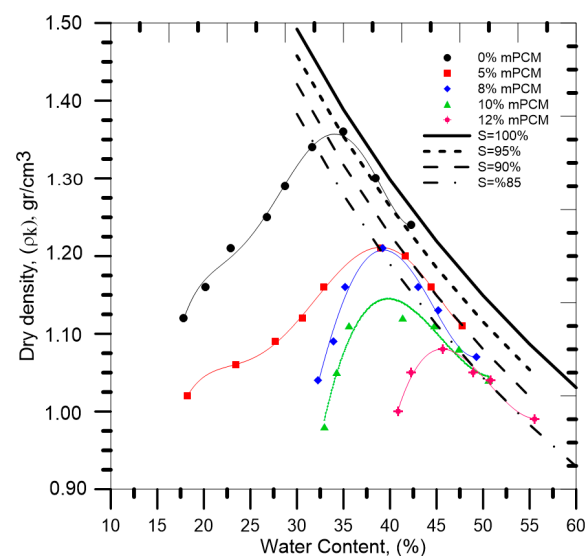


Figure 2. Standard compaction curves.

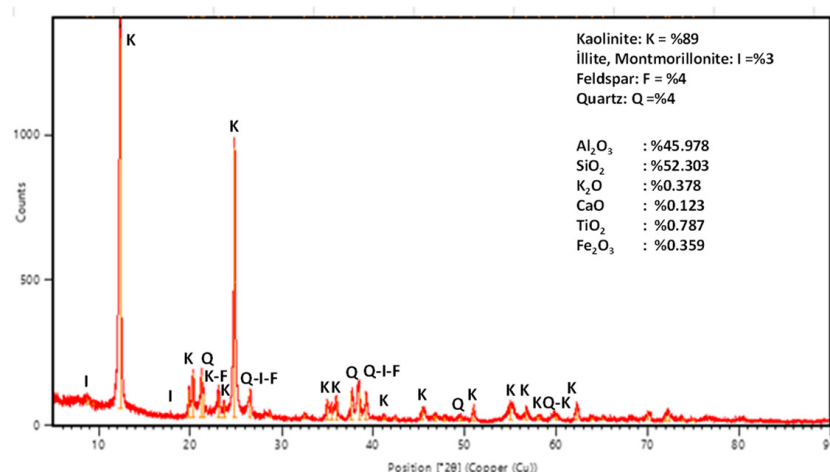


Figure 3. XRF and XRD analysis result.

The particle size distributions of MH and mPCM are shown in Figure 1a,b. The particle size of the soil is between 0.075 and 0.002 mm. The analysis performed on mPCM established that 10% of the material was below 0.02 mm, and 90% was below 0.4 mm. mPCM is a slightly off-white powder. The small size of the mPCM and almost particle-sized soil, along with the large specific surface area and the strong surface adsorption capacity, played an essential role in the homogeneous mixing of the microcapsules with the soil. The images of the MH and mPCM are presented in Figure 1a.

2.1.2. Phase Change Material (PCM)

In this study, paraffin-based organic PCM, which can transition into the liquid phase without phase separation and to the solid phase without re-phase separation, is environmentally friendly, non-corrosive, chemically stable, as well as cost-effective; liquid at room temperature was preferred.

Mahedi et al. [25] mixed liquid PCM with loess soil to improve the F-T properties of the soil and determined that the high content of PCM caused significant seepage, leading to failure. It showed the importance of choosing an appropriate additive ratio for the PCM–soil interaction. Studies have confirmed that liquid paraffin should not be used without microencapsulation [3,25,38]. Microencapsulation is the process of coating PCM with a shell material [43]. Microcapsules allow volume changes of PCM, hindering its reaction and interaction with the external environment. In the encapsulation process, there is no risk of seepage due to the small volume of coating of PCMs [44]. More specifically, mPCM has a core–shell structure. Therefore, during the phase transition from solid to liquid state, the capsule’s core materials change from solid to liquid, yet the outer layer remains solid. Moreover, mPCM has been reported to be advantageous in terms of thermodynamic properties, volume stability and mechanical properties [3].

This study used a non-irritating and odorless white-colored mPCM with a paraffin core material exhibiting phase change between the applied freeze ($-20\text{ }^{\circ}\text{C}$) and thaw ($+20\text{ }^{\circ}\text{C}$) boundary conditions. Due to the failure of liquid PCM in achieving soil improvement, the liquid PCM was synthesized and microencapsulated in the Chemistry Laboratory. To investigate the thermal properties of the mPCM used and liquid PCM, differential scanning calorimetry (DSC) analysis was performed on the mPCM (Figure 4a–c and Table 2). NETZSCH DSC 3500 was used to determine the thermal properties of the liquid PCM, mPCM and 10% mPCM-added soil (P10).

The sample was prepared by weighing 4–10 mg on a high-precision (10^{-4}) balance for DSC analysis. Analyses were made using nitrogen gas; the temperature range applied is between $-20\text{ }^{\circ}\text{C}$ and $+20\text{ }^{\circ}\text{C}$. The test was started by keeping the sample at $-20\text{ }^{\circ}\text{C}$ for 2 min and then heating it to $+20\text{ }^{\circ}\text{C}$. Afterwards, the sample was held at $+20\text{ }^{\circ}\text{C}$ for 2 min

and cooled back down to -20°C . The heating and cooling processes were carried out at a $2^{\circ}\text{C}/\text{min}$ rate.

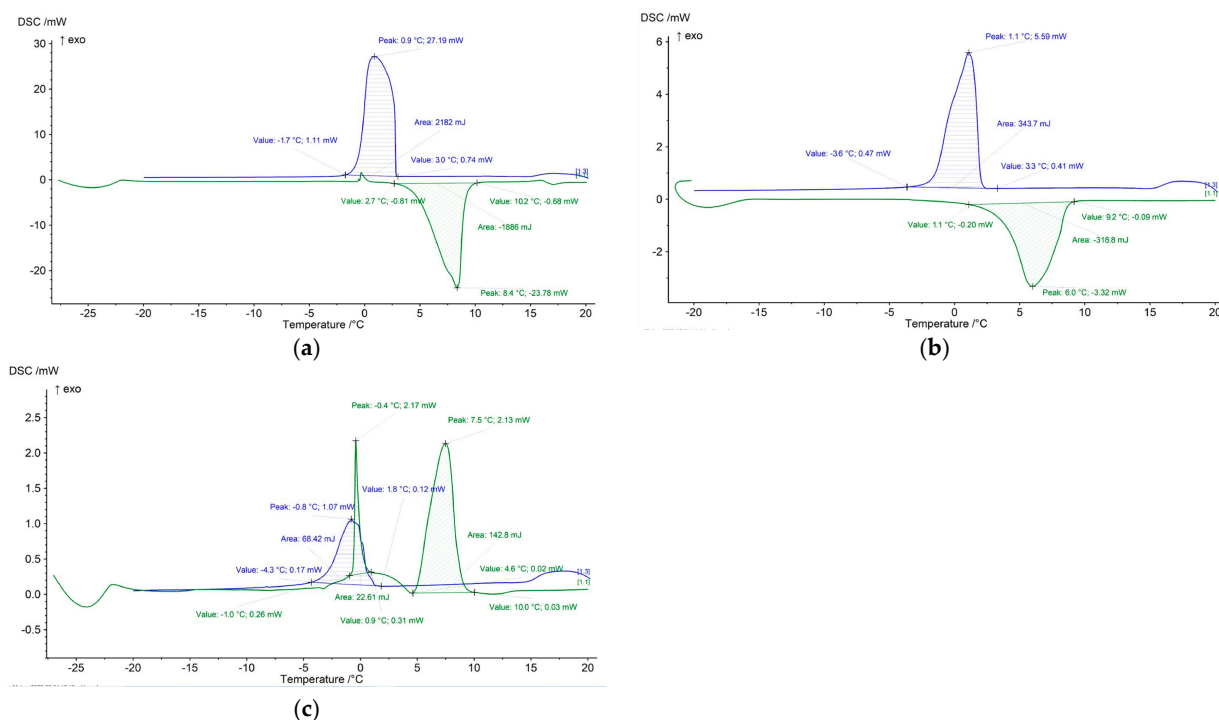


Figure 4. DSC analysis of liquid PCM, mPCM and 10% mPCM added to the soil (P10). (a) Liquid PCM; (b) mPCM; (c) 10% mPCM added soil (P10).

Table 2. Thermal properties of liquid PCM, mPCM, and 10% mPCM added to the soil (P10).

		Liquid PCM	mPCM	10% mPCM Added to the Soil (P10)
Melting Process	Temperature range ($^{\circ}\text{C}$)	2.7 to 10.2	1.1 to 9.2	-1 to 0.9 and 4.6 to 10.0
	Peak temperature ($^{\circ}\text{C}$)	8.4	6.0	-0.4 and 7.5
	Heat of fusion (mJ)	1886	318.8	22.61 and 142.8
	Enthalpy (J/g)	200.6	81.75	5.138 and 32.46
Solidification process	Temperature range ($^{\circ}\text{C}$)	-1.7 to 3	-3.6 to 3.3	-4.3 to 1.8
	Peak temperature ($^{\circ}\text{C}$)	0.9	1.1	-0.8
	Heat of fusion (mJ)	2182	343.7	68.42
	Enthalpy (J/g)	232.1	88.12	15.55

As shown in Figure 4a and Table 2, the temperature ranges where liquid PCM is active are between 2.7 and 10.2°C for an endothermic reaction, and between -1.7 and 3°C for an exothermic reaction. However, at 2.7 and 10.2°C , the liquid PCM experienced a phase transition from solid to liquid, with a peak phase transition temperature (melting point) of 8.4°C . During the melting process, its enthalpy was 200.6 J/g . Between -1.7°C and 3°C , the liquid PCM transitioned from the liquid to the solid phase, with a peak phase change temperature of 0.9°C . During the crystallization (solidification) process, its enthalpy was 232.1 J/g . During the transition from solid to liquid, the liquid PCM completes melting at a temperature of 7.5°C , while its transition from liquid to solid phase occurs at 4.7°C .

As shown in Figure 4b and Table 2, the temperature ranges where mPCM is active are between 1.1 and 9.2°C for an endothermic reaction and -3.6 and 3.3°C for an exothermic reaction. However, at 1.1 and 9.2°C , mPCM experienced a phase change from solid to liquid with a peak phase change temperature (melting point) of 6°C . During the melting process, its enthalpy was 81.75 J/g . Between -3.6°C and 3.3°C , the mPCM transitioned

from the liquid to the solid phase, with a peak phase change temperature of 1.1 °C. During the crystallization (solidification) process, its enthalpy was 88.12 J/g. During the transition from solid to liquid, the mPCM completes melting at an amplitude of 8.1 °C, while its transition from liquid to solid phase occurs at 6.9 °C.

FT-IR spectroscopy is one of the most common methods of analyses used to determine the chemical structures of organic compounds depending on the vibrations of atoms [45]. In this study, atoms with different functional groups and chemical bonds were determined by FT-IR spectroscopy. The FT-IR analysis results for the soil, mPCM and 10% mPCM added to the soil (P10) are presented in Figure 5.

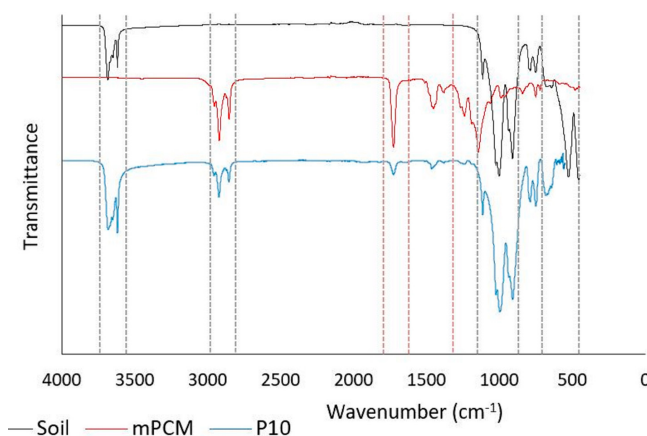


Figure 5. FT-IR spectra of soil, mPCM, and 10% mPCM added to soil (P10).

The MH is kaolin, and the kaolin structures are silicate-based materials. The chemical formula of kaolin is $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. As seen in the XRD analysis results in Figure 3, the soil comprises Al_2O_3 and SiO_2 . Since it is a natural soil, it also contains oxides of K, Ca, Ti, and Fe. These elements form bonds in Si–O–Al, Si–O–Ti, and Si–O–Fe structures. FT-IR spectra and XRD analysis results support this claim. The peaks of the MH at 3600 cm^{-1} (3651, 3619, 3685) in the FT-IR spectrum given in Figure 5 are due to the OH stresses in the kaolin structure. The peaks at 1000 cm^{-1} (1147, 1024, 1001) are Si–O, the peaks at 550 cm^{-1} are Al–O, the peaks at 495 cm^{-1} are Ti–O and the peaks at 580 cm^{-1} are Fe–O. The 500 cm^{-1} peaks are the interaction frequencies of Si, Al, K, Ca, Ti, and Fe in kaolin with oxygen.

There are various forms of PCM material available. In this study, a PCM additive in an organic-based tetradecane structure with a general formula of $(\text{C}_n\text{H}_{2n+2})$ was used. CH stretching frequencies and CH bending frequencies are observed in the FT-IR spectrum, as expected. In Figure 5, the peak CH at 2938 cm^{-1} , the peak CH^3 at 2922 cm^{-1} , and the peak CH^2 at 2853 cm^{-1} in the FT-IR spectrum of mPCM are due to the C–H vibrations of the groups, as well as the 1454 cm^{-1} CH bending, 1381 cm^{-1} methyl groups bending, 1450 cm^{-1} C–H bending, and 700 cm^{-1} bending frequency of the same peak. These peaks in frequencies can easily be seen in the spectrum curves. There is also a sharp peak at 1726 cm^{-1} in the spectrum caused by carbonyl derived from PCM synthesis.

2.2. Sample Preparation

To examine the effects of mPCM on the strength and compression behavior of MH exposed to F-T cycles when the ambient temperature changes, unconfined compression in addition to one-dimensional compression tests were carried out. The soil sample, which passed through a No. 200 sieve, was kept in an oven at 105 °C for 24 h. After being removed from the oven, it was cured for 24 h by adding distilled water at the optimum water content (sealed bag, aluminium foil, stretch film and desiccator) once it reached room temperature.

Dry soil and mPCM were mixed together homogeneously at contents of 5%, 8%, 10% and 12% of dry soil weight. Distilled water at the OWC was added and the mixture was

mixed for 15 min until the surfaces of all the particles were uniform. After this pre-treatment stage, it was compacted at standard compaction energy level.

Cylindrical samples with a diameter of 37.5 mm and a length of 75 mm were used for the unconfined compression tests, and samples with a diameter of 50 mm and a thickness of 20 mm were used for the one-dimensional compression tests. The compaction mold layer information (bottom, middle, top) of the samples used for one-dimensional compression tests was noted. The test samples were wrapped in stretch film so they did not lose their water content and were placed in the F-T cabinet to be subjected to the relevant F-T cycles. The samples were prepared by the same method without adding mPCM (pure soil, 0% mPCM, P0) and with addition (5, 8, 10, and 12% mPCM, P5, P8, P10, P12). Figure 2 and Table 3 shows standard compaction test results for P0, P5, P8, P10 and P12.

Table 3. Compaction test results of mPCM addition.

mPCM Additive (%)	P0	P5	P8	P10	P12
ρ_{kmax} (g/cm ³)	1.36	1.21	1.19	1.15	1.08
OWC (%)	34.80	39.00	39.00	40.00	46.00

Considering the test results presented in Figure 2 and Table 3, the increase in the content of mPCM, which has a lighter specific gravity than the soil, decreased the maximum dry density of the soil and increased the OWC. Lecompte et al. [46] attributed this to the greater water absorption capacity of mPCM. The initial rise in the OWC for mPCM was due to the increase in the void ratio. However, after the initial rise, the OWC remained constant with increasing mPCM ratio, then showed a significant increase. The OWC of soil and mPCM mixtures may have increased due to the explosion of microcapsules during compaction. The relationship between the OWC and maximum dry unit volume obtained by raising the mPCM ratio also supports the results of Mahedi et al. [25] Mahedi [25], Chen [38], and Rao [3] used mPCM by adding 4, 5, 8, and 10% to the soil in the studies. In this study, unlike others, a 12% mPCM content was also mixed with soil to determine the optimum mPCM ratio.

To determine the change in liquid limit and plastic limit, Atterberg limit tests were performed on P5, P8, P10 and P12 and are displayed in Figure 6. According to the relationship between the mPCM content and the liquid and plastic limits shown in Figure 6, the rise in the mPCM ratio increased the liquid limit and plastic limit. The reason for this is that the high-water-absorption-capacity mPCM's microcapsules may have exploded. Adding different ratios (5, 8, 10, 12) of mPCM did not change the soil class, and the soil class remained MH.

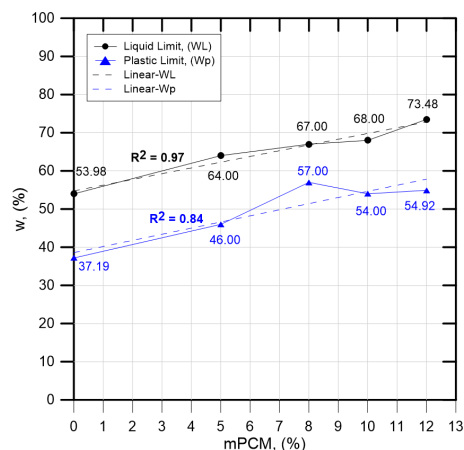


Figure 6. Atterberg limit tests of P0, P5, P8, P10, and P12.

3. Freeze and Thaw Tests

Unconfined compression strength (UCS) and one-dimensional compression tests were carried out on the test samples (P0, P5, P8, P10, P12) prepared according to the above procedure without (reference) and with applying the F-T cycle. In the article, proportional comparisons are presented according to the results of the reference test.

In the literature, one F-T cycle was completed in the experimental studies by keeping the samples at $-20\text{ }^{\circ}\text{C}$ for 24 h and at $+20\text{ }^{\circ}\text{C}$ for 24 h [8,19,25,47]. This study used a cabinet ranging from $-20\text{ }^{\circ}\text{C}$ to $+20\text{ }^{\circ}\text{C}$ for freezing and thawing cycles. Samples kept at $-20\text{ }^{\circ}\text{C}$ for 24 h completed the freeze process, while samples at $+20\text{ }^{\circ}\text{C}$ for 24 h completed the thaw process, and one F-T cycle was completed. For 11 F-T cycles, test samples were kept in an F-T cabinet for 22 days. The UCS and one-dimensional compression tests performed on the samples are named in Table 4. The specimens are abbreviated by numerals representing the content of mPCM and the number of applied F-T cycles. The first term of the specimens represents the content of mPCM added to the soil, and the second term represents the number of F-T cycles. Accordingly, “P8-5” means that the 8% mPCM added test sample was exposed to five F-T cycles. UCS and one-dimensional compression tests were performed on the test specimens after the relevant F-T cycles were completed.

Table 4. Tests performed on samples.

Tests	Additive mPCM Content (%)	Number of Freeze–Thaw (F-T) Cycles						
		0	1	3	5	7	9	11
Undrained Compression Strength and One Dimensional Compression Tests	0	P0-0	P0-1	P0-3	P0-5	P0-7	P0-9	P0-11
	5	P5-0	P5-1	P5-3	P5-5	P5-7	P5-9	P5-11
	8	P8-0	P8-1	P8-3	P8-5	P8-7	P8-9	P8-11
	10	P10-0	P10-1	P10-3	P10-5	P10-7	P10-9	P10-11
	12	P12-0	P12-1	P12-3	P12-5	P12-7	P12-9	P12-11

UCS tests according to the ASTM D2166 [48] standard, applying the relevant F-T cycles (0, 1, 3, 5, 7, 9, 11) to the test specimens (P0, P5, P8, P10 and P12), were carried out. In the test, the sample’s unconfined compressive strength (q_u) was determined by applying a constant shear rate of 1.27 mm/min.

One-dimensional compression tests were carried out according to ASTM D2435 [49] by applying the relevant F-T cycles (0, 1, 3, 5, 7, 9, 11) on test specimens (P0, P5, P8, P10 and P12). In the one-dimensional compression tests, the loading and unloading steps are considered as 50, 100, 200, 400, 800, 1600 kPa. In the tests, water was added to the sample at the first minute of the 50 kPa loading step, and step-by-step loading was carried out until the 1600 kPa stress was reached. In the tests, 24 h passed between each load step. The test was completed by reading the value at the end of 24 h during loading.

4. Results and Discussion

4.1. Undrained Compression Strength (USC) Tests

It was observed that the stress–strain behaviors of the P0, P5, P8, P10 and P12 samples shown in Figure 7 are quite similar. The rough surfaces at both ends of the sample at the beginning of the curves did not make good contact with the loading head, so they do not reflect the fundamental mechanical properties. Afterwards, it was seen that there was an approximately linear increase in stress with an increase in axial strains in the range of 8–18%. After the elastic region, the rate of stress rose, before this rise gradually slowed, reaching the peak stress and decreasing progressively as the strain increased.

In all the stress–strain curves, strain softening behavior was observed in reaching peak strength after elastic behavior. As the number of F-T cycles increased, the rate of stress increase in the elastic part decreased. The samples were damaged due to the F-T processes, so the plastic deformation rate increased.

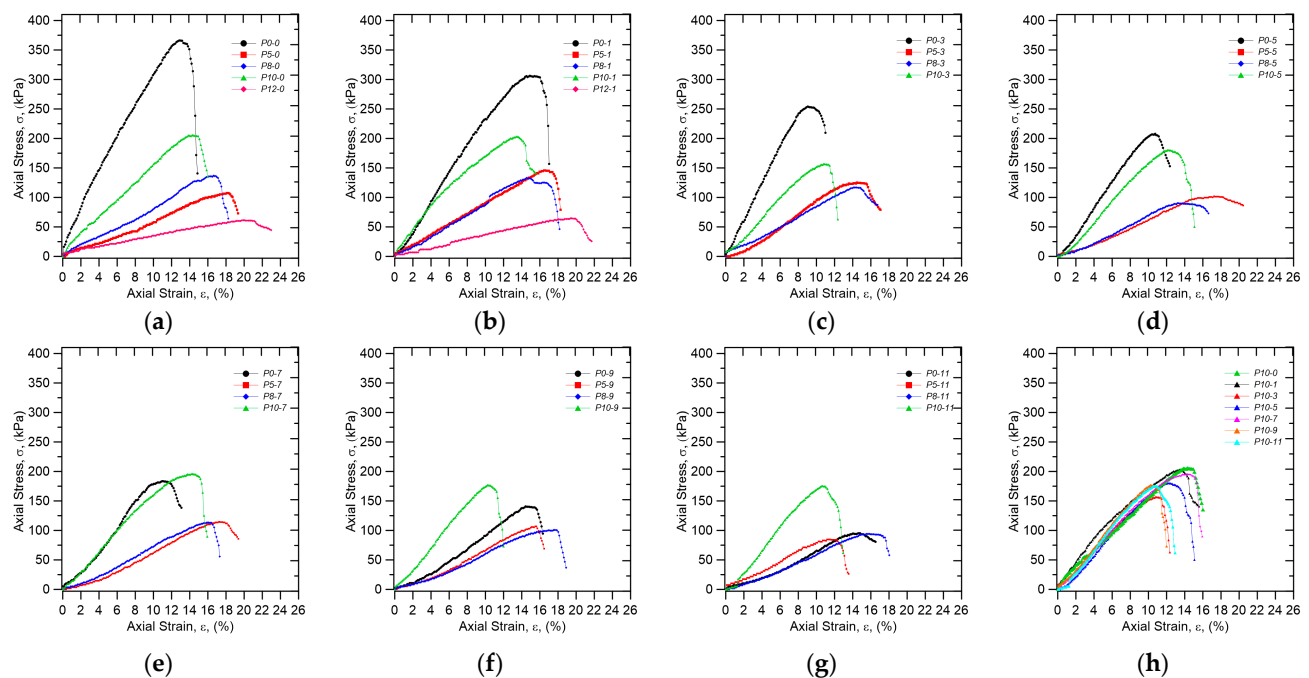


Figure 7. Undrained compression strength (UCS) tests. (a) Without F-T; (b) 1 cycle; (c) 3 cycles; (d) 5 cycles; (e) 7 cycles; (f) 9 cycles; (g) 11 cycles; (h) P10.

4.1.1. Test Results for Samples without Freeze–Thaw Cycling

Figure 7a shows the UCS of samples not subjected to F-T cycles. The UCS appeared to decrease with increasing mPCM addition. Due to the lower specific gravity of mPCM, the soil's maximum dry density and strength fell with the rising additive ratio. Lecompte et al. [46] also stated that the presence of mPCM in soil significantly decreased the mechanical strength, and concluded that mPCM behaved as voids that reduced the strength. Rao [3] stated that the low specific gravity of mPCM is essential in reducing strength.

In the curves in Figure 7a, the peak values of the UCS occurred in the 13–20% axial strain range. The strength was decreased in P5 and P8; mPCM was added to the soil, and the UCS tended to increase at P10. On the other hand, the UCS of P12 showed lower strength compared to P5, P8 and P10.

4.1.2. Test Results for Samples after Freeze–Thaw Cycles

Figure 7b–g show the changes in UCS of P0, P5, P8, P10 and P12, including specimens exposed to different F-T cycles. It was observed that P12 significantly (approximately 84%) reduced the strength of the soil. Considering the ability of the mPCM to keep the strength of the soil constant throughout the F-T cycles, the 12% additive ratio tests were performed without a cycle (0) and under one cycle. The F-T cycles did not progress due to the low strength caused by the mPCM content.

It was observed that increasing F-T cycles significantly reduced the UCS of the pure soil. It was concluded that the mPCM additive kept the strength of the soil stable throughout the increasing F-T cycles. The initial strength of the soil decreased with the addition of mPCM, while it preserved its strength in F-T cycles. Increasing the mPCM content in the soil increased the strength, and it was determined that the sample performed better in the F-T processes, especially with a 10% mPCM content. In Figure 7h, it was determined that the specimens remain in the elastic ranges until 10% axial deformation in all F-T cycles (1, 3, 5, 7, 9 and 11) at 10% mPCM contribution. It was seen that a 10% mPCM additive content improved the strength of the soil compared to other additive ratios, and kept it constant throughout the F-T cycles. A similar trend was identified in previous studies [3,25,27,38].

In the study conducted by Chen [38], it was stated that with the increase in the mPCM content in the CH soil used, the failure became brittle and the plastic properties

weakened at the damage stage. This behavior became more evident with the increase in the amount of additives. In particular, when the mPCM ratio is 10%, it has been stated that the strength decreases linearly until it completely disappears [38]. On the contrary, the strength remained stable in the 10% mPCM-added samples in MH soil used in this article. These results prove that the soil and the mPCM additive should be determined by each other. The factors explaining this can be listed as the type of soil used, the mineralogical structure of the soil, the particle size distribution, and the mPCM type. Figure 8a shows the relationship between the unconfined compressive strength (q_u) determined in soils with mPCM added and the number of F-T cycles applied. When Figures 7a and 8a are evaluated together, it is clear that the UCS of the P5-0, P8-0, P10-0, and P12-0 samples decreased by 71, 63, 44, and 84%, respectively, compared to the P0-0 sample (pure soil). The unconfined compressive strength in pure soil was 366.54 kPa. After 1, 3, 5, 7, 9, and 11 F-T cycles, the strength decreased by 16%, 31%, 43%, 50%, 62%, and 74%, respectively, and a significant reduction occurred.

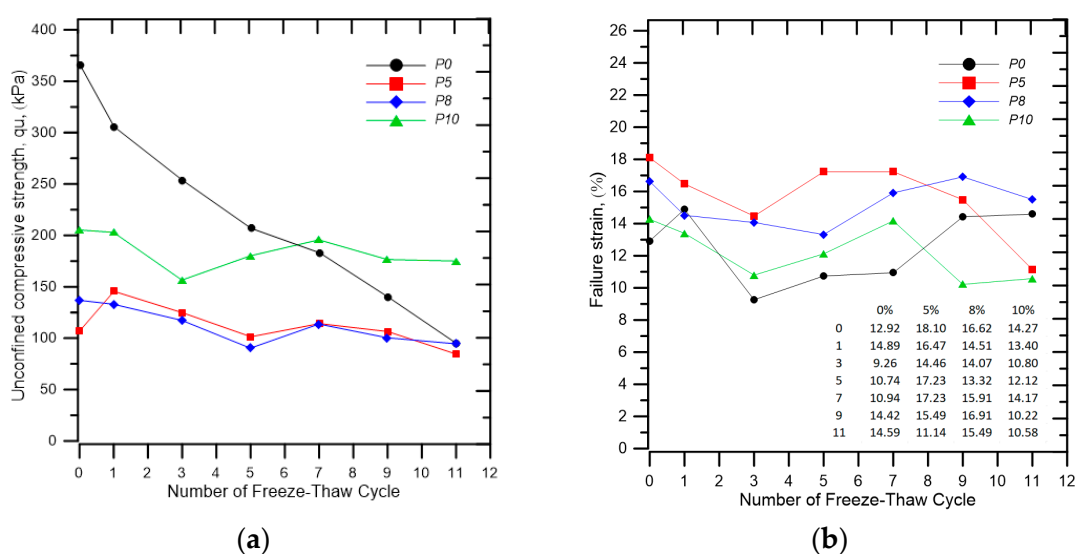


Figure 8. The relationship between the number of freeze–thaw cycles and the (a) q_u and (b) the failure strain.

The strength behaviors of P5 and P8 were very similar. This is because the OWC values of the P5 and P8 samples were the same (39%) and on the 85% water saturation curve (Figure 2). The strength behaviors of P5 and P8 prepared in the same OWC were not affected by the increase in the mPCM content from 5% to 8%. Thus, the behaviors of the P5 and P8 test samples were similar throughout the F-T cycles. The strengths of the P5-11 and P8-11 samples were the same as those of the P0-11 samples. While it is seen that the P0-11 specimen lost 74% of its value at the end of 11 cycles, P5-11 and P8-11 remained nearly constant in all processes. This shows that the strength of the mPCM-added soil can be kept constant without being affected by temperature fluctuations. Thus, incorporating mPCM into the soil can maintain the strength unaffected during F-T cycles.

In addition, it was determined that when the mPCM ratio was 10%, the strength increased compared to other contents, and showed better behavior in terms of F-T cycles. It was observed that the P10-11 sample showed a steadier performance than the other additive ratios, with a maximum strength reduction of 14%. These results indicate that the mPCM additive effectively reduced the negative changes in the heat-giving and -receiving property and the strength of the soil during F-T cycles. This is because, although P10 was similar to the P5 and P8 samples in terms of OWC (39–40%), the OWC value was on the 80% water saturation curve (Figure 2). The strength behavior of the P10 test specimens prepared with similar OWCs was positively affected by the increased mPCM content. The increase in the amount of mPCM filled the voids of the soil, preventing the free water from

freezing and the volume from changing. Thus, the UCS behavior improved compared to the P5 and P8 samples.

The strain corresponding to the maximum stress is the failure strain. The failure strain is an important parameter in the stress–strain behavior of the soil. Figure 8b shows the relationship between the failure strain and the number of F-T cycles. It demonstrates that F-T processes cause significant changes in the failure strain behavior of pure soil and soil with added mPCM. In all soil mixtures, the first and third F-T cycles caused a decrease in the failure strain, while the fifth and seventh F-T cycles caused an increase. The 9th and 11th cycles affected the strains differently. In general, increasing the mPCM ratio decreased the failure strain.

Chen [38] stated that the first F-T cycle significantly affected the strain behavior of the samples without added mPCM, while the strain did not differ much in other processes. The failure strain in the samples with added mPCM was unaffected by F-T cycles and remained almost constant. Chen [38] also stated that the failure strain decreases with increased mPCM content. He explained this behavior via the fact that the mPCM shell is a polymer material with a certain degree of brittleness [50], and the compressive strength of the PCM is small, increasing the brittleness of the soil.

In Figure 9, the photos taken before and after the UCS test of the samples subjected to 11 F-T cycles (the cycle in which the greatest strength reduction occurs) are presented. As seen in Figure 9, the F-T cycles caused wear fatigue in the samples. For this reason, after the samples were taken from the cabinet after completing their cycles, they were carefully removed from the stretch film they were wrapped in. In the post-experiment photos shown in Figure 9, it is clear that the failure of the soil changed with the contribution of mPCM. With the increasing mPCM ratio, the soil was exfoliated and failed. In addition, the white-colored mPCM particles are also clearly seen. This proves that mPCM mixes homogeneously into the soil.



Figure 9. View of specimens before and after unconfined pressure test; (a) 0% mPCM, (b) 5% mPCM, (c) 8% mPCM, (d) 10% mPCM.

Rao [3] stated that increasing the mPCM ratio caused significant fragility in soil samples. In this study, fragility was not observed in the samples with increasing mPCM ratios.

The reason for this is that the OWC was high (35–40%) in the soils with and without additives. The OWC used by Rao [3] was 15%. Due to the increasing water content and increasing mPCM ratio, the failure of the soil sample changed from brittle to ductile. This caused the soil to be exfoliated.

Before and after being subjected to the F-T cycles of the samples used in the unconfined pressure experiments, diameter (D), length (L), and volume change (ΔV) controls were made. Lu et al. [51] suggested a reference method for calculating the volumetric change, as shown in Figure 10a. The diameter of the sample was measured three times along 1/3 of its height, and the height of the sample was measured with a digital caliper from three different places. The average diameter and height values obtained are substituted in Equations (1) and (2), and the volume is calculated as in Equation (3). The initial and final volumes were obtained using the diameter and height values before and after exposure to

F-T cycles. The relationships between the volumetric changes obtained from the unconfined compression tests and the applied number of F-T cycles are presented in Figure 10b.

$$D_{ort} = \frac{1}{3} (D_1 + D_2 + D_3) \quad (1)$$

$$H_{ort} = \frac{1}{3} (H_1 + H_2 + H_3) \quad (2)$$

$$V = \frac{1}{4} \times \pi \times D_{ort}^2 \times H_{ort} \quad (3)$$

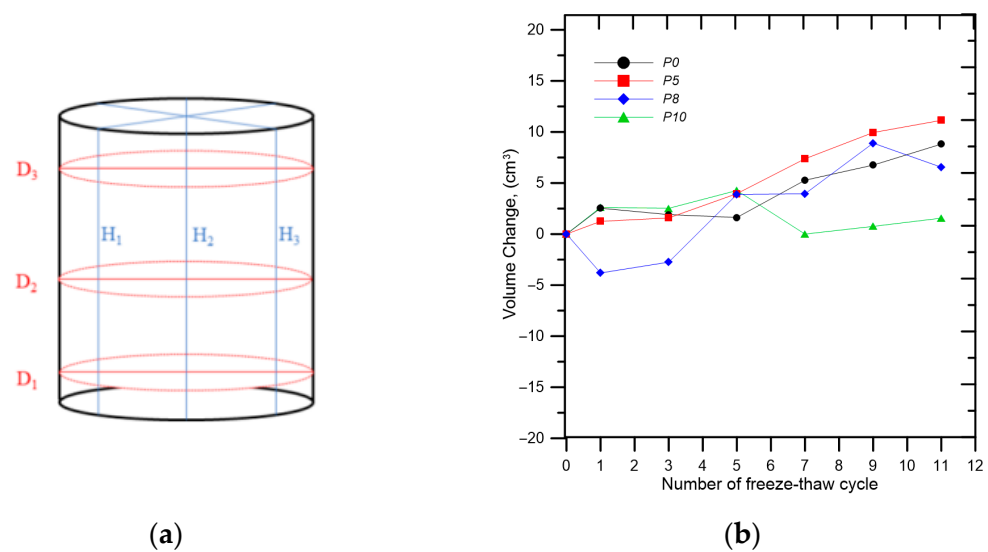


Figure 10. (a) Volume change calculation (Lu. et al. [51]). (b) ΔV —Freeze–thaw cycle number relationship.

The increase in F-T cycles caused a change in the volumes of P0, P5, P8 and P10. According to the calculated volume changes, at the 11th cycle, 9, 11, 7, and 2 cm³ volumetric changes occurred in P0-11, P5-11, P8-11, and P10-11, respectively. With the addition of the 10% mPCM, it is seen that fewer volume changes occurred in total. In this study, the optimum mPCM ratio was 10%, and higher strength and less volumetric change were observed during F-T cycles.

During the F-T cycle, the mPCM undergoes a phase change to generate and store heat. When the free water content of the sample is high, if the mPCM content is low, the energy released by the mPCM is insufficient to resist (prevent) the volumetric change of the soil particles from expanding with external temperature change. Therefore, when mPCM is used, it is important to experimentally examine its performance in relation to the soil. mPCM behaved similarly to soil voids, reducing free water migration and freezing in soil voids.

When Figures 8a and 10b are evaluated together, it is seen that the without-mPCM sample exhibits a more significant volumetric change with increasing F-T cycles, and the increasing volumetric change behavior decreases the strength. It was found that increasing mPCM content changed the volumetric change less, and kept the strength constant throughout the F-T cycles. This behavior shows that the increase in the mPCM additive content has an improving effect on the strength behavior of the soil.

It was checked whether there was a change in the weights of the samples in each F-T cycle. For this purpose, the weights were weighed before they were put into the cabinet and removed from the cabinet, and the differences between the two weights were noted (Figure 11).

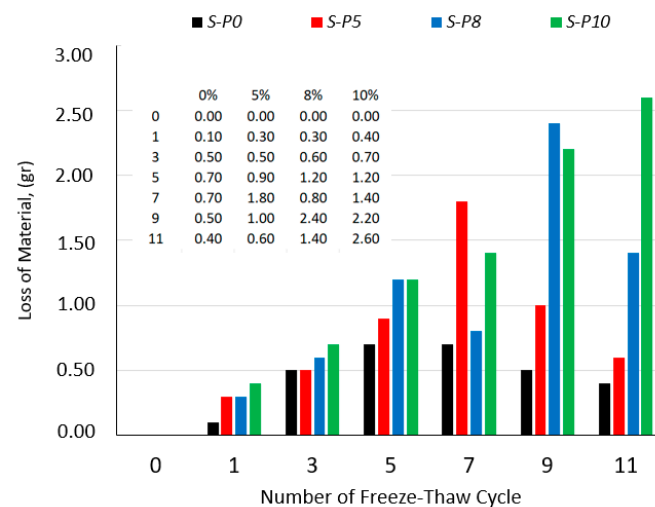


Figure 11. The relationship between the material loss (gr) and the number of freeze–thaw cycles.

F-T cycles cause losses of material. Due to the softening during the thawing process (waiting at 20 °C for 24 h), some soil remained smeared on the stretch film. The amount of lost material generally increased with the increasing number of F-T cycles. The maximum amounts of lost material in P0, P5, P8 and P10 were calculated as 0.70, 1.80, 2.40, and 2.60 g, respectively. These amounts of smeared material are minimal and did not change the water content of the test samples.

To determine the long-term effects of mPCM additive on strength, in addition to the experiments, test samples with and without mPCM additive were tested in three groups. The first group's samples were subjected to 11 cycles and then kept in a desiccator for 30 days, and the unconfined compressive strength (q_u) was measured. The second and third group's samples were prepared in the relevant optimum water content without applying an F-T cycle and kept in the desiccator for 30 and 60 days, respectively. Then an unconfined pressure test was performed.

The first, second and third groups of specimens are labeled as P (0, 5, 8, 10% mPCM)-11-30, P (0, 5, 8, 10% mPCM)-0-30 and P (0, 5, 8, 10% mPCM)-0-60, respectively. The first term represents the mPCM ratio, the second term represents the number of F-T cycles, and the third term represents the curing time in the desiccator. The obtained unconfined compression test results are presented in Figure 12a–c. Furthermore, the non-cycle and 11-cycle test results with and without mPCM samples are shown in Figure 12d, together with the results derived from these three groups of experiments.

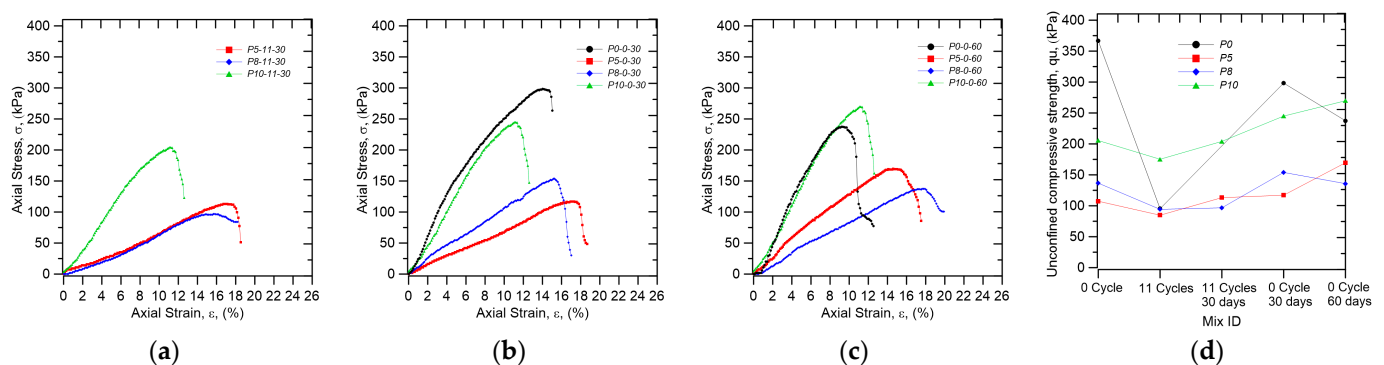


Figure 12. Unconfined compression test results of samples with waiting for 30–60 days; (a) 30 days waiting after 11 cycles (group 1); (b) 30 days waiting without cycle (group 2); (c) 60 days waiting without cycle (group 3); (d) unconfined compression test results for group tests.

Figure 12a shows the results obtained from the first group of experimental samples. At the end of the 30 days, an unconfined compression test was performed on P5, P8 and P10, but unconfined compression tests could not be performed on the pure soil (P0). Pure soil showed a more sensitive behavior and it was damaged after the F-T cycles, and the samples were broken. According to the unconfined compression test results performed on P5, P8 and P10, the unconfined compressive strengths (q_u) of the P5-11-30 and P5-0 samples were quite close to each other. The strength of the P8-11-30 sample decreased compared to the P8-0 sample. The same unconfined compression strength was obtained in the P10-11-30 and P10-0 samples.

The results obtained from the second group's test samples are presented in Figure 12b. Strength reduction occurred in the P0-0-30 sample compared to the P0-0 sample. The P5-0-30, P8-0-30 and P10-0-30 samples had higher strengths than the P5-0, P8-0, and P10-0 samples, respectively.

The results obtained from the third group's test samples are presented in Figure 12c. Strength reduction occurred in the P0-0-60 sample compared to the P0-0 sample. In the P5-0-60 sample, a higher strength was obtained than in the P5-0 sample. The P8-0 and P8-0-60 samples showed the same strength values. A higher strength was obtained in the P10-0-60 sample compared to the P10-0 sample.

In Figure 12d, the highest and the lowest strengths obtained were in the P0-0 and P5-0 samples, at 355.64 kPa and 107.33 kPa, respectively. The P0-11, P5-11, and P8-11 samples showed the same strengths. The highest strength in 11 cycles was obtained in the P10-11 sample. After the no cycling and 11th F-T cycle, the behaviors of the sample in the desiccator for 30 days and 60 days differed depending on the additive ratio. It was observed that there was no significant change in the strength of P5-11-30 and P8-11-30 samples. The P10-11-30 sample increased in strength. While the 5% mPCM additive increased the strength over time, the 8% mPCM additive showed the opposite behavior. The 10% mPCM added samples showed better performance and increased strength.

Rao [3] defined a dimensionless parameter, K , which is shown in Equation (4), to evaluate the antifreeze performance of mPCM-added soil. In Equation (4), the strength obtained from the unconfined compression tests and the antifreeze performance of mPCM were evaluated. The dimensionless parameter of K is formulated as:

$$K = \frac{\sigma_n - \sigma_0}{\sigma_0} \times 100\% \quad (4)$$

In Equation (4), in σ_0 and σ_n (the UCS values of the sample not subjected to F-T cycles and after the F-T cycles), n is the number of F-T cycles (1, 3, 5, 7, 9, 11).

The relationship between the K value and the applied F-T cycles is shown in Figure 13. With the increase in the mPCM ratio, less fluctuation occurred in the calculated K value. This shows that the mPCM additive used effectively prevents the reduction in strength in clayey silty (MH) soils after F-T cycles.

4.2. One-Dimensional Compression Tests (ASTM D2435 [49])

One-dimensional compression tests were performed on the pure soil and with mPCM-added samples subjected and not subjected to F-T cycles, and the results are presented in Figure 14 for each F-T cycle.

It is seen that the vertical effective stress-compression behaviors of the pure soil and with mPCM-added samples shown in Figure 14 are quite similar. Compression is rapid at the initial stress applied in the test. As the time progresses, the speed of compression slows down. After a certain period, the settlement under that load slows down to a negligible extent and ends. When settlement in a loading step is completed, the stress level is increased. Thus, this behavior repeats itself at each loading step.

It was checked whether there was a change in the weights of the samples in the F-T cabinet at the end of the relevant F-T cycles. For this purpose, the pre-cabin and post-cabin

weights were weighed, and the differences between both weights were determined. No material loss was observed in any of the test samples.

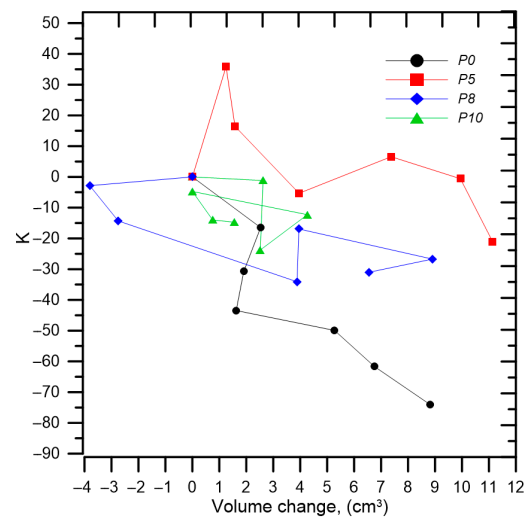


Figure 13. K—Freeze–thaw cycle number relationship.

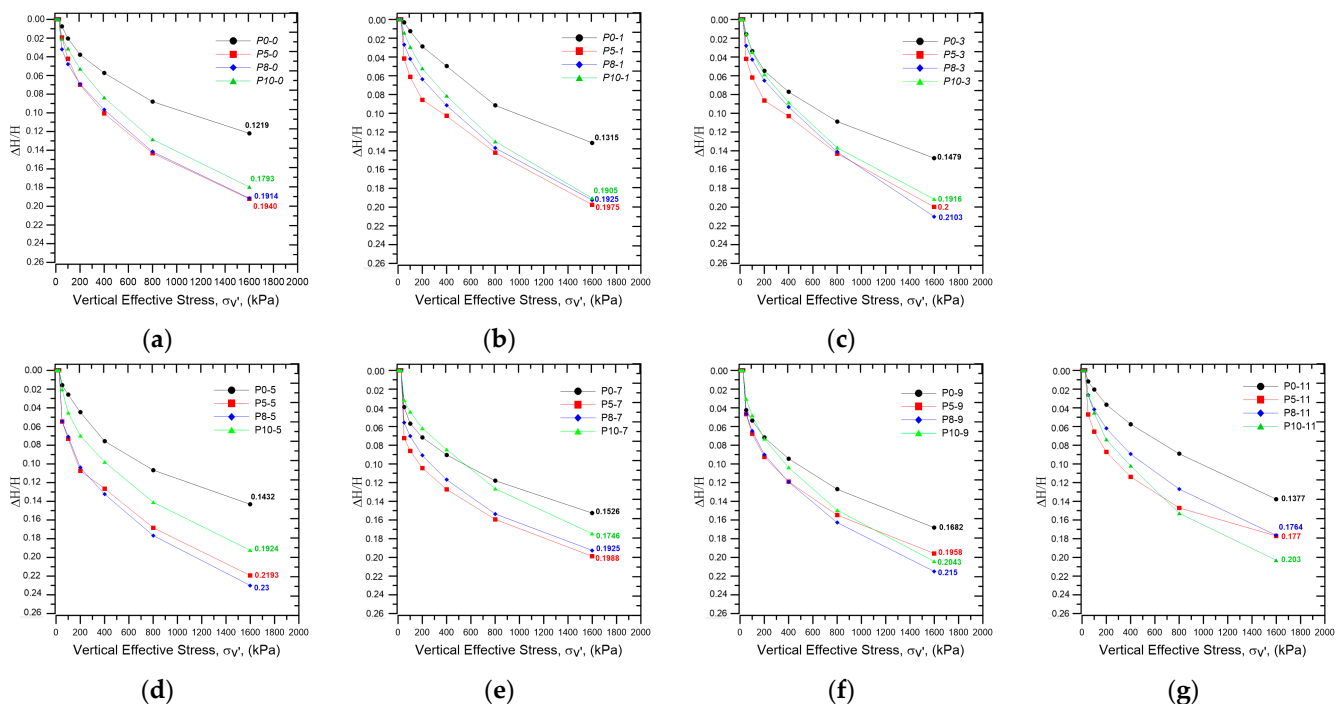


Figure 14. One-dimensional compression tests. (a) Without F-T cycle; (b) 1st cycle; (c) 3rd cycle; (d) 5th cycle; (e) 7th cycle; (f) 9th cycle; (g) 11th cycle.

Figure 14a shows the one-dimensional compression behavior of P0-0, P5-0, P8-0 and P10-0. In the curves in Figure 14a, $\Delta H/H$ at 1600 kPa, the final stress step, occurred between the ranges of 0.1219 and 0.1920. The compression behaviors of P5-0 and P8-0 samples were the same. The $\Delta H/H$ values for P5-0, P8-0 and P10-0 are higher than that of P0-0.

Figure 14b–g show the changes in the $\Delta H/H$ value of pure soil and mPCM-added samples subjected to different F-T cycles. The increasing F-T cycles significantly increased the settlement (0.1219–0.1682) of pure soil. In each cycle, the settlements of P5, P8 and P10 samples were more significant than that of pure soil. The reason for the changes occurring under increasing consolidation stresses is the explosion of microcapsules. Therefore, the mPCM additive kept the soil settlement constant with increasing F-T cycles. Generally, the

settlements in P10 samples were less than those in P5 and P8 samples. Also, in Figure 14a–g, the trends in the $\Delta H/H$ (vertical effective stress curves) of the mPCM-added samples are quite similar under different mPCM contents.

As a result, increasing F-T cycles caused a significant increase in $\Delta H/H$ for pure soil. Although the initial settlement value of the sample increased with the mPCM-additive, the settlements in the F-T cycles remained approximately the same. Increasing the mPCM additive ratio decreased settlements, and it was determined that the sample performed better in F-T cycles, especially with the 10% mPCM additive. It was observed that the P10-11 sample preserved its performance more notably than the other samples with different additive contents, with a maximum of 13% increased settlement. From the results obtained, it has been determined that the mPCM additive effectively reduces the adverse effect in sample settlement during the F-T cycles with its heat transfer-removal property. It shows that the 10% mPCM additive is compatible with the MH soil, and the settlement of the sample can remain constant without being affected by temperature fluctuations.

Figure 15a–c show the relationships between the $\Delta H/H$ obtained at 50 kPa, 100 kPa, and 200 kPa stresses applied in the one-dimensional compression tests performed on P0, P5, P8 and P10 samples.

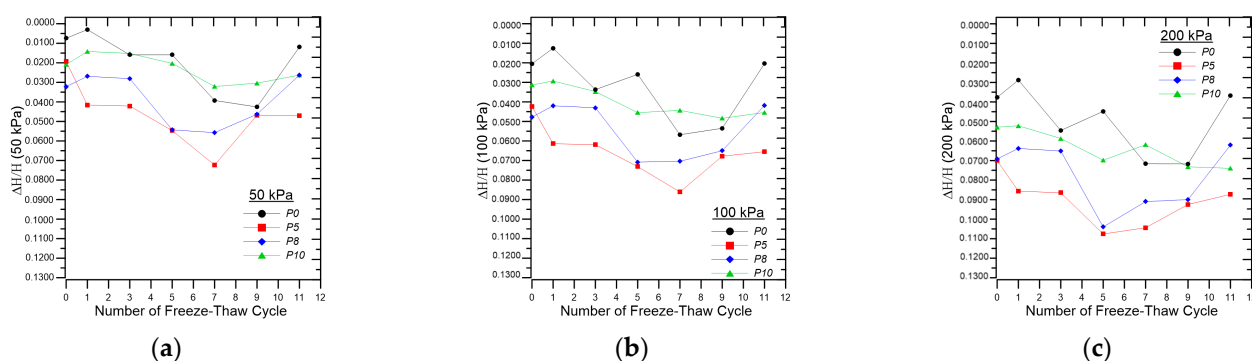


Figure 15. The variation in $\Delta H/H$ with freeze–thaw cycle number relationship.

When the $\Delta H/H$ values obtained at different stresses (50 kPa, 100 kPa, 200 kPa) are lower than that at the final stress (1600 kPa), the $\Delta H/H$ values of the mPCM additive are narrow. They appear to vary within a range, and this shows that the mPCM additive affects the adverse changes in soil settlement during F-T cycles under low consolidation stresses (50–200 kPa).

4.3. Microscopic Properties

When the UCS test results are evaluated, the P10 samples can be seen to have performed better than P5 and P8 in relation to the behavior of pure soil after F-T cycles. In this case, mixing the high-plasticity clayey silt soil (MH) used in this study with 10% mPCM is recommended, as this results in the best behavior. The high-plasticity clayey silt soil (MH), mPCM and P10 used in the tests were examined in SEM analysis with different levels of magnifications. However, since a representative sample could not be obtained, SEM, DSC, and FT-IR analyses could not be performed on the P5 and P8 samples. The SEM, DSC, and FT-IR analysis results of soil, mPCM, and P10 are presented in Figures 4, 5 and 16, respectively. It is seen that the high-plasticity clayey silt soil (MH), mPCM, and P10, whose photos are given in Figure 16, were homogeneously mixed.

Figure 16a shows that the MH soil has a layered structure and voids between the layers; the particle sizes are widely variable, the soil is not homogeneous, and there is roughness on the surface of the soil. In addition, there are clusters in certain regions. The shapes of the clustered groups are close to spherical, but the dimensions vary between 12.66 μm and 61.36 μm . With the help of 10,000-times magnification, it is seen that the

clustered structures also have layers. Soil particle diameter distribution was determined by hydrometer analysis, and is presented in Figure 1a. The SEM results also support this.

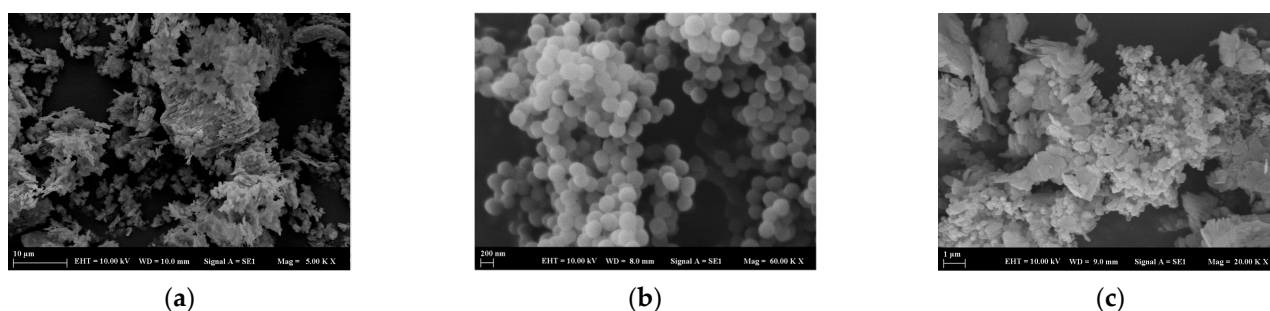


Figure 16. SEM analysis images: (a) MH soil; (b) mPCM; (c) P10.

Figure 16b shows that the mPCM is spherical and homogeneous. There are no cracks or cavities on the mPCM surface. There are clusters amongst the particles, and these clusters consist of nested spherical structures. There are tunnel-shaped spaces between the spherical assemblages. The particle sizes in the mPCM structure are small compared to the soil. Particle sizes are presented in Figure 1b.

In Figure 16c, it is seen that spherical mPCM structures penetrated the layered soil in P10, and the sphericity was slightly impaired. While the soil particle sizes in the kaolin structure were around 50 µm on average, the particle size decreased to the nm range when mixed with mPCM. However, while the diameters of the spherical structures of mPCM were around 50 nm at most, the spherical structures seen in the soil reached the dimensions of 150–220 nm. With the addition of mPCM to the soil, it can be seen that the particle size becomes smaller than that of the soil.

When the DSC analysis results given in Figure 4c and Table 2 for P10 are examined, it is seen that there are two different peak temperatures in the melting process and only one peak temperature in the solidification processes. The temperature ranges where P10 is active are between -1 and 0.9 and 4.6 and 10 °C for an endothermic reaction and -4.3 and 1.8 °C for an exothermic reaction. However, in the ranges of -1 to 0.9 and 4.6 to 10 °C, mPCM showed a phase change from solid to liquid with peak phase change temperatures (melting point) of -0.4 and 7.5 °C, respectively. The enthalpy values were 5.138 and 32.46 J/g during this phase change. Between -4.3 °C and 1.8 °C, the mPCM transitioned from the liquid to the solid phase, with a peak phase change temperature of -0.8 °C. During this phase change, its enthalpy was 15.55 J/g.

When the FT-IR analysis result given in Figure 5 of P10 is examined, the peaks belonging to the soil and the peaks originating from the mPCM can be seen on the spectrum. Here, there is a peak at 1726 cm^{-1} . mPCM did not interact chemically with the soil. Only the mPCM in the nanosphere structure was placed in the silicate material, which had a layered structure.

4.4. Temperature Distribution

Test samples prepared for unconfined compression and one-dimensional consolidation tests were subjected to freeze and thaw in a cabinet at -20 °C to $+20$ °C. The temperatures were measured for P0, P5, P8 and P10 test samples (Figure 17). For this purpose, a K-type industrial thermocouple that can measure between -50 °C and 1300 °C was used. Measurements were made in the middle (~ 35 mm), bottom (~ 65 mm), and top (~ 15 mm) of the unconfined compression test sample ($37.5\text{ mm} \times 76\text{ mm}$). Since the sample height (76 mm) was small, no difference was observed between the measured values of the sample's middle, bottom, and top. Figure 17 shows the measurements taken from the midpoint of the test samples.

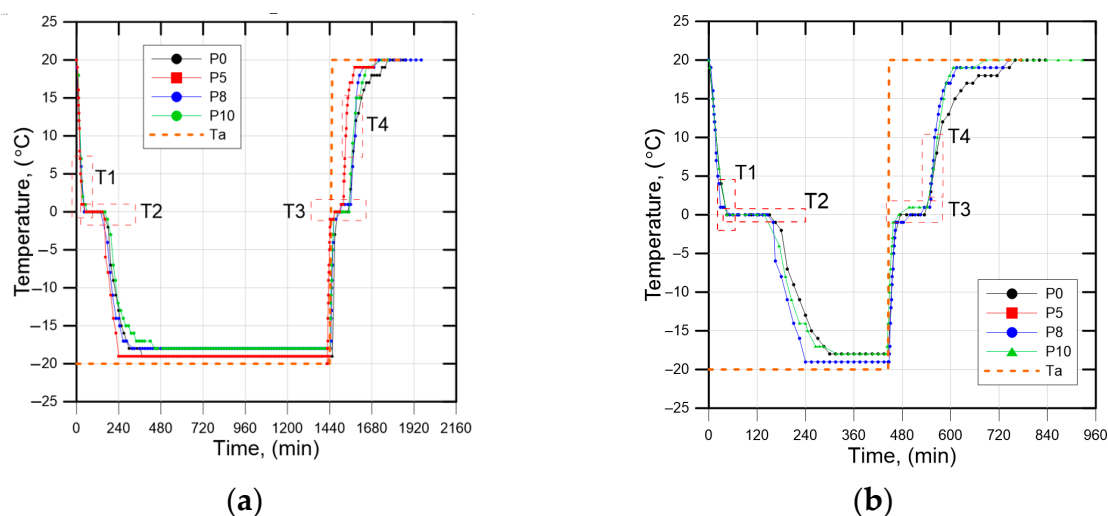


Figure 17. Temperature variations of samples (a) with 24 h waiting time and (b) without 24 h waiting time.

In Figure 17, ambient temperature is indicated by T_a . The ambient temperature was constant at $-20\text{ }^{\circ}\text{C}$ for 24 h and $+20\text{ }^{\circ}\text{C}$ for 24 h. Figure 17a shows data obtained on samples subjected to F-T cycles (at $-20\text{ }^{\circ}\text{C}$ for 24 h and $+20\text{ }^{\circ}\text{C}$ for 24 h). Figure 17b shows the data obtained when the ambient temperature changed to $+20\text{ }^{\circ}\text{C}$ immediately after the samples reached $-20\text{ }^{\circ}\text{C}$. The same values were obtained in both cases. During testing, the temperature curves of the samples containing the mPCM developed in four phases as the ambient temperature decreased and increased. These phases are shown as T1 (solidification–freezing transition period), T2 (freezing period), T3 (thawing transition period), and T4 (thawing period).

Pure soil (P0) completed the T1 period in 45 min, while P5, P8 and P10 completed this period in 52, 45, and 60 min, respectively. Samples P0, P5, P8, and P10 started to freeze (reached $0\text{ }^{\circ}\text{C}$) 45, 52, 45, and 60 min after the start of the test, respectively. $0\text{ }^{\circ}\text{C}$ is the beginning of the T2 period (freezing period). It is seen that the T1 times are close for the P0, P5, and P8 samples. However, there was a noticeable increase in T1 time for P10.

The time elapsed during T2 decreased with increasing mPCM ratio. The elapsed times of the P0, P5, P8 and P10 in the T2 period were 105, 93, 95, and 100 min, respectively. Adding mPCM reduced the soil's freezing point to varying degrees (9.5%, 9.5%, 5%). P0, P5, P8 and P10 reached the end of the T2 period 150, 145, 138, and 160 min from the beginning of the test, respectively. The completion times of the T1 and T2 periods are close to each other for all samples. However, the duration of the periods (T1 and T2) is thought to affect the working mechanism of mPCM.

T3 represents the waiting time at $0\text{ }^{\circ}\text{C}$ in the transition to the thawing period. With the increase in the mPCM ratio, a decrease in the time taken during T3 was observed. The T3 period times of P0, P5, P8 and P10 are 60, 25, 15, and 12 min, respectively. The waiting time (transition period to thaw) of the samples at $0\text{ }^{\circ}\text{C}$ was relatively short.

While the P0 reached $0\text{ }^{\circ}\text{C}$ to $+20\text{ }^{\circ}\text{C}$ (between T3–T4) in 225 min, P5, P8 and P10 completed this part in 215, 205, and 140 min, respectively. With the increase in the mPCM ratio, a decrease in the time taken during T4 was observed. The times in the T3 and T4 periods were very short for P10. As the T_a increased, the P0 changed in a much shorter time compared to P5, P8 and P10, especially in the T3 and T4 periods.

The time required for the P0 to drop from $20\text{ }^{\circ}\text{C}$ to $-20\text{ }^{\circ}\text{C}$ was 375 min. In soil with P5, P8 and P10, these times were 300, 330, and 445 min, respectively. The time required for the temperature to drop from $20\text{ }^{\circ}\text{C}$ to $-20\text{ }^{\circ}\text{C}$ generally increased with the increasing mPCM ratio of the sample. This provides further evidence for the suitable thermal effects of mPCM.

The addition of mPCM to the soil has a significant effect on the temperature behavior of the soil mix. For example, at the 25th minute of the test, the temperature of the P0 was

9 °C, while the temperatures of the P5, P8 and P10 were 4, 6, and 7 °C, respectively. As the heat generation from the mPCM during the phase change caused a thawing effect in the soil, the mPCM caused a slowdown in the freezing process of the soil. In addition, the increase in the additive content led the soil temperatures to decrease further.

5. Conclusions

This study investigated the effects of the mPCM additive on the strength and compressibility behavior of soil to be used in road embankments in cold regions under freeze–thaw cycles. For this purpose, an encapsulated mPCM core material, paraffin, and high-plasticity clayey silt (MH) soil with high kaolin content sensitive to freezing were used. The mineralogical, morphological, thermal, and chemical properties of MH and mPCM were investigated by SEM, FT-IR, XRD, XRF, DSC, and particle size analyses. With the addition of mPCM to the MH soil at different rates (5, 8, 10, 12%), the volumetric change and strength behavior of the infill soil when exposed to different freeze–thaw cycles (1, 3, 5, 7, 9, 11) were investigated. In this context, the findings obtained from the compaction, Atterberg limits, unconfined compression strength, and one-dimensional compressibility experiments are as follows:

- The closeness of the grain sizes of mPCM and soil (MH) ensured the homogeneous mixing of soil and mPCM. mPCM is embedded into the silicate material, which has a layered structure. However, MH and mPCM did not interact chemically. It has been proven by SEM, particle size, DSC and FT-IR analyses that mPCM maintains its stable structure during phase changes and has organic-based PCM properties;
- With the increase in mPCM ratio, higher OWC, higher Atterberg limits and lower maximum dry unit weight values were obtained. This is because the microcapsules exploded during the experiment;
- With increasing freeze–thaw cycles, the strength of the pure soil decreased and settlements increased. mPCM affected the mechanical behavior of the soil with its thermal properties, decreased the strength (USC) of the soil and increased its settlement. However, it kept the strength and settlements constant throughout the freeze–thaw cycles;
- mPCM mainly prevents volume changes by dispersing in the soil voids, reducing the passage of free water in the voids and freezing (affecting the thermal properties of the soil). In particular, it was determined that the 10% additive ratio was the most suitable ratio for volume change, strength and settlement behavior during freeze–thaw cycles (from −20 °C to +20 °C).

In this study, a soil with an OWC value of 35%, a kaolin content of 89% and a high freeze–thaw sensitivity was used. Freezing and thawing negatively affects the mechanical behavior of the soil. In this context, the interoperability of the mPCM and MH used was examined. Although mPCM negatively affected the soil's high OWC, settlement and strength behavior compared to the initial conditions, it kept its mechanical properties constant throughout the F-T cycles as a result of its physical interaction with the soil. Studies carried out in this context have been promising for improving the mechanical properties of soils exposed to freeze–thaw.

6. Limitations and Recommendations

- In this study, a single soil and a single mPCM type were used. Therefore, the results of this study should not be generalized. More studies should be conducted using different mPCM and soil types.
- In applications of mPCM with the soil, the type of mPCM, compatibility with the soil, chemical stability, thermal properties, and environmental protection should be considered. In addition, PCM with appropriate phase changes should be selected according to regional climatic conditions and project target temperatures.
- Studies have proven that mPCM reduces the temperature fluctuation of the soil during freeze–thaw cycles and keeps a stable behavior. Due to its working mechanism, mPCM can control the internal temperature of the soil, reduce volume changes and improve

soil behavior. In geotechnical applications, large quantities of soil will require large amounts of mPCM. These applications should be made after determining the optimum additive content in mPCM mixed earthworks to be carried out in cold regions. In using mPCM, it should be considered that the water content may increase due to the explosion of microcapsules under high compaction energy and high stresses.

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