

ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

**EVALUATING THE EFFICACY OF POLYETHYLENE GLYCOL AND
MAGNESIUM CHLORIDE AS ANTI-FREEZE AGENTS ON THE MECHANICAL
PROPERTIES OF CLAYS SUBJECTED TO FREEZE-THAW CYCLES**



Ph.D. THESIS

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Department of Civil Engineering

Soil Mechanics and Geotechnical Engineering Programme

JULY 2024

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ

**POLİETİLEN GLİKOL VE MAGNEZYUM KlorÜRÜN ANTİFRİZ MADDELERİ
OLARAK DONMA-ÇÖZÜLME DÖNGÜLERİNE MARUZ KALAN KİLLERİN
MEKANİK ÖZELLİKLERİ ÜZERİNDEKİ ETKİNLİĞİNİN DEĞERLENDİRİLMESİ**

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To my spouse and family,



FOREWORD

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ABBREVIATIONS

AASHTO	:American Association of State Highway and Transportation Officials
ASTM	:American Society for Testing and Materials
C	:Cement
CBR	:California Bearing Ratio
CRREL	:Cold Regions Research and Engineering Laboratory
DDL	:Diffuse Double Layer
DI	:Durability Index
F-T	:Freeze-Thaw
FTIR	:Fourier Transform Infrared Spectroscopy
IR	:Infrared Radiation
LL	:Liquid Limit
MDD	:Maximum Dry Density
MEG	:Monoethylene Glycol
MgCl₂	:Magnesium Chloride
OWC	:Optimum Water Content
P-XRD	:Powder X-ray Diffraction
PAM	:Polyacrylamide
PEG	:Polyethylene Glycol
PI	:Plasticity Index
PL	:Plastic Limit
SEM	:Scanning Electron Microscopy
TRIP	:The Road Information Program
USACE	:United States Army Corps of Engineers
USCS	:Unified Soil Classification System
XRD	:X-ray Diffraction



SYMBOLS

d	:Inner-Planar Spacing Distance
e_{res}	:Equilibrium Pore Space Ratio
e_f	:Frozen Void Ratio Before Thawing Starts
e_{th}	:Thawed Void Ratio
K_{ff}	:Conductivity of Frozen Fring
n	:Diffraction Order
N_{res}	:Number of Freeze-Thaw Cycles to Reach Equilibrium
q_u	:Unconfined Compression Strength
T_1	:Temperature at the base of the active ice lens
T_f	:In-situ temperature
γ_d	:Dry Unit Weight
θ	:Critical Angle of Incidence on the Crystal Plane
λ	:Wavelength of the X-rays
σ_0	:Field Load



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EVALUATING THE EFFICACY OF POLYETHYLENE GLYCOL AND MAGNESIUM CHLORIDE AS ANTI-FREEZE AGENTS ON THE MECHANICAL PROPERTIES OF CLAYS SUBJECTED TO FREEZE-THAW CYCLES

SUMMARY

The construction industry frequently encounters significant challenges due to adverse weather conditions, leading to project delays and increased costs. Approximately 45% of global construction projects are interrupted by weather-related incidents, resulting in billions of dollars in additional expenditures annually. One critical issue in colder climates is the impact of freeze-thaw (F-T) cycles, which disrupt soil stability and permeability, causing cracks, expanding pore spaces, and affecting water retention and drainage. These cycles reduce soil strength and cohesion, increasing the risk of erosion and runoff and compromising the structural integrity of construction projects.

Traditional soil stabilization methods, such as mixing soils with cement or lime, have limitations, particularly in mitigating the effects of F-T cycles. These methods often fail to prevent the loss of soil strength and volume changes after successive F-T cycles, necessitating the development of more robust solutions.

This study investigates the potential of Polyethylene Glycol (PEG 400) and magnesium chloride (MgCl_2) solutions as antifreeze and stabilizing agents for fine-grained soils. The research evaluates the effects of these solutions on soil properties, including plasticity, strength, and durability, under both unfrozen conditions and after F-T cycles. By understanding these effects, the study seeks to identify effective methods to mitigate the detrimental impact of F-T cycles on soil embankments in cold climates.

Initial experiments with PEG 400 included Atterberg limits tests, compaction tests, unconfined compressive strength (UCS) tests, and a single F-T test at -20°C . The results indicated that higher concentrations of PEG increased the soil's Atterberg limits, decreased the maximum dry density, and increased the optimum water content. Although PEG acted as an antifreeze by preventing strength loss after one F-T cycle at higher concentrations, it significantly reduced soil strength initially due to its hydrophilic properties, rendering it ineffective as a stabilizing additive.

Further tests on a clay-cement mixture with PEG showed that PEG significantly reduced the strength of the mix. Strength tests after one F-T cycle at -5°C revealed that higher concentrations of PEG could not prevent strength loss and further decreased the mixture's strength. This indicated that PEG negatively affected the cement hydration process, making it unsuitable for soil stabilization in cold climates.

In contrast, MgCl_2 showed more promising results. A comprehensive range of tests, including Atterberg limits tests, UCS tests, compaction tests, volume change measurements, F-T cycle tests, and microstructural analyses using FTIR, SEM, and XRD, demonstrated that higher concentrations of MgCl_2 (14% solution) significantly lowered the soil's Atterberg limits, improved compaction by increasing the maximum

dry density and reducing the optimum water content, and enhanced soil strength even without curing. Curing showed minor effects, with higher MgCl_2 concentrations further improving UCS. MgCl_2 also demonstrated durability under F-T cycles, maintaining soil strength and volume stability.

Durability index (DI) values quantified the stability of MgCl_2 -treated soils after F-T cycles. At -10°C , soils treated with 9% and 14% MgCl_2 solutions maintained their strength through seven F-T cycles, whereas untreated and 4% MgCl_2 -treated soils showed significant strength reductions. At -20°C , untreated and 4% MgCl_2 -treated soils lost considerable strength, while 9% MgCl_2 -treated samples exhibited smaller strength reductions, and 14% MgCl_2 -treated samples maintained consistent strength across seven cycles. DI values confirmed that precise MgCl_2 concentration adjustments are crucial for soil stability in cold climates.

Volume stability tests showed that untreated and 4% MgCl_2 -treated samples expanded with F-T cycles, while 9% and 14% MgCl_2 -treated samples showed no significant volume changes, indicating effective stabilization. Microstructural analyses revealed that MgCl_2 promoted clay flocculation and particle size enlargement, aligning with observed improvements in soil properties.

Comparative analysis of MgCl_2 and PEG under F-T conditions highlighted MgCl_2 's superior performance in enhancing soil strength, durability, and volume stability. MgCl_2 's ability to improve soil properties and maintain structural integrity under challenging environmental conditions makes it a more effective antifreeze and stabilizing agent than PEG.

The findings suggest that adopting MgCl_2 as a stabilizing and antifreeze agent can significantly improve the performance of soil embankments in cold climates. By preventing the adverse effects of F-T cycles, MgCl_2 ensures that construction projects can continue during colder periods, reducing delays and associated costs. This research provides a foundation for future studies to explore the full potential of MgCl_2 in various geotechnical applications and contribute to the development of more resilient infrastructure in regions prone to harsh weather conditions.

In summary, this thesis provides compelling evidence that MgCl_2 is an effective antifreeze and stabilizing agent for fine-grained soils in cold climates. Its ability to improve soil properties, maintain structural integrity, and enhance durability under F-T cycles makes it a valuable additive for the construction industry. Future research should build on these findings to optimize MgCl_2 concentrations for different soil types and climatic conditions, ultimately contributing to the development of more resilient and sustainable infrastructure.

POLİETİLEN GLİKOL VE MAGNEZYUM Klorürün ANTİFRİZ MADDELERİ OLARAK DONMA-ÇÖZÜLME DÖNGÜLERİNE MARUZ KALAN KİLLERİN MEKANİK ÖZELLİKLERİ ÜZERİNDEKİ ETKİNLİĞİNİN DEĞERLENDİRİLMESİ

ÖZET

İnşaat sektörü, olumsuz hava koşulları nedeniyle ciddi zorluklarla karşı karşıya kalmaktadır, bu da proje takvimlerini ve maliyetlerini önemli ölçüde etkilemektedir. Dünya genelinde inşaat projelerinin yaklaşık %45'i hava koşullarından kaynaklanan aksaklıklar nedeniyle gecikmekte ve her yıl milyarlarca dolarlık ek maliyetlere neden olmaktadır. Bu zorluklar, özellikle donma-çözülme (D-Ç) etkisinin belirgin olduğu yol yapım projelerinde daha da belirgindir. Donma-çözülme döngüleri, mühendislik amaçları için kritik olan zemin özelliklerini önemli ölçüde değiştirmekte ve yol yapımında ciddi riskler oluşturmaktadır. Bu döngüler, zeminin stabilitesini ve dayanıklılığını tehlikeye atarak, özellikle orta ve yüksek enlemler ile yüksek rakımlı bölgelerde belirgin değişikliklere yol açmaktadır.

Donma-çözülme döngüleri, zemin stabilitesi ve geçirgenliğini bozarak, kuruma çatlakları ve gözenek genişlemesine neden olur ve bu da su tutma ve drenaj problemlerine yol açar. Ayrıca, bu döngüler zemin mukavemetini ve kohezyonunu azaltarak, erozyon ve yüzey akışına karşı zemini daha hassas hale getirir. Donma-çözülme döngüleri, gevşek zeminlerin hacmini azaltırken, yoğun zeminlerin hacmini arttırarak zemin yapılarına zarar verir. Bu durum, aynı zamanda, düzensiz donma kabarması, çözülme oturmaları ve temel bütünlüğünün bozulması gibi yapısal arızaları hızlandırır.

Bu zorlukların üstesinden gelmek için geleneksel yöntemler, zeminleri çimento veya kireçle karıştırmayı içerir. Ancak, son yıllarda araştırmacılar, zararları en aza indirmek için çeşitli yenilikçi katkı maddelerini incelemiştir. Bu alternatifler arasında uçucu kül, alkali ile aktive edilmiş volkanik kül ve cüruf, mikrobiyal indüklenmiş biyopolimerler, faz değişim malzemeleri, geri dönüştürülmüş polimer ve doğal lifler, kimyasallar ve polimerler gibi maddeler bulunmaktadır. Ancak, tüm bu iyileştirme yöntemlerinin ortak bir özelliği, D-Ç döngüleri sonrasında nihai zemin mukavemetinin, D-Ç döngülerinden önceki başlangıç mukavemetine göre azalması ve önemli ölçüde hacim değişikliklerine maruz kalmalarıdır.

Donma-çözülme döngülerinin neden olduğu hasarları önlemenin bir diğer yolu, zemine antifriz maddelerle iyileştirmektir. Bu yöntem, zemin neminin donmasını önleyerek buz mercceklerinin oluşumunu ve dolayısıyla çatlamayı engellerken, sıkıştırılmış zemin yapısının bütünlüğünü korur.

Çeşitli antifrizlerin zeminler üzerindeki etkisi ile ilgili araştırmalar nispeten sınırlıdır. Bu maddelerden biri, çeşitli uygulamaları olan bir tür Etilen Glikol olan Monoetilen Glikol'dür (MEG). Ancak, MEG'in antifriz olarak kullanımı üzerine yapılan bir çalışma, MEG'in kil üzerindeki olumsuz etkileri nedeniyle antifriz olarak uygun bir malzeme olmayabileceğini önermektedir. Bu çalışmanın bulguları, MEG ile

kariřtırılmıř zeminin dayanım ve sertlięinin kariřım oranı arttıķķa azaldıęını g stermektedir.

Dolayısıyla, soęuk havalarda antifriz olarak bilinen Monoetilen Glikol' n (MEG) kullanımı, olumsuz etkileri nedeniyle uygun olmayabilir. Bu tezde,  nemli antifriz  zelliklerine sahip olduęu bilinen Polietilen Glikol (PEG 400) kullanılarak alternatif ve daha etkili bir antifriz arayıřında deneyler yapılmıřtır. Ancak, Atterberg limitleri, standart proktor ve serbest basınē testleri sonrası, PEG 400' n uygun olmadıęı sonucuna varıldı. PEG 400, Atterberg limitlerini  nemli  lē de artırdı ve zemin mukavemetini ciddi řekilde azalttı. Sonuē olarak, killi zeminin dayanımını etkilemeden antifriz etkisi saęlayacak alternatif bir malzeme arayıřına girildi ve Magnezyum Klor r ($MgCl_2$) umut verici bir   z m olarak belirlendi.

Magnezyum Klor r ($MgCl_2$)   zeltisinin zeminlerin mekanik  zellikleri  zerindeki etkilerini stabilizat r olarak inceleyen  alıřmalar, zemin m hendislik  zelliklerinde iyileřmeler g stermiřtir. Ancak, $MgCl_2$ 'nin donma-  z lme kořullarında antifriz maddesi olarak zemin  zelliklerine etkisi hen z arařtırılmamıřtır. Bu  alıřma, suda   z n r magnezyum klor r ($MgCl_2$)   zeltisinin kil zeminin fiziksel ve mekanik  zellikleri  zerindeki etkilerini, donmamıř kořullarda ve donma-  z lme d ng leri sonrasında incelemeyi amaēlamaktadır.

Bu doęrultuda,  alıřmada,  eřitli deneyler gerēekleřtirilmiř; Atterberg limitleri, standart proktor, serbest basınē dayanımı (UCS) testleri ve $MgCl_2$   zeltisi ile iyileřtirilmiř zemin numuneleri  zerinde donma-  z lme (D- ) d ng leri uygulanmıřtır. Ayrıca, $MgCl_2$   zeltisinin zeminle etkileřimini mikroyapısal d zeyde anlamak amacıyla taramalı elektron mikroskobu (SEM), X-ıřını difraksiyonu (XRD) ve Fourier d n ř m kızıl tesi spektroskopisi (FTIR) kullanılarak mikroyapısal analizler de yapılmıřtır.

PEG   zeltisi ile ilgili test sonuēları, PEG konsantrasyonlarının artmasıyla birlikte Atterberg limitlerinde s rekli bir artıř g zlemlenmiřtir. PEG ile kariřtırılmıř zemin numuneleri iēin sıvı limit (L.L) %37, plastik limit (P.L) %19 ve plastiklik indeksi (P.I) %18 olan deęerler, %50 PEG   zeltisi ile sırasıyla %55, %22 ve %32 olmuřtur.

PEG konsantrasyonlarının artmasıyla birlikte maksimum kuru yoęunluk 17.5 kN/m^3 'ten 15.2 kN/m^3 'e d řm ř ve optimum su muhtevası %19'dan %28'e y kselmiřtir. PEG konsantrasyonlarının artması, zemin dayanımında azalmaya yol aēmıřtır. Ancak, PEG'in kil  zerinde antifriz etkisini incelemek amacıyla, kariřtırılmamıř numuneler ve %10, %20, %30, %40 ve %50 PEG   zeltileri ile kariřtırılmıř numuneler  zerinde -20°C 'de tek bir donma-  z lme d ng s  uygulanmıřtır. Sonuēlar, y ksek PEG konsantrasyonlarının kilin mukavemetini  nemli  lē de azalttıęını g sterdi; %50 PEG   z m  mukavemeti %68 oranında azalttı. Bu sonuē PEG'in hidrofilik yapısından dolayı bekleniyordu.  te yandan, %40 ve %50 konsantrasyonlarındaki PEG, -20°C 'de bir donma-  z lme d ng s nden sonra mukavemet kaybını  nlerken, kariřtırılmamıř numunede %27'lik bir mukavemet azalması yařandı. Bu, y ksek PEG konsantrasyonlarının antifriz olarak buz merceęi oluřumunu  nledięini g stermektedir. Ancak, PEG'in su tutma  zellikleri nedeniyle zemin yapısını olumsuz etkiledięi ve bařlangıēta dayanımı  nemli  lē de azalttıęı iēin PEG'in kil stabilizat r  olarak uygun olmadıęı sonucuna varıldı.

PEG'in kil mukavemetini azaltması nedeniyle, PEG'in kil-ēimento kariřımı  zerindeki etkileri de incelendi. Ancak, sonuēlar, PEG'in kil-ēimento kariřımının mukavemetini de  nemli  lē de azalttıęını g stermiřtir. Donma-  z lme d ng s  sonrası yapılan mukavemet testlerindedir, y ksek PEG konsantrasyonlarının bile mukavemet kaybını

önleyemediği ve kil-çimento karışımının mukavemetinin daha da azaldığı görülmüştür. Bu, PEG'in çimento hidrasyon sürecini olumsuz etkilediğini ve genel olarak mukavemeti azalttığını göstermektedir.

MgCl₂ çözeltisi ile yapılan testlerin sonuçları ise oldukça olumludur. Yüksek MgCl₂ konsantrasyonları, zeminin Atterberg limitlerini önemli ölçüde azaltmıştır. %14 MgCl₂ konsantrasyonunda, Likit limit %14, plastik limit %9 ve plastisite indeksi %19 oranında azalmıştır. Bu azalma, çift değerlikli magnezyum iyonlarının katyon değişimi süreci ile ilişkilidir ve difüze çift katman kalınlığının azalması sonucu meydana gelmiştir.

Ayrıca, MgCl₂'nin zeminin kompaksiyon değerlerini de iyileştirdiği gözlemlendi; maksimum kuru yoğunluk 17,4 kN/m³'ten 17,89 kN/m³'e yükseldi ve optimum su muhtevası %19'dan %17,7'ye düştü. Bu değişiklikler, hızlı katyon değişimi sonucu zemin parçacıklarının topaklaşması ve birikmesi nedeniyle meydana gelmiş, bu da kilin su emilimini azaltmış ve kuru yoğunlukları artırmıştır. MgCl₂ çözeltisi eklenmesi, zemin dayanımını da önemli ölçüde artırmıştır. %4, %9 ve %14 MgCl₂ konsantrasyonları için gözlemlenen dayanım artışları sırasıyla %9, %26 ve %38 olmuştur. Kürleme süresi dayanım üzerinde küçük bir etki göstermiştir, %14 MgCl₂ çözeltisi ile iyileştirilmiş numunelerde sadece %5 dayanım artışı görülmüştür.

Donma-çözülme döngüleri altında dayanıklılık testleri, -10°C ve -20°C'de kürlenmemiş ve 7 ile 28 gün süreyle kürlenmiş numuneler üzerinde gerçekleştirilmiştir. Bu testler, MgCl₂'nin kil üzerindeki antifriz ve stabilizasyon etkilerini değerlendirmiş ve soğuk koşullarda bütünlüğü korumak için optimal konsantrasyonları belirlemiştir. Dayanıklılık indeksi (DI), donma-çözülme döngüleri öncesi ve sonrası UCS oranlarını karşılaştırarak iyileştirme sonrası stabiliteyi ölçmek için kullanılmıştır. -10°C'de, %9 ve %14 MgCl₂ çözeltisi ile iyileştirilmiş zeminler, 7 donma-çözülme döngüsü boyunca dayanım ve DI değerlerini korumuş, %4 MgCl₂ çözeltisi ise daha az etkili olmuştur. İyileştirilmemiş numuneler, 7 döngü sonrasında DI'nin 0.41'e düşmesiyle dayanım kaybı yaşamış, %4 MgCl₂ çözeltisi ile iyileştirilmiş numuneler ise 0.59'a düşmüştür. -20°C'de, iyileştirilmemiş ve %4 MgCl₂ çözeltisi ile iyileştirilmiş zeminler önemli ölçüde dayanım kaybı yaşamış, %9 çözeltisi ile iyileştirilmiş numuneler daha küçük bir dayanım kaybı sergilemiş ve %14 çözeltisi ile iyileştirilmiş numuneler ise 7 döngü boyunca dayanımlarını korumuştur. -20°C'de DI değerleri, iyileştirilmemiş numunelerde 0.31'e, %4 ve %9 MgCl₂ çözeltisi ile iyileştirilmiş numunelerde ise sırasıyla 0.55 ve 0.70'e düşmüştür. Bu bulgular, soğuk iklimlerde zemin stabilitesi için MgCl₂ konsantrasyonlarının hassas ayarlanması gerektiğini vurgulamakta ve katkı maddesi konsantrasyonu, sıcaklık ve zemin dayanıklılığı arasındaki ilişkiyi ortaya koymaktadır.

Hacim değişimi testleri, -10°C ve -20°C'de gerçekleştirilmiştir. -10°C'de, iyileştirilmemiş ve %4 MgCl₂ çözeltisi ile iyileştirilmiş numuneler donma-çözülme döngüleri ile genişlemiştir. Ancak, %4 MgCl₂ çözeltisi ile iyileştirilmiş numuneler daha az genişlemiştir. %9 ve %14 MgCl₂ ile iyileştirilmiş numuneler ise hacim değişikliği göstermemiştir. -20°C'de, %9 MgCl₂ çözeltisi ile iyileştirilmiş numuneler genişleme göstermiş, ancak iyileştirilmemiş ve %4 MgCl₂ ile iyileştirilmiş numunelere kıyasla daha az genişleme göstermiştir. %14 MgCl₂ ile iyileştirilmiş numuneler ise hacimlerini korumuştur.

Mikroyapısal analizler, MgCl₂'nin zemin yapısında önemli değişiklikler meydana getirdiğini göstermiştir. XRD ve FTIR analizleri, iyileştirilmemiş ve %14 MgCl₂ çözeltisi ile iyileştirilmiş zemin numuneleri arasında difraksiyon desenlerinde kayda

değer bir fark olmadığını ve yeni kimyasal bağların tespit edilmediğini göstermiştir. Bu sonuçlar, $MgCl_2$ 'nin zemin üzerindeki işlevsel gruplar üzerinde minimal bir etkiye sahip olduğunu önermektedir. SEM analizleri, katkı maddesi eklenmesiyle zemin flokülasyonunun ve sonrasında partikül boyutu büyümesinin net kanıtlarını sağlamıştır. Bu mikroyapısal değişiklikler, zeminlerin gözlemlenen geoteknik özelliklerindeki değişimlerle uyumlu olmuştur.

Donma-çözülme koşulları altında $MgCl_2$ ve PEG'in karşılaştırmalı analizi, $MgCl_2$ 'nin zemin mukavemetini, dayanıklılığını ve hacim stabilitesini arttırmada daha iyi performans gösterdiğini ortaya çıkardı. $MgCl_2$ 'nin etkinliği, buz merceği oluşumunu önleyen ve yapısal bütünlüğü koruyan katyon değişimi ve antifriz özelliklerinden kaynaklanmaktadır. Buna karşılık PEG, su tutma oranını artırarak kuru birim hacim ağırlığı ve genel zemin mukavemetini azaltır. Ek olarak, yüksek $MgCl_2$ konsantrasyonları zeminin sıkışmasını iyileştirir, maksimum kuru yoğunluğu artırır ve zemin parçacıklarının topaklaşması ve toplanması nedeniyle optimum nem içeriğini azaltır.

Bu bulgular, $MgCl_2$ çözeltilerinin uygun konsantrasyonlarda kullanıldığında PEG'den daha etkili antifriz ve stabilizatör olduğunu, zorlu koşullar altında zemin stabilizasyonunda tercih edildiğini göstermektedir.

Ancak gelecekteki araştırmalar, $MgCl_2$ konsantrasyonlarını yerel iklim ve zemin özelliklerine göre ayarlamaya ve pratik uygulamalarını daha iyi anlamak ve gerçek dünyadaki performansını doğrulamak için geniş ölçekli saha testleri yapmaya odaklanmalıdır.

Son olarak, bu çalışma $MgCl_2$ 'nin soğuk iklimlerde zemin stabilizasyonu için etkili bir çözüm olduğunu, inşaat projelerinin ve zemin dolgularının dayanıklılığını ve güvenilirliğini artırma potansiyeli olduğunu vurguluyor ve $MgCl_2$ 'nin pratik uygulamalarını genişletmek için daha fazla araştırma öneriyor.

1. INTRODUCTION

The construction sector encounters considerable hurdles because of unfavorable weather conditions, which profoundly affect project schedules and expenses. Roughly 45% of construction ventures worldwide suffer interruptions due to weather-related incidents, resulting in billions of dollars in extra expenditures yearly. These obstacles are compounded by specific factors, like the necessity to suspend ground activities during subfreezing temperatures, thereby complicating project execution (Alshebani & Wedawatta, 2014; Sebt et al., 2007; Senouci & Mubarak, 2016).

A particular concern is the effect of freeze-thaw (F-T) cycles, especially pronounced in the construction of roads. This phenomenon presents a significant risk in colder climates, potentially leading to project delays or even suspension to avoid issues related to freezing and thawing. This highlights the persistent challenge faced by the industry due to adverse weather conditions, particularly in subfreezing environments. These disruptions not only result in project setbacks but also escalate expenses as construction teams contend with the complexities brought about by unfavorable weather, underscoring the significance of adopting effective measures to reduce weather-related risks in construction (Daniels et al., 2010; Effect of Weather on Highway Construction, 1978).

Freeze-thaw (F-T) cycles profoundly impact soil properties, which are vital for engineering purposes, particularly in geotechnical engineering and the construction of roads (Ma et al., 2019). These cycles pose threats to the stability (Zhang et al., 2022) and endurance of embankment soils, mainly in districts with mid and high altitudes, resulting in notable alterations in geotechnical characteristics (Sigrun H. Kværnø & Lillian Øygarden, 2006; Six et al., 2004). Research conducted over 60 years has clarified the effects of these cycles on soil structure, particularly in the United States and Canada, since the 1950s (Logsdail & Webber, 1959). They cause desiccation fractures in the soil, which disrupts its permeability and stability. They also significantly increase the amount of pore space in the soil, which makes drainage and water retention extremely difficult. Moreover, F-T cycles weaken soil strength and

cohesion, heightening its vulnerability to erosion and runoff (Cruse et al., 2001; Dalla Santa et al., 2019; Xu et al., 2018).

Additionally, freeze-thaw (F-T) cycles significantly contribute to soil embankment damage by causing alterations in the volume and density of vulnerable soil. Dense soils expand in volume, whereas loose soils shrink, heightening the risk to soil structures (Qi et al., 2008; Viklander, 1998b). This occurrence also speeds up the process of weathering, which can cause structural problems like uneven frost heave (Miao et al., 2020), thaw settling (Wang et al., 2015), compromised foundation integrity (Li et al., 2016), and slope destabilization (Gowthaman et al., 2020). Repeated freeze-thaw (F-T) cycles significantly vary material characteristics, such as elastic modulus, failure strength, shear strength, and stress-strain behavior. The harmful consequences of cyclic F-T processes were highlighted by research by Kamei et al., which showed a significant drop in both the strength and durability index of soft soils subjected to these cycles (Kamei et al., 2012). Similar to this, Lu et al. found that expansive soils' resilient modulus decreased noticeably as the frequency of F-T cycles increased, which in turn affected the soils' deformation properties and overall stability (Lu et al., 2019). Therefore, engineering design must carefully take freeze-thaw (F-T) cycles into account because of their complicated impacts. This is particularly true for fine-grained soils with low shear strength and bearing capacity, making them unsuitable for construction (Eigenbrod, 1996; Orakoglu & Liu, 2017).

To mitigate the detrimental freeze-thaw (F-T) cycles' effects on the mechanical properties of soil, traditional methods involve mixing soils with cement (Lu et al., 2020) or lime (Nguyen et al., 2019; Yıldız & Soğancı, 2012). Furthermore, researchers have extensively investigated non-traditional additives in recent years to minimize damage. These alternatives include utilizing fly ash (Dayioglu et al., 2017), alkali-activated volcanic ash and slag (Miraki et al., 2022), biological tactics such as microbial-induced biopolymers (Bonal et al., 2022), phase change material (PCM) (Chen et al., 2023), recycled ash-natural fibers mix (Tiwari et al., 2021), fibers-cement-soil mix (Tao et al., 2022), using polymers like PAM (Polyacrylamide) (Soltani-Jigheh et al., 2019), utilizing recycled materials (Kamei et al., 2012), or integrating special additives like rice husk ash (Li et al., 2024). Nevertheless, although these methods enhance the ultimate soil strength which subjected to F-T cycles compared to untreated soils, one disadvantage they all have in common is that they all lose strength after each cycle compared to their starting strength prior to F-T cycles.

This weakening indicates that persistent volume fluctuations during F-T cycles affect and change the embankment soil structure, resulting in a decrease in strength. Accompanied by volume changes, it poses considerable problems to the stability of infrastructure and soil embankments.

Given these limitations, another strategy to tackle the issues caused by freeze-thaw (F-T) cycles is incorporating antifreeze materials into the soil. By preventing soil moisture from freezing, this method helps avert the development of ice lenses and the following induced cracking, thus preserving the compacted soil's integrity. There has been relatively little research into the impact of various antifreeze compounds on soil. Among these substances is Monoethylene Glycol (MEG), a form of Ethylene Glycol with numerous applications. Ethylene Glycols, in general, are utilized across multiple industries; however, Monoethylene Glycol has garnered significant interest from the scientific community, particularly for its role as an antifreeze agent. This heightened focus on MEG is due to its unique properties and potential environmental implications when used in soil (Akram et al., 2020; Dye, 2001; Yue et al., 2012). However, a study has indicated that MEG may not be suitable as an antifreeze agent due to its detrimental effects on clays, resulting in a drop in compressive strength. The study's findings demonstrate that the strength and stiffness of soil polluted with MEG decrease gradually with increasing contamination levels (Estabragh et al., 2016). Subsequently, utilizing MEG in cold weather conditions appears impractical due to these negative effects.

Therefore, this study aims to propose a novel solution by identifying a new non-toxic additive that, unlike conventional stabilizers—which lose effectiveness after multiple F-T cycles—and antifreeze agents that degrade soil properties, can preserve clay's strength and volume after repeated F-T cycles. For this purpose, we initially considered polyethylene glycol (PEG 400), a non-toxic antifreeze agent recognized for its strong antifreeze properties, to assess its impact on clay. However, results from Atterberg limits, compaction, and unconfined compression tests indicated that PEG 400 was unsuitable, as it significantly increased the Atterberg limits and substantially reduced the soil's strength.

Following these findings, it was essential to find another material that can satisfy the goal of this study. After extensive research, magnesium chloride (MgCl_2), a type of salt, emerged as a potential solution due to its ability to significantly lower the freezing point when dissolved in water. A comprehensive literature review revealed that the

effects of MgCl_2 on soil's mechanical properties, especially its role as a stabilizer, have been explored in several studies, consistently showing that MgCl_2 significantly enhances soil's engineering properties (Bahloul et al., 2019; Muhammad et al., 2018; Tang et al., 2021; Turkoz et al., 2014). However, a notable gap remained in the literature regarding its antifreeze effects and impact on soils subjected to freeze-thaw (F-T) cycles, particularly under varying subzero temperature conditions.

To address this gap and evaluate the potential effectiveness of MgCl_2 , this research investigated its effects on the mechanical and physical properties of clay soils before, during, and after freeze-thaw (F-T) cycles at different subzero temperatures. A series of laboratory experiments were conducted on soil specimens treated with varying MgCl_2 concentrations. The tests included Atterberg limits, standard Proctor compaction, unconfined compressive strength (UCS), and freeze-thaw (F-T) cycles at -10°C and -20°C with MgCl_2 concentrations of 4%, 9%, and 14%, where samples were subjected to 0, 1, 3, 5, and 7 F-T cycles to evaluate changes over time. Additionally, we incorporated advanced microstructural analysis techniques to gain a deeper understanding of how clay and MgCl_2 solution interact. Also, the microstructure of the soil was visualized using scanning electron microscopy (SEM), mineralogical changes were identified with the aid of X-ray diffraction (XRD), and the presence of chemical bonds and functional groups in the treated soil was revealed with Fourier-transform infrared spectroscopy (FTIR). This comprehensive approach allowed us to assess the ability of MgCl_2 to mitigate F-T cycle effects without compromising soil strength and evaluate the significance of dosage under varying freezing conditions. The results demonstrated that MgCl_2 is effective in mitigating the deteriorative effects of F-T cycles on clay soils. Specifically, a 9% MgCl_2 solution proved promising in preventing strength loss and maintaining the volume at -10°C , while a 14% solution was effective at -20°C . These findings indicate that the optimal dosage of MgCl_2 is dependent on the severity of the freezing conditions, highlighting the necessity to adjust treatment concentrations based on environmental temperatures. This not only confirms the potential of MgCl_2 as an antifreeze additive but also underscores its practical applicability in varying cold climates.

In conclusion, this research represents a pioneering effort to introduce MgCl_2 as an antifreeze agent for soil stabilization under different temperature regimes. By demonstrating the ability of MgCl_2 to prevent freezing and maintain soil strength during F-T cycles, this study opens avenues for future research on antifreeze materials

in geotechnical engineering. Future studies could focus on testing MgCl_2 on different types of soils, under various environmental conditions, or in combination with other stabilizing agents. Ultimately, this work aspires to contribute to the development of more resilient infrastructure in cold regions by proposing an innovative approach to soil treatment that both mitigates F-T effects and enhances soil stability.

The subsequent sections of this thesis will provide a comprehensive presentation and detailed discussion of the test methods employed and the results gained, mainly focusing on the effects of PEG 400 and magnesium chloride (MgCl_2) on soil properties before and after freeze-thaw cycles. Each test method will be thoroughly explained, and the results will be analyzed and interpreted to draw meaningful conclusions regarding the suitability of these additives.





2. LITERATURE REVIEW

This chapter reviews key research on frost heave and freeze-thaw effects, focusing on their impact on soil mechanics and infrastructure. It covers historical developments, theoretical advances, and experimental findings, providing a foundation for understanding frost heave mechanisms and strategies to mitigate cold climate challenges in infrastructure design.

2.1 Background of Frost Heave Research

Understanding the phenomenon of frost heave requires not only a grasp of its underlying mechanics but also an appreciation of the historical context and the pioneering work of scientists who laid the groundwork for current knowledge. The exploration of frost heave dates back to early studies that observed the effects of freezing on soil and infrastructure. Over the decades, numerous researchers have contributed to unraveling the complexities of this process. With this historical foundation, we can delve into a detailed explanation of frost heave, exploring the mechanisms that drive this phenomenon and its implications for engineering and construction.

Since the late 1600s, scientists have been aware of the phenomenon known as frost heave, in which soil expands and contracts due to freezing and thawing. Urban Hjärne first documented this process in 1694. By the mid-1700s, the connection between frost heave and ground freezing was more precise, with E.O. Runeberg noting the presence of ice granules within frozen soil (Runeberg, 1765). However, soil weakening during thawing, a significant contributor to structural damage, was not yet understood.

Simon Johnson found in 1914 that during the freezing process, water migrates to freezing layers, causing an increase in the volume of soil water (Johansson, 1914). The theory that frost heave was caused by the expansion of freezing water was refuted by Stephen Taber in 1929. His experiments demonstrated ice lens growth in soil would continue as long as liquid water was available at the base of the soil column (Taber, 1929). Concurrently, G.J. Bouyoucos made significant theoretical contributions,

discovering that water in the soil does not freeze uniformly at a single temperature (Bouyoucos, 1920).

In 1925, Sweden led the way in comprehensively addressing frost heave, hosting a significant conference in Luleå. This event spurred collaborative investigations, resulting in numerous publications from 1928 to 1933. Taber's experiments in 1929 highlighted both vapor and capillary transport of water to the freezing front (Taber, 1929).

Casagrande found a correlation between frost heave and the proportion of fine soil particles in 1931. He did not, however, intend for these findings to be taken as a rigid definition of frost susceptibility (Casagrande, 1931). In 1935, R.K. Schofield's pF scale linked soil water's free energy to freezing-point depression, but this approach was later considered superficial compared to thermodynamic methods (Schofield, 1935).

During the 1950s, the U.S. Arctic Construction and Facilities Engineering Laboratories conducted systematic tests on frost heave. Despite using equipment similar to Taber's, they struggled to improve upon Casagrande's findings. Theories on frost heave processes emerged in the late 1950s, with K. Jackson and B. Chalmers proposing an irreversible process involving supercooled water (Jackson & Chalmers, 1958) and L. Gold suggesting a model based on the ice–water interface's surface tension (Gold, 1957).

In the 1960s, R.D. Miller and his students made significant strides in advancing the theoretical foundation of frost heave, validating the Clapeyron equation through an osmotic model of unfrozen water films. Building on this work, segregation potential was introduced by Konrad and Morgenstern, who combined the concepts of thermodynamic equilibrium at the base of the ice lens with unfrozen pore water suction (Konrad & Morgenstern, 1980, 1981).

The research conducted by Péwé and Paige on the Alaska Railroad in 1959 documented significant frost jacking issues, prompting the implementation of corrective measures and offering valuable early insights how frost heave affects infrastructure (Péwé & Paige, 1959). Differential frost heaving, frost jacking, and thaw weakening were the subjects of further research in the 1970s and 1980s, emphasizing choosing suitable soil gradations to reduce the effects of frost action. In 1983, Penner and Crawford established a connection between climate conditions, seasonal frost

penetration depths, and soil frost susceptibility (Penner & Crawford, 1983). Esch et al. (1981) identified key soil parameters crucial for pavement performance based on their research findings (Esch et al., 1981).

Additionally, several studies have focused on preventing frost heave in soil embankments. For instance, Esch pioneered insulation techniques to control frost heave in the 1960s. These techniques evolved into successful applications using extruded insulation, implemented by the Alaska Department of Transportation. However, differential icing issues led to some challenges, with later research showing that placing insulation approximately 3 feet beneath the pavement surface could effectively control frost action while minimizing differential heaving (Esch, 1969).

2.2 Freeze-Thaw Damage

Researchers have extensively studied two significant problems associated with soil freeze-thaw cycles: frost heaving and thaw weakening. These phenomena, collectively known as frost action, arise from the annual freeze-thaw cycles and can result in significant pavement damage, increased maintenance costs, and degraded ride quality (Christopher et al., 2006; Linell, 1953). The effects of frost action on roads are visually manifested as pavement heave, surface cracking, frost boils, noticeable weakening, deflection, and water pumping in cracks.

Frost heaving occurs when soil water freezes and expands, drawing water from unfrozen layers upwards due to a water potential gradient (Mitchell & Soga, 2005; Penner, 1970). This process can damage road surfaces, reducing their functional performance (Simonsen & Isacsson, 1999). Thaw weakening happens when the frozen subgrade melts, but the meltwater is trapped by still-frozen layers, saturating the soil and reducing its strength. The period of highest risk for the road spans from a few days to three weeks after the onset of thawing (Linell, 1953). Thaw weakening is influenced by several factors, including soil type and conditions, frost penetration properties, temperature variations during the freeze-thaw cycle, traffic load type and volume, water availability, and drainage conditions. As the depth of frost penetration increases, the critical weakening period extends. Additionally, snow-covered shoulders can restrict drainage, further complicating the thawing process by impeding water flow, similar to the impact of frozen sublayers.

Uneven soil heaving below asphalt pavements can cause periodic bumps or waves, as illustrated in Figure 2.1. In concrete pavements, frost heave can cause cracks in concrete slabs or lift one slab relatively higher than another. The bearing capacity of pavements experiences seasonal variations: it increases during freezing and decreases significantly during spring thaw. Typical damage due to thaw weakening includes pothole formation in asphalt pavements and uplift in concrete pavements. In the first situation, the asphalt pavement breaks up because it cannot support the weight of passing vehicles after the melted water softens the sub-base material. In the second case, the "liquidy soil" underneath the pavement is ejected through joints or cracks as the concrete slab moves downward under heavy traffic loads. Consequently, load restrictions are imposed to prevent structural damage to pavements.



Figure 2.1 : Visual Signs of Pavement Damage Caused by Freeze-Thaw Cycles: (1-a) frost heave in Norway (Ystenes, 2011), (1-b) thaw weakening on an unpaved road (Shoop et al., 2008), (1-c) alligator cracking ("Frost Action,"), and (1-d) frost boils on an asphalt road ("Frost boils – the reason for emergency road restrictions | Whatcom News," 2017).

About a third of the U.S. is located within cold regions. It has been estimated by The Road Information Program (TRIP) that rebuilding the roads damaged in the U.S. due to frost action every winter would require more than \$2 billion (DiMillio, 1999), not counting the cost of maintenance. The true cost of the damage is higher, and it includes the higher cost of transportation because of closure and detouring.

Actually, frost action causes damage to more than just pavements. Structures with foundations placed above the frost line could fail when the soils undergo seasonal freezing and thawing. Retaining walls, culverts, storage tanks of liquid gases, chilled buried gas pipelines, and hot-oil pipelines in permafrost are all vulnerable to frost damage if the surrounding soils are frost susceptible.

Understanding mechanisms of thaw weakening and frost-heave is crucial for mitigating their impact on infrastructure. These freeze-thaw processes not only affect pavements but also various structures, leading to significant economic costs and functional disruptions.

2.3 Freeze-Thaw Weathering and the Phenomenon of Frost Heaving

In cold regions, especially during the late fall and winter months, soil embankments and landfills are frequently subjected to freeze-thaw weathering. This cyclical process of freezing and thawing can lead to the mechanical breakdown of soil and rock, weakening their structure. Moreover, the formation of ice lenses during freeze-thaw weathering contributes to frost heave, causing the upward displacement of soil. Indeed, frost heave is a type of frost action, which is a physical weathering process that involves the repeated freezing and thawing of water within soil or rock. Consequently, gaining insight into the mechanisms underlying frost heave and soil weakening due to thawing is essential for developing effective engineering solutions to address freeze-thaw challenges. This understanding becomes particularly crucial when designing and constructing infrastructure on soils that are susceptible to frost, especially in cold climates. Indeed, when soil expands or deforms due to the freezing of water within its pores, this phenomenon is known as frost heave. This phenomenon creates upward pressure, causing the soil to increase in volume. In the field, this pressure is released through the upward movement of the ground surface, which is why the term "frost heaving" is used. As noted by Kaplar and Mitchell (1970), frost heave takes place when three particular conditions are present. These conditions include having a source

of water, soils that freeze easily and sustained freezing temperatures. The variability of these conditions across different areas often results in uneven heaving instead of heaving uniformly. This irregular heaving can result in damage to foundations, utilities, pavements, and various other structures (Kaplar, 1970; Mitchell, 1993).

When temperatures rise during certain seasons, the frozen ground begins to thaw from the surface as the air warms up. In the initial stages of thawing, the melted water from the top layers of soil often gets trapped by the layers below, which are still frozen solid. This entrapment can lead to excessive saturation of the soil, significantly reducing its ability to bear weight, a phenomenon known as thaw weakening (Doré & Zubeck, 2009; Guthrie & Zhan, 2002; Janoo & Berg, 1990). In such situations, soil might become inadequate for supporting structures, potentially leading to infrastructural damage.

The extent of soil displacement due to frost heave and the subsequent weakening during the thawing process is contingent upon the amount and spatial distribution of ice crystals formed within the soil matrix (Stephen, 1930). Soil displacement caused by frost heaving is primarily attributed to two distinct mechanisms: the freezing of water that moves towards the freezing-front due to thermodynamic processes and the solidification of water already present within the soil pores (Penner, 1986). The formation of ice crystals in soil occurs through two primary mechanisms: in-situ freezing and segregational freezing.

The process of in-situ freezing involves the direct solidification of water molecules present within the pore spaces between soil particles. This phenomenon is more prevalent during instances of rapid soil freezing (Research & Konrad, 1984). The initial formation of ice crystals takes place at the core of the pore space, subsequently expanding outward until the entire pore is filled. Despite the fact that water experiences an approximate 9% volumetric expansion upon freezing, the resulting upward displacement of soil (frost heave) caused by in-situ freezing in unsaturated soils is generally less than 3% of the depth of the frozen zone (McCarthy, 1988). Accordingly, the heaving pressure exerted by in-situ freezing is relatively minor in magnitude (Perfect et al., 1988).

On the other hand, the segregational freezing process can potentially result in substantial vertical displacement of soil and generate significant heaving pressures. The magnitude of these heaving pressures is influenced by the counteracting

overburden pressures exerted by the overlying soil layers and surface loads (Fowler, 1989). This process is particularly concerning for the integrity of infrastructure. Segregational freezing occurs when water migrates from unfrozen areas into the freezing zone in frost-susceptible soils that freeze at a slower rate. When water is available, it is typically drawn from deeper soil layers toward the freezing front, forming ice lenses at or just above this front (see Figure 2.2). Ice lenses develop through the segregational freezing process when the rate of heat removal from the soil matches the rate of heat supply to the freezing front. As the freezing front progressively advances deeper into the soil profile, over time, several ice lenses can form. The total thickness of these created ice lenses directly determines the amount of vertical displacement measured at the ground's surface.

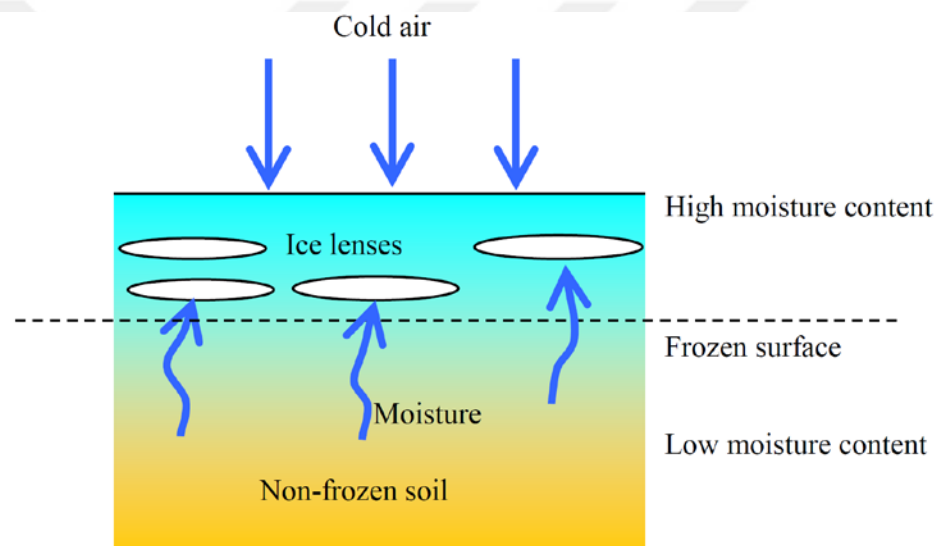


Figure 2.2 : Schematic of frost heave, (Sato et al., 2019).

Water migration during freezing is driven by two key phenomena. The first phenomenon involves the movement of water due to temperature differences, where water flows from warmer areas to colder areas within the soil. The second phenomenon is the generation of suction during the freezing process, which further draws water towards the freezing front. These combined effects contribute to the overall migration of water in the soil during freezing conditions.

2.3.1 Mechanism of frost heave

When soil freezes, the heat flow and water movement play a critical role in forming ice lenses. Water in the soil begins to freeze as the temperature decreases, releasing latent heat. This heat creates a thermal gradient that influences water migration within

the soil. Also, water moves towards the freezing front due to capillary action and soil suction. This accumulation of water at the freezing front leads to the formation of ice lenses. These ice lenses cause the soil to heave, a process known as frost heave, which can impact construction and infrastructure stability. Therefore, understanding the dynamics of the freezing front and ice lens formation is crucial for predicting and mitigating these effects.

2.3.1.1 Dynamics of the freezing front and ice lens formation

Figure 2.3 provides a schematic representation of the ice segregation process. At a given time t , the frost line has descended to a depth d beneath the surface of the soil sample. By this stage, several ice lenses have already developed. Directly below the lowest of these lenses, known as the "active" ice lens, lies a zone of soil referred to as the frozen fringe.

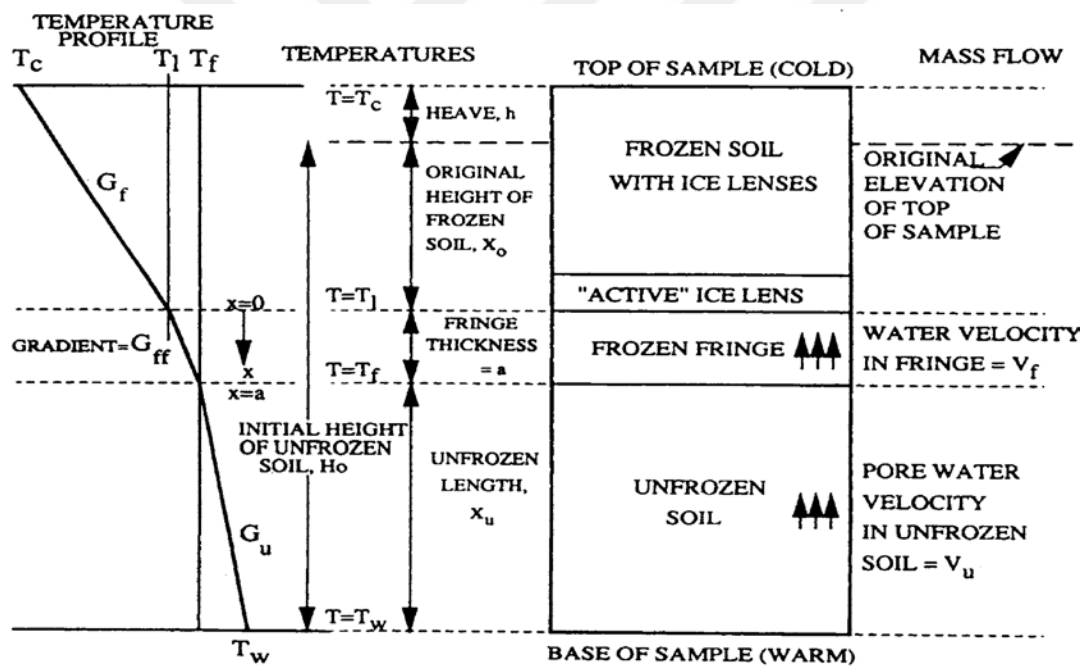


Figure 2.3 : Frost heave in a hypothetical one-dimensional soil column, (Andersland & Ladanyi, 2004; Nixon, 1991).

The segregation freezing temperature, which is the temperature at the base of the active ice lens (T_l), is slightly below the in-situ temperature (T_f) at which ice can form in soil pores (Svec, 1989). The frozen fringe, the partly frozen region between T_l and T_f , has reduced hydraulic conductivity due to the presence of pore ice. This fringe exists because pore fluid does not freeze at a single temperature; freezing occurs over a range

due to the salinity of the pore fluid and the surface activity of clay particles (Konrad, 1989b, 1994).

Water from the underlying soil is drawn through the frozen fringe by suction at the base of the active ice lens. As long as there is a connection between suction and the frozen fringe's (K_{FF}) hydraulic conductivity that permits water flow, this migration will continue. During active ice lens growth, the segregation freezing temperature and ice lens location remain constant. In contrast, the freezing front (T_f) moves, increasing the width of the frozen fringe and decreasing its hydraulic conductivity. When the work required to transport water through the frozen fringe exceeds the latent heat-derived work, the fringe moves deeper, and a new ice lens forms below the old one. In other words, the freezing front moves deeper into the soil because as the frozen fringe widens, its hydraulic conductivity decreases, making it increasingly difficult for water to move through it. This increased resistance means that the energy required to transport water through the frozen fringe becomes more significant over time. Initially, the latent heat released during the freezing process helps keep the water moving through the fringe. This heat, which is created during the liquid-solid phase transition, helps transport water through the frozen fringe by keeping it moving through the increasingly blocked pores. However, as the resistance continues to rise, there comes a point when the latent heat energy is no longer sufficient to overcome this resistance. So, the system needs to find a new path to continue the freezing process. Therefore, the freezing front shifts deeper into the soil where conditions are more favorable, such as higher hydraulic conductivity and less resistance. This allows the creation of a new ice lens in these deeper layers, ensuring the freezing process can continue effectively.

This process of "rhythmic" ice lensing will continue until a thermal steady-state state is touched and the last ice lens is formed (Konrad & Morgenstern, 1980; Svec, 1989).

The formation of ice lenses and the resultant extent of frost heaving are influenced by multiple factors. External factors encompass the rate of frost penetration, overburden pressure, and water availability (Konrad, 1988; Othman & Benson, 1992; Penner, 1986). Additionally, soil-specific characteristics such as unfrozen hydraulic conductivity, mineralogical composition, and pore size distribution significantly impact the frost-heaving process (Konrad, 1994). These factors will be explained in detail in the following sections. When the rate of frost penetration is high, the water in the soil pores freezes rapidly. This rapid freezing creates a small hydraulic

conductivity of frozen fringe (K_{FF}), which restricts the movement of water needed for ice lens formation. In other words, a quick freeze prevents the soil moisture from moving. On the other hand, when the frost penetration rate is low, it allows more time for soil moisture to move, facilitating the growth of ice lenses (Chamberlain & Gow, 1979; Konrad & McCammon, 1990).

For an ice lens to form, the ice lens pressure must exceed the soil's tensile strength and any overburden pressure. High overburden pressures prevent discrete ice lens formation, resulting in diffuse ice and soil bands (Penner & Goodrich, 1982). For an ice lens to form, some of the energy released during freezing must be used to lift the soil. This means the pressure within the ice needs to be slightly greater than the overburden pressure. If the overburden pressure exceeds the ice pressure, additional energy must be released by freezing unfrozen water. Lower freezing temperatures release more energy to counteract the overburden pressure (Nixon, 1991).

Significant frost heaving requires access to an external water source. When water is continually available during frost heaving, it is termed an open system. Conversely, a closed system has no access to external water. Figure 2.4 illustrates the difference between these two systems.

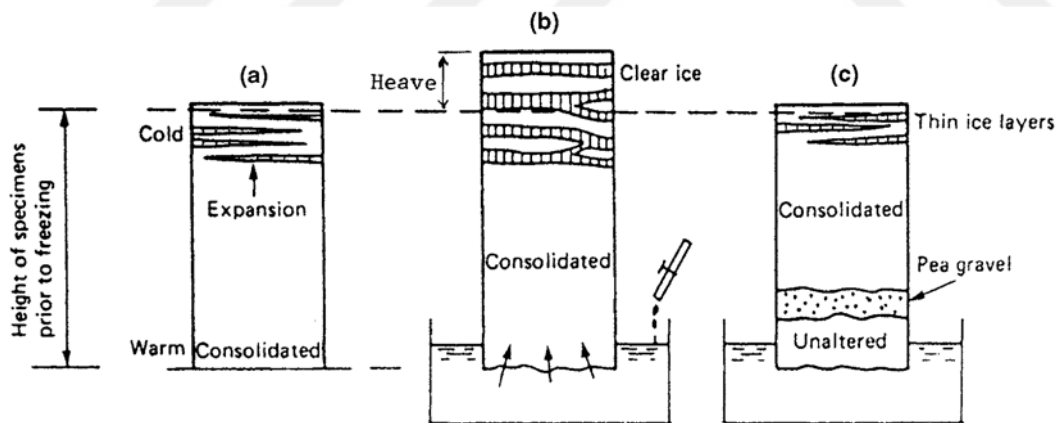


Figure 2.4 : Ice formation in soils: (a) closed system; (b) open system; (c) pea gravel layer changes upper part of specimen into a closed system, (Andersland & Ladanyi, 2004).

An open system is typically defined as a soil system with a continuous supply of water from below throughout the freezing process. This type of system can exchange water, heat, and energy with its surroundings (Nixon, 1991). In contrast, a closed system lacks access to more free water. While it can exchange heat and energy, it cannot exchange

water. As a result, if ice lenses form in a closed system, they will generally be much smaller.

2.3.1.2 Water migration during ice lensing

Heat flow-driven water migration

A temperature gradient forms within the soil layers when heat is transferred from the warmer soil to the colder air above by air that is cooler than the ground. This gradient causes water to migrate from the warmer to the cooler regions due to thermodynamic differences (Mitchell, 1976; Takagi, 1963). This movement of water can occur even in the absence of ice. The freezing front moves further into the soil as a result of the soil losing more heat and the water inside its pores starting to freeze. At the air-soil interface, a notable temperature gradient frequently arises because air temperatures can be significantly lower than ground temperatures. Penner points out that the rate of heat transfer across this contact is precisely proportional to the magnitude of the temperature differential. As a result, sharp changes in temperature can cause the freezing boundary to quickly move through the upper layers of soil. This leads to the in situ freezing of water within the pores between soil particles, causing a small amount of upward expansion of the soil, known as frost heave (Penner, 1960, 1972).

When the advancement of the freezing boundary slows down and reaches an equilibrium state, in which the heat extracted from the soil near the freezing front is equal to the heat provided by the soil beneath it, the process of segregational freezing can begin. As long as the overburden pressure stays below the frost-heave pressure, this process results in the production of ice lenses that grow perpendicular to the direction of frost penetration (Fredlund et al., 1991). Conduction and convection are the two ways that heat moves through soil to the freezing front. Thermal conductivity determines the efficiency of heat conduction through the soil matrix, while hydraulic conductivity influences heat convection, which involves the movement of heat through the transport of water from the underlying soil to the freezing zone. This dual process ensures that both solid-state heat transfer and fluid movement contribute to the overall thermal dynamics at the freezing front. Additionally, the incoming water changes from liquid to solid at the freezing front, releasing its latent heat of fusion, which is another critical heat source.

Provided that the equilibrium conditions persist, an ice lens can continue to increase in thickness. However, changes in air or soil conditions can disrupt this balance, leading to irregular formation of ice lenses (Fredlund et al., 1991; Penner, 1962). For instance, if the air temperature drops further or there is less water available just below the freezing point, it can interrupt the heat flow and halt the formation of ice lenses. This cycle continues until conditions once again favor ice lens formation. As this process continues over time, it results in the formation of multiple ice lenses interspersed with layers of frozen soil in situ. The ice lenses tend to become thicker and more widely spaced as depth increases (Harris, 1995; Penner, 1962).

Suction-driven water migration in frozen fringe and ice lens formation

In a system of freezing soil, water can come from multiple sources. The distance of these water sources to the freezing front is critical in determining the extent and speed of frost heaving. Frost heaving happens when soil pores fill with ice or supercooled water, leading to soil expansion (Hermansson & Guthrie, 2005; Miller, 1972). Closer water sources lead to higher soil saturation, increasing the frost-heaving rate due to the abundance of freezable water (McGaw, 1972). In open systems, continuous ice lens growth requires an external water source, although uniform soil saturation is not necessary, as water can migrate to the freezing front (Dirksen & Miller, 1966). Frost heave can be produced in closed systems by redistributing water upward to the freezing front without the requirement for an external water supply (Guthrie & Hermansson, 2003). The movement of water through soil, driven by the rate of heat removal, ultimately controls ice lens growth in freezing soil, illustrating the complex interactions between water availability, soil saturation, and temperature in frost heaving.

When ice develops among the particles of soil, it causes the capillaries' effective diameter within the network of unfrozen water to shrink, which results in a decreased amount of liquid water. This process induces a phenomenon known as cryosuction, which is characterized by a negative pressure on the pore water due to freezing. This pressure, in turn, pulls both the liquid water and water vapor upward from the soil beneath it toward the advancing layer of ice (Beskow, 1947; Black & Hardenberg, 1991; Guthrie et al., 2006). Osmotic suction and matric suction are the two main parts of cryosuction (Fredlund et al., 1991).

Osmotic suction arises because of the solutes or ions present in the pore water, whose concentrations rise during the formation of ice. At first, ice crystals emerge within the tiny spaces between soil particles. As these crystals expand, they force the dissolved ions into the adjacent areas of water that have not frozen (Hanks & Ashcroft, 1980). This movement of ions occurs because ice excludes impurities, including ions, leading to higher ion concentrations in the remaining unfrozen water (Cary & Mayland, 1972; Guthrie & Zhan, 2002; Panday & Corapcioglu, 1991). This increased concentration of ions decreases the freezing point of the remaining liquid water, leading to the presence of supercooled liquid water within the soil that is already frozen. Located in the frozen fringe—the area that sits between the advancing edge of the freeze and the base of the closest ice lens— (Konrad & Morgenstern, 1982)— unfrozen water layers remain, enabling the flow of water and aiding in the upward movement of soil during freezing (frost heaving). Variations in the concentration of ions generate gradients of osmotic pressure, which drive water from places with fewer ions to areas with more ions through these thin layers of unfrozen water, facilitating a steady provision of water to the advancing freeze line (Guthrie et al., 2002).

Matric suction arises from air-water menisci in the soil, influenced by surface tension. These menisci are curved surfaces formed where water meets air within soil pores due to water's surface tension (Weast et al. 1989). Matric suction is increased by smaller meniscus radii, lower contact angles, and higher surface tension (Guthrie et al., 2002). This force draws water into small spaces between soil particles, enhanced by higher ion concentrations, which increase surface tension. Smaller particles create smaller menisci, intensifying this suction force.

Materials with coarse grains, such as gravels, feature large particles and menisci radii, leading to reduced matric suction and increased vulnerability to frost (Freitag & McFadden, 1997). Clayey soils, despite high potential matric suction due to small particles and high capillary rise, are less frost-susceptible due to low permeability, preventing sustained ice lens growth (Freitag & McFadden, 1997). Silts are very vulnerable to frost heave because they have the proper particle-size distribution and adequate permeability for water transfer, which makes them ideal for matric suction (Freitag & McFadden, 1997; Lay, 2005). This vulnerability arises from their ability to generate strong suction while allowing water movement, facilitating ice lens formation and frost heave.

Water migrates from regions of low total suction to areas with high total suction, whether in liquid or vapor state (Jabro, 2009). Osmotic and matric suction are combined in total suction, and the water flow rate through the soil is determined by the suction gradient as well as the water or vapor conductivity of the soil, which is impacted by the saturation level and structure of the soil. Liquid and vapor flow rates are increased by higher porosity and decreased tortuosity (fewer twists in routes) (Hanks and Ashcroft, 1980). Increased soil saturation increases the flow of liquid water but reduces the flow of water vapor.

In freezing soil, as temperatures decline, more pore water transitions into ice, diminishing the amount of unfrozen water between soil particles and substantially elevating matric suction. Nevertheless, as freezing advances, the soil's hydraulic conductivity experiences a rapid decrease (Hanks & Ashcroft, 1980). Liquid water migrates from warmer regions of the soil to colder areas, especially toward an active ice lens. As it moves, the paths through which the water flows become increasingly convoluted and narrow. This cycle repeats until the water flow ultimately stops at the base of the latest ice lens that has formed (as shown in Figure 2.5).

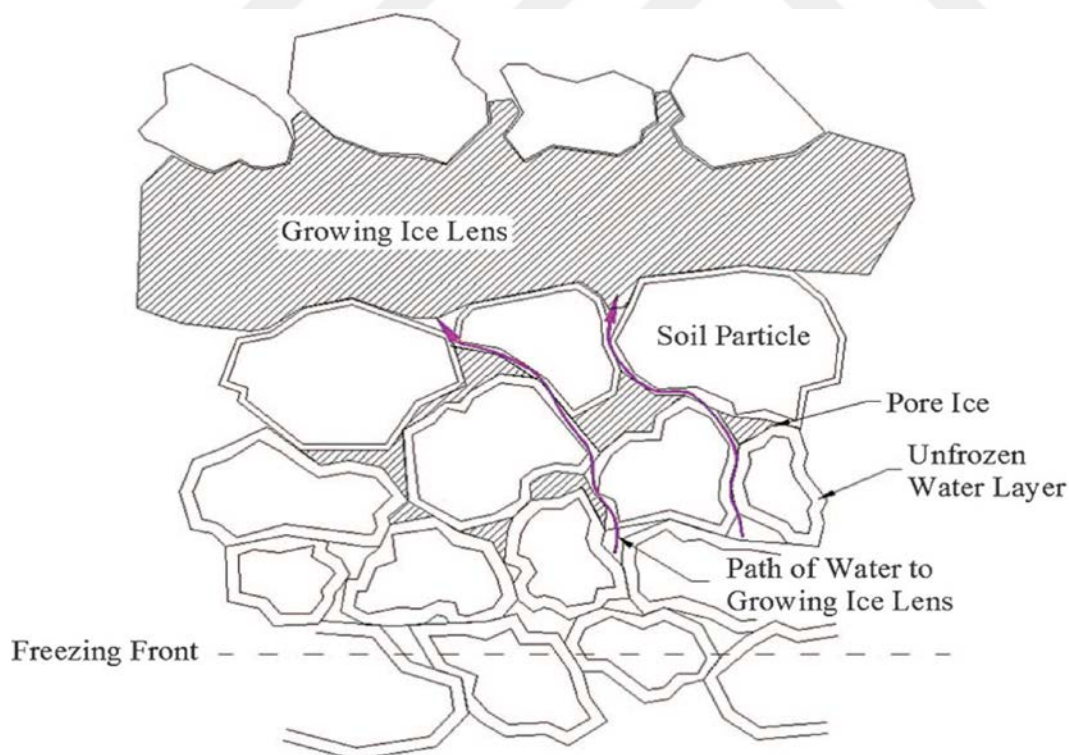


Figure 2.5 : Diagram showing the development of an ice lens, (Lay, 2005).

As the soil thaws and the ice within it melts, the liquid water content increases. This rise can lead to supersaturation, especially in frost-prone soils like silts. The liquefying

ice at the frozen soil's base can infiltrate into the layers underneath, with the rate of drainage largely dependent on the soil's hydraulic conductivity. Alternatively, the meltwater from the upper portion of the frozen soil can become confined over the still-frozen, almost impervious layer below. Because of the positive pore water pressures created by the trapped water, the stiffness and strength of the soil are weakened by the reduction of interparticle friction (Doré & Zubeck, 2009; Guthrie & Zhan, 2002; Janoo & Berg, 1990).

Thaw-induced weakening, regarded as the greatest crucial frost-related degradation system, substantially impacts soil stability and structural soundness (Berg, 1988; Janoo & Berg, 1990; White & Coree, 1990). The process takes place as a result of trapped meltwater increasing pore water pressure, which reduces the soil's load-bearing capacity. As a result, the soil becomes more prone to deformation and damage when subjected to loads. A more detailed exploration of thaw weakening will be provided in section 2.5.

2.3.2 Frost susceptibility

In terms of frost susceptibility, among gravel, silt, and clay, silt exhibits the highest vulnerability due to its small particle size, which renders it prone to being rejected by gradually developing ice crystals. Unlike gravel, silt's hydraulic conductivity is sufficiently high to maintain a constant supply of water. Conversely, gravel has a very low hydraulic conductivity at the low pore water pressure typical of the freezing front, and its larger particle size prevents displacement by ice crystals. Likewise, clay also has low hydraulic conductivity, leading to restricted water flow and a potential imbalance between heat removal and water influx. Consequently, the freezing front may rapidly penetrate the freezing clay.

2.3.2.1 Types of classification

The concept of frost susceptibility serves as a method of classifying soils based on their propensity to experience segregated freezing, which involves the formation of ice lenses. Various transportation agencies around the world have adopted different criteria for this classification. Generally, three levels of classification have been established (Chamberlain, 1984). Table 2.1 illustrates these categories and the corresponding data requirements. Presently, Ontario utilizes the frost susceptibility criteria depicted in Table 2.2 specifically for subgrade soils. According to this

criterion, non-frost-susceptible subbase and base materials must contain no more than 10% fines smaller than 0.075 mm and exhibit a plasticity index of zero, aligning with the Type II classification system.

Table 2.1 : Categories of frost susceptibility and necessary information, (Chamberlain, 1984).

Test level	Type of Information Required
Type I	Specified particle size, uniformity
Type II	Soil type of classification, Atterberg limits, capillarity, permeability, mineralogy, uniformity coefficient, sand equivalent, moisture conditions, density, saturated CBR, etc.
Type III	Frost heave, heave rate, thaw CBR, laboratory freezing test data, etc.

Table 2.2 : Ontario frost susceptibility criteria for subgrade soils, (Chamberlain, 1984).

Percent of Particles Between 0.005 and 0.075mm	Frost Susceptibility
<40	Low
40-55	Borderline
55-100	High

2.3.2.2 Standards for classification in the CRREL II test

The CRREL II (Cold Regions Research and Engineering Laboratory) test assesses the frost susceptibility of soils using two main criteria: frost-heave followed by thaw-weakening.

Frost-Heave Criterion: This involves measuring the frost heave rates during the first and second freeze-thaw cycles within the initial eight hours. The chosen heave rate—whether from the first or second freezing cycle—depends on the site's freezing conditions. For sites with frequent freeze-thaw cycles and a water table close to the freezing and thawing zone, the heave rate from the second freeze is selected. In contrast, areas with severe winter climates and continuous frost penetration rely on the heave rate from the first freeze (Chamberlain, 1987).

Thaw Weakening Criterion: This is determined by the thawed CBR (California Bearing Ratio) value, which differs from the standard CBR test used in non-frost conditions. Tester and Gaskin (1996) referred to this as the "thawed bearing value" to distinguish it from typical CBR tests (Tester & Gaskin, 1996). The susceptibility of soils to frost-related impacts can be assessed by comparing the eight-hour heave rate

and the thawed California Bearing Ratio (CBR) value, as outlined in Table 2.3. The classification criteria may vary depending on the soil type, as some materials may exhibit substantial thaw weakening despite relatively low heave rates. In such scenarios, the classification is based on the thaw weakening susceptibility, as determined by the CRREL II test, taking a cautious approach.

Importantly, the CRREL II test uses the actual thawed CBR value rather than the percentage reduction in CBR for the thaw weakening criterion. Thus, soils with inherently weak bearing strength are classified as highly frost-susceptible, regardless of whether the weakness is due to frost or other factors. This necessitates careful application of the frost susceptibility criterion in this study.

Table 2.3 : Frost susceptibility criteria for the CRREL II test, (Chamberlain, 1987).

Frost Susceptibility Classification	Frost Heave Criteria 8-hour heave rate (mm/day)	Thaw Weakening Criteria Thaw CBR
Negligible	<1	>20
Very Low	1-2	20-15
Low	2-4	15-10
Medium	4-8	10-5
High	8-16	5-2
Very High	>16	<2

2.4 Thawing Process

The frozen ground contains ice in various configurations, including coatings on soil particles and isolated inclusions, and is mixed within the soil matrix. When thawing occurs, this ice transitions into the water, necessitating adjustments in the soil's structural equilibrium under the prevailing overburden pressure (Andersland & Ladanyi, 2014). If the ground heaves, the excess water must be drained, with the drainage rate being influenced by thermal factors and described by Terzaghi's consolidation theory (Harris, 1995).

The thawing process can lead to increased moisture in the supporting layers, thereby weakening the pavement (Janoo & Berg, 1998). Throughout spring, ice melting can saturate these structures, significantly diminishing their load-bearing capacity (Simonsen & Isacsson, 1999). Milder climates, such as those in Scotland, sometimes experience this phenomenon, which is linked to road deterioration caused by frequent freeze-thaw cycles (Aho & Saarenketo, 2006).

Research by Ballantyne and Harris suggests that the geotechnical characteristics of soil during thawing are influenced by its textural composition and clay content. Soils with coarse-grained textures, such as those containing sand and silt, have low P.L. and L.L., resulting in low plasticity. These coarse-grained soils primarily derive their strength from intergranular friction, with minimal contribution from cohesion. Conversely, clay-based soils exhibit higher plastic and liquid limit values, leading to increased plasticity, reduced intergranular friction, and substantial cohesive properties. In contrast, soils comprising sand and silt possess lower void ratios and reduced saturation moisture contents compared to clay-dominant counterparts. Consequently, these coarse-grained soils require less water to reach saturation levels. Furthermore, due to their low P.L. and L.L. characteristics, sandy and silty soils are highly sensitive to fluctuations in moisture content and may transition into a flow state if the moisture level surpasses the liquid limit. On the other hand, because of the strong pore-water pressures brought on by thaw consolidation, clay soils frequently fail over distinct slip surfaces (Ballantyne & Harris, 1994).

2.5 Thaw Weakening

The thawing process in frost-susceptible soils can lead to a significant reduction in the bearing capacity of pavement structures. As the frozen soil thaws in the spring, ice lenses within the soil begin to melt. This can result in the accumulation of meltwater above the still-frozen underlying soil, causing supersaturation conditions. If the rate of ice melting exceeds the rate of water drainage, the soil can become severely weakened, decreasing the interparticle friction and effective strength. This condition is further intensified by traffic loads (Mactutis et al., 1998; Seppälä, 1999).

As a result, the soil consolidates under its own weight and external loads. Field tests conducted by the US Army Corps of Engineers (USACE) indicate that frost-susceptible base materials can experience a reduction in bearing capacity ranging from 13 to 62 percent during the thaw-weakening period (Janoo, 2002). Likewise, research in Japan has demonstrated that the layer coefficients used in the American Association of State and Highway Officials (AASHTO) pavement design method can decrease by 16 to 35 percent during this period (Kubo & Takeichi, 1992). After that, as water dissipates, the soil skeleton will adjust itself to a new equilibrium void ratio. The strength of the soil will be gained back gradually. However, severe damage to

structures, such as pavements, could occur during the early Spring due to thaw weakening. The worst damage typically occurs in fine-grained soils which are frost-susceptible. In non-frost-susceptible soils, no excess water is present during thawing since no ice lenses form in such soils, and the frozen water does not weaken the soil upon thawing. Thawing in frost-susceptible soils is accompanied by thaw settlement. A minor portion of the settlement can be ascribed to the volumetric reduction that occurs when ice transforms into liquid water. That being said, the majority of the settlement is from the drainage of melted water during thaw consolidation. The total settlement is dependent primarily on the amount of ice formed during freezing. The rate of settlement is affected by the soil type, the drainage condition, and the rate of thawing (Phukan, 1985; Simonsen & Isacsson, 1999). The entire process of thaw consolidation may take a relatively long time due to the low permeability of the still-frozen soil underneath and/or a poor drainage system. A typical thaw consolidation test result is shown in Figure 2.6.

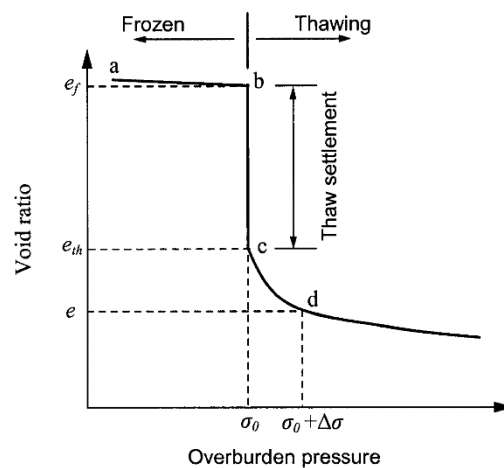


Figure 2.6 : A typical thaw consolidation test result, (after Tsytovich, 1960).

The frozen soil sample is placed in a conventional consolidometer. It is first loaded to an overburden pressure which is equivalent to the field load (σ_0). There is a small decrease in the void ratio under this initial loading process (points a to b). The sample is then thawed from the top to down, and a large change in the void ratio occurs due to the phase change from ice to water and drainage of the excess melted water (points b to c). This portion of the curve is the thaw settlement. Additional load is applied after thaw is completed, and consolidation under the initial load is nearly completed. Further drainage occurs in the same manner as a normal consolidation process (points c to d).

In Figure 2.6, e_f denotes the frozen void ratio before thawing starts, and e_{th} denotes the thawed void ratio.

Additionally, the American Society for Testing and Materials (ASTM, 2013) can be used to determine both the frost-heave and thaw-weakening susceptibility of soil. Estimating the time required for the soil to drain from 100% to 80% saturation is another suggested technique for figuring out a soil's capacity to thaw (Janoo, 2002). Granular materials have been demonstrated to become unstable under repeated loadings when the saturation level exceeds 80% (Janoo, 2002).

2.6 Experimental Studies on F-T Cycles on Soil Behavior

Many researchers have looked closely at how different soil qualities are affected by freeze-thaw cycles. These studies have covered aspects such as soil structure (Aldaoood et al., 2014; Chamberlain & Gow, 1979; Cui et al., 2014; Eigenbrod et al., 1996; Zheng et al., 2015), water permeability (Chamberlain et al., 1990; Jamshidi & Lake, 2015; Konrad & Samson, 2000; Makusa et al., 2014), shear resistance characteristics (Cui et al., 2014; Simonsen & Isacsson, 2001), and unconfined compressive strength (Aldaoood et al., 2014; Jamshidi & Lake, 2015; Solanki et al., 2013).

When freeze-thaw cycles occur, soils with an important fine fraction show changes in a number of characteristics. These modifications include shifts in pore water redistribution, densification, volume, strength, compressibility, and microstructural changes such as particle rearrangement and crack development (Kim & Daniel, 1992; Konrad, 1989a). Furthermore, these processes influence the Atterberg limits and reduce bearing capacity due to increased pore water pressure during thawing (Viklander & Knutsson, 1997).

Soil structure is mainly affected by the cyclical nature of freeze-thaw events, which are caused by the cryogenic suction and pressure created during freezing (Thomas et al., 2009; Wang et al., 2016) and the development of ice lenses (Taber, 1930; Viklander, 1998b). These changes can significantly affect the soil's engineering properties. The kind of soil and its stress history determine the deformation brought on by frost heave and thaw settling (Chamberlain, 1981).

Within delicate soil compositions containing minimal argillaceous substances, exemplified by silty clays, the more substantial silt components take precedence in the

ground's structure. Meanwhile, the minuscule clay elements form aggregated clusters capable of shifting through the interstitial spaces (Figure 2.7a). In such scenarios, the bulkier granules predominantly dictate the soil's capacity to compress, whereas the fine clay components largely determine how easily fluids can pass through. After undergoing cycles of freezing and thawing, the space between particles remains largely unaltered, as the icy conditions limit the reorganization of larger grains. Nonetheless, the soil's ability to transmit liquids improves. This occurs because the clay particles simultaneously compact and spread out, resulting in broader pathways that facilitate enhanced fluid movement.

Conversely, in fine soils with a higher content of clay minerals, such as silty clay, flocculated clay particles form a matrix in which larger grains float without direct contact (refer to Figure 2.7b). In this scenario, clay particles control both compressibility and permeability. After undergoing freeze-thaw cycles, clay packets rearrange into a denser and more dispersed state, resulting in a significant decrease in the void ratio. Contrary to expectations, even as the soil becomes more compact, its ability to allow fluid passage actually improves. This paradoxical effect stems from the emergence of minute shrinkage fissures within the soil structure, a direct consequence of repeated freezing and thawing episodes.

Research conducted by Graham and Au in 1985 revealed that the freezing process leads to the development of distinct cracks within the soil. Their findings also indicated that when moisture is present, the soil's softening under minimal pressure results in less significant effects (Graham & Au, 1985).

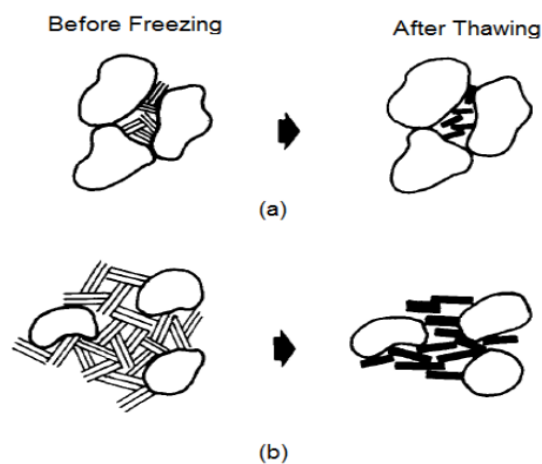


Figure 2.7 : Particle orientation after F-T cycles: (a) Clayey silts, (b) Silty clay, (Chamberlain & Gow, 1979).

Chamberlain investigated how freezing induces soil overconsolidation through suction forces. He developed a technique to calculate the peak suction experienced during the freezing process. His research uncovered a direct correlation between the extent of soil settlement upon thawing and the proportion of initial moisture content to the soil's plastic limit. Notably, Chamberlain observed that soil subjected to a freeze-thaw cycle exhibited a decrease in volume compared to its pre-freezing state (Chamberlain, 1981).

Konrad's research on clayey silts with different levels of overconsolidation yielded intriguing results. He discovered that lightly overconsolidated soils experienced a reduction in void ratio after thawing, whereas heavily overconsolidated soils showed an increase. His observations also indicated a decline in segregation potential following each freeze-thaw cycle, with the most significant changes occurring within the initial three cycles. Utilizing X-ray technology, Konrad was able to visualize alterations in soil density and structure within the frozen zone, providing visual evidence of freezing-induced changes (Konrad, 1989a, 1989b). The experiments corroborated a rise in water permeability following freeze-thaw cycles, consistent with the earlier discoveries of Chamberlain and Gow (Chamberlain & Gow, 1979).

Chamberlain's research focused on how freeze-thaw cycles affect the water flow and internal arrangement of densely packed soils containing silt, loam, and clay. Their findings revealed a minor expansion in the soil's pore space, attributed to water turning into ice. Although changes in water conductivity varied, they observed a marked rise in the soil's moisture saturation level after repeated freezing and thawing (Chamberlain et al., 1990).

Eigenbrod conducted cyclic one-dimensional open-system freeze-thaw tests on soft, fine-grained soils, noting volume changes up to 30% after thawing, depending on initial moisture content and plasticity. In soft clay samples exposed to freeze-thaw cycles, researchers observed a relationship between the highest negative pore pressure and soil compression, a process they referred to as freeze-thaw consolidation. However, this consolidation effect was absent in stiff clay samples with moisture levels close to their plastic limit (Eigenbrod et al., 1996).

Under cyclic freeze-thaw tests, Viklander and Eigenbrod investigated sandy, silty soils with inclusions of stone. They found that at the ideal compaction water content, soil, and stone heave were at their maximum and decreased with increasing compaction water content. When moisture levels surpassed the optimal content by 5-6%,

researchers noted an absence of heave or shifting of stone particles (Viklander & Eigenbrod, 2000).

In a study focusing on Qinghai-Tibet clay, Wang investigated the effects of 21 freeze-thaw cycles in a closed system. The research examined alterations in the clay's vertical dimension, moisture content, and physical characteristics. They found an increase in specimen height and a decrease in water content during the first seven cycles, after which both parameters stabilized (Wang et al., 2007).

In their research on silty soil exposed to one freeze-thaw event, Qi and colleagues discovered a pivotal dry density threshold. Beyond this point, they observed reductions in soil density, cohesive properties, and the pressure required for pre-consolidation (Qin et al., 2008). Research by Hui and Ping (2009) examined the effects of frost-heave and repetitive freezing-thawing cycles on reddish silt-rich clay samples from the Qinghai-Tibet rail corridor. Their findings revealed that as the number of freeze-thaw episodes increased, the soil's tendency to heave due to frost diminished. Additionally, they noted shifts in moisture distribution, with an uptick in water content within frozen zones and a corresponding reduction in unfrozen areas. The study also highlighted an increase in soil dry density, particularly in the lower parts of the samples (Hui & Ping, 2009).

Konrad's investigation delved into the impact of repeated freezing and thawing on water permeability in glacial sediment obtained from Péribonka, Quebec. The researcher utilized two sample types: water-saturated, liquefied, and consolidated specimens, as well as samples compacted below optimal moisture content. These underwent a series of closed-system freezing, thawing, and steady-pressure permeability assessments. The study revealed that alterations in water flow capacity were counterbalanced by settling that occurred during thawing. This phenomenon resulted in a reduction of the void ratio, which was primarily influenced by the initial void ratio before the freezing process began (Konrad, 2010).

Aldaoood and his team studied the impact of repetitive freezing and thawing on the structural integrity and mineral composition of gypsum-rich soils reinforced with lime. The research focused on evaluating the durability and performance of these lime-treated soils under cyclical temperature fluctuations near the freezing point. They noted significant water content increases in the first cycle and slight increases in

subsequent cycles across all samples, with volume change continuously increasing with each cycle (Aldood et al., 2014).

Wang and colleagues studied the frost-heave and thawing settlement in sediment reinforced with Portland cement, lime, and fly ash. They found these treated samples had 20% less volumetric fluctuation compared to untreated loess (Wang et al., 2016).

2.6.1 Baseline parameters and freeze-thaw behavior

When soils are repeatedly frozen and thawed, tiny structural adjustments take place. These modifications are shaped mainly by the pre-existing void ratio and soil compression level. This phenomenon is visually represented in Figure 2.8, illustrating the contrasting behaviors of initially unconsolidated and tightly packed soils. Viklander's research revealed that in loosely arranged soils, the freezing process induces volumetric expansion as ice crystals form distinct layers. Conversely, during the thawing phase, these soils experience a reduction in volume as the soil framework settles, resulting in heightened density and a more intimate arrangement of larger sediment particles.

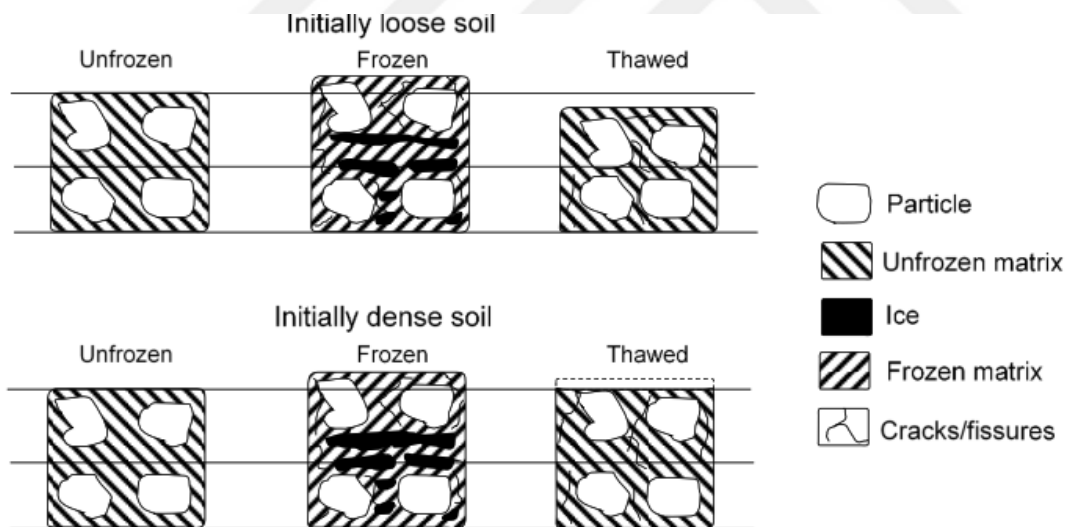


Figure 2.8 : Microstructural effect due to freezing and thawing, (Viklander, 1998b).

In contrast, for initially dense soils, while volume increases during freezing, much of this increase dissipates during thawing when the soil's matrix solidifies. Ultimately, the volume remains roughly the same as it was before freezing, with minimal microstructural alterations. Soil particles largely retain their positions after freeze-

thaw cycles, resulting in a slight loosening of the soil structure compared to its initial state (Viklander, 1998a).

Viklander's study on fine-textured glacial deposits undergoing eighteen freeze-thaw cycles found that high-porosity samples ($e=0.56$) decreased in pore space while low-porosity samples ($e=0.25$) increased. After one to three frost cycles, both types converged to similar void ratios between 0.31 and 0.40 (Viklander, 1998a).

In a subsequent investigation, Viklander utilized X-ray imaging to monitor the displacement of an implanted rock fragment within densified glacial sediment subjected to ten F-T cycles. This research corroborated the theory of an equilibrium pore space ratio (e_{res}) emerging till after a specific quantity of freeze-thaw iterations (N_{res}). Soils initially exhibiting low porosity tend to expand, with their void ratio gravitating towards the equilibrium value. Conversely, initially loose soils experience compaction, similarly converging on the equilibrium void ratio. This phenomenon is graphically depicted in Figure 2.9 (Viklander, 1998b).

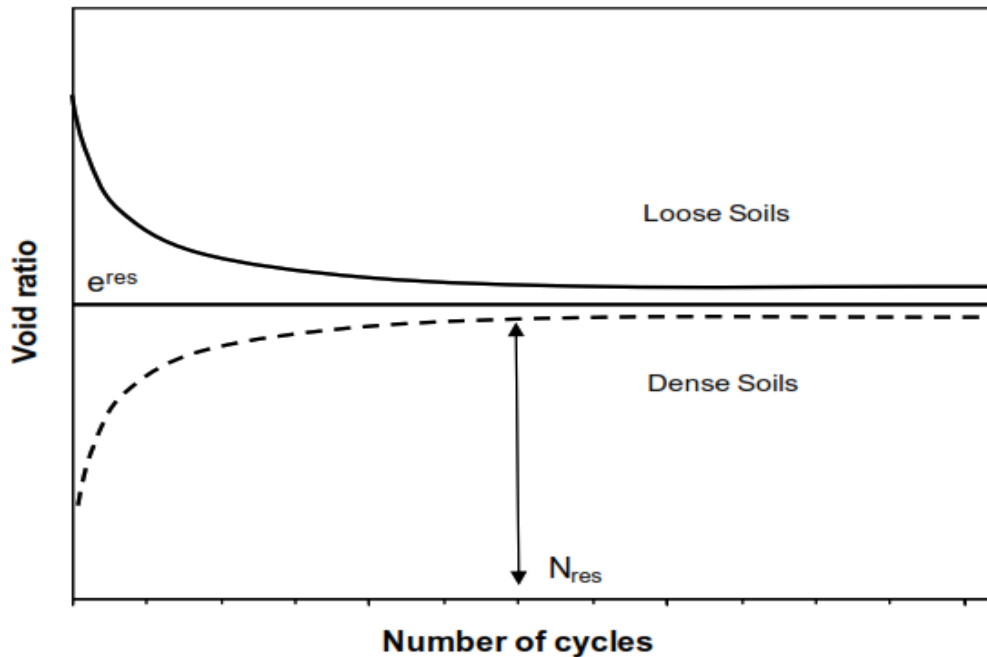


Figure 2.9 : Residual void ratio relative to number of F-T cycles, (Viklander, 1998b).

Qi and colleagues studied densified loessial samples from Lanzhou with dry unit weights ratios of 15.3 to 17.3 kN/m³ under F-T cycling. They identified a critical dry density threshold that dictates the soil's response to freeze-thaw cycles. Samples below this threshold compacted further, while those above it decreased in density. Samples

at the critical dry unit weights maintained their ratio after the F-T cycles (Qin et al., 2008).

2.6.2 Variables influencing soil volume change due to freeze-thaw

In addition to the impacts of soil types and classifications on frost-heave and thaw-settlement, the rate of freezing, or cooling rate, is a crucial factor influencing the volumetric behavior of soils during freeze-thaw cycles (Benson & Othman, 1993; Konrad, 1988; Penner, 1986). Svec states that a higher heave rate is produced by a smaller freezing fringe and a lower segregation temperature. On the other hand, with high frost penetration rates, the quick freezing process does not give enough time for appreciable water flow between consecutive ice segregation sites, leading to a reduced heave rate (Svec, 1989).

Ice segregation occurs when the pore pressure reaches a critical value, triggering the genesis of a fresh crystalline water layer oriented along the thermal gradient (Nixon, 1991; O'Neill & Miller, 1985). This process, along with the volumetric expansion caused by the phase change of pore water in both completely frozen soils and ice lenses, leads to frost heave (Thomas et al., 2009). To facilitate unidirectional thermal extraction, investigators have utilized an array of insulation (Eigenbrod et al., 1996; Viklander, 1998a, 1998b).

2.7 Effects of Freeze-Thaw Cycles on Soil Strength and Deformation

Understanding how freeze-thaw cycles affect soil properties is essential for various geotechnical applications, particularly in regions experiencing seasonal freezing and thawing. These cycles can significantly modify the mechanical behavior and stability of soils, influencing construction, agriculture, and infrastructure projects. Over the past few decades, a large number of researchers have examined how freeze-thaw cycles affect soil strength, deformation, and overall stability (Nagare et al., 2012; Oztas & Fayetorbay, 2003; Simonsen & Isacsson, 1999). However, research results on the impact of F-T cycling on soil permeability, heat transfer, and mechanical properties such as particle cohesion, shear resistance, volumetric mass changes, and load-bearing capacity remain inconsistent.

To address issues related to aggregate stability, laboratory tests were performed on soil samples from Erzurum Province, Turkey. These experiments confirmed that hydration

levels significantly impact soil resilience. Additionally, granular substrates with particles larger than 1 mm showed improved structural integrity after F-T cycling (Oztas & Fayetorbay, 2003). Kværnø and Øygarden studied the effect of F-T cycling on the cohesion of fine-grained soils, including silt, clay loam, and silty-clay-loam, using Norwegian protocols. They found that freeze-thaw cycles reduced structural integrity in all types, with silt being the most vulnerable (Sigrun Hjalmarsdottir Kværnø & Lillian Øygarden, 2006).

Qi and collaborators studied loess sediment from Lanzhou with dry unit-weight ratios of 15.3 to 17.3 kN/m³ under a thermal gradient of -20°C to +1°C. Lower-density samples were compressed after F-T cycling, while higher-density samples expanded and loosened. Cohesive forces decreased, the internal angle of friction slightly increased, and the resilient modulus declined universally. Notably, for a specific critical dry unit weight (γ_d), the soil's engineering properties, including its dry density, remained unchanged after the cycles (Qi et al., 2008).

Wang and colleagues conducted F-T cycling experiments on clay from the Qinghai-Tibet region, focusing on F-T cycles frequency. They found that volumetric expansion due to frost heave stabilized after seven freeze-thaw cycles. Using an 18-hour period at -7°C in a closed system without moisture sources, they observed declining particle cohesion and a resilient modulus that initially decreased, then slightly increased, and stabilized after the seventh cycle (Wang et al., 2007).

Triaxial compression tests at negative temperatures were conducted to better understand the strength properties of frozen soils (Brouchkov, 2000; Brouchkov, 2002; Hazirbaba et al., 2011; Sinitsyn & Løset, 2011). The stress-strain behavior of silty clay specimens changed from strong strain softening with a peak in deviator stress to weaker strain inclinations as confining pressures increased (Zhang et al., 2007). Zhang and colleagues proposed refinements in triaxial methods by advocating for a volumetric quantification approach to enhance precision, considering the volumetric and compressive properties of geomaterials below freezing conditions (Zhang et al., 2013).

Most research shows that F-T cycles in an open-system with water ingress negatively impact load-bearing capacity and resilient modulus (Tang & Xu, 2016; Wang et al., 2007). But, mechanical properties remain largely unchanged in a closed-system with limited pore water passage (Kweon & Hwang, 2013). Furthermore, the bearing

capacity deteriorates under dynamic loads from vibrations. For instance, Cui and Zhang discovered that clay samples subjected to F-T cycles and subway vibrations experienced a 26.7% increase in strain and a 53% rise in excess pore pressure compared to undisturbed samples (Cui & Zhang, 2015). More energy is transferred to the soil at lower vibration frequencies, causing a higher accumulation of plastic strain (Cui et al., 2014; Tang & Xu, 2016).

2.8 Frost-Heave Mitigation

Two main strategies can be utilized to protect pavements from the F-T cycle-induced problems. The first strategy involves reducing frost heave, which helps control the development of roughness over both the short and long term. By reducing frost heaving, this technique addresses concerns like frost-related cracking and deterioration as temperatures rise. Another approach enhances the pavement structure's durability, increasing its resilience to frost heave and thaw-induced weakening.

If a pavement structure does not adhere to the required frost protection standards, it must be upgraded to safeguard its longevity. Various techniques are at hand to minimize the impacts of frost and elevate the operational efficiency of pavements in cold environments. Frost heaving typically occurs under three conditions: the subgrade soil is exposed to prolonged freezing, the soil is prone to frost, and water is present near the freezing point, supporting ice lens formation. Enhancing pavement performance in cold regions involves effectively addressing any of these factors. The following parts will offer a concise overview of some successful strategies for mitigation.

2.8.1 Reducing the duration of exposure to freezing temperatures

To mitigate the effects of frost action, a common method involves restricting the subgrade soil's exposure to freezing temperatures, which can be achieved by adding an insulating layer to the pavement construction and widening the granular layers.

2.8.1.1 Enhancing granular layer thickness

An effective method to reduce frost heaving and preserve pavement load-bearing ability through spring thaw is to augment the width of granular layers. This strategy involves introducing or improving a frost-resistant granular subbase layer that is situated between the frost-prone subgrade soil and the base layer. In cold and damp

conditions, this subbase layer is crucial because it helps distribute loads, reduce differential movements, and minimize frost action by lowering the subgrade's water content and temperature through better drainage.

Most pavement design methodologies recommend a specific total pavement thickness to counteract freezing effects. Traffic loads are used to determine the thickness of the bonded surface and base layers; the thickness of the subbase layer is adjusted to reach the total thickness. This subbase layer is usually composed of cost-effective, non-frost-susceptible granular materials.

In situations where the pavement's surface elevation must adhere to strict limits, the "remove-and-replace" method is employed. This involves excavating the frost-susceptible subgrade soil to a depth that meets frost-protection requirements and replacing it with a non-frost-susceptible granular layer. This technique is particularly useful for localized areas with highly frost-susceptible soils, ensuring adequate pavement protection without raising the road's final profile. Proper drainage of the sub-excavated area is crucial in all cases.

A specific application of the remove-and-replace method is the construction of longitudinal transition wedges. Transition zones are employed to mitigate issues associated with differential frost heave in areas where a road traverses between regions of low and high frost susceptibility. As illustrated in Figure 2.10, a typical transition wedge design includes the following principles (Dysli, 1991):

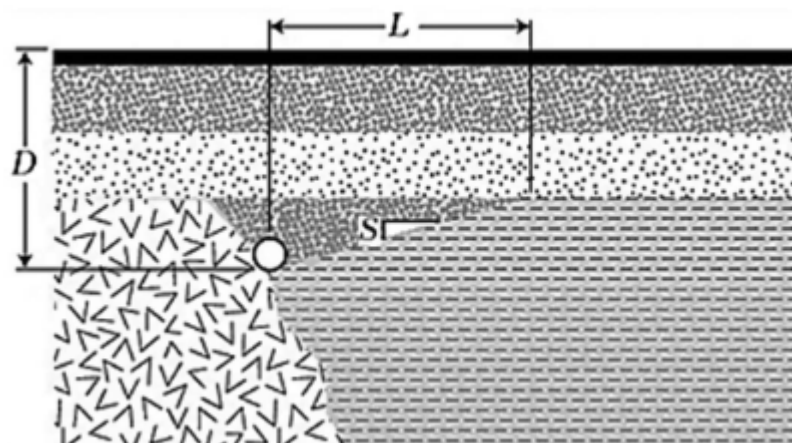


Figure 2.10 : A longitudinal transition wedge is used to reduce differential behavior in road crossings from bedrock (left) to frost-susceptible soil (right) (Doré & Zubeck, 2009).

- **Maximum Thickness:** The wedge's maximum thickness should be designed to guarantee that the total granular protection thickness at the interface between the two soil units is greater than or equal to the anticipated maximum frost penetration depth.
- The transition zone's length should effectively mitigate differential frost heave between soil units. This length (L) must be calculated based on the expected traffic speed and the anticipated frost heave variations between adjacent soil units. The recommended gradient for these transitions is 20 horizontal units to 1 vertical unit (20H:1V), and the minimum transition length should not be less than 20 meters.
- **Effective Drainage:** Ensuring proper drainage of the transition wedge is vital.

In regions where geological contacts between soils with contrasting frost-susceptibility are infrequent, transition wedges offer a cost-effective solution for mitigating severe differential frost heave between adjacent soil units.

By following these principles, engineers can design effective pavement structures that resist frost heave and maintain their integrity during seasonal freeze-thaw cycles.

2.8.1.2 Techniques and materials for pavement insulation

In regions susceptible to severe and uneven frost-heave, insulation for paved surfaces is crucial. Contrary to common perception, the insulation doesn't halt the progression of the cold front through the pavement structure. Instead, it blocks heat transfer when the pavement surface is unprotected from freezing air, which reduces heat loss from the pavement. The layer assists in preserving the soil temperature above the freezing point during the winter months. This, in turn, helps to effectively minimize or even eliminate the occurrence of frost-heave, uneven frost-heave, and frost penetration within the pavement system.

Insulation, often made of mechanically weak materials, requires meticulous design of the overlying pavement layers. Furthermore, the incorporation of a sufficient thickness of granular materials above the insulation layer is crucial to help alleviate the risk of differential icing on the pavement surface. The required width of this layer, which is granular, may vary depending on the specific practices and standards of the relevant agency or organization.

Road builders employ a diverse array of thermal barriers in their projects. The majority of these protective layers function by encapsulating tiny air pockets within their

structure, diminishing heat transfer, and offering robust temperature regulation. Frequently utilized options encompass inflated and molded polystyrene variants, as well as substances derived from urethane compounds. Alternatively, certain materials leverage their ability to absorb moisture, thereby accumulating substantial amounts of latent heat. This property impedes the advancement of freezing temperatures upon contact with the insulating stratum. In pavement insulation applications, byproducts from timber processing and materials like peat have found utility due to their capacity to store latent heat effectively.

Panels crafted from polystyrene dominate the field of roadway thermal protection, owing to their superior heat-resistant properties, considerable load-bearing capacity, and resilience in pavement settings over extended periods. Among these, the extruded variant stands out as the preferred choice, delivering optimal performance. In contrast, the expanded form offers a more budget-friendly option, albeit with heightened susceptibility to moisture absorption, which compromises its insulating efficacy. To counteract this drawback, engineers typically augment the thickness of expanded polystyrene layers by approximately 20 percent, ensuring adequate thermal barrier functionality despite its increased water sensitivity (Saint-Laurent, 2006). The labor-intensive placing method of polystyrene poses a major problem, as it necessitates careful attention to the joints between boards and their coating with granular materials. Heat loss at the joints can be reduced by using two layers of boards with staggered joints.

Alternative thermal barriers for road construction encompass aerated concrete and low-density granular substances, which offer a comparatively rigid stratum with reduced heat transfer properties and can be readily implemented using conventional construction machinery. Innovative insulating materials gaining traction include expanded clay products and aerated glass particles. Nevertheless, these substances require safeguarding against fine particle infiltration through the application of a separating geotextile layer. Additionally, they exhibit vulnerability to moisture absorption. Researchers and engineers continue to assess the enduring structural integrity and heat-resistant capabilities of these materials, as their long-term efficacy remains under scrutiny in real-world applications.

Researchers have explored various repurposed and reclaimed substances as potential thermal barriers in road construction. Experimental studies, both in controlled

environments and on-site, have investigated the efficacy of arboreal byproducts (such as tree bark, wood shavings, and timber fragments), decomposed plant matter, pulverized and chunked rubber, as well as plastic remnants. Though these materials generally demonstrate commendable heat-resistant qualities, they often fall short in terms of structural robustness. Their practicality in roadway insulation becomes apparent when they are readily accessible in substantial volumes at minimal expense. Typically, these alternative insulators find application in the development or renovation of less-trafficked community thoroughfares. To ensure adequate load-bearing capacity, engineers must incorporate a sufficiently thick layer of granular materials atop these recycled insulators.

2.8.2 Reducing the susceptibility of subgrade soils to frost

Combating the detrimental effects of frost on soil substrates necessitates a multifaceted approach to diminish their freezing susceptibility. Engineers employ a range of methodologies, including chemical soil fortification, mechanical modification of soil, and subgrade augmentation. These interventions serve a dual purpose: they not only mitigate the occurrence and irregularity of frost heave but also enhance the structural integrity of roadways during the critical spring thaw phase. By implementing these strategies, road builders can significantly improve the resilience and longevity of pavement structures in frost-prone regions.

2.8.2.1 Stabilization with chemicals

Chemical soil modification techniques have proven highly effective in diminishing the freeze-thaw vulnerability of subgrade materials. These treatments are generally concentrated on the uppermost 300 mm of the subgrade layer. In his comprehensive analysis, Dysli highlights two predominant methodologies: cement-based stabilization and lime-based stabilization (Dysli, 1991).

Stabilization with cement functions by creating a cohesive matrix among soil particles, preserving their inherent mineral characteristics. This process significantly reduces the soil's hydraulic conductivity, thereby restricting water migration towards potential ice lens formation zones. Moreover, it bolsters the soil's mechanical resilience against frost heave forces and enhances its structural rigidity during the critical spring thaw period. In his research, Dysli reports a remarkable finding: incorporating cement at a

ratio of 4% to 8% by weight can diminish frost heaving by as much as 90% within the treated soil stratum (Dysli, 1991).

Stabilization with lime, in contrast, operates by modifying the hydrophilic properties of clay particles, fostering inter-particle bonding and diminishing soil plasticity. This technique necessitates an extended curing period, typically spanning several months, to achieve full efficacy. Owing to its capacity to permeate fine-grained matrices, lime stabilization proves especially advantageous for silty clay and clay-rich soils. To ensure optimal outcomes, it is imperative to conduct comprehensive freezing tests, which serve to determine the precise lime dosage required for effective treatment.

2.8.2.2 Mechanical homogenization

Mechanical homogenization is a strategic intervention aimed at mitigating uneven frost heave through the uniform mixing of the uppermost subgrade soil layer. This process employs specialized equipment known as pulvimixers, which thoroughly intermix the soil to a substantial depth of 900 mm. By creating a more consistent soil matrix, this technique significantly reduces the risk of uneven frost heaving, which can be particularly detrimental to pavement structures. The efficacy of this method has been recognized and implemented by the state of Minnesota, as evidenced by its inclusion in their official engineering standards. This approach not only enhances frost resistance but also promotes more uniform pavement performance across varying soil conditions, contributing to extended infrastructure longevity in frost-susceptible regions (Forsyth, 1993).

2.8.2.3 Improvement of subgrade

The strategic replacement or augmentation of the current subgrade soil with properly chosen borrow materials is included in the enhanced subgrade process. These imported materials are chosen for their reduced frost susceptibility and enhanced mechanical characteristics. This concept has been notably integrated into the French pavement design philosophy, where it plays a crucial role in creating resilient road structures. In this approach, engineers meticulously select and treat an engineered soil layer to meet precise specifications for bearing capacity and frost resistance. The selection criteria and treatment processes are tailored to the specific road classification, ensuring that each pavement structure is optimized for its intended use and local environmental conditions (Peyronne & Caroff, 1984).

2.8.3 Reducing water availability

Mitigating water availability is a critical strategy in the battle against frost heave and its detrimental effects on pavement integrity. This objective can be accomplished through two primary approaches. First, by implementing sophisticated drainage solutions within the pavement structure, excess water can be efficiently channeled away from frost-susceptible layers. Second, the implementation of a capillary barrier involves placing a layer of coarse-grained material beneath frost-susceptible soil layers. The stark contrast in pore sizes between the fine-grained soil and the coarse-grained barrier disrupts capillary action, effectively preventing upward water migration.

2.8.3.1 Enhancing drainage

Enhancing drainage systems plays a pivotal role in mitigating frost action through dual mechanisms. The primary effect is the reduction of water content within both the pavement structure and subgrade by effectively lowering the water table. However, it's important to note that while improved drainage undoubtedly contributes to decreased frost heaving, its efficacy is not absolute. This limitation stems from the powerful suction forces generated at the segregation front during the freezing process. These forces are capable of drawing water from considerable depths, often several meters below the forming ice lens, thus potentially circumventing some of the benefits provided by enhanced surface and near-surface drainage.

Efficient drainage systems play a critical role during the spring thaw period, a time of heightened vulnerability for pavement structures. As temperatures rise and ice lenses begin to melt, well-designed drainage becomes instrumental in two key aspects. Firstly, it enables the swift dissipation of excess pore pressure generated by the liquefaction of ice lenses. Secondly, adequate drainage rapidly reduces the water content in both soil and pavement materials, accelerating the restoration of the structure's load-bearing capacity.

2.8.3.2 Utilizing capillary break systems

Capillary break systems employ materials characterized by large pore sizes, such as clean coarse sand, pea gravel, or advanced geocomposites combining geonets with geotextile separators. These carefully selected materials are strategically placed above

the water table to effectively impede the upward movement of unsaturated water flow (Henry & Holtz, 2001). The effectiveness of capillary break systems stems from their unique hydraulic properties. These materials exhibit minimal capillary action and possess significantly lower unsaturated hydraulic conductivity compared to the surrounding soil matrix. This stark contrast in hydraulic characteristics creates a functional barrier that substantially impedes the upward migration of water. Moreover, these systems offer a dual benefit: while blocking upward water movement, they simultaneously facilitate the redirection of downward-infiltrating surface water towards engineered drainage systems (Henry et al., 2005; Zornberg et al., 2010).

The design of capillary break systems leverages the strategic use of non-frost-susceptible materials, which forms the core of their functionality. As elucidated by Henry, this approach allows for a cost-effective layering strategy in pavement construction (Henry et al., 2005). The efficacy of a capillary break system hinges critically on its precise placement relative to the water table. To achieve optimal performance, it is imperative that the capillary break layer is situated at an elevation sufficiently above the highest anticipated groundwater level.



3. MATERIALS AND METHODS

This research explored the characteristics of fine-grained soils, particularly clay, by evaluating their Atterberg limits, including the Liquid Limit (LL), Plastic Limit (PL), and Plasticity Index (PI). The valuation adhered to the established ASTM D4318 standards (ASTM, 2018). The study further incorporated the standard Proctor compaction tests, adhering to the ASTM D698 (ASTM, 2021b) guidelines, performed on soil samples treated with varying concentrations of Polyethylene Glycol (PEG) and Magnesium Chloride (MgCl_2) solutions. These tests were designed to determine the maximum dry density (MDD) and optimum water content (OWC) of the treated samples. Additionally, the strength of the treated samples was assessed through unconfined compressive strength (UCS) tests, following the ASTM D2166M (ASTM, 2016a) protocol, on samples cured for different durations and uncured samples for comparative analysis.

Initially, PEG was chosen for its antifreeze properties, aiming to prevent frost heave in cold environments. We conducted a series of tests, including Atterberg limits, standard Proctor compaction tests, UCS, and one freeze-thaw (F-T) cycle at -20°C , with varying concentrations of PEG (10%, 20%, 30%, 40%, 50%). However, the results showed that PEG caused a significant increase in the Atterberg limits, indicating higher soil plasticity, while drastically reducing the mechanical strength of the clay. Additionally, the introduction of PEG resulted in an increase in the Maximum Dry Density (MDD) and a decrease in the Optimum Water Content (OWC), which further complicated its effectiveness in soil stabilization. This suggested that although PEG exhibited antifreeze properties, it was not suitable for use in clay stabilization due to its negative impact on the soil's mechanical properties.

In response, we experimented with the addition of cement as a strength-enhancing agent. We hypothesized that combining cement with PEG would enable us to leverage PEG's antifreeze capabilities while maintaining or improving the soil's strength through the cement. However, after conducting UCS tests and one freeze-thaw cycle at -20°C , we observed that while cement increased the clay's strength, the addition of

PEG still caused a reduction in overall mechanical strength. This led us to explore alternative materials. After testing several materials with antifreeze properties, we identified MgCl_2 as a promising alternative.

the next step was determining the optimal dosage of MgCl_2 , we conducted practical experiments by preparing MgCl_2 -clay mixtures with various concentrations (4%, 9%, 14%) and placing them in a freezer at -10°C and -20°C . Using a thermometer, we observed that MgCl_2 concentrations of 4%, 9%, and 14% were suitable for our study's goals. Specifically, the 9% and 14% concentrations acted as effective antifreeze agents at -10°C , while the 14% concentration could withstand freezing at -20°C , as their freezing points were lower than these temperatures. Based on these observations, we proceeded with further testing using MgCl_2 dosages of 4%, 9%, and 14%, including Atterberg limits, standard Proctor compaction tests, and UCS tests.

The next phase of the study examined how freeze-thaw (F-T) cycles affect the mechanical properties of soil stabilized with different concentrations of MgCl_2 solution under freezing temperatures of -20°C and -10°C . The experimental methodology followed ASTM D560 standards (ASTM, 2016b). The primary objective was to ascertain whether modifications in the MgCl_2 solution concentration are necessary to enhance the effectiveness of soil stabilization under varying temperature exposures and to evaluate the soil's response to environmental freeze conditions.

Two sets of soil specimens, one treated with MgCl_2 and the other untreated, were prepared and underwent a 28-day curing period prior to the F-T cycling test. The specimen parameters like diameter, weight, and height were measured to analyze volume changes and evaluate the mechanical behavior after each F-T cycle.

The experimental protocol incorporated two distinct freeze-thaw (F-T) regimes to simulate varying degrees of winter severity. One set of specimens underwent a moderate freeze condition, with exposure to -10°C for a 24-hour duration. Concurrently, a second set was subjected to a more rigorous freeze phase at -20°C , replicating severe winter conditions. Following each freeze interval, all specimens were transferred to a controlled environment chamber maintained at 23°C with 100% relative humidity for an additional 24 hours. This transition constituted the thawing phase, culminating in a complete 48-hour F-T cycle. To comprehensively assess the cumulative effects of repeated F-T exposure, this cyclical process was iterated up to seven times for both temperature regimes.

Unconfined compressive strength tests were performed at predetermined intervals—specifically after the 1st, 3rd, 5th, and 7th cycles—to quantify the evolution of mechanical properties throughout the F-T cycles. Concurrently, volumetric measurements were recorded following each individual freeze and thaw phase, providing a comprehensive profile of soil volume fluctuations in response to temperature variations. This multifaceted approach aimed to elucidate the efficacy of MgCl₂ solution as a soil stabilization agent under diverse thermal conditions.

To complement the macroscopic analysis, a comprehensive microstructural investigation was conducted on a representative subset of samples. This in-depth examination employed a suite of advanced analytical techniques, including Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and Fourier Transform Infrared Spectroscopy (FTIR). SEM imaging provided high-resolution visualizations of the soil fabric, elucidating changes in particle arrangement, pore structure, and the formation of potential ice lenses. XRD analysis offered insights into any mineralogical transformations or alterations in crystalline structures resulting from the freeze-thaw (F-T) cycles. FTIR spectroscopy facilitated the identification of chemical bonds and functional groups, potentially revealing the formation of new compounds or alterations in existing molecular structures due to F-T processes and MgCl₂ interactions.

In this chapter, the details of each test method and the materials used in this study are explained under Section 3.1 and Section 3.2, respectively. At the end of each test method, the sample preparation for our specific experiments is also detailed. Moreover, the detailed results and discussions of these experiments are presented in Chapter 4.

3.1 Experimental Test Methods

3.1.1 Particle size distribution

In this study, the particle size distribution of soil samples was determined using a hydrometer analysis, which is essential for understanding the proportions of various soil fractions such as silt and clay. Approximately 50 grams of dried soil were carefully measured and treated with a dispersing agent, typically a solution of sodium hexametaphosphate, to facilitate the separation of soil particles. This treatment was conducted over 18 hours to ensure thorough dispersion of the soil aggregates,

preventing any agglomeration that could affect the accuracy of the particle size analysis.

Following the dispersion process, the hydrometer analysis was performed. The dispersed soil sample was suspended in a graduated cylinder filled with distilled water, which was then gently agitated to ensure an even distribution of soil particles. A hydrometer was inserted into the suspension to measure the density at specified intervals, based on Stokes' Law, which describes the settling velocities of particles in a fluid. Initial and subsequent readings were taken at intervals of 2, 5, 15, 30, 60, 120, and 1440 minutes, allowing for the calculation of the sedimentation rates of different soil fractions and providing a comprehensive profile of the sedimentation process over time.

The data obtained from the hydrometer analysis was used to plot a particle size distribution curve, representing the cumulative percentage of soil particles finer than a given size against the particle diameter. This curve offers valuable insights into the soil's textural composition, which is crucial for predicting and managing soil behavior under different environmental and loading conditions. Thus, the hydrometer analysis provided a detailed assessment of the soil's particle size distribution, ensuring accurate and reliable results that contribute to a comprehensive understanding of the soil's characteristics (ASTM, 2021a). Details are described at section 3.2.1.

3.1.2 Specific gravity

To accurately assess the density of the soil particles, researchers employed a volumetric flask technique. The process began by selecting a predetermined mass of soil that had been thoroughly dehydrated. This soil sample underwent a rigorous desiccation procedure involving exposure to a constant temperature of 105°C in a laboratory oven for a full day. Subsequently, the mass of the dehydrated soil was meticulously documented using a high-precision scale designed for analytical purposes.

Next, a flask (Figure 3.1) containing 50gr dry soil sample was filled with distilled water until it was full. To eliminate any trapped air bubbles, which could affect the accuracy of the measurement, the flask was gently agitated and then subjected to a vacuum to ensure all air was removed. The weight of the flask, now containing both the soil and water, was recorded.

To determine the weight of the displaced water, the weight of the flask containing only water was measured separately under the same conditions. By comparing the weight of the flask with soil and water to the weight of the flask with only water, the weight of the displaced water was calculated. This step is crucial as it provides the volume of water equivalent to the volume of the soil particles.

The specific gravity of the soil solids was then calculated by dividing the weight of the dry soil by the weight of the displaced water. This specific gravity value is an essential parameter in geotechnical engineering, as it helps in understanding the soil's density and its behavior under various environmental conditions. It also plays a critical role in further analyses, such as the determination of void ratios, porosity, and the overall stability of the soil in freeze-thaw cycles (ASTM, 2023).

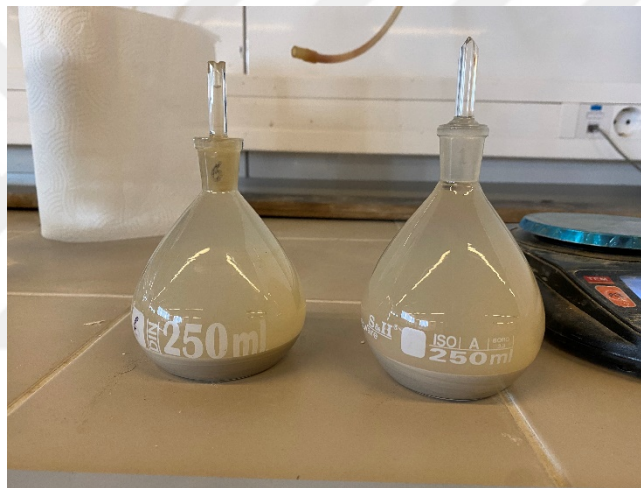


Figure 3.1 : Specific gravity test.

3.1.3 Dosage calculation

In the initial phase of the study, we conducted tests with Polyethylene Glycol (PEG 400) as an antifreeze additive. PEG 400 is a liquid, which is why we prepared the solutions based on volume. We tested concentrations of 10%, 20%, 30%, 40%, and 50% on clay. The volume-based approach was used to ensure accurate control over the mixture's consistency and to allow for even distribution of the liquid additive throughout the soil matrix. This method is particularly important for liquid additives like PEG 400, which must be thoroughly mixed with water to form a homogenous solution before being incorporated into the soil.

The concentration of PEG 400 was calculated using equation 3.1:

$$\text{Concentration (\%)} = \left(\frac{\text{Volume of PEG}}{\text{Volume of PEG} + \text{Volume of Water}} \right) \times 100 \quad (3.1)$$

These solutions were then mixed with soil samples to evaluate the effects of PEG 400 on the mechanical properties of the soil.

Following the use of PEG 400, the other material employed in this study was magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$). Unlike PEG, magnesium chloride hexahydrate is a solid flake, so we calculated its solution concentration based on weight rather than volume. This method ensures precision in the dosage, as the solid must first dissolve fully into water before being mixed with the soil.

The weight-based concentration was calculated using equation 3.2:

$$\text{Concentration (\%)} = \left(\frac{\text{Weight of MgCl}_2}{\text{Weight of MgCl}_2 \cdot 6\text{H}_2\text{O} + \text{Weight of Water}} \right) \times 100 \quad (3.2)$$

However, there is a subtle point that must be considered when calculating the concentration of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. Since the flakes are $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, they contain water molecules as part of their crystalline structure. To accurately calculate the concentration, it is essential to use the ratio of the solid weight of MgCl_2 to the total weight of the solution.

According to the specifications for $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, which will be discussed in detail in Section 3.2.3, the flakes contain approximately 47% solid MgCl_2 and 53% water. This ratio is standard and does not vary, as the $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ flakes are consistently produced in this composition. Therefore, when calculating the concentration of the solution, we must first determine the solid weight of MgCl_2 :

This can be calculated using equation 3.3:

$$\text{Weight of MgCl}_2 = (\text{Weight of MgCl}_2 \cdot 6\text{H}_2\text{O}) \times 47\% \quad (3.3)$$

The reason is, after dissolution in water, the $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ flakes lose their crystalline structure, and the water molecules that were part of the hexahydrate are released. As a result, the solution no longer contains the hexahydrate form; it consists only of water and dissolved MgCl_2 particles. Therefore, from this point on, we refer to it simply as MgCl_2 solution rather than $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ solution.

As an example, to prepare a 14% MgCl_2 solution, we dissolve 43 g of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ in 100 g of water. Since the flakes contain 47% solid MgCl_2 , this results in approximately a 14% MgCl_2 solution.

3.1.4 Determination of optimal dosages using freezing point measurements

Before conducting the detailed thermometer and physical tests, we prepared two initial small samples to observe the preliminary effects of MgCl_2 . One sample was treated with 15% MgCl_2 solution, and the other was left untreated. Both samples were placed in a -20°C environment for 24 hours, as shown in Figure 3.2. In the untreated sample (Figure 3.2a), ice slush formed on the surface, whereas the sample treated with MgCl_2 (Figure 3.2b) showed no signs of ice formation.

We observed that the untreated sample was completely frozen and solid, while the treated sample retained the same soft texture as before being placed in the freezer. This initial observation strongly indicated that MgCl_2 could serve as an effective antifreeze agent. Following this finding, we proceeded with the detailed thermometer tests and freeze-thaw cycles to confirm and expand upon these results.

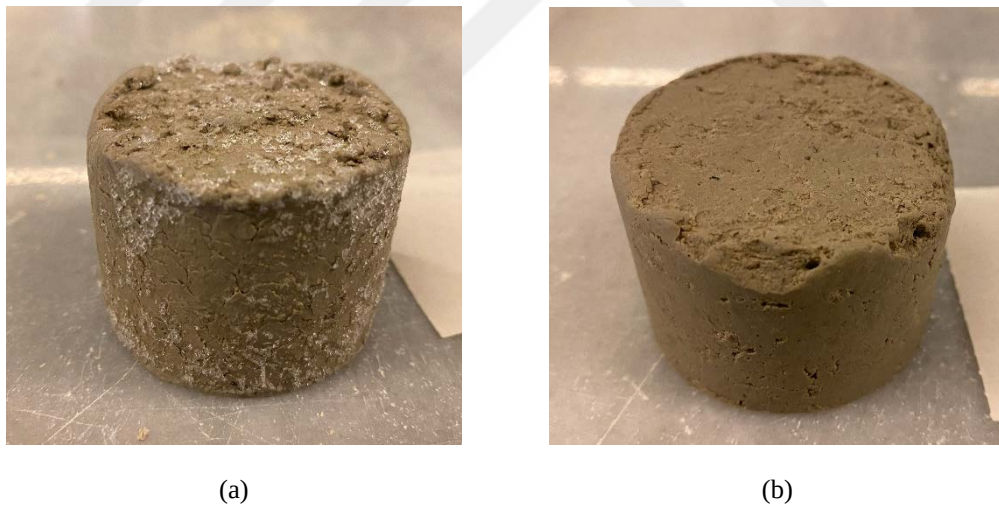


Figure 3.2 : Samples subjected to freezing at -20°C for 24 hours: (a) untreated sample with ice slush formation on the surface; (b) 15% MgCl_2 -treated sample without ice formation.

To determine the optimal dosage of MgCl_2 for soil stabilization, we conducted a series of practical experiments. We prepared MgCl_2 -clay mixtures with various concentrations, including 4%, 5%, 8%, 9%, 10%, 13%, 14%, and 15%, and placed them in a freezer at -10°C and -20°C . The objective was to physically observe which concentration could withstand these temperatures without freezing.

While higher concentrations of MgCl_2 can endure lower temperatures, we aimed to select an optimal dosage by considering both environmental impact and cost efficiency. Based on these criteria, we identified 4%, 9%, and 14% as the most suitable concentrations for further testing. With the help of a thermometer, we observed that the 4%, 9%, and 14% concentrations met the objectives of our study. Specifically, the 9% and 14% solutions acted as effective antifreeze agents at -10°C , while the 14% concentration also provided protection at -20°C , as their freezing points were lower than these temperatures. These results confirmed that the selected concentrations aligned with the goals of our study. Figure 3.3 shows the physical samples prepared for this test.

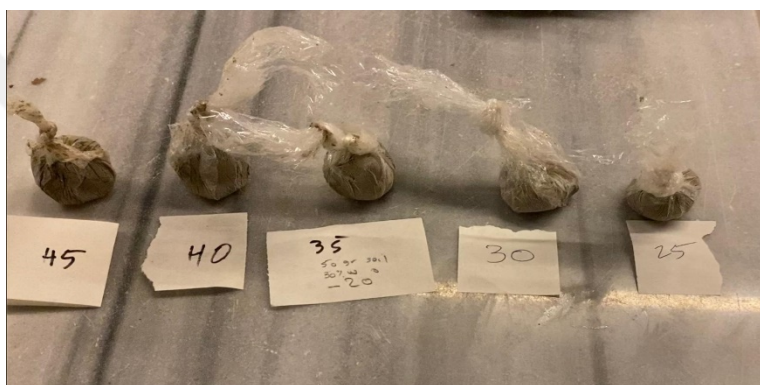


Figure 3.3 : MgCl_2 -clay mixtures prepared for freezing point testing.

After confirming the effectiveness of these concentrations at -10°C and -20°C , we proceeded to further testing to validate the antifreeze performance under more extreme conditions. For this, additional MgCl_2 -treated clay samples were prepared, with a thermometer probe inserted into the center of each sample, and the samples were placed in a freezer at -27°C . This allowed us to precisely monitor internal temperature changes and confirm that the selected concentrations were effective in preventing freezing at even lower temperatures. The results demonstrated that 4%, 9%, and 14% MgCl_2 concentrations were optimal for use in freeze-thaw cycle tests under varying conditions. Figure 3.4 illustrates the thermometer device used for these measurements.

Following these results, we continued with additional testing using these concentrations, including Atterberg limits and other mechanical tests. The detailed results from the thermometer tests and freezing point determinations are discussed in Section 4.2.2.



Figure 3.4 : Thermometer device used for freezing point measurements.

3.1.5 Atterberg limits

Representative samples from the soil underwent Atterberg limits testing to assess its plasticity. To evaluate the plasticity characteristics of the soil, representative samples were collected and subjected to Atterberg limits tests, which include determining the liquid limit (LL) and plastic limit (PL). The liquid limit for the soil was determined using an Atterberg limits device on material that passed through a No. 40 sieve. This test was conducted according to standardized procedures (ASTM, 2018) employing an Atterberg limits instrument, which is made expressly to gauge the moisture content at which soil changes from a plastic to a liquid condition. Soil samples passing through a No. 40 sieve (425 μm opening) were used to ensure uniformity and consistency in particle size.

Soil that had passed through a No. 40 sieve was rolled into 3-mm-diameter threads until they cracked in order to ascertain the plastic limit. On a glass plate, soil samples were hand-rolled into threads about 3 mm in diameter to determine the plastic limit. The soil was considered to have reached its plastic limit when the threads began to crumble and crack. This method, also specified in (ASTM, 2018), provides a measure of the lowest water content at which the soil remains in a plastic state. The plasticity index (PI), which quantifies the range of water content over which the soil exhibits plastic behavior, was calculated based on the obtained liquid and plastic limits. These values were then used to classify the soil according to its liquid limit and plasticity index. This classification helped in identifying the soil type and understanding its

potential behavior under freeze-thaw conditions, which is critical for determining its suitability as an antifreeze and stabilizing agent.

In addition to using the Atterberg limits for soil classification, further Atterberg limits tests were conducted to investigate the effects of PEG and MgCl_2 on the clay's Atterberg limits. The detailed results of these tests are discussed in Section 4.1.2 for PEG and Section 4.2.3 for MgCl_2 , respectively.

3.1.6 Soil classification

The soil was classified according to the Unified Soil Classification System (USCS), a standardized method extensively used in geotechnical engineering to categorize soils based on their engineering properties. The laboratory procedures included a detailed particle size distribution analysis and the determination of Atterberg limits, providing a robust framework for soil classification according to the USCS. Accurate classification is essential for predicting soil behavior under various environmental conditions and for assessing its suitability for use in geotechnical applications, particularly those involving freeze-thaw cycles. Details are described at section 3.2.1.

3.1.7 Standard proctor test with Harvard miniature apparatus

The compactness of cohesive soil masses, whether natural or placed by humans, is often evaluated based on two critical criteria: optimum moisture content and maximum dry unit weight. However, the current testing method has two significant drawbacks: it requires a large amount of material, approximately 5 kg, and it is time-consuming. Considering the scarcity of resources and the necessity to perform multiple experiments, researchers opted for the compact Harvard apparatus to maximize efficiency and minimize the quantity of soil and supplements needed for testing. Both the Harvard Miniature Method and the conventional Standard Proctor Procedure apply an identical compressive energy of 600 kN-m/m^3 to soil samples.

The traditional Proctor compaction evaluation involves creating a moisture-density graph by compressing a series of soil specimens at specified water contents. The corresponding dry density for each moisture level is then calculated, resulting in a comprehensive moisture-density relationship graph.

The compact Harvard compaction device (Figure 3.5), conceptualized by Wilson, has found applications in preparing samples for triaxial testing and establishing moisture-density correlations for various soils. This apparatus enables swift density

determinations at different moisture levels and requires merely 1 to 2 kilograms of material. Consequently, it produces moisture-density curves more rapidly than the Proctor method while utilizing only a small fraction of the soil sample (Wilson, 1970; Wilson, 1950). Comparative results have demonstrated that the compact Harvard technique can yield results equivalent to those of conventional compaction methods when specific parameters are fine-tuned based on the soil's characteristics. These adjustable factors include the elevation and mass of the tamping instrument, the quantity of soil strata, and the frequency of compressive impacts per stratum. Opting for the miniaturized Harvard device over the traditional Proctor apparatus offers significant advantages in terms of resource conservation and efficiency. This approach not only expedites the testing process but also conserves valuable soil samples.

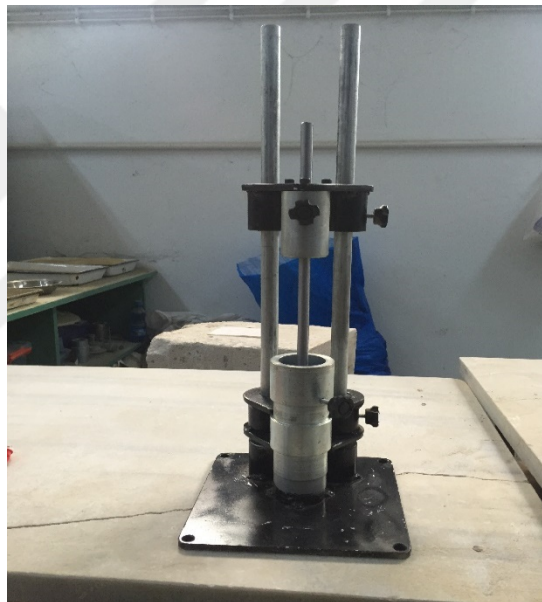


Figure 3.5 : Harvard miniature compaction apparatus.

Consequently, it facilitates more extensive research across diverse soil types, enabling the creation of a comprehensive repository for precise moisture-density correlations. Such a database proves invaluable for ensuring high-quality assessments of soil compaction properties in various geotechnical applications. By adopting a hammer height of 23 cm and 17 blows per layer over three layers, the study was able to obtain the same compaction energy as the Standard Proctor test, ensuring consistent compaction effort and supporting the use of the Harvard miniature device for particular needs. For the treated samples, the results of the standard Proctor test for PEG-treated clays will be discussed in detail in Section 4.1.3, and for MgCl_2 -treated clays, the results are presented in Section 4.2.4.

3.1.8 Unconfined compressive strength (UCS) test

Compacted soil compressive strength is commonly determined by the unconfined compression test, which is conducted in accordance with ASTM D2166-00 criteria. This test is carried out under strain-controlled conditions, wherein the soil sample is positioned between a pair of compression plates, and force is exerted to compact the specimen. Figure 3.6 shows a representative apparatus utilized for conducting this assessment.



Figure 3.6 : Unconfined compression device.

Before the test, the soil specimens (untreated and treated with PEG or MgCl_2) were prepared by adding water and the corresponding additives to the soil mixtures. The samples were then allowed to rest for 24 hours to ensure that the clay particles had enough time to absorb the water and additives, creating a more homogenized mixture. This step was crucial for achieving uniformity in the treated samples and ensuring that the effects of the additives on the soil properties were accurately reflected in the test results. After this absorption period, the samples were compacted, and the curing process began.

The procedure begins by preparing a cylindrical soil specimen, ensuring it is uniform and free of cracks. The specimen is placed between the loading platens of the testing machine, which applies stress at a controlled rate (typically 1 mm per minute) until failure, indicated by a drop in load or visible cracks.

Load and displacement data are recorded throughout the test, and the results are converted into stress vs. strain plots. Stress is calculated from the applied load and cross-sectional area, while strain is based on the displacement and original height of the specimen.

The key parameters obtained from the stress vs. strain plot include:

Unconfined Compressive Strength (q_u): This is the highest stress observed on the stress vs. strain curve. It represents the maximum axial compressive stress the soil specimen can withstand without any lateral confinement.

Strain at Failure: This is the strain corresponding to the unconfined compressive strength, indicating how much the specimen deformed at the point of failure.

The unconfined compressive strength (q_u) is an essential parameter for geotechnical engineering as it provides a measure of the soil's ability to withstand axial loads.

The strain at failure provides insight into the ductility and brittleness of the soil. Soils that exhibit high strain at failure are considered ductile, meaning they can undergo significant deformation before failure. In contrast, soils with low strain at failure are brittle and fail with minimal deformation.

The unconfined compression test is widely used in the design and analysis of foundations, embankments, and other soil structures. It is a relatively simple and quick test that provides valuable information about the strength and behavior of cohesive soils under axial loading.

However, this test also has limitations, as it does not apply confining pressure, making it unsuitable for non-cohesive soils like sands and gravels, which require lateral support. Additionally, it does not replicate real field conditions where soils experience both axial and lateral stresses. Despite these drawbacks, it remains a vital tool in geotechnical engineering for assessing the strength of cohesive soils and informing the design of stable soil structures (ASTM, 2016a).

It should be noted that, for the samples treated with PEG and PEG-cement, they were cured for only 1 day after compaction before the unconfined compression test. This short curing time was chosen to assess the antifreeze effect immediately after compaction, simulating real-world conditions where there is limited time for curing. After preparation, the samples were wrapped in plastic film to prevent moisture loss, and for curing, they were placed in a humidity room with 100% relative humidity. The results of these tests are discussed in detail in Section 4.1.4.

Moreover, after determining that MgCl_2 works effectively as an antifreeze, the curing durations were defined to evaluate how the strength of the treated soil evolves over time. For the MgCl_2 -treated samples, the specimens were cured for 0, 7, and 28 days following compaction. This was done to observe whether the strength decreases, remains intact, or increases with different curing durations. Similar to the PEG samples, after preparation, the MgCl_2 -treated samples were wrapped in plastic film and cured in a humidity room with 100% relative humidity. The results of these tests are discussed in detail in Section 4.2.5.

3.1.9 Freeze-thaw cycle test

The freeze-thaw test was conducted to assess the impact of repeated freeze-thaw cycles on soil samples, focusing on the effects of Magnesium Chloride (MgCl_2) treatment at different concentrations. Prior to testing with MgCl_2 , samples treated with PEG and PEG-cement underwent freeze-thaw cycles for one cycle, but due to unsatisfactory results, further testing was discontinued. Additionally, PEG and PEG-cement-treated samples were only cured for 1 day before testing. This was done to simulate real-world conditions where, after compaction, the weather is getting colder, and there is limited time for curing. If the antifreeze effect worked, curing could continue naturally in such conditions.

For the MgCl_2 -treated samples, the soil was prepared by incorporating MgCl_2 solutions at concentrations of 4%, 9%, and 14%. These samples were cured for 0, 7, and 28 days before undergoing freeze-thaw cycles at -10°C and -20°C . The extended curing durations for MgCl_2 -treated samples were intended to observe how the antifreeze properties affect soil behavior over time under freeze-thaw cycles.

To do this test, the samples were prepared by molding them into standard shapes and sizes and categorized into untreated, 4% treated, 9% treated, and 14% treated groups.

Each sample was carefully wrapped in plastic film to maintain moisture content and prevent contamination.

The freeze-thaw cycles were conducted under controlled conditions. Each cycle consisted of 24 hours of freezing followed by 24 hours of thawing at 23°C in a humid room to simulate natural conditions. This process was repeated for up to 7 cycles. Unconfined compression tests were conducted after 1, 3, 5, and 7 cycles to evaluate the mechanical properties of the samples and monitor changes in soil stability and strength over time.

The results for PEG-treated samples and MgCl₂-treated samples are discussed in Section 4.1.4 and Section 4.2.6, respectively. Additionally, after each freeze-thaw cycle, volume changes in the MgCl₂-treated samples were recorded for both -10°C and -20°C cycles, with detailed discussions presented in Section 4.2.7.

3.1.10 Microstructural characterization

The following analytical techniques were employed to investigate the micro-fabrics of both treated and untreated clay samples. However, these tests were only performed on MgCl₂-treated samples, and the details are discussed in section 4.2.8.

3.1.10.1 FTIR analysis test

Fourier Transform Infrared Spectroscopy (FTIR) measures the infrared region of the electromagnetic spectrum, characterized by longer wavelengths and lower frequencies than visible light. In FTIR analysis, infrared radiation (IR) is applied to a sample, causing bonds between different atoms to absorb light at various frequencies. An infrared spectrometer measures the light and produces an FTIR spectrum that graphs infrared light absorbance. This method determines a material's molecular composition and structure by measuring its ability to absorb IR light at different wavelengths.

FTIR analysis can identify unknown materials by comparing the IR spectrum against a reference database and quantify materials by creating a standard curve of known concentrations. The technique is employed to identify unknown substances, additives within polymers, and surface contamination. An interferometer produces an optical signal with encoded IR frequencies, which is measured and decoded using Fourier transformation.

When infrared radiation is absorbed by a soil sample, a Fourier Transform Infrared (FTIR) spectrum in the mid-infrared region is produced, revealing the overall chemical profile, including information about the soil's organic and mineral compounds. The IR spectrum absorption bands ($400\text{--}4000\text{ cm}^{-1}$) correspond to the basic vibrations of the functional groups present in the sample, enabling detailed interpretation of the nature and proportion of organic matter and minerals in clay samples.

FTIR is a preferred technique due to its minimal material requirement, quick and cost-effective analysis, and versatility in identifying solids, liquids, or gases. With access to hundreds of thousands of reference spectra, FTIR can reliably identify the primary components of a compound.

3.1.10.2 SEM

Scanning Electron Microscopy (SEM) employs a focused beam of high-energy electrons to generate signals from the surface of solid specimens, revealing detailed information about external morphology, chemical composition, crystalline structure, and particle orientation. SEM techniques allow imaging of areas ranging from 1 cm to 5 microns in width, at magnifications from 20x to 30,000x, and with spatial resolutions of 50 to 100 nm. The data collected over selected surface areas produce two-dimensional high-resolution images that illustrate spatial variations in these properties.

SEM generates high-resolution images, provides spatial variations in chemical compositions, and identifies phases based on qualitative chemical analysis or crystalline structure. It precisely measures features down to 50 nm, with backscattered electron images enabling rapid phase discrimination in multiphase samples.

3.1.10.3 XRD analysis

The X-ray diffraction analyzer is an advanced imaging tool that uses intense electromagnetic radiation to study crystalline structures, unlike traditional optical instruments. When a monochromatic X-ray beam with wavelength (λ) hits the ordered atomic planes of a crystal at a specific angle (θ), scattering occurs. By adjusting the incident angle (θ), different interplanar distances (d) align with Bragg's principle, producing a graph that shows the relationship between the angle and the intensity of diffracted peaks. This graph serves as a unique signature for each mineral, reflecting their atomic arrangements and indicating changes in mineral composition.

This technique enables researchers to examine the internal atomic structure of materials, revealing information about crystal symmetry, lattice parameters, and phase identification. Its non-destructive nature is particularly useful in materials science, geology, and pharmaceuticals, where understanding atomic arrangements is crucial for predicting material properties.

This phenomenon is governed by Bragg's law equation 3.4:

$$n\lambda = 2d \sin \theta \quad (3.4)$$

In this equation, d symbolizes the distance between atomic planes (expressed as a function of θ), n represents the diffraction order, θ indicates the crucial angle at which X-rays strike the crystalline surface, and λ denotes the X-ray wavelength. The research harnessed advanced diffraction analysis to accomplish multiple objectives related to soil composition.

This technique was implemented to elucidate the structural arrangement and atomic spacing within soil crystallites, detect and verify the existence of specific clay-based minerals, and evaluate transformations in clay mineral structures resulting from the introduction of varying concentrations of magnesium chloride solution. By using XRD, the study aimed to provide valuable insights into the nature of soil minerals and their composition, contributing to a comprehensive understanding of soil's physical properties and behavior.

To determine the mineralogical composition of the samples, X-ray Diffraction (XRD) measurements were performed. The XRD analyses were conducted using powder samples prepared from dried specimens which ground to below 75 microns. Approximately 0.5-10 grams of the powdered sample were placed into the sample holder to ensure random particle orientation.

The measurements were carried out in the XRD Laboratory of the ITU Department of Geology, using a "Bruker D8 Advance" model powder X-ray diffractometer (P-XRD). The instrument operated with a current of 40 mA and a voltage of 40 kV, utilizing Cu K α radiation without a filter. The scanning range was set from 0° to 72° 2 θ , with a step size of 2° 2 θ /min, and employed a Lynxeye detector. The resulting X-ray diffractograms were analyzed using the "Jade 6.5" data evaluation program (MDI, California, USA) with the "PDF-2" database.

3.2 Materials

3.2.1 Soil

The study's fine-grained soil samples were taken from coal mines near the Kilyos district, located north of Istanbul. A number of studies were carried out, including hydrometer analysis (ASTM, 2021a) (Figure 3.7), Atterberg limits (ASTM, 2018), and specific gravity tests (ASTM, 2023) to fully characterize the soil. Figure 3.8 shows the outcomes of crystallographic analysis, specifically the diffraction pattern produced by X-rays.

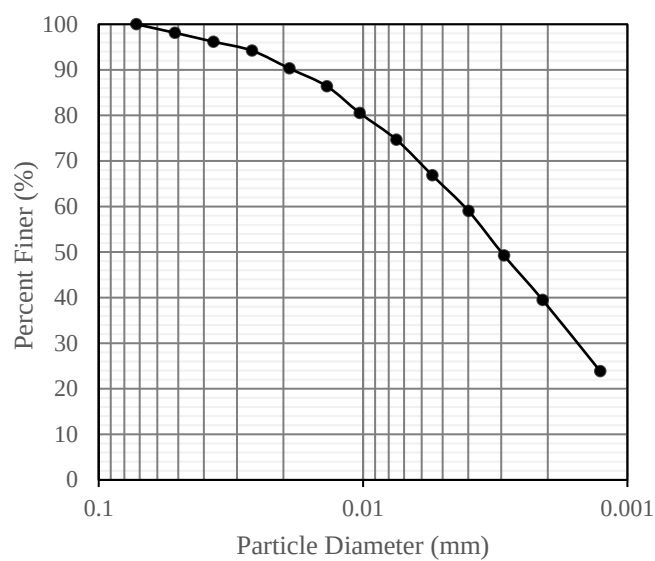


Figure 3.7 : Grain size distribution of clay sample using hydrometer analysis.

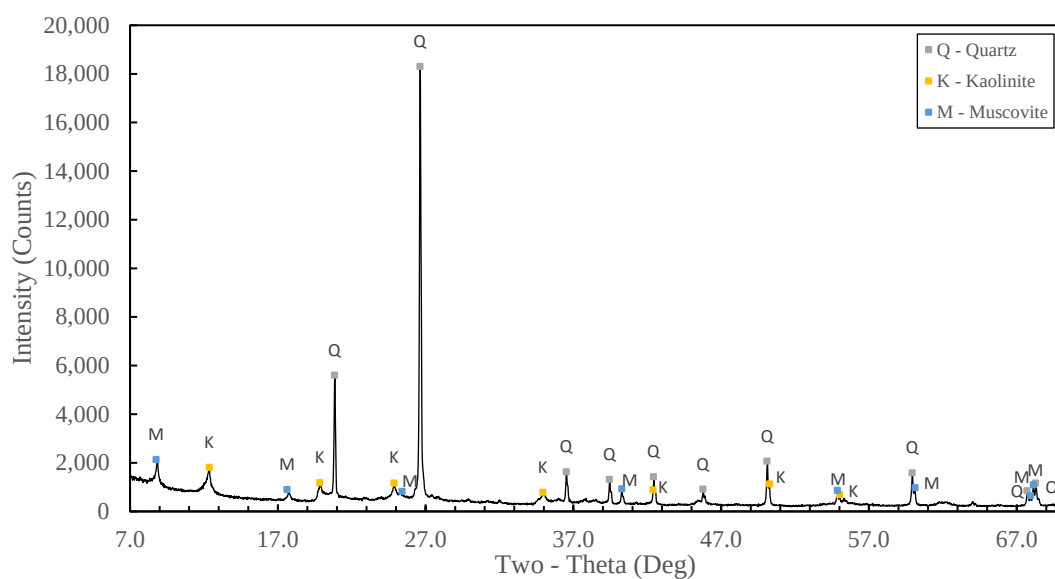


Figure 3.8 : X-ray diffraction (XRD) pattern.

This data reveals the predominant components of the sample: Quartz crystals, identifiable by distinctive peaks at specific angles (20.8°, 26.6°, 36.5°, 39.4°, 42.4°, 45.7°, 50.1°, 59.9°, 67.7°, and 68.2° on the 2 θ scale), and Kaolinite identifiable by distinctive peaks at specific angles (12.3°, 19.8°, 24.8°, 34.9°, 42.3°, 50.2°, and 55° on the 2 θ scale). Additionally, trace amounts of Muscovite is detected (8.7°, 17.6°, 25.4°, 40.2°, 54.8°, 60.1°, 67.9°, and 68.1° on the 2 θ scale). Furthermore, the sample falls under the category of low plasticity clay (CL) as per the unified soil classification system (ASTM, 2020). Table 3.1 presents the detailed physical properties of the soil used in this research. Figure 3.9 shows the clay samples prior to testing.

Table 3.1 : Physical properties of soil.

Physical Properties	Values
Silt (%)	61
Clay (%)	39
Specific gravity	2.72
Maximum dry density (kN/m ³)	17.4
Optimum water content (%)	19
Liquid limit (%)	37.2
Plastic limit (%)	19.7
Plasticity index (%)	17.5
pH	8.1



Figure 3.9 : Clay samples.

3.2.2 Polyethylene glycol (PEG 400)

Polyethylene glycol (PEG) is a versatile polyether compound used in various applications depending on its molecular weight. It is produced by polymerizing ethylene glycol, a key ingredient in antifreeze solutions. The basic structure of PEG is related to the polymer ethylene oxide, with the general formula $\text{H}(\text{OCH}_2\text{CH}_2)_n\text{OH}$, where n indicates the average number of oxyethylene units.

PEG-400, a commonly used water-soluble polymer, also dissolves in numerous organic solvents, such as aromatic hydrocarbons. Figure 3.10 also illustrates the appearance of PEG-400, which is widely used as a plasticizer to improve lubricity, enhance workability, and act as a water retention agent in products like concrete mixtures, adhesives, binders, and soldering fluxes. The hydrophilic properties and water retention ability of PEG contribute to its effectiveness in these roles. PEG is characterized by numerical suffixes that denote its average molecular weights, which can range from 300 g/mol to 1×10^7 g/mol (Bell-Young, 2018; Kumar et al., 2018).

In addition to these properties, PEG is also known for its antifreeze capabilities, as high concentrations can reduce the freezing point of a water-PEG solution to as low as -70°C (Zobrist et al., 2003). For this study, PEG was selected for its antifreeze properties and the lack of prior studies on its effects on soils, prompting this research. The results indicated that PEG had a negative impact on the soil's mechanical strength. A related study involving monoethylene glycol (MEG) similarly demonstrated a detrimental effect on soil strength. (Estabragh et al., 2016).



Figure 3.10 : Appearance of polyethylene glycol (PEG-400).

Table 3.2 displays the general characteristics of PEG400 used in this investigation, as provided by the data sheet from Söz Kimya Ltd.

Table 3.2 : General properties of PEG400.

Physical Properties	Values
Molecular Weight	401
Appearance (25 °C)	Liquid
Color (50 °C)	7
pH	6.9
Hydroxyl Value	279.5
Water Content	0.01
Dioxane	2
Free E0	4.0
Viscosity (25 °C)	92.3

3.2.3 Magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$)

Because of its exceptional water solubility, magnesium chloride is frequently utilized as an antifreeze agent in a variety of sectors. It works very well for managing dust and deicing roadways. When applied to roads in liquid or solid form, magnesium chloride prevents snow and ice from accumulating, improving safety in freezing conditions (Frisman, 2014). Figure 3.11 shows the appearance of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ flakes, the solid form used in this study. Solutions of magnesium chloride with a 21.6% concentration exhibit exceptional stability, withstanding temperatures as low as -33°C (Minsk et al., 1996). Figure 3.12 illustrates the freezing points of MgCl_2 at different concentrations, demonstrating the effectiveness of MgCl_2 as an antifreeze in various environmental conditions (Minsk et al., 1996). This ability makes magnesium chloride an invaluable material for applications in freezing environments.



Figure 3.11 : The appearance of 3.2.3 magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) flakes.

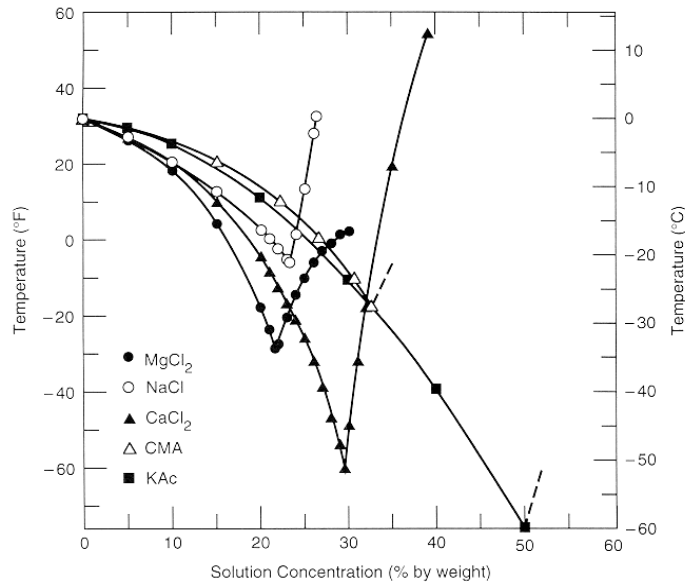


Figure 3.12 : Freezing point vs. concentration of five chemicals including MgCl₂, (Minsk et al., 1996).

In this study, magnesium chloride hexahydrate (MgCl₂·6H₂O) was used. MgCl₂·6H₂O is a solid flake that contains water molecules as part of its crystalline structure. Upon dissolving in water, the crystalline structure is lost, and the water molecules that were part of the hexahydrate are released. As a result, after dissolution, the solution is referred to as simply MgCl₂ solution rather than MgCl₂·6H₂O solution, as it contains only MgCl₂ ions dissolved in water.

Although the effects of MgCl₂ on soil's mechanical properties, particularly its role as a stabilizer, have been explored in several studies—consistently showing that MgCl₂ significantly enhances soil's engineering properties (Bahloul et al., 2019; Muhammad et al., 2018; Tang et al., 2021; Turkoz et al., 2014)—there has been no research specifically investigating the use of MgCl₂ as an antifreeze in soils subjected to freeze-thaw cycles (F-T cycles). At the time this study was published, no research had introduced an antifreeze agent for soil, as most studies focused solely on stabilizing the soil by increasing its strength against F-T cycles. However, even when stabilized, soils often lose strength after several F-T cycles.

This gap in the research led to the aim of this study, which was to find a solution that not only stabilizes the soil but also prevents the strength loss that typically occurs due to repeated freeze-thaw (F-T) cycles. Through our investigation, MgCl₂ emerged as a potential solution to address this problem and represents a significant step toward filling this gap in the existing literature.

The sample used in this study was sourced from Dead Sea Works Ltd., and its general properties are detailed in Table 3.3, according to the manufacturer's data sheet.

Table 3.3 : General properties of MgCl₂

Physical Properties	Values
Magnesium Chloride (MgCl ₂)	Min 46.5%
Calcium Chloride (CaCl ₂)	Max 2.2%
Sodium Chloride (NaCl)	Max 0.9%
Potassium Chloride (KCl)	Max 0.6%
Water of Crystallization	Balance to 100%
Sulfates (SO ₄)	Max 0.025%
Iron (Fe)	Max 5 ppm
Mercury (Hg)	Max 0.2 ppm
Lead (Pb)	Max 0.1 ppm
Arsenic (As)	Max 0.1 ppm
Cadmium (Cd)	Max 0.1 ppm
pH of aqueous Solution	8.0 - 9.5

3.3 Summary of Experimental Test Plan and Procedures

The following section provides a concise overview of the experimental procedures and conditions used throughout the study. A detailed summary of the test samples, treatments, and conditions is presented in Table 3.4. This table outlines the key steps undertaken during the investigation, serving as a reference for the tests conducted in this research.

Table 3.4 : Summary of experimental procedures and conditions for test samples.

Step No.	Test/Procedure	Material(s) Used	Treatment/Concentration	Parameters Measured	Curing Time & F-T Conditions
1	Soil Characterization	Clay	N/A	Particle size distribution, Atterberg limits, specific gravity	N/A
2	Atterberg Limits Tests	Clay + PEG	PEG (0%, 10%, 20%, 30%, 40%, 50%)	LL, PL, and PI	N/A
3	Compaction Tests	Clay + PEG	PEG (0%, 10%, 20%, 30%, 40%, 50%)	Maximum Dry Density, Optimum Water Content	N/A
4	UCS Test (PEG)	Clay + PEG	PEG (0%, 10%, 20%, 30%, 40%, 50%)	Stress-strain behavior, UCS values	N/A
5	F-T Cycle Test (PEG)	Clay + PEG	PEG (0%, 10%, 20%, 30%, 40%, 50%)	UCS before and after one freeze-thaw cycle	Cured for 1 day; F-T at -20°C
6	UCS Test (PEG-Cement)	Clay + PEG + Cement	PEG (0%, 1%, 3%, 5%, 10%, 20%) + 7% Cement	Stress-strain behavior, UCS values	N/A
7	F-T Cycle Test (PEG-Cement)	Clay + PEG + Cement	PEG (0%, 1%, 3%, 5%, 10%, 20%) + 7% Cement	UCS before and after one freeze-thaw cycle	Cured for 1 day; F-T at -5°C
8	Freezing Point Determination	Clay + MgCl ₂	MgCl ₂ (4%, 9%, 14%)	Freezing point measurement using a thermometer	N/A
9	Atterberg Limits Tests	Clay + MgCl ₂	MgCl ₂ (0%, 4%, 9%, 14%)	LL, PL, and PI	N/A
10	Compaction Tests	Clay + MgCl ₂	MgCl ₂ (0%, 4%, 9%, 14%)	Maximum Dry Density, Optimum Water Content	N/A
11	UCS Test	Clay + MgCl ₂	MgCl ₂ (0%, 4%, 9%, 14%)	Stress-strain behavior, UCS values	N/A
12	F-T Cycle Test	Clay + MgCl ₂	MgCl ₂ (0%, 4%, 9%, 14%)	UCS before and after freeze-thaw 1,3,5,7 cycles	Cured 0, 7, 28 days; F-T at -10°C and -20°C
13	Volume change test	Clay + MgCl ₂	MgCl ₂ (0%, 4%, 9%, 14%)	Volume change after each F-T cycle	N/A
14	Microstructural Analysis (SEM, XRD, FTIR)	Clay + MgCl ₂	MgCl ₂ (0%, 14%)	SEM imaging, XRD diffraction, FTIR spectroscopy	N/A

4. RESULTS AND DISCUSSION

This chapter presents and analyzes the experimental results on the effects of Polyethylene Glycol (PEG) and Magnesium Chloride (MgCl_2) on the mechanical and physical properties of clay, particularly under freeze-thaw conditions. Key findings related to soil characterization, Atterberg limits, compaction properties, unconfined compressive strength (UCS), freeze-thaw resistance, volume changes, and microstructural analysis are discussed. These results provide valuable insights into the effect of these additives in soil stabilization, particularly highlighting the potential applications of MgCl_2 in geotechnical engineering to prevent the negative effects of freeze-thaw cycles, mitigate strength loss, and reduce volume changes. The detailed test results for PEG-treated soils are presented and discussed in section 4.1, while the findings for MgCl_2 -treated soils are thoroughly examined and discussed in section 4.2.

4.1 Test Results for PEG-Treated Clay

4.1.1 Soil characterization

In this thesis, the soil sample described in detail in Section 3.2.1 has been used for all conducted tests. This ensures consistency and reliability across the various analyses performed, including hydrometer analysis, Atterberg limits, specific gravity tests, and crystallographic analysis. By using the same sample throughout, the results are directly comparable, providing a comprehensive and accurate characterization of the soil's properties.

4.1.2 Atterberg limits tests

As seen in Figure 4.1, this study examined the effects of various PEG solution concentrations (10%, 20%, 30%, 40%, and 50%) on the soil's plastic limit (PL), plasticity index (PI), and liquid limit (LL). Results indicated a steady rise in these parameters with increasing PEG concentration. Notably, soil mixtures treated with a 50% PEG solution experienced significant increases: a 48.6% boost in the liquid limit, a 21% uplift in the plastic limit, and a 77.7% surge in the plasticity index. Detailed outcomes are presented in Table 4.1. The rising trend in Atterberg limits with higher

PEG concentrations is primarily due to PEG's hydrophilic properties, its interaction with soil particles, increased viscosity, and the resulting modification of soil structure (Kumar et al., 2018).

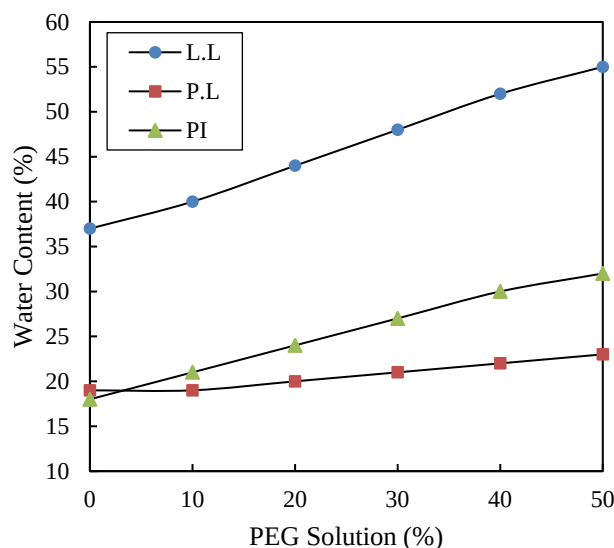


Figure 4.1 : Effect of PEG on Atterberg limits of soil.

Table 4.1 : Atterberg limit test results for various concentrations of PEG solution.

PEG Solution Concentration (%)	L.L. (%)	P.L. (%)	P.I. (%)
0	37	19	18
10	40	19	21
20	44	20	24
30	48	21	27
40	52	22	30
50	55	23	32

4.1.3 Compaction tests

As illustrated in Figure 4.2, the findings reveal a consistent change in the compaction parameters, specifically the maximum dry density and optimum water content, as the concentration of PEG increases. The graph shows that with the increasing concentration of PEG solution, there is a noticeable decrease in the maximum dry density of the soil. For instance, untreated soil (0% PEG) achieves a peak dry density of approximately 17.5 kN/m³ at an optimum water content of around 18%. In contrast, soil treated with a 50% PEG solution reaches a lower peak dry density of about 15 kN/m³ at a higher optimum water content of approximately 26%, Table 4.2.

The changes in maximum dry density and optimum water content with increasing PEG concentrations highlight the significant impact of PEG on soil compaction characteristics. This effect is primarily driven by PEG's hydrophilic properties, its interaction with soil particles, increased viscosity, and the resulting modification of soil structure.

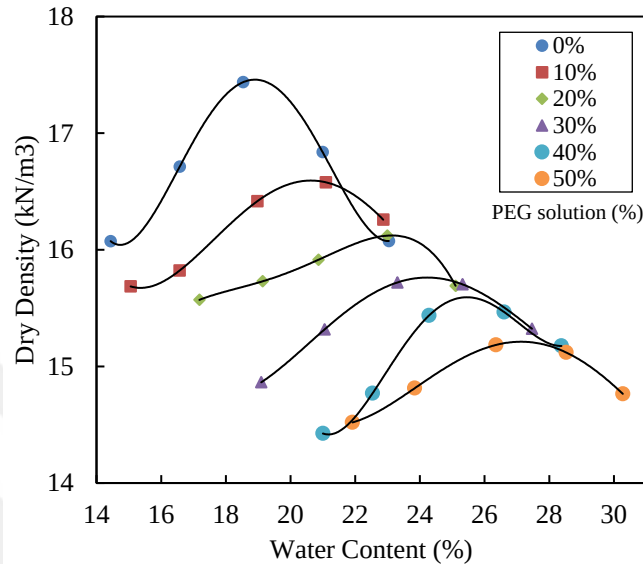


Figure 4.2 : The impact of PEG solution on the maximum dry density and water content of clay.

Table 4.2 : OWC and MDD of soil with different PEG solution concentrations.

PEG Solution Concentration (%)	Optimum Water Content (%)	Maximum Dry Density (kN/m ³)
0	19	17.5
10	21	16.6
20	23	16.1
30	24	15.8
40	26	15.6
50	28	15.2

4.1.4 Unconfined compression tests

4.1.4.1 Results for clay-PEG mixture

The unconfined compressive strength (UCS) of clay-PEG mixtures, as depicted in the accompanying stress-strain curves, reveals a consistent decline in UCS as the concentration of PEG increases (Figure 4.3).

The stress-strain curves show that the peak stress (the unconfined compressive strength), decreases progressively with higher concentrations of PEG solution.

The untreated soil (0% PEG) achieves the highest UCS, while the soil treated with a 50% PEG solution exhibits the lowest UCS. Detailed results are summarized in Table 4.3.

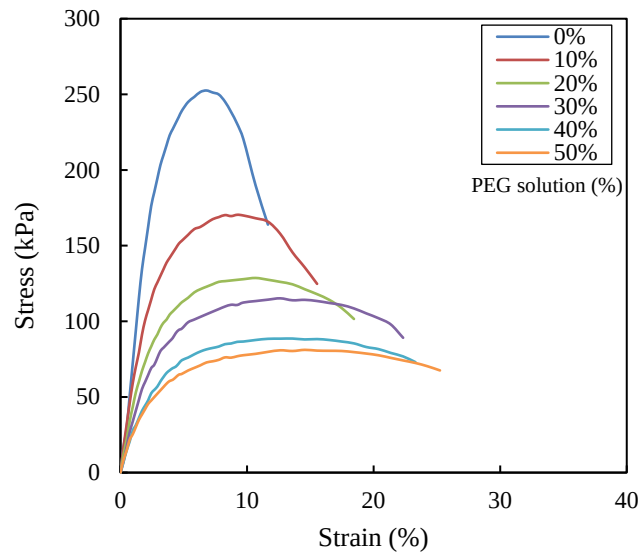


Figure 4.3 : Stress-strain curves for untreated and PEG-treated clay samples at varying concentrations.

Table 4.3 : UCS results for clay samples treated with varying PEG concentrations.

PEG Solution Concentration (%)	Strain (%)	Stress
0	6.80	252.54
10	9.22	170.40
20	10.68	128.64
30	12.62	115.18
40	13.59	88.59
50	14.56	81.13

Figure 4.4 presents the UCS results for clay-PEG mixtures after one freeze-thaw cycle at -20°C. The graph shows that, although the overall trend of strength loss aligns with previous findings, the clay samples exhibit a more pronounced reduction in strength after undergoing one freeze-thaw cycle (Table 4.4).

Table 4.5 further highlights the stress differences between soils subjected to freeze-thaw cycles and those that did not undergo any cycles. Notably, the samples treated with 40% and 50% PEG solution showed no significant strength loss after one freeze-thaw cycle, indicating that PEG functions effectively as an antifreeze agent. This antifreeze capability prevents strength degradation by minimizing ice formation and reducing internal stresses caused by freeze-thaw cycles.

However, despite this beneficial effect in mitigating freeze-thaw damage, the overall unconfined compressive strength (UCS) of the clay samples treated with PEG still decreases when compared to untreated samples.

This reduction in unconfined compressive strength (UCS) is likely due to the hydrophilic nature of polyethylene glycol (PEG). PEG has a strong affinity for water, which results in an increased water content within the soil. This excess moisture alters the viscosity of the soil, making it more fluid, and modifies the interactions between soil particles. Consequently, these changes negatively affect the soil's mechanical structure, reducing its overall strength and stability. The PEG solution infiltrates the soil matrix, forming a more lubricated structure, which weakens the bonding between soil particles. These changes result in a loss of soil rigidity and strength, despite its ability to resist freeze-thaw damage.

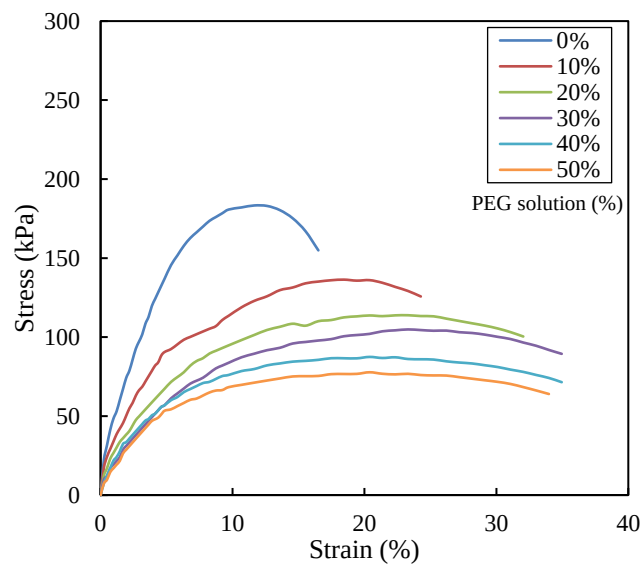


Figure 4.4 : Stress-strain curves for untreated and PEG-treated samples after one F-T cycle at -20°C.

Table 4.4 : UCS results for clay-PEG mixture after one F-T cycle at -20°C.

PEG Solution Concentration (%)	Strain (%)	Stress(kPa)
0	11.65	183.32
10	18.45	136.37
20	23.30	113.83
30	23.30	104.85
40	20.39	87.45
50	20.39	80.43

Table 4.5 : Comparative analysis of stress reduction in clay-PEG mixtures after one F-T cycle at -20°C.

PEG Solution Concentration (%)	Stress Difference	Stress loss (%)
0	69.22	27
10	34.03	20
20	14.81	12
30	10.33	9
40	1.14	1
50	0.70	1

As illustrated in Figure 4.5, a comparison between two samples (sample a, treated with 20% PEG, and sample b, treated with 20% PEG but subjected to one freeze-thaw cycle) shows clear visual evidence of this effect. Sample b exhibits significant bulging compared to sample a, indicating that the freeze-thaw cycle leads to structural deformation despite PEG treatment. Therefore, while higher PEG concentrations offer some protection against freeze-thaw effects, they simultaneously compromise the mechanical integrity of the soil, reducing its load-bearing capacity.

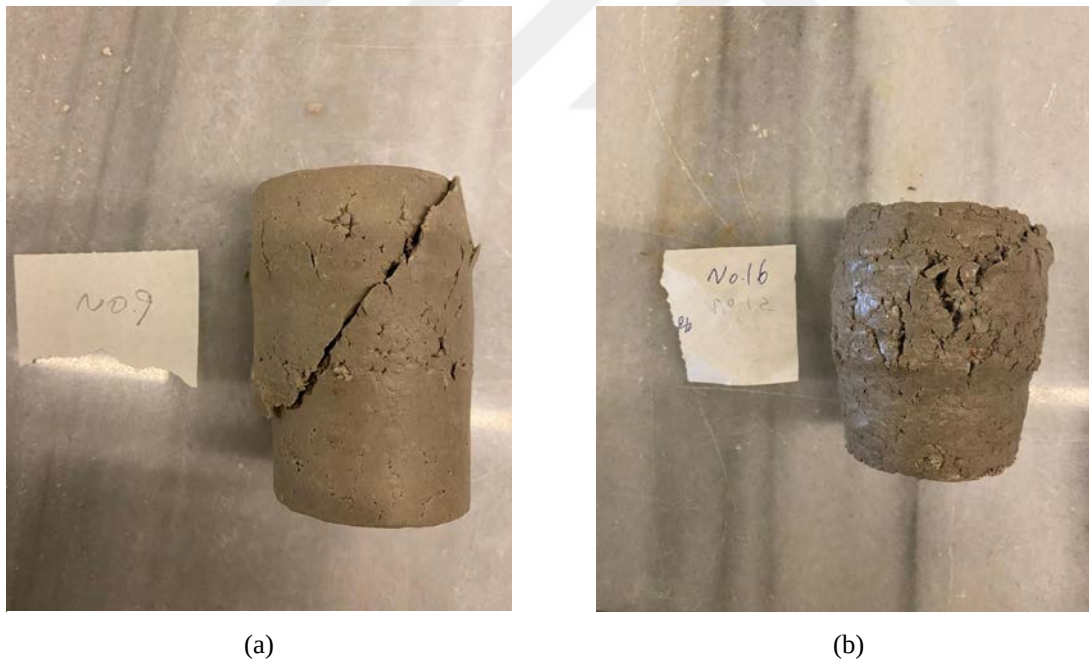


Figure 4.5 : (a) 20% PEG-treated sample, (b) 20% PEG-treated sample after one freeze-thaw cycle.

4.1.4.2 Results for the clay-PEG-cement mixture

After observing that PEG significantly reduced the strength of clay even before undergoing freeze-thaw cycles, we hypothesized that adding cement might help

counteract this strength loss. The aim was to investigate whether cement could mitigate the weakening effect of PEG while also leveraging PEG's antifreeze properties to prevent the clay-cement mixture from freezing. To test this hypothesis, we fixed the cement concentration at 7% and varied the PEG concentrations at 1%, 3%, 5%, 10%, and 20%.

Figure 4.6 and Table 4.6 show the stress-strain behavior of these mixtures. Untreated clay exhibits the lowest UCS (210.62 kPa). The addition of 7% cement significantly enhanced the UCS to 1298.45 kPa, indicating that cement effectively strengthens the clay. With adding of 1% and 3% PEG, the UCS increased further, reaching a peak of 1413.32 kPa at 3% PEG.

However, beyond 5% PEG, the UCS began to decline, with the lowest UCS observed at 20% PEG (1063.73 kPa). This suggests that small amounts of PEG improve the clay-cement mixture's strength, but higher concentrations diminish it, likely due to PEG's hydrophilic nature, which increases water absorption and alters the soil structure.

In conclusion, the optimal mixture for maximizing UCS was observed with 3% PEG and 7% cement. Beyond this concentration, the adverse effects of PEG on strength became more apparent, highlighting the importance of balancing PEG content to maintain both antifreeze benefits and mechanical strength.

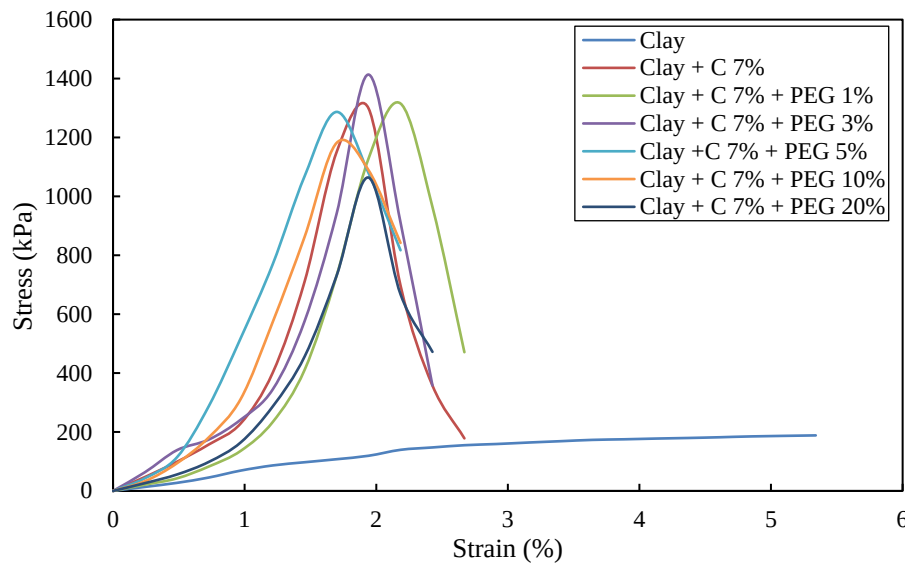


Figure 4.6 : Stress-strain curves for untreated and PEG-cement-treated clay samples at varying concentrations.

Table 4.6 : UCS results for clay samples treated with cement and varying PEG concentrations.

PEG Solution Concentration (%)	Strain (%)	Stress (kPa)
Clay	10.68	210.62
Clay + 7% cement	1.94	1298.45
Clay + 7% cement + 1% PEG	2.18	1315.17
Clay + 7% cement + 3% PEG	1.94	1413.32
Clay + 7% cement + 5% PEG	1.7	1286.65
Clay + 7% cement + 10% PEG	1.7	1181.51
Clay + 7% cement + 20% PEG	1.94	1063.73

To assess the behavior of clay-PEG-cement mixtures under freeze-thaw (F-T) conditions at -20°C, samples were subjected to one F-T cycle. The UCS after one F-T cycle for untreated clay was 161.07 kPa, while adding 7% cement raised it to 765.98 kPa, as shown in Figure 4.7 and Table 4.7. However, cement-treated samples lost up to 41% of their initial strength after the F-T cycle, as detailed in Table 4.8.

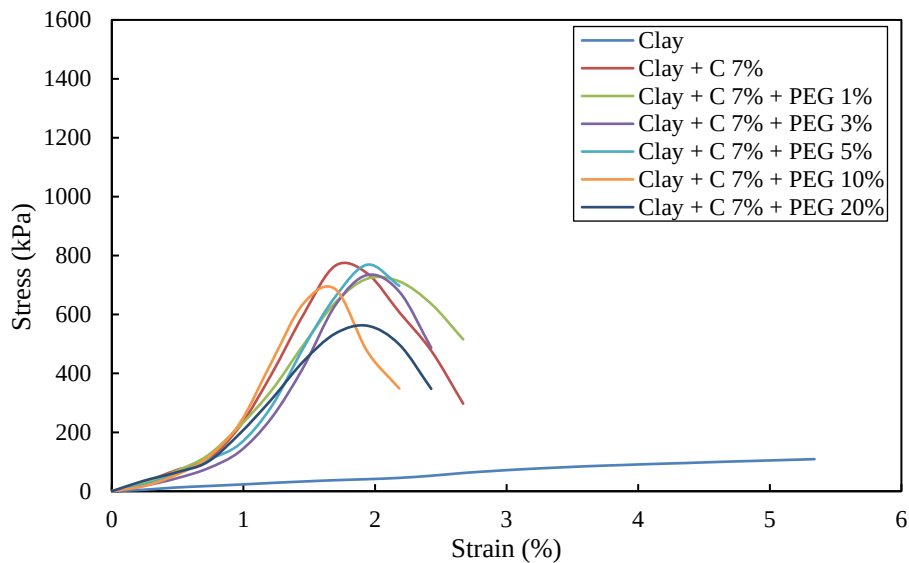


Figure 4.7 : Stress-strain curves for untreated and PEG-cement-treated clay samples after one F-T cycle at -20°C.

The addition of PEG had varying impacts on the UCS of the clay-cement mixtures. A PEG concentration of 5% was found to be optimal, resulting in the highest UCS of 769.08 kPa after one freeze-thaw cycle, as shown in Table 4.7. However, based on the results in Table 4.8, PEG failed to demonstrate a significant antifreeze effect on the clay-cement mixtures. It became evident that PEG did not work effectively with this combination, as even increasing PEG concentrations could not prevent the strength loss after the freeze-thaw cycle.

Table 4.7 : UCS results for clay-PEG- cement mixture after one F-T cycle at -20.

PEG Solution Concentration (%)	Strain (%)	Stress (kPa)
Clay	13.59	161.07
Clay + 7% cement	1.70	765.98
Clay + 7% cement + 1% PEG	1.94	721.64
Clay + 7% cement + 3% PEG	1.94	734.13
Clay + 7% cement + 5% PEG	1.94	769.08
Clay + 7% cement + 10% PEG	1.70	685.88
Clay + 7% cement + 20% PEG	1.94	561.83

Table 4.8 : Comparative analysis of stress reduction in clay-PEG-cement mixtures after one F-T cycle at -20°C.

PEG Solution Concentration (%)	Stress Difference	Stress loss (%)
Clay	49.55	24
Clay + 7% cement	532.47	41
Clay + 7% cement + 1% PEG	593.53	45
Clay + 7% cement + 3% PEG	679.19	48
Clay + 7% cement + 5% PEG	517.57	40
Clay + 7% cement + 10% PEG	495.63	42
Clay + 7% cement + 20% PEG	501.90	47

In conclusion, although PEG showed some benefits, neither PEG nor cement alone could preserve the mixture's strength under freeze-thaw conditions at -20°C. Therefore, alternative stabilizing agents are needed to prevent strength loss.

An example of PEG-cement-treated clay is shown in Figure 4.8, where the sample treated with 7% cement and 20% PEG.

**Figure 4.8 :** PEG-cement-treated clay sample with 7% cement and 20% PEG.

4.2 Test Results for MgCl_2 -Treated Clay

4.2.1 Soil characterization

In this section, the same soil sample described in detail in Section 3.2.1, which was also used for the PEG tests, was used for the MgCl_2 tests. This ensures consistent results across all analyses, including hydrometer analysis, Atterberg limits, specific gravity tests, and crystallographic analysis, allowing for direct comparison of the effects of MgCl_2 on the soil's properties.

4.2.2 Freezing point determination

Following the initial tests described in Section 3.1.4, we conducted detailed thermometer-based measurements to accurately determine the freezing points of MgCl_2 -treated clay samples. Samples treated with 4%, 9%, and 14% MgCl_2 solutions, as well as an untreated control, were subjected to a freezing environment at -27°C , and temperature changes were continuously recorded using thermometers placed within the samples.

To determine the freezing point from the thermometer data, we analyzed the slope of each temperature curve (Figure 4.9). The freezing point is where the curve flattens before descending again, indicating the release of latent heat during the phase change from liquid to solid. Once fully frozen, the temperature drops rapidly, forming a steeper slope after.

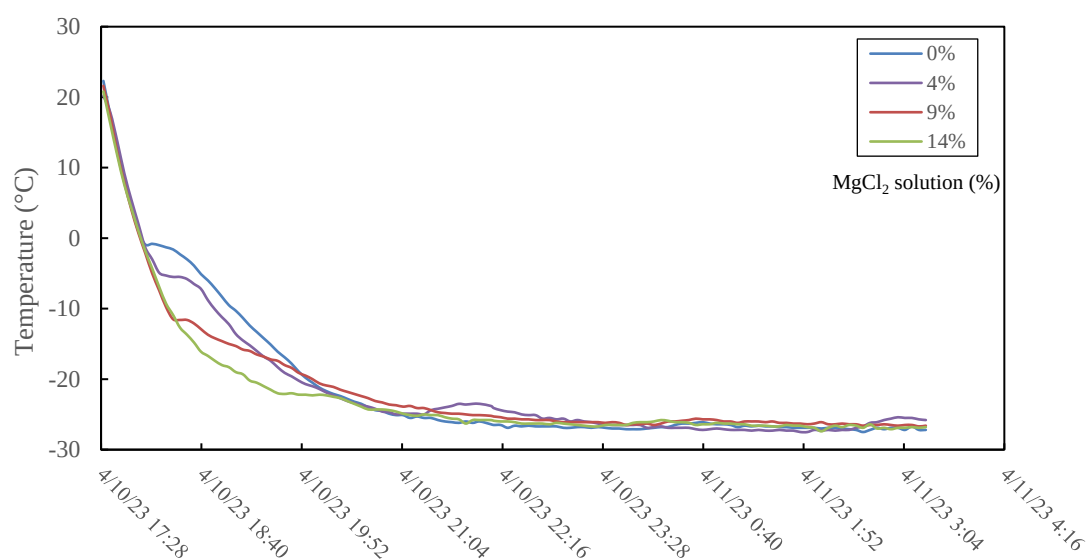


Figure 4.9 : Temperature profiles of untreated and MgCl_2 -treated clay samples.

In the untreated sample, this flattening occurred around -1°C . The 4% MgCl_2 -treated sample froze at approximately -5°C , showing a delay compared to the untreated sample but still freezing at lower temperatures. The 9% MgCl_2 -treated sample froze near -12°C , providing a more effective delay, while the 14% sample showed the best antifreeze performance, freezing near -23°C . The freezing points for each concentration are summarized in Table 4.9.

Table 4.9 : Freezing points of untreated and MgCl_2 -treated samples.

MgCl_2 Solution Concentration (%)	Freezing point
0	-1°C
4	-5°C
9	-12°C
14	-23°C

These results confirm that the freezing point decreases with increasing MgCl_2 concentration. Based on these findings, 4%, 9%, and 14% concentrations were selected for further testing in freeze-thaw cycles at both -10°C and -20°C . This selection allowed for a systematic comparison of the performance of MgCl_2 -clay mixtures under varying temperature conditions, providing insight into the antifreeze effectiveness of each concentration and its potential for use in mitigating freeze-thaw damage in soils.

4.2.3 Atterberg limits tests

This investigation delved into the impacts of varying concentrations of MgCl_2 solution (4%, 9%, and 14%) on the liquid limit (LL), plastic limit (PL), and plasticity index (PI) of the soil, Figure 4.10, Table 4.10. The findings illustrate a consistent decrease in these parameters as the concentration of MgCl_2 increases. Particularly noteworthy reductions were observed in soil blends treated with a 14% MgCl_2 solution, showcasing decreases of 14% in the liquid limit, 9% in the plastic limit, and 19% in the plasticity index. These outcomes are in concordance with prior investigations conducted by Venkatabor Rad (Venkatabor Rad & Rechi, 1977), indicating that the decline is likely attributable to a thinning of the diffuse double layer (DDL). This phenomenon is believed to be instigated by the cation exchange with divalent magnesium ions and a consequent escalation in electrolyte concentration, given the complete solubility of MgCl_2 in water.

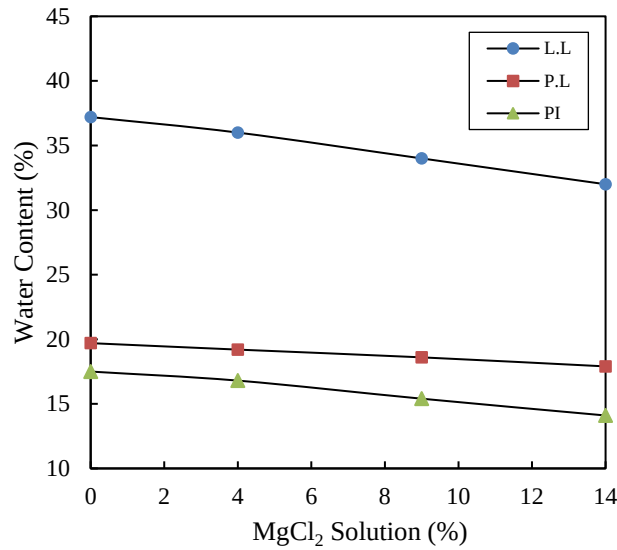


Figure 4.10 : Effect of MgCl₂ solution on the Atterberg limits of soil.

Table 4.10 : Atterberg limit test results for various concentrations of MgCl₂ solution.

MgCl ₂ Solution Concentration (%)	L.L. (%)	P.L. (%)	P.I. (%)
0	37.2	19.7	17.5
4	36	19.2	16.8
9	34	18.6	15.4
14	32	17.9	14.1

4.2.4 Compaction tests

The impact of varying concentrations of MgCl₂ solution on the compaction characteristics of the soil is depicted in Figure 4.11 and summarized in Table 4.11. It is observed that the addition of MgCl₂ solution leads to an enhancement in the maximum dry density (MDD) while reducing the optimum water content (OWC), particularly evident at higher MgCl₂ solution concentrations. These findings are consistent with observations made by Randolph (Randolph, 1997). The augmentation in dry density is ascribed to particle flocculation and aggregation, consequent to rapid cation exchange occurring within the soil- MgCl₂ solution amalgam. Notably, the application of a 14% MgCl₂ solution elevated the MDD to 17.89 kN/m³ from the initial value of 17.4 kN/m³ observed in untreated soil while concurrently diminishing the OWC from 19% in untreated soil to 17.7%. These results indicate that augmenting the concentration of MgCl₂ solution and thus accelerating the rate of cation exchange

within the soil- MgCl_2 solution mixture diminishes the capacity of clay surfaces to absorb water, thereby resulting in an observed elevation in dry density values.

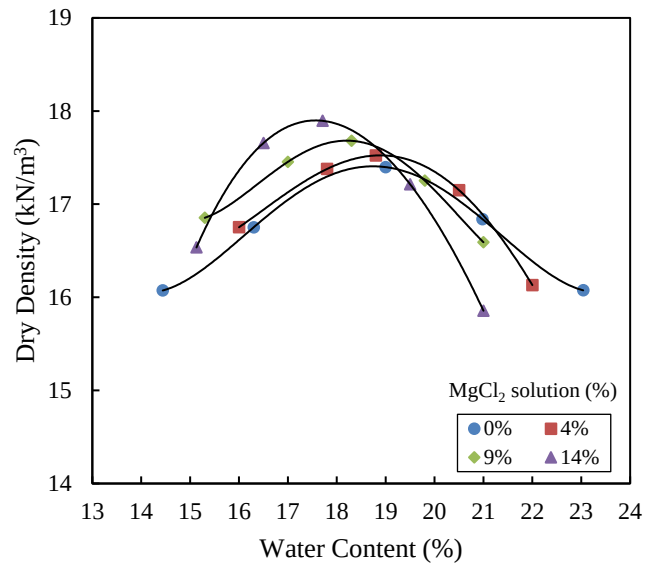


Figure 4.11 : The impact of MgCl_2 solution on the dry density and water content of soil.

Table 4.11 : OWC and MDD of soil with different MgCl_2 solution concentrations.

MgCl ₂ Solution Concentration (%)	Optimum Water Content (%)	Maximum Dry Density (kN/m ³)
0	19	17.4
4	18.9	17.52
9	18.3	17.68
14	17.71	17.89

4.2.5 Unconfined compression tests

The unconfined compression test was executed to evaluate the efficacy of magnesium chloride (MgCl_2) solution in enhancing the compressive strength of the soil. Illustrated in Figure 4.12 is the stress-strain behavior of both untreated and MgCl_2 -treated soil samples, encompassing curing periods of 7 and 28 days, as well as samples without any curing. The findings indicate that the incorporation of MgCl_2 solution generally results in a notable augmentation of soil strength across all curing durations, with this improvement being directly correlated to the concentration of the MgCl_2 solution. Moreover, the test outcomes suggest that higher concentrations of MgCl_2 lead to more significant gains in compressive strength, underscoring the additive's role in stabilizing soil over time, particularly after extended curing periods.

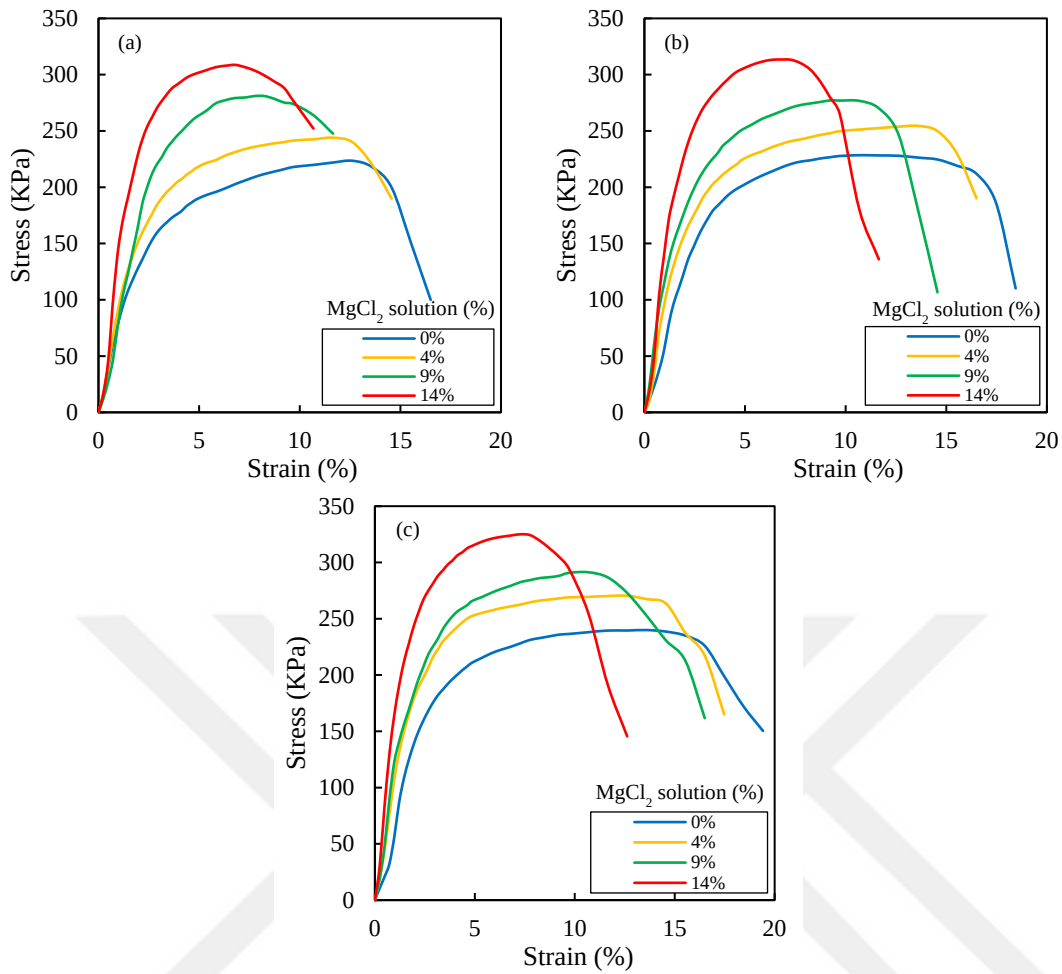


Figure 4.12 : Stress-strain diagrams for untreated and treated samples at different curing periods: (a) uncured, (b) cured for 7 days, and (c) cured for 28 days.

Figure. 4.13a delineates the enhancements in Unconfined Compressive Strength (UCS) with increasing $MgCl_2$ solution concentration, elucidating the impact of curing time on treated samples with varying concentrations. For instance, untreated samples exhibited UCS enhancements of 9%, 26%, and 38% with concentrations of 4%, 9%, and 14% $MgCl_2$ solution, respectively, leading to UCS values escalating from 223.5 kPa to 244 kPa, 281 kPa, and 308 kPa. Furthermore, the data suggests a marginal augmentation in UCS values associated with prolonged curing periods. For instance, a 28-day curing period of soil treated with a 14% $MgCl_2$ solution resulted in a 5% enhancement in UCS values. Additionally, as depicted in Figure 4.13b, an escalation in $MgCl_2$ concentration diminishes failure strain for treated soils. Consequently, soils treated with higher levels of $MgCl_2$ exhibit reduced deformability.

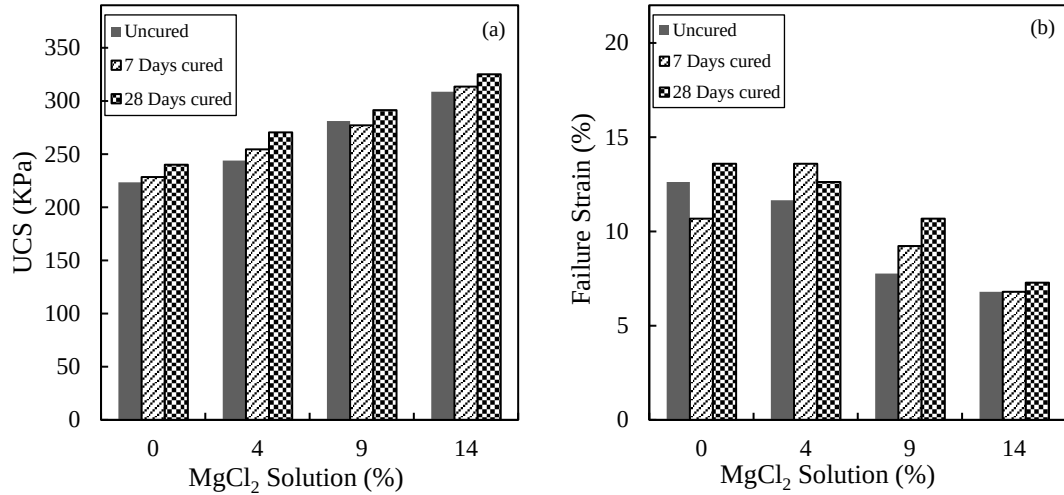


Figure 4.13 : Effect of $MgCl_2$ solution on: (a) unconfined compressive strength (UCS) values and (b) strain at failure.

Tables 4.12 and 4.13 present the maximum UCS and strain values at failure for both cured and uncured $MgCl_2$ -treated samples.

Table 4.12 : Maximum UCS values of cured and uncured $MgCl_2$ Solution samples.

$MgCl_2$ Solution Concentration (%)	Uncured (kPa)	7 days cured (kPa)	28 days cured (kPa)
0%	223.51	228.48	239.96
4%	244.00	254.49	270.44
9%	281.05	277.07	291.29
14%	308.70	313.45	325.05

Table 4.13 : Maximum strain values of cured and uncured $MgCl_2$ Solution samples at failure.

$MgCl_2$ Solution Concentration (%)	Uncured (%)	7 days cured (%)	28 days cured (%)
0%	12.62	10.68	13.59
4%	11.65	13.59	12.62
9%	7.77	9.22	10.68
14%	6.80	6.80	7.28

4.2.6 Freeze-thaw cycles tests

To investigate the antifreeze and stabilizing effects of $MgCl_2$ on clay, Frost-Thaw (F-T) cycle tests were conducted to assess its impact on the stress-strain behavior of treated soils. These tests were carried out at $-10^{\circ}C$ and $-20^{\circ}C$ on samples cured for 28 days to determine if $MgCl_2$ concentrations require adjustment for different temperatures. The aim was to understand how varying concentrations of $MgCl_2$

maintain soil integrity under both mild and extreme cold conditions, providing insights into their effectiveness against thermal stresses. Additionally, an index was established to evaluate the durability of treated samples, calculated as the ratio of the unconfined compressive strength (UCS) after freeze-thaw cycles (UCS_i) to UCS after curing (UCS_0), as shown in equation 4.1 (Shibi & Kamei, 2014). This metric is essential for assessing the stability of the material post-treatment under F-T conditions.

$$DI = \frac{UCS_i}{UCS_0} \quad (4.1)$$

4.2.6.1 Uncured samples:

Results at -10°C:

Figure 4.14 shows the effect of freeze-thaw (F-T) cycles at -10°C on the stress-strain behavior of untreated and treated soil samples. The data indicate a significant reduction in strength for untreated samples exposed to F-T cycles (Figure 4.14a). In contrast, Figures 4.14c and 4.14d show that treating samples with 9% and 14% MgCl_2 solutions significantly mitigates this strength loss, maintaining the stress-strain behavior. A lower concentration of 4% MgCl_2 also demonstrates a noticeable, albeit smaller, antifreeze effect by slightly reducing strength loss and exhibiting a lesser decrease in strength after seven cycles compared to untreated samples (Figure 4.14b).

The variation in unconfined compressive strength (UCS) with the addition of MgCl_2 solutions indicates that the stabilizer enhances soil strength during F-T cycles (Figure 4.15a). For instance, after three F-T cycles, the untreated soil has a strength of 130.56 kPa, whereas soils treated with 4%, 9%, and 14% MgCl_2 solutions exhibit increased strengths of 185.91 kPa, 285.54 kPa, and 301.10 kPa, respectively. The effectiveness of the 4% MgCl_2 solution in enhancing soil strength diminishes over successive cycles. However, the strength gains from 9% and 14% solutions remain substantial even as the cycles progress.

Figure 4.15b shows the changes in the durability index (DI). Both untreated and 4% MgCl_2 -treated samples experience a significant decrease in DI, particularly after the first F-T cycle. This underscores the importance of the initial F-T cycle in impacting soil durability. After seven cycles, the DI drops to 0.38 for untreated samples and to 0.50 for those treated with a 4% MgCl_2 solution, highlighting the severe effects of F-T cycles on soil durability. In contrast, the DI for samples treated with 9% and 14% MgCl_2 remains stable before and after the seven F-T cycles.

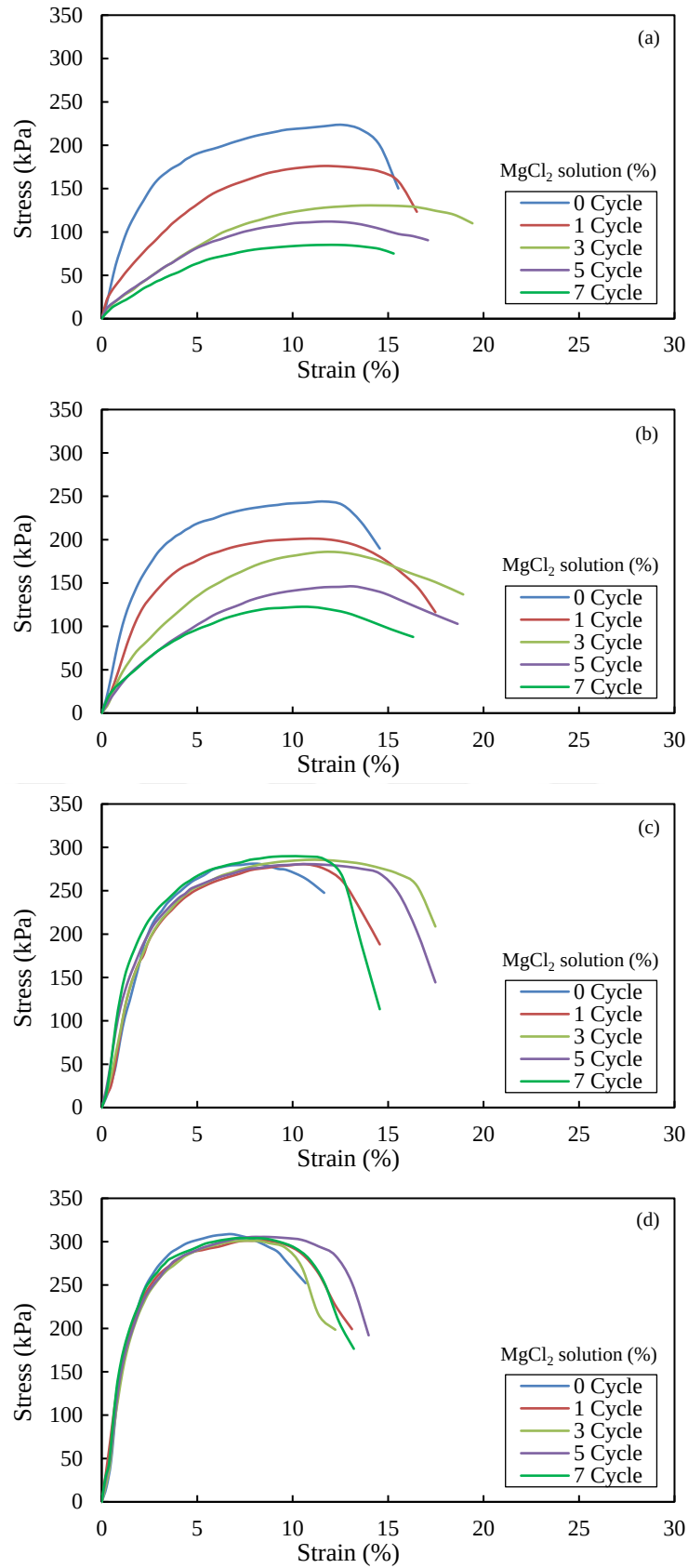


Figure 4.14 : Stress-strain relationships at -10°C for uncured samples with different MgCl_2 treatments: (a) Untreated, (b) 4% MgCl_2 , (c) 9% MgCl_2 , (d) 14% MgCl_2 .

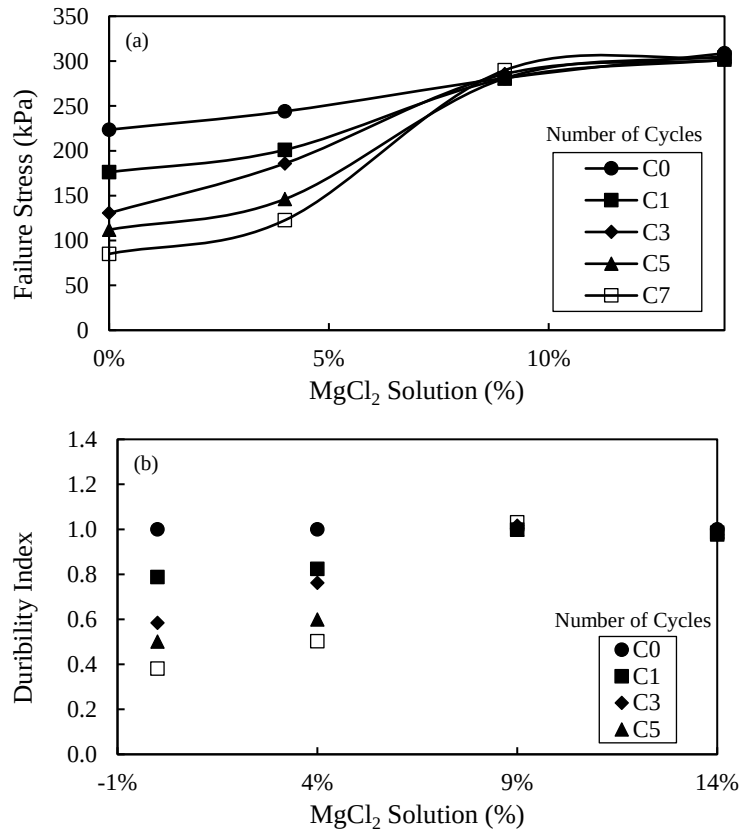


Figure 4.15 : The impact of MgCl₂ solution at -10°C on: (a) UCS and (b) durability index values for uncured samples.

Detailed experimental results are presented in Table 4.14 and Table 4.15.

Table 4.14 : Failure stress at -10°C for uncured samples.

F-T cycles	0% MgCl ₂ solution (kPa)	4% MgCl ₂ solution (kPa)	9% MgCl ₂ solution (kPa)	14% MgCl ₂ solution (kPa)
0	223.51	244.00	281.05	308.70
1	176.12	201.05	280.55	301.55
3	130.56	185.91	285.54	301.10
5	112.01	146.18	280.74	305.45
7	85.10	122.63	289.79	304.05

Table 4.15 : Durability Index of uncured samples at -10°C .

F-T cycles	0% MgCl ₂ solution	4% MgCl ₂ solution	9% MgCl ₂ solution	14% MgCl ₂ solution
0	1.00	1.00	1.00	1.00
1	0.79	0.82	1.00	0.98
3	0.58	0.76	1.02	0.98
5	0.50	0.60	1.00	0.99
7	0.38	0.50	1.03	0.98

Results at -20°C:

To evaluate the effectiveness of MgCl_2 solutions as antifreeze agents, freeze-thaw (F-T) tests were conducted at -20°C . The stress-strain behavior of untreated and treated samples over 7 F-T cycles is shown in Figure 4.16. Untreated samples and those with 4% MgCl_2 showed significant strength loss (Figures 4.16a, 4.16b). In contrast, samples treated with 9% MgCl_2 had a smaller strength decline (Figure 4.16c), while those with 14% MgCl_2 maintained consistent strength throughout the cycles (Figure 4.16d).

At -20°C , similar to observations at -10°C , treated samples consistently display higher unconfined compressive strength (UCS) compared to untreated samples across all F-T cycles, as shown in Figure 4.17a. For instance, after the third cycle, the untreated soil had a strength of 121.88 kPa, while soil treated with 4%, 9%, and 14% MgCl_2 solutions had strengths of 146.96 kPa, 213.85 kPa, and 313.70 kPa, respectively. A notable distinction at -20°C is the behavior of samples treated with a 9% MgCl_2 solution, which, unlike at -10°C , showed a reduction in strength with more cycles, though this decline was less severe compared to the 4% treated and untreated samples. The 14% MgCl_2 solution-treated samples retained consistent strength throughout the cycles.

Regarding durability index (DI) values, a different trend emerged at -20°C compared to -10°C . At -10°C , DI values for samples treated with a 9% MgCl_2 solution remained stable across seven F-T cycles. However, at -20°C (Figure 4.17b), both untreated samples and those treated with 4% and 9% MgCl_2 solutions showed a decrease in DI. After 7 F-T cycles, DI values dropped to 0.38 for untreated samples and to 0.45 and 0.64 for samples treated with 4% and 9% MgCl_2 solutions, respectively. In contrast, DI values for samples treated with a 14% MgCl_2 solution remained stable, similar to the results at -10°C , maintaining consistent levels before and after 7 F-T cycles. This variation highlights the critical impact of MgCl_2 concentration on enhancing soil stability under severe thermal conditions, emphasizing the need to tailor antifreeze treatments to specific environmental requirements. The data underscore the complex relationship between additive concentration and soil durability.

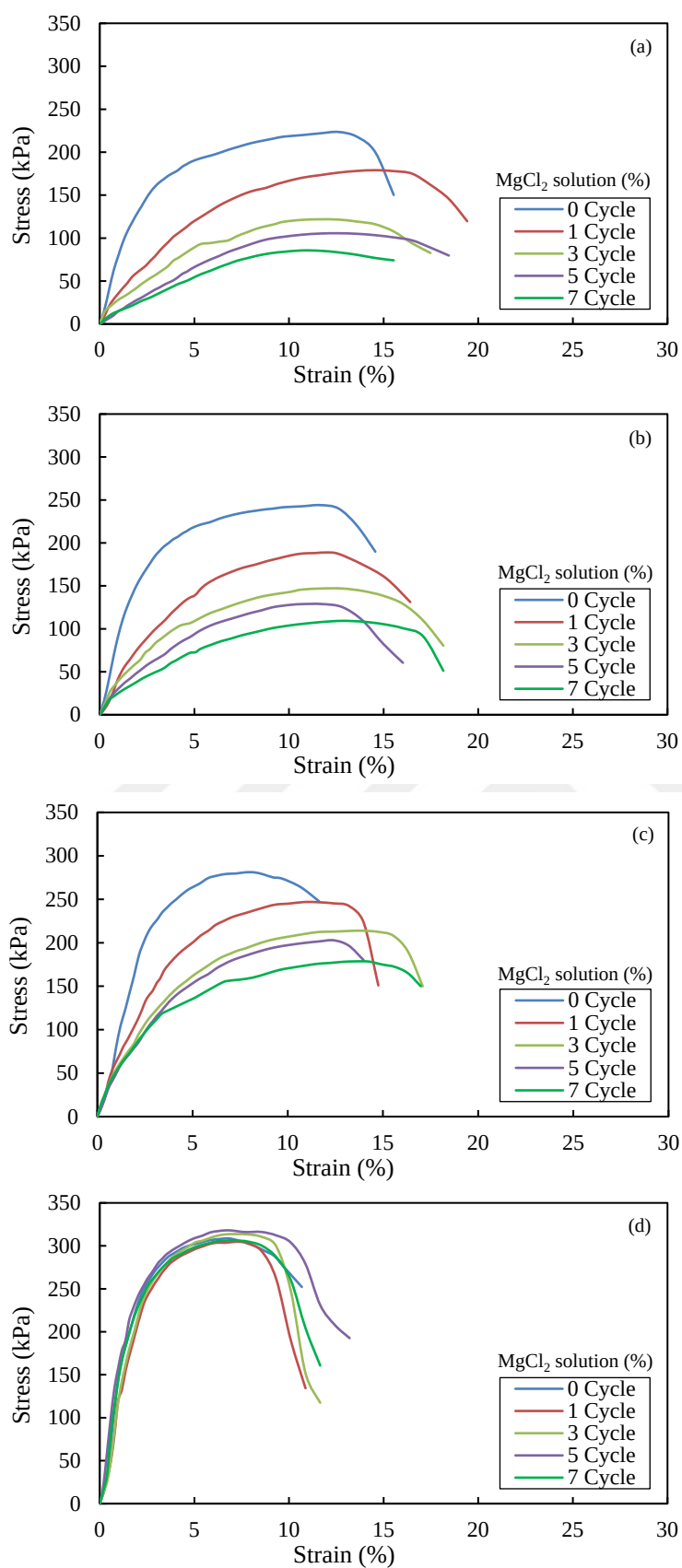


Figure 4.16 : Stress-strain relationships at -20°C for uncured samples with different MgCl_2 treatments: (a) Untreated, (b) 4% MgCl_2 , (c) 9% MgCl_2 , (d) 14% MgCl_2 .

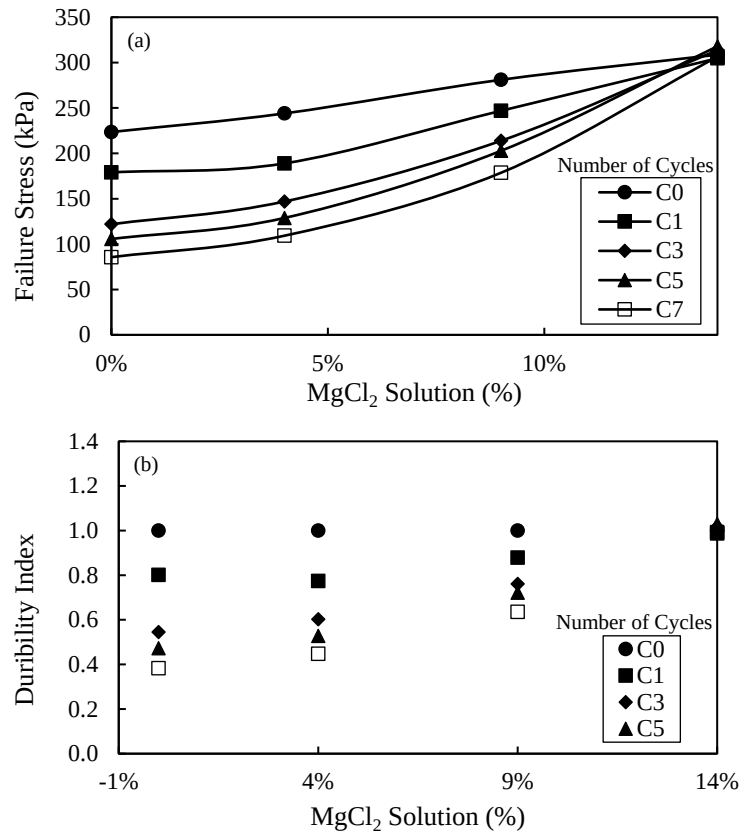


Figure 4.17 : The impact of MgCl₂ solution at -20°C on: (a) UCS and (b) durability index values for uncured samples.

Detailed experimental results, including failure stress and durability index for uncured samples at -20°C , are presented in Table 4.16 and Table 4.17.

Table 4.16 : Failure stress at -20°C for uncured samples.

F-T cycles	0% MgCl ₂ solution (kPa)	4% MgCl ₂ solution (kPa)	9% MgCl ₂ solution (kPa)	14% MgCl ₂ solution (kPa)
0	223.51	244.00	281.05	308.70
1	179.01	188.91	246.88	304.76
3	121.88	146.96	213.85	313.70
5	105.64	128.84	202.80	317.98
7	85.62	109.28	178.61	306.44

Table 4.17 : Durability Index of uncured samples at -20°C .

F-T cycles	0% MgCl ₂ solution	4% MgCl ₂ solution	9% MgCl ₂ solution	14% MgCl ₂ solution
0	1.00	1.00	1.00	1.00
1	0.80	0.77	0.88	0.99
3	0.55	0.60	0.76	1.02
5	0.47	0.53	0.72	1.03
7	0.38	0.45	0.64	0.99

4.2.6.2 Samples with 7 days cured:

Results at -10°C:

The effects of Freeze-Thaw (F-T) cycles at -10°C on the stress-strain characteristics of samples that have been cured for seven days are shown in Figure 4.18. It is evident that untreated samples experience a significant reduction in strength during these F-T cycles (Figure 4.18a). In contrast, Figures 4.18c and 4.18d reveal that the application of 9% and 14% MgCl₂ solutions provides a significant antifreeze effect, maintaining the stress-strain characteristics of the samples. Even a lower concentration of 4% MgCl₂ solution has a modest but noticeable effect, slightly reducing the strength loss and showing a less pronounced decline in strength after 7 cycles compared to untreated samples (Figure 4.18b).

The variations in unconfined compressive strength (UCS) values with the addition of MgCl₂ solution suggest that, for a specific Freeze-Thaw (F-T) cycle, the stabilizer improves the soil's strength (see Figure 4.19a). For example, after the third freeze-thaw cycle, the untreated soil exhibits a strength of 138.44 kPa, while soil samples treated with 4%, 9%, and 14% MgCl₂ solutions show increased strengths of 185.82 kPa, 295.36 kPa, and 324.37 kPa, respectively. The strengthening effect of the 4% MgCl₂ solution diminishes with continued cycles. However, the strength enhancements from the 9% and 14% solutions remain effective as the cycles progress.

The changes in the durability index (DI), illustrated in Figure 4.19b, indicate a notable decrease in DI values for both untreated and 4% MgCl₂-treated samples. Untreated samples experience a sharp decline, particularly during the initial F-T cycle, which underscores the critical impact of the first F-T cycle on the overall durability of the soil. After 7 F-T cycles, the DI value drops significantly, reaching 0.41 for untreated samples and 0.52 for those treated with 4% MgCl₂, emphasizing the substantial effect of repeated F-T cycles on soil durability.

In contrast, samples treated with 9% and 14% MgCl₂ solutions exhibit much greater stability, with DI values remaining consistent both before and after the 7 F-T cycles, highlighting the effectiveness of higher MgCl₂ concentrations in maintaining soil durability under freezing conditions.

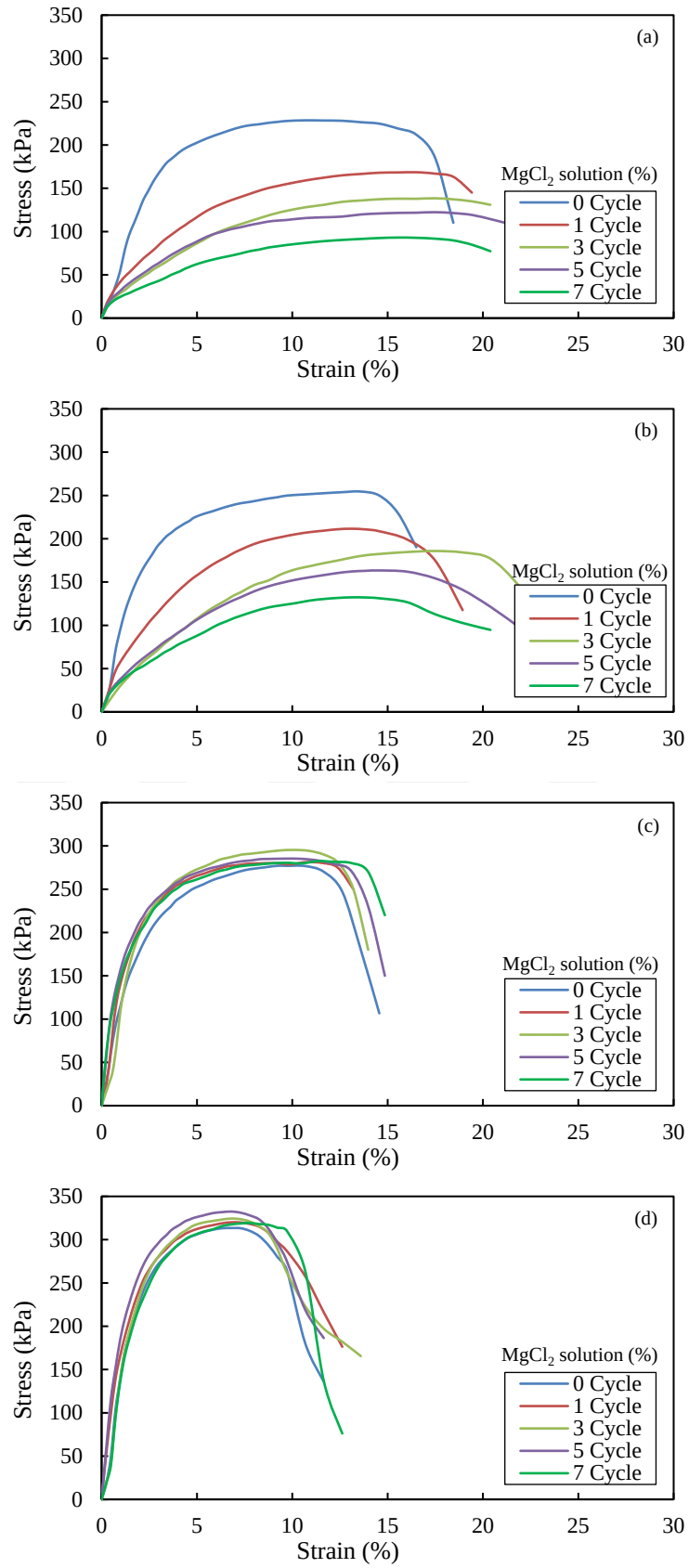


Figure 4.18 : Stress-strain relationships at -10°C for 7 days cured samples with different MgCl_2 treatments: (a) Untreated, (b) 4% MgCl_2 , (c) 9% MgCl_2 , (d) 14% MgCl_2 .

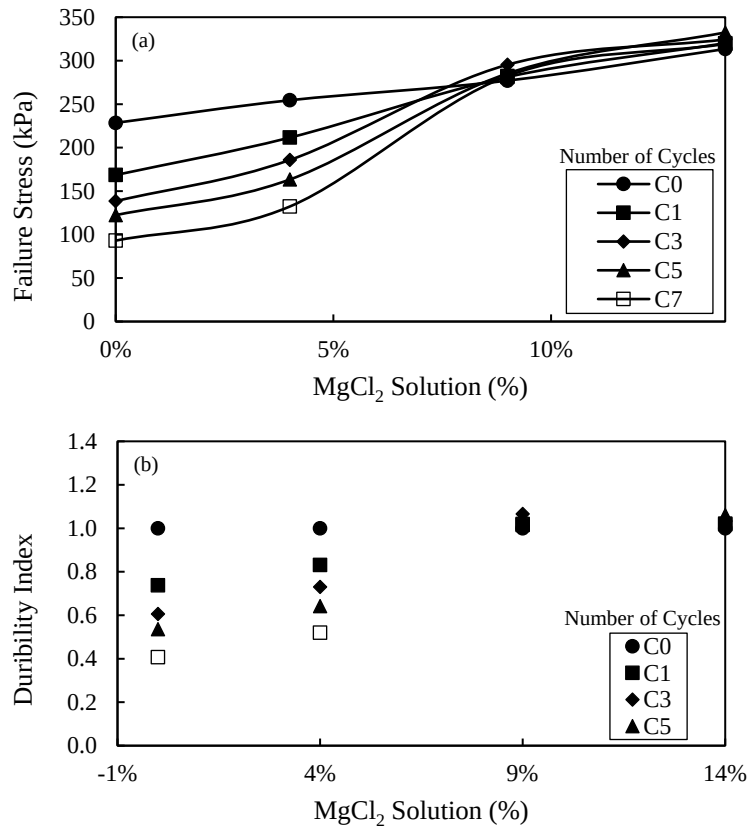


Figure 4.19 : The impact of MgCl₂ solution at -10°C on: (a) UCS and (b) durability index values for 7 days cured samples.

Below are detailed experimental results provided in Table 4.18 and Table 4.19, highlighting the failure stress and durability index for 7days cured samples at -10°C .

Table 4.18 : Failure stress at -10°C for 7 days cured samples.

F-T cycles	0% MgCl ₂ solution (kPa)	4% MgCl ₂ solution (kPa)	9% MgCl ₂ solution (kPa)	14% MgCl ₂ solution (kPa)
0	228.48	254.49	277.07	313.45
1	168.51	211.55	281.27	320.10
3	138.44	185.82	295.36	324.37
5	122.38	163.36	285.30	332.45
7	93.07	132.30	282.40	319.23

Table 4.19 : Durability Index of 7 days cured samples at -10°C .

F-T cycles	0% MgCl ₂ solution	4% MgCl ₂ solution	9% MgCl ₂ solution	14% MgCl ₂ solution
0	1.00	1.00	1.00	1.00
1	0.74	0.83	1.02	1.02
3	0.61	0.73	1.07	1.03
5	0.54	0.64	1.03	1.06
7	0.41	0.52	1.02	1.02

Results at -20°C:

To evaluate the effectiveness of MgCl₂ solutions as antifreeze agents, freeze-thaw (F-T) tests were conducted at -20°C. The stress-strain behavior of untreated and treated samples over 7 F-T cycles is shown in Figure 4.20. Untreated samples and those with 4% MgCl₂ showed significant strength loss (Figures 4.20a, 4.20b). In contrast, samples treated with 9% MgCl₂ had a smaller strength decline (Figure 4.20c), while those with 14% MgCl₂ maintained consistent strength throughout the cycles (Figure 4.20d).

At -20 °C, similar to observations at -10 °C, treated samples consistently display higher unconfined compressive strength (UCS) compared to untreated samples across all F-T cycles, as shown in Figure 4.21a. For instance, after the third cycle, the untreated soil had a strength of 119.37 kPa, while soil treated with 4%, 9%, and 14% MgCl₂ solutions had strengths of 159.42 kPa, 232.37 kPa, and 305.35 kPa, respectively. A notable distinction at -20 °C is the behavior of samples treated with a 9% MgCl₂ solution, which, unlike at -10 °C, showed a reduction in strength with more cycles, though this decline was less severe compared to the 4% treated and untreated samples. The 14% MgCl₂ solution-treated samples retained consistent strength throughout the cycles.

Regarding durability index (DI) values, a different trend emerged at -20 °C compared to -10 °C. At -10 °C, DI values for samples treated with a 9% MgCl₂ solution remained stable across seven F-T cycles. However, at -20 °C (Figure 4.21b), both untreated samples and those treated with 4% and 9% MgCl₂ solutions showed a decrease in DI. After 7 F-T cycles, DI values dropped to 0.33 for untreated samples and to 0.42 and 0.68 for samples treated with 4% and 9% MgCl₂ solutions, respectively. In contrast, DI values for samples treated with a 14% MgCl₂ solution remained stable, similar to the results at -10 °C, maintaining consistent levels before and after 7 F-T cycles. This variation highlights the critical impact of MgCl₂ concentration on enhancing soil stability under severe thermal conditions, emphasizing the need to tailor antifreeze treatments to specific environmental requirements. The data underscore the complex relationship between additive concentration and soil durability.

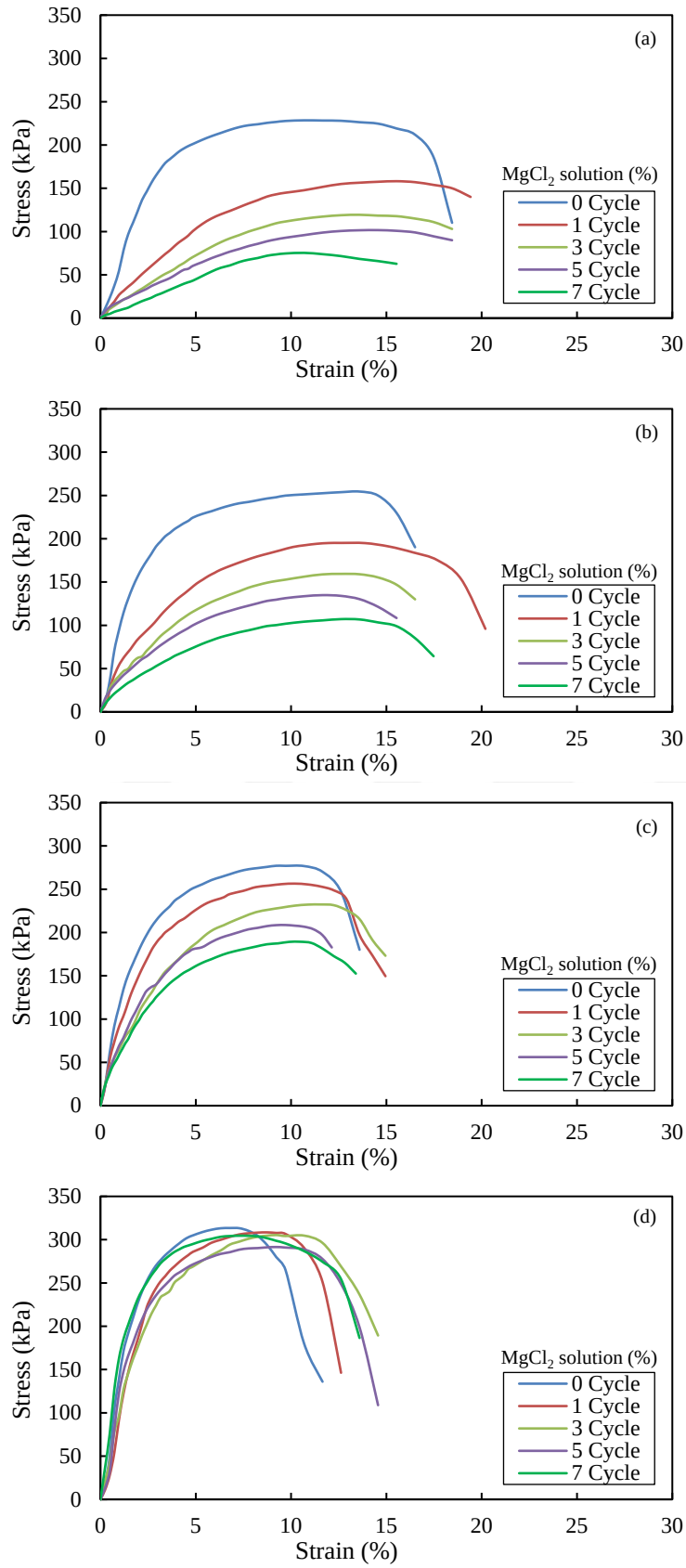


Figure 4.20 : Stress-strain relationships at -20°C for 7 days cured samples with different MgCl_2 treatments: (a) Untreated, (b) 4% MgCl_2 , (c) 9% MgCl_2 , (d) 14% MgCl_2 .

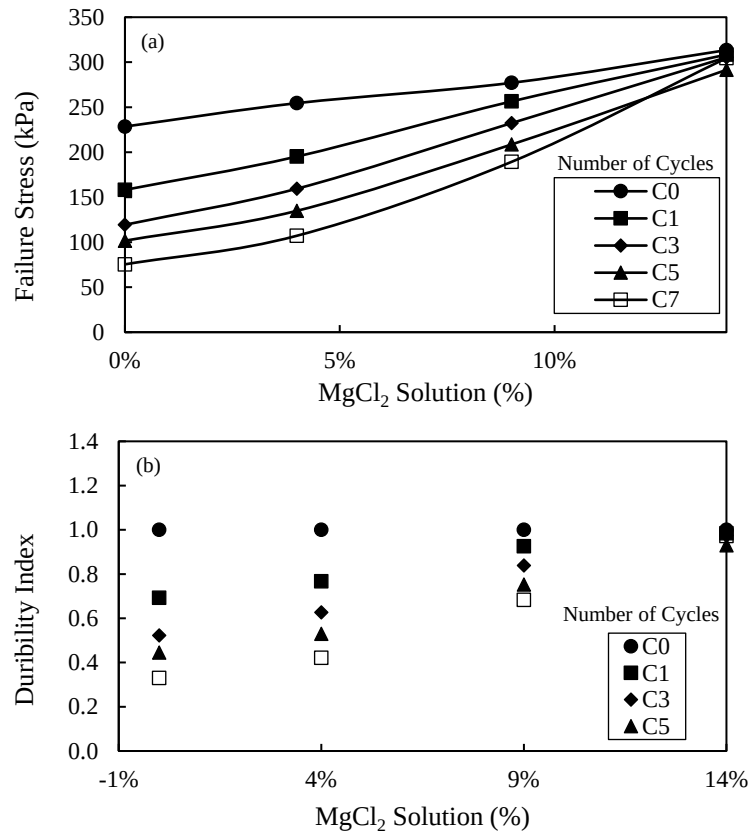


Figure 4.21 : The impact of MgCl₂ solution at -20°C on: (a) UCS and (b) durability index values for 7 days cured samples.

Below are detailed experimental results provided in Table 4.20 and Table 4.21, highlighting the failure stress and durability index for 7 days cured samples at -20°C .

Table 4.20 : Failure stress at -20°C for 7 days cured samples.

F-T cycles	0% MgCl ₂ solution (kPa)	4% MgCl ₂ solution (kPa)	9% MgCl ₂ solution (kPa)	14% MgCl ₂ solution (kPa)
0	228.48	254.49	277.07	313.45
1	158.17	195.15	256.45	308.43
3	119.37	159.42	232.37	305.35
5	101.67	134.84	208.62	291.50
7	75.40	107.15	189.30	304.56

Table 4.21 : Durability Index of 7 days cured samples at -20°C .

F-T cycles	0% MgCl ₂ solution	4% MgCl ₂ solution	9% MgCl ₂ solution	14% MgCl ₂ solution
0	1.00	1.00	1.00	1.00
1	0.69	0.77	0.93	0.98
3	0.52	0.63	0.84	0.97
5	0.44	0.53	0.75	0.93
7	0.33	0.42	0.68	0.97

4.2.6.3 Samples with 28 days cured:

Results at -10°C:

Figure 4.22 illustrates the effect of freeze-thaw (F-T) cycles at -10°C on the stress-strain properties of both untreated and treated samples cured for 28 days. The untreated samples show a significant decline in strength during the F-T cycles (Figure 4.22a). However, Figures 4.22c and 4.22d reveal that treating samples with 9% and 14% MgCl_2 solutions effectively mitigates this strength reduction, preserving the stress-strain characteristics. The 4% MgCl_2 solution shows a modest yet noticeable impact, slightly reducing the strength loss and displaying a lesser decrease in strength after 7 cycles compared to the untreated samples (Figure 4.22b).

The variation in unconfined compressive strength (UCS) values with the addition of MgCl_2 solutions demonstrates that the stabilizer significantly enhances the soil's strength during freeze-thaw cycles, as shown in Figure 4.23a. For example, after the third freeze-thaw cycle, the untreated soil has a UCS of 145.84 kPa, while the samples treated with 4%, 9%, and 14% MgCl_2 solutions show marked improvements, with strengths of 200.89 kPa, 303.54 kPa, and 327.62 kPa, respectively. The strengthening effect of the 4% MgCl_2 solution begins to diminish as the freeze-thaw cycles progress, suggesting a limited long-term impact. However, the strength gains from the 9% and 14% MgCl_2 solutions remain consistent throughout the cycles, indicating their greater efficacy in preserving soil strength under continuous freeze-thaw conditions. This shows that higher MgCl_2 concentrations offer more reliable and sustained reinforcement of soil structure, particularly in prolonged exposure to freezing conditions.

The change in the durability index (DI), represented in Figure 4.23b, shows an apparent decrease in DI values for both untreated and 4% treated samples, with untreated samples exhibiting a sharp decline during the first F-T cycle. This highlights the critical role of the initial F-T cycle in affecting the durability of soil samples. After 7 F-T cycles, the DI decreases to 0.42 for untreated samples and to 0.59 for samples treated with a 4% MgCl_2 solution, underscoring the severe impact of F-T cycles on soil durability. In contrast, DI values for samples treated with 9% and 14% MgCl_2 solutions remain stable, showing consistency before and after 7 F-T cycles.

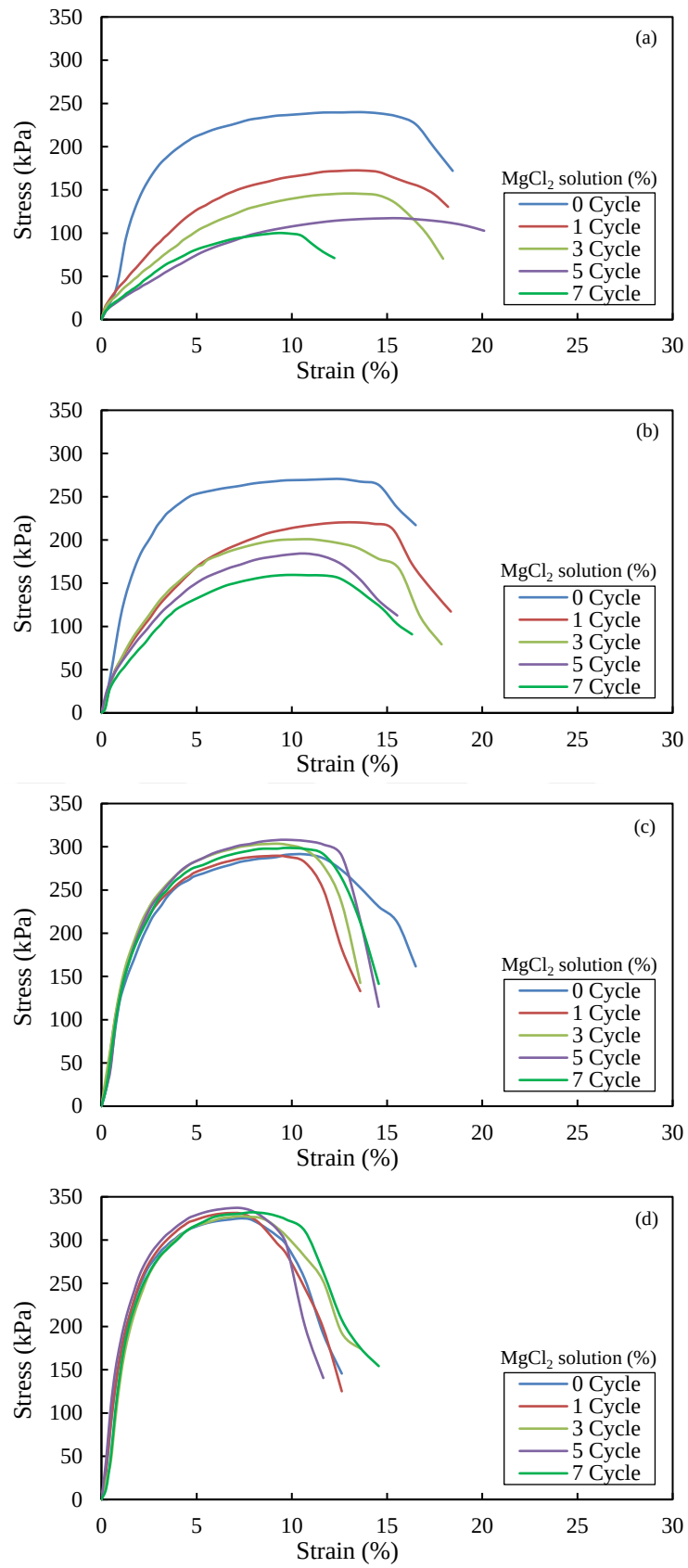


Figure 4.22 : Stress-strain relationships at $-10\text{ }^{\circ}\text{C}$ for 28 days cured samples with different MgCl_2 treatments: (a) Untreated, (b) 4% MgCl_2 , (c) 9% MgCl_2 , (d) 14% MgCl_2 .

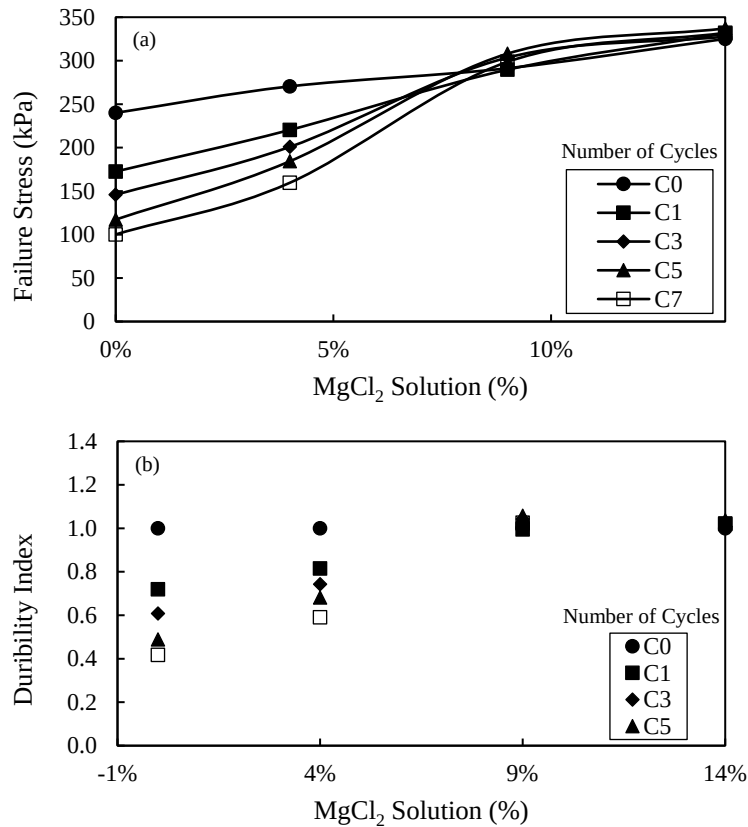


Figure 4.23 : The impact of MgCl₂ solution at -10°C on: (a) UCS and (b) durability index values for 28 days cured samples.

Below are detailed experimental results provided in Table 4.22 and Table 4.23, highlighting the failure stress and durability index for 28 days cured samples at -10°C .

Table 4.22 : Failure stress at -10°C for 28 days cured samples.

F-T cycles	0% MgCl ₂ solution (kPa)	4% MgCl ₂ solution (kPa)	9% MgCl ₂ solution (kPa)	14% MgCl ₂ solution (kPa)
0	239.96	270.44	291.29	325.05
1	172.50	220.35	289.62	331.24
3	145.84	200.89	303.54	327.62
5	117.26	184.32	308.08	337.10
7	100.18	159.60	298.66	332.04

Table 4.23 : Durability Index of 28 days cured samples at -10°C .

F-T cycles	0% MgCl ₂ solution	4% MgCl ₂ solution	9% MgCl ₂ solution	14% MgCl ₂ solution
0	1.00	1.00	1.00	1.00
1	0.72	0.81	0.99	1.02
3	0.61	0.74	1.04	1.01
5	0.49	0.68	1.06	1.04
7	0.42	0.59	1.03	1.02

Results at -20°C:

To assess the effectiveness of MgCl_2 solutions as antifreeze agents, freeze-thaw (F-T) tests were performed at -20°C . The stress-strain responses of untreated and treated samples over seven F-T cycles are presented in Figure 4.24. Untreated samples, as well as those treated with 4% MgCl_2 , experienced a notable loss in strength (Figures 4.20a, 4.24b). In contrast, samples treated with 9% MgCl_2 experienced a smaller reduction in strength over the freeze-thaw cycles (Figure 4.24c), indicating better resistance compared to untreated samples. Meanwhile, those treated with 14% MgCl_2 consistently maintained stable strength throughout all cycles (Figure 4.24d). This demonstrates the increasing effectiveness of MgCl_2 with higher concentrations in preserving soil strength.

At -20°C , as observed at -10°C , treated samples consistently exhibited higher unconfined compressive strength (UCS) compared to untreated samples across all F-T cycles, as illustrated in Figure 4.25a. For example, after the third F-T cycle, the untreated soil had a strength of 121.42 kPa, while soils treated with 4%, 9%, and 14% MgCl_2 showed strengths of 202.52 kPa, 238.03 kPa, and 318.38 kPa, respectively. A key difference at -20°C is that samples treated with 9% MgCl_2 exhibited a decline in strength with additional cycles, unlike at -10°C . However, this reduction was less severe compared to the 4% treated and untreated samples. The 14% MgCl_2 -treated samples maintained consistent strength throughout all F-T cycles.

At -20°C , a different trend in durability index (DI) values emerged compared to -10°C . While DI values for samples treated with 9% MgCl_2 remained stable across seven F-T cycles at -10°C , a decrease was observed at -20°C (Figure 4.25b). After seven F-T cycles, the DI dropped to 0.31 for untreated samples and to 0.62 and 0.70 for samples treated with 4% and 9% MgCl_2 , respectively. This shows that higher MgCl_2 concentrations provide better durability under more extreme conditions. Conversely, DI values for samples treated with 14% MgCl_2 remained stable, similar to the results at -10°C , maintaining consistent levels before and after seven F-T cycles. This consistency highlights the significant role of MgCl_2 concentration in enhancing soil stability under extreme thermal conditions, stressing the importance of tailoring antifreeze treatments to specific environmental needs. The results underline the complex relationship between additive concentration and soil durability, showing that higher concentrations are more effective in preserving soil integrity.

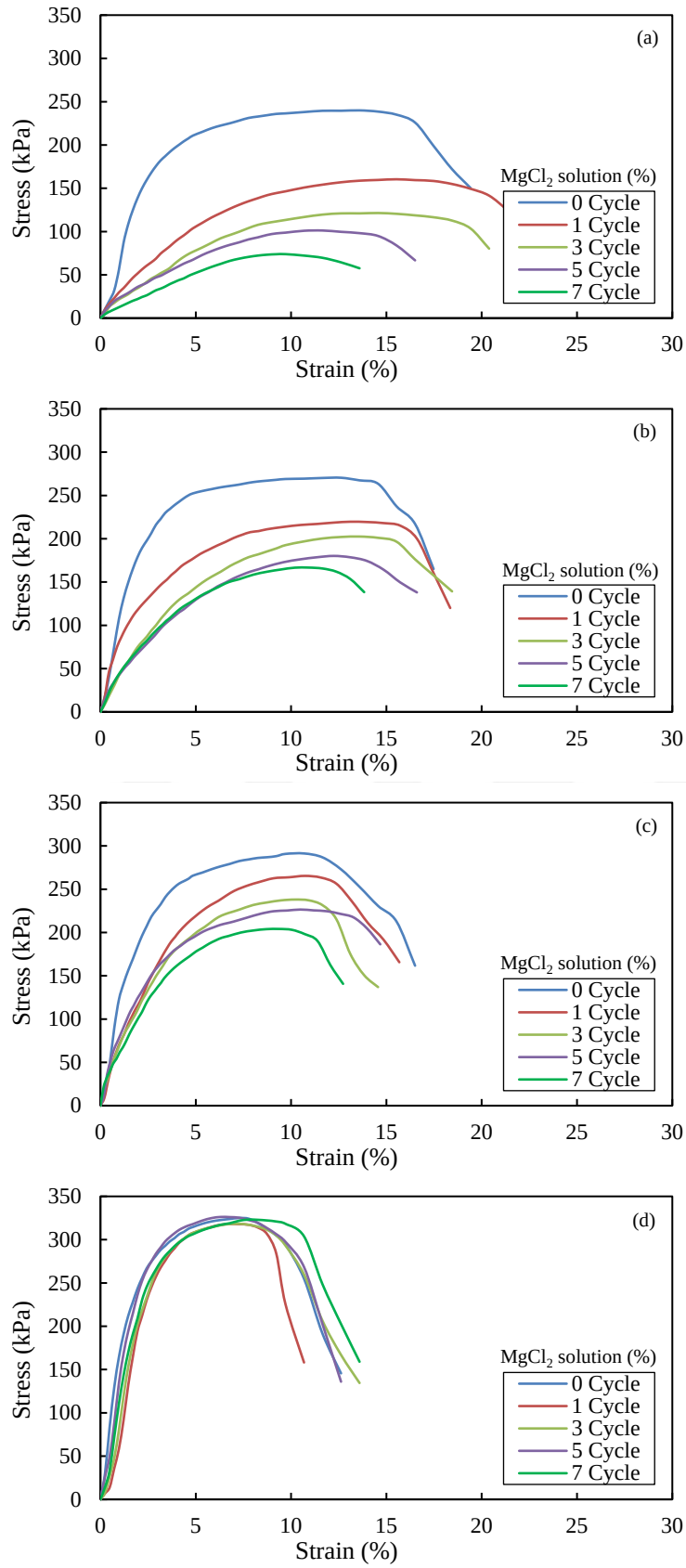


Figure 4.24 : Stress-strain relationships at $-20\text{ }^{\circ}\text{C}$ for 28 days cured samples with different MgCl_2 treatments: (a) Untreated, (b) 4% MgCl_2 , (c) 9% MgCl_2 , (d) 14% MgCl_2 .

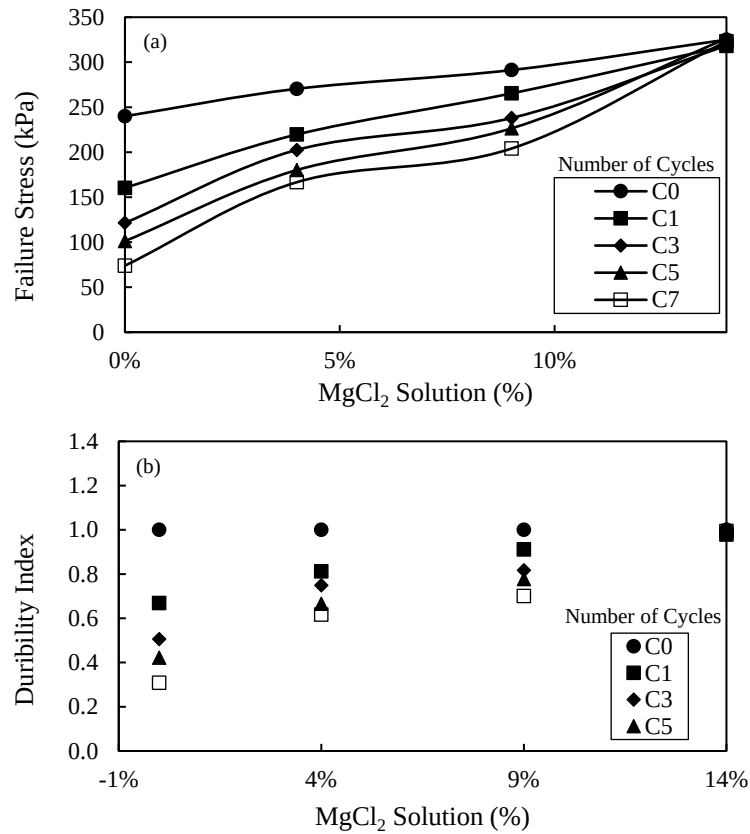


Figure 4.25 : The impact of MgCl₂ solution at -20°C on: (a) UCS and (b) durability index values for 28 days cured samples.

Below are detailed experimental results provided in Table 4.24 and Table 4.25, highlighting the failure stress and durability index for 28 days cured samples at -20°C .

Table 4.24 : Failure stress at -20°C for 28 days cured samples.

F-T cycles	0% MgCl ₂ solution (kPa)	4% MgCl ₂ solution (kPa)	9% MgCl ₂ solution (kPa)	14% MgCl ₂ solution (kPa)
0	239.96	270.44	291.29	325.05
1	160.39	219.65	265.38	318.05
3	121.42	202.52	238.03	318.38
5	101.22	180.15	226.50	326.24
7	73.91	166.67	204.08	323.14

Table 4.25 : Durability Index of 28 days cured samples at -20°C .

F-T cycles	0% MgCl ₂ solution	4% MgCl ₂ solution	9% MgCl ₂ solution	14% MgCl ₂ solution
0	1.00	1.00	1.00	1.00
1	0.67	0.81	0.91	0.98
3	0.51	0.75	0.82	0.98
5	0.42	0.67	0.78	1.00
7	0.31	0.62	0.70	0.99

4.2.7 Effect of freeze-thaw cycles on E modulus of MgCl₂-treated clay

The elastic modulus measurements after freeze-thaw cycles for untreated and MgCl₂-treated samples (4%, 9%, and 14%) at -10°C and -20°C provide valuable insights into their behavior under freezing conditions. At -10°C, the untreated soil showed significant degradation, with the elastic modulus dropping from 7.7 MPa to 3.3 MPa after one cycle, and further to 2.0 MPa by the seventh cycle. The 4% MgCl₂-treated samples performed better but still decreased to 3.9 MPa by the seventh cycle. The 9% and 14% MgCl₂-treated samples displayed much stronger performance, with the 9% samples maintaining around 10.9 MPa, and the 14% samples peaking at 13.3 MPa after seven cycles, demonstrating excellent resistance to freeze-thaw effects (table 4.26).

At -20°C, untreated samples experienced more severe strength loss, with the modulus dropping from 7.7 MPa to 2.7 MPa after the first cycle, and further to 1.2 MPa after seven cycles. The 4% MgCl₂-treated samples also declined to 3.9 MPa by the seventh cycle, while the 9% samples reduced to 5.1 MPa. However, the 14% MgCl₂-treated samples showed the best resistance, with a slight drop from 12.9 MPa to 11.9 MPa after seven cycles, indicating strong protection at lower temperatures (Table 4.27).

In addition to mitigating freeze-thaw effects, the initial addition of MgCl₂ increased the elastic modulus of the treated samples. The untreated sample started at 7.7 MPa, whereas the 4%, 9%, and 14% treated samples began at 8.7 MPa, 10.0 MPa, and 12.9 MPa, respectively, showing an initial strengthening effect.

In conclusion, while 4% and 9% MgCl₂ provided some improvement, the 14% concentration was most effective in minimizing strength loss, especially at extreme temperatures. These findings highlight the importance of selecting the appropriate MgCl₂ concentration for stabilizing soils exposed to freeze-thaw conditions.

Table 4.26 : Elastic modulus of MgCl₂-treated clay samples after F-T cycles at -10°C.

F-T cycles	0% MgCl ₂ solution (MPa)	4% MgCl ₂ solution (MPa)	9% MgCl ₂ solution (MPa)	14% MgCl ₂ solution (MPa)
0	7.7	8.7	10.0	12.9
1	3.3	5.2	10.9	13.6
3	2.6	5.2	11.3	13.4
5	1.9	4.5	11.1	14.2
7	2.0	3.9	10.9	13.3

Table 4.27 : Elastic modulus of MgCl₂-treated clay samples after F-T cycles at -20°C.

F-T cycles	0% MgCl ₂ solution (MPa)	4% MgCl ₂ solution (MPa)	9% MgCl ₂ solution (MPa)	14% MgCl ₂ solution (MPa)
0	7.7	8.7	10.0	12.9
1	2.7	6.4	6.3	12.1
3	1.8	4.0	6.0	13.4
5	1.9	3.6	6.7	12.8
7	1.2	3.9	5.1	11.9

It should be noted that, for this analysis, only 28-day cured samples were used, as curing time was found to have little effect on the overall behavior, making 28 days the benchmark for comparison.

4.2.8 Effect of freeze-thaw cycles on volume changes in MgCl₂-treated clay

This research explores the volume changes in materials subjected to freeze-thaw (F-T) cycles, focusing on the ameliorative impact of MgCl₂. During freezing, the transformation of water into ice causes a volume expansion of about 9%, displacing soil particles. Upon thawing, the original volume isn't fully restored, leading to larger pore sizes and reduced failure strength. This irreversible expansion and strength reduction are attributed to structural changes in the sample matrix, as discussed by Wang et al. (Wang et al., 2007) and further supported by Lu et al. (Lu et al., 2019) and Jamshidi et al. (Jamshidi et al., 2023).

Below are detailed graphical results provided in Figure 4.26, illustrating the effects of freeze-thaw (F-T) cycles on volume alteration of samples at two different temperatures.

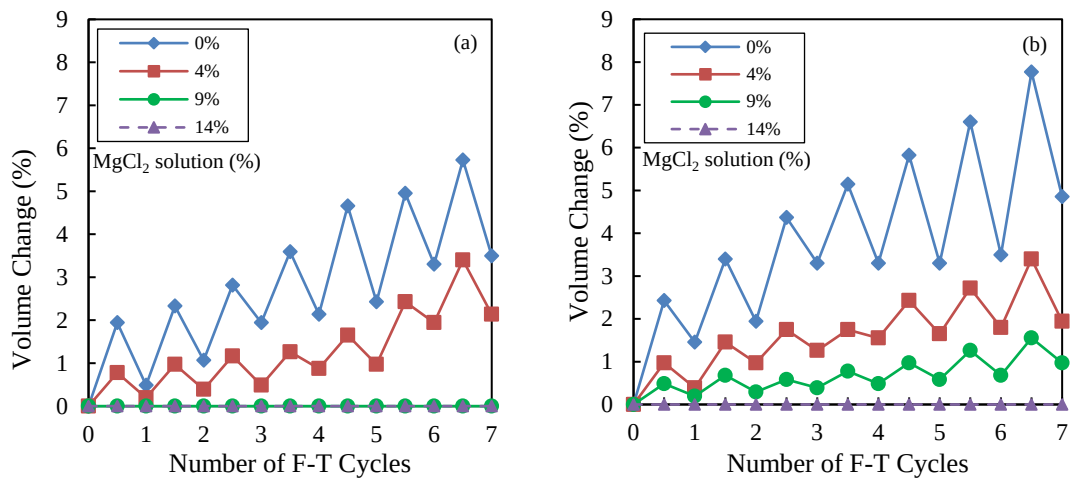


Figure 4.26 : F-T cycle effects on samples' volume alteration at: (a) -10 °C and (b) -20 °C.

Moreover, as an illustration of the damaging effects of freeze-thaw cycles on soil structure, Figure 4.27 presents photographs of untreated and 14% MgCl_2 -treated samples taken before the seventh cycle, with the untreated sample shown in Figure 4.27(a) and the treated sample in Figure 4.27(b). The untreated sample in Figure 4.27(a) visibly exhibits cracks, providing a clear example of the structural degradation caused by freeze-thaw processes. These cracks contribute to the volume changes and significant reduction in strength, demonstrating how freeze-thaw cycles can severely compromise soil stability. In contrast, the 14% MgCl_2 -treated sample in Figure 4.27(b) shows no visible cracking, illustrating its ability to preserve soil integrity and resist damage throughout the cycles. This comparison highlights the critical role of MgCl_2 in protecting soil from freeze-thaw-induced damage.



Figure 4.27 : Untreated (a) and 14% MgCl_2 -treated (b) soil samples before the 7th freeze-thaw cycle.

4.2.9 Microstructural analyses

4.2.9.1 XRD and FTIR analysis

X-ray diffraction (XRD) analysis is an essential technique for explaining changes in material properties, interactions between interlayers, and the effects of added particles, offering a comprehensive data set for interpreting soil mineralogy. Figure 4.28 presents the XRD results for both untreated samples and those treated with a 14% MgCl_2 solution. Upon examination, the diffraction patterns of the untreated and treated samples show no significant differences. Specifically, there are no new peaks in the

diffraction patterns of the treated soil, and the peak positions, denoted by the 2θ values, remain consistent across both sample sets. This uniformity indicates that the interlayer spacing was preserved, no new chemical bonds were formed, and the MgCl_2 solution did not substitute the interlayer cations in the clay structure. The primary mechanism underlying the observed phenomena is likely due to electrostatic interactions between the clay particles and the charged species of the MgCl_2 solution, which contributes to an enhancement in soil strength.

Fourier-transform infrared (FTIR) spectroscopy was utilized to identify functional groups within soil minerals. Comparisons of FTIR spectra from untreated soil and soil treated with a 14% MgCl_2 solution revealed no significant differences, suggesting that MgCl_2 does not substantially alter the soil's functional groups. This lack of change in the spectra indicates that the mechanism of treatment is primarily based on physical (electrostatic) interactions, as depicted in Figure 4.29.

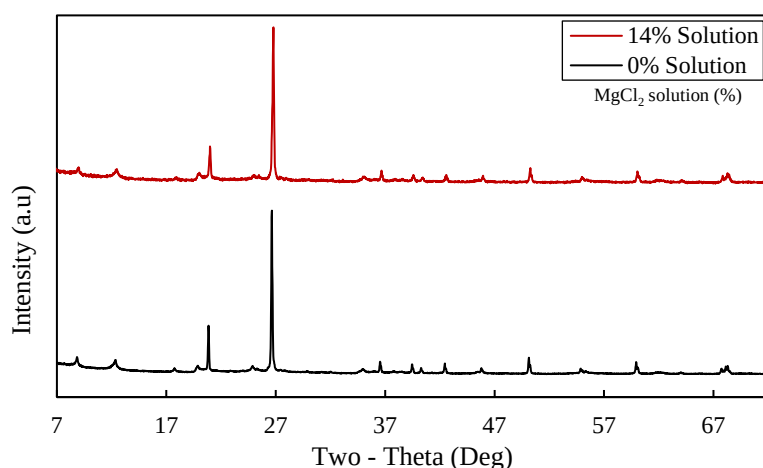


Figure 4.28 : XRD profiles for untreated and 14% MgCl_2 -treated soil.

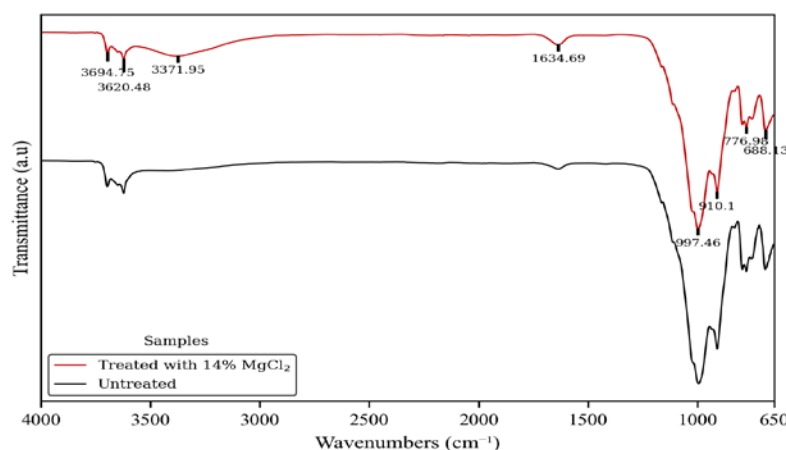


Figure 4.29 : FTIR spectrums for untreated and 14% MgCl_2 -treated soil.

4.2.10 SEM analysis

Scanning electron microscopy (SEM) was employed to investigate microstructural modifications in the composition of soil samples. Figure 4.30 presents SEM images of both untreated and 14% MgCl_2 treated soil samples following 28 days of curing. The application of a 14% MgCl_2 solution facilitated a reorganization of particles, leading to a more cohesive structure and enhanced structural integrity compared to the untreated samples. This particle aggregation altered the geotechnical properties by decreasing interactions between surface areas and water, with the extent of changes being proportional to the MgCl_2 solution concentration. These modifications are likely due to the increased salt concentration from the added MgCl_2 solution, which promoted rapid flocculation and a significant increase in particle size. Consequently, the sample treated with a 14% MgCl_2 solution demonstrated the highest structural strengths, indicative of improved structural integrity and larger particle size.

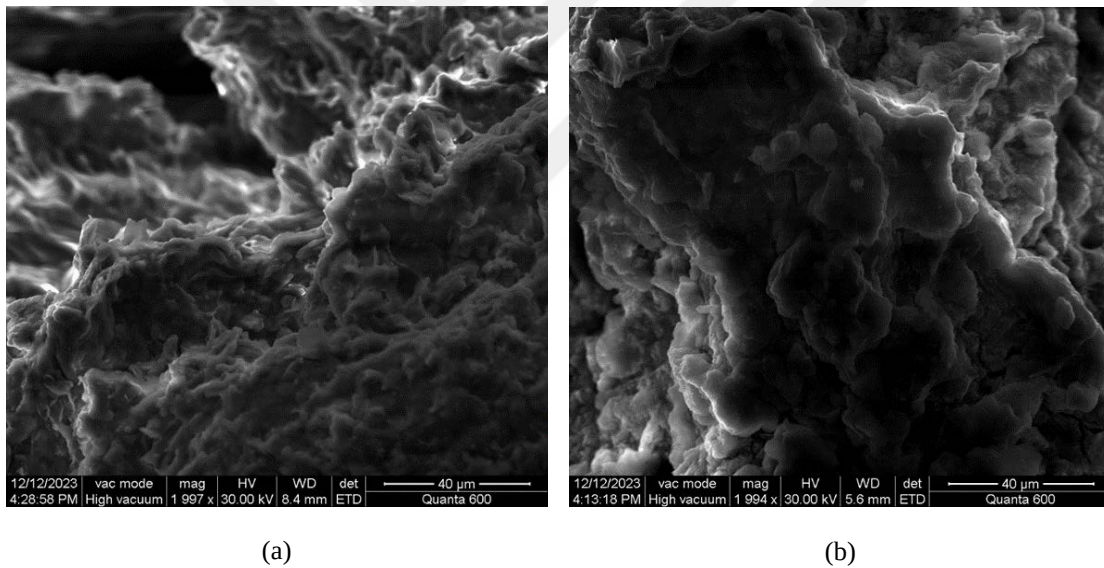


Figure 4.30 : SEM images: (a) 28 days cured untreated sample and (b) 28 days cured MgCl_2 14% solution treated sample.

5. CONCLUSION

Stabilized soil embankments in cold climates are inherently vulnerable to freeze-thaw (F-T) cycles, which ultimately cause strength loss and volume change. This challenge necessitates the exploration of additives capable of mitigating freezing to preserve strength and volume stability. Addressing this issue enhances the durability and reliability of soil embankments under cold weather conditions, enabling projects to continue during colder periods and yielding considerable savings in both costs and project timelines.

In response to this challenge, this study embarked on a comprehensive experimental investigation to assess the efficacy of Polyethylene Glycol (PEG 400) and MgCl_2 solutions as both antifreeze and stabilizing agents for fine-grained soils. The thesis focused on evaluating the effects of these solutions on the soils' plasticity, strength, and durability properties.

For the PEG solution, the tests conducted included Atterberg limits tests, compaction tests, unconfined compressive strength (UCS) tests, and a single freeze-thaw (F-T) test at -20°C . Additionally, the effect of PEG on a clay-7% cement mixture was examined to investigate any practical positive impact, given that PEG alone reduced the compressive strength of the clay, as explained in previous sections.

For the MgCl_2 solution, a more extensive range of tests was performed, including Atterberg limits tests, UCS tests, compaction tests, volume change measurements, evaluations under different freeze-thaw cycles, and microstructural analysis including FTIR, SEM, and XRD tests.

The limited tests for the PEG solution were due to the significant reduction in clay strength caused by PEG. Consequently, it was deemed unnecessary to conduct further freeze-thaw cycle tests with PEG. Following this observation, the focus shifted to identifying a more effective additive, leading to the selection of MgCl_2 .

For the PEG solution, the following conclusions were drawn based on test results:

- Atterberg Limits: Increasing PEG concentrations consistently resulted in higher Atterberg limits. The results demonstrate that untreated soil with a liquid

limit (L.L) of 37%, plastic limit (P.L) of 19%, and plasticity index (P.I) of 18% will yield values of 55%, 23%, and 32%, respectively, with a 50% PEG solution.

- **Compaction Characteristics:** With higher PEG concentrations, the maximum dry density decreased from 17.5 kN/m³ to 15.2 kN/m³, and the optimum water content increased from 19% to 28%.
- **Unconfined Compressive Strength:** Higher concentrations of PEG led to a decrease in soil strength. However, to investigate the antifreeze effect of PEG on clay under freeze-thaw (F-T) cycles, an F-T test with only one cycle was conducted on untreated samples and samples treated with 10%, 20%, 30%, 40%, and 50% PEG solutions. The results showed that PEG at higher concentrations significantly reduced the clay strength, with a 50% PEG solution decreasing strength by 68% and increasing the failure strain from 6.8% to 14.56%, which was expected due to the hydrophilic aspect of PEG. On the other hand, PEG at 40% and 50% concentrations prevented strength loss after one F-T cycle at -20°C, whereas the untreated sample experienced a 27% strength reduction. This indicates that higher PEG concentrations acted as an antifreeze by preventing ice lens formation. However, the substantial reduction in initial clay strength due to the hydrophilic water retention properties of PEG and its impact on soil structure rendered PEG ineffective as a stabilizing additive for clay.

Additionally, since PEG alone caused a reduction in clay strength, we investigated its effect on a clay-cement mixture to see if cement could compensate for the negative effects of PEG. Unfortunately, the results showed that PEG significantly reduced the strength of the clay-cement mixture as well. Strength tests were repeated after one F-T cycle at -5°C, and interestingly, even higher concentrations of PEG could not prevent strength loss and further decreased the strength of the clay-cement mixture. This indicates that PEG can negatively affect the cement hydration process, impeding it and resulting in overall reduced strength.

The conclusions of the study for the MgCl₂ solution were as follows:

Uncured samples, along with those subjected to 7 and 28 days of curing, displayed comparable patterns in their outcomes, albeit with minor distinctions. Notably, the

samples cured for 28 days exhibited the most promising results, indicating the efficacy of prolonged curing periods in enhancing the desired effects of MgCl_2 solution.

- **Atterberg Limits:** Higher MgCl_2 concentrations significantly lower the soil's Atterberg limits, particularly at 14%, decreasing the liquid limit by 14%, plastic limit by 9%, and plasticity index by 19%. This reduction is due to the cation exchange process involving divalent magnesium ions, which reduces the diffuse double-layer thickness.
- **Compaction Characteristics:** MgCl_2 improves soil compaction, increasing the maximum dry density (MDD) to 17.89 kN/m^3 from 17.4 kN/m^3 and lowering the optimum water content (OWC) from 19% to 17.71% at a 14% concentration. These changes result from soil particle flocculation and accumulation due to rapid cation exchange, reducing clay's water absorption and enhancing dry densities.
- **Unconfined Compressive Strength:** Adding MgCl_2 solution enhances soil strength significantly at higher concentrations, with observed UCS increases of 9%, 26%, and 38% for 4%, 9%, and 14% MgCl_2 concentrations, respectively, even without curing. Curing shows a minor effect, with only a 5% UCS improvement in samples treated with a 14% MgCl_2 solution. However, this increased strength comes with reduced failure strain, indicating decreased deformability in soils treated with MgCl_2 solution.
- **Durability Under Freeze-Thaw Cycles:** F-T cycle tests conducted on 28-day cured samples at -10°C and -20°C evaluated the antifreeze and stabilization effects of MgCl_2 on clay, determining the optimal concentrations for maintaining integrity under cold conditions. A durability index (DI) was used to quantify the post-treatment stability by comparing the UCS ratios before and after F-T cycles. At -10°C , soils treated with 9% and 14% MgCl_2 solutions maintained strength and DI values through seven F-T cycles, whereas a 4% MgCl_2 solution was less effective. Untreated samples showed a drop in DI to 0.42, and those treated with 4% MgCl_2 solution to 0.59 after seven cycles. At -20°C , untreated and 4% MgCl_2 -treated soils lost strength significantly, while 9% solution-treated samples exhibited a smaller strength reduction, and 14% solution-treated samples maintained consistent strength across seven cycles. DI values showed reductions in all treatments except the 14% solution, decreasing to 0.31 in untreated samples and 0.62 and 0.70 in samples treated

with 4% and 9% MgCl_2 solutions, respectively. This emphasizes the need for precise MgCl_2 concentration adjustments for soil stability in cold climates, highlighting the relationship between additive concentration, temperature, and soil durability.

- **Elastic Modulus:** The results show that MgCl_2 significantly improved the elastic modulus of clay samples exposed to freeze-thaw cycles. Untreated samples experienced severe reductions in modulus, dropping from 7.7 MPa to 2.0 MPa at -10°C and from 7.7 MPa to 1.2 MPa at -20°C after seven cycles. The 4% MgCl_2 -treated samples showed moderate improvement, with modulus values decreasing to 3.9 MPa at both temperatures. However, the 9% and 14% MgCl_2 -treated samples demonstrated much greater resistance, particularly at 14%, where the modulus remained at 13.3 MPa at -10°C and 11.9 MPa at -20°C after seven cycles. These findings highlight MgCl_2 's effectiveness in preserving the elastic modulus and improving soil stability under freezing conditions.
- **Volume Stability:** At -10°C , untreated and 4% MgCl_2 -treated samples expanded with freeze-thaw cycles. However, the samples treated with 4% MgCl_2 exhibited reduced expansion. Samples treated with 9% and 14% MgCl_2 showed no change. At -20°C , samples treated with 9% MgCl_2 exhibited expansion, albeit less than untreated and 4% MgCl_2 -treated samples. Samples treated with 14% MgCl_2 maintained their volume.
- **Microstructural Analysis:** XRD and FTIR analyses of untreated and MgCl_2 -treated soil samples (14% solution) revealed no notable differences in diffraction patterns, with no new chemical bonds detected. These results suggest that MgCl_2 has minimal impact on the soil's functional groups. SEM analyses provided clear evidence of clay flocculation and subsequent particle size enlargement upon adding the additive. These microstructural changes align with observed shifts in the soil's geotechnical properties.

Regarding the limitations of magnesium chloride (MgCl_2) in this study, one key aspect is that the concentration of MgCl_2 was selected based on its ability to prevent freezing under controlled laboratory conditions. However, field conditions can vary greatly, and factors such as soil type, moisture content, and temperature fluctuations in real-world environments could influence the performance of MgCl_2 . Thus, further research

is needed to assess the scalability of the laboratory findings in diverse environmental settings.

Additionally, the relatively short-term nature of the freeze-thaw cycles tested in this study presents another limitation. The study was able to demonstrate MgCl_2 's effectiveness over several cycles, but the long-term durability and potential degradation of the soil- MgCl_2 mixture under extended freeze-thaw cycles, beyond the scope of this research, remain unknown.

5.1 Comparison of MgCl_2 Over PEG:

The comparative analysis of MgCl_2 and PEG under freeze-thaw conditions demonstrates the superior performance of MgCl_2 in enhancing soil strength, durability, and volume stability. The effectiveness of MgCl_2 is primarily attributed to its cation exchange capabilities and antifreeze properties, which prevent ice lens formation and maintain structural integrity.

In contrast, PEG tends to increase water retention, which reduces dry density and overall soil strength. Its hydrophilic nature leads to heightened water absorption and increases its susceptibility.

In conclusion, the evidence strongly supports using MgCl_2 solutions at appropriate concentrations based on weather temperature as a more effective antifreeze and stabilizing agent compared to PEG. This preference for MgCl_2 is due to its ability to improve the strength and durability of soils under challenging environmental conditions, ensuring greater volume stability and making it the preferred choice for soil stabilization in cold climates.

This thesis highlights MgCl_2 as a reliable antifreeze and stabilizing agent that significantly improves the durability and reliability of soil embankments in cold climates. Future research should focus on site-specific adjustments of MgCl_2 concentrations based on local climatic conditions and soil properties. Additionally, large-scale field experiments are recommended to deepen our understanding of MgCl_2 's practical applications and to further validate its efficacy in real-world scenarios.



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