ENGINEERING PROPERTIES OF CEMENT-TREATED SILTY SAND SUBJECTED TO FREEZE THAW CYCLES

Nazerke Sagidullina, Bachelor Degree in Civil and Environmental Engineering

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53 Kabanbay Batyr Avenue,
Astana, Kazakhstan, 010000

Supervisor: Sung-Woo Moon
Co-supervisor: Jong Kim

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Declaration Form

DECLARATION
I hereby, declare that this manuscript, entitled "influence of freeze-thaw cycles on physical and mechanical properties of cement-treated silty sand*",* is the result of my own work except for quotations and citations which have been duly acknowledged.
I also declare that, to the best of my knowledge and belief, it has not been previously or concurrently submitted, in whole or in part, for any other degree or diploma at Nazarbayev University or any other national or international institution.

Name: Nazerke Sagidullina
Date: 08/04/2022

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Abstract

The problem of soft ground is currently of great interest, as with the rapid development of infrastructure, researchers are trying to cope with the improvement of properties of problematic soil to build structures on it since any structures built on weak soils can be easily damaged. In cold regions, the problem of weak soils is further exacerbated by freeze-thaw cycling, resulting in reduced soil strength. For the improvement of soil properties, the soil stabilization method can be used. For such purposes, ordinary Portland Cement (OPC) is commonly used. However, despite the effectiveness of OPC cement as a binding material, it produces a significant amount of carbon dioxide emission. As an alternative to OPC, calcium sulfoaluminate (CSA) cement can be used. Therefore, the purpose of this research study is to present the results from laboratory experiments to evaluate the effectiveness of the soil treatment method using CSA cement for the improvement of the properties of silty sand. The unconfined compressive strength (UCS) and ultrasonic pulse velocity (UPV) testing conducted on the soil samples that were cured for 3, 7, and 14 days and subjected to 0, 1, 3, 5, 7, 10, and 15 freeze-thaw cycles. The water content is defined from the optimum moisture content and three different cement content were used, 3%, 5%, and 7%. Applying the results from the unconfined compressive strength (UCS) test, the strength loss/gain and resilient modulus parameters were obtained. The findings of the study show that the strength and pulse velocity values decreased with the exposure of soil specimens to cyclic freezing and thawing. However, improvement in soil performance can be observed with the use of CSA cement. Overall, the application of CSA cement for treatment purposes could be an effective method to enhance soil performance and meet the subgrade design requirements.

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Chapter 1 – Introduction

1.1 Introduction

In regions with a cold climate, soils undergo freeze-thaw cycles, which subsequently leads to a change in soil properties, which further causes a potential loss of soil strength. The influence of freeze-thaw action on soil, especially on problematic soils, can reduce the ultimate strength, and affects the resilient modulus, volume, compressibility, bearing capacity, and microstructure of soil. Such soil damage causes the collapse of the infrastructure built on them.

The characteristics of soil can be improved by applying the soil stabilization method, which includes various methods applied to modify soil properties for better engineering performance [1-3]. There are two main types of soil stabilization methods, which are physical and chemical soil stabilization. Physical methods are based on the application of physical processes to enhance soil properties, while the chemical soil stabilization method uses chemicals, emulsions, and various binders [4]. Several research papers have examined the effectiveness of various additives using chemical stabilization techniques in improving soil performance by evaluating the mechanical and physical characteristics of treated soil under freeze-thaw cycles. Among the existing additives, Ordinary Portland cement (OPC) in combination with other binding materials (e.g., fibers, fly ash, and slags) is extensively used. Although OPC has been widely utilized in geotechnical engineering, there is a concern due to the emission of greenhouse gases from cement production. Therefore, there is a need for an environmentally friendly alternative that could replace OPC. One type of sustainable cement is Calcium Sulfoaluminate cement (CSA), which releases less carbon than OPC, and it is already widely used in the concrete industry for various purposes, such as bridge construction and pipe manufacturing. Moreover, many research works were carried out to test the CSA cement for ground improvement purposes under various conditions. The results of experimental and numerical studies proved the rapid strength gain property of CSA cement while comparing it with other binding materials [5-9]. Furthermore, Jumassultan et al [10] investigated the influence of cyclic freezing and thawing on the CSA-treated quartz sand, where CSA-treated sand has shown better performance with insignificant changes after cyclic freezing and thawing. However, there are limited studies conducted for problematic soils treated with CSA cement under cyclic freezing and thawing.

1.2 Definition of the problem

The silty sand is considered a poor filler material for the subgrade and cannot be used for engineering construction purposes, since it has low strength, poor stability, and can be easily deformed, which makes it difficult to meet the requirements for the subgrade design of highway and railways. In addition, the weather conditions of our country also affect the properties of the soil. Exposing the soil to continuous cyclic freezing and thawing results in loss of strength and weakening of soil properties. Therefore, this type of soil cannot be used directly for construction purposes. There is a need to improve the quality of subgrade soil with the fast growth of industrialization, and the construction of bridges and roads in Kazakhstan. It can be reached by the application of the soil stabilization technique.

1.3 Research question and hypothesis

There are two main *research questions***.**

1. Does the use of CSA cement in stabilizing weak soils could be effective, and can it replace conventional binders?
2. How do freeze-thaw cycles affect CSA stabilized weak soil?

Hypothesis:

The treatment of soil using the CSA cement could improve the weak soil properties to meet the requirements of subgrade design under cyclic freezing and thawing.

1.4 Objective

The main purpose of the study is to examine the effectiveness of CSA cement treatment to stabilize weak soil under the action of freeze-thaw cycles.

Scope

Laboratory investigation for characterization of silty sand treated with CSA cement through unconfined compressive strength and ultrasonic pulse velocity test under cyclic freezing and thawing.

Chapter 2 – Literature review

2.1 Introduction

Two types of soil stabilization methods, which are physical and chemical soil stabilization, are the most commonly applied techniques. The chemical stabilization method is widely used for the improvement of subgrade soil properties. Stabilizers used for chemical stabilization purposes can be categorized as traditional, by-products of various materials, and non-traditional [11]. Moreover, many researchers have investigated the effect of cyclic freezing and thawing on the soil properties. Therefore, this chapter will describe the previous research work conducted on the stabilization of soil exposed to cyclic freeze-thaw action. Furthermore, since the focus of this study is the application of CSA cement, some studies conducted on the investigation of soil stabilization with CSA cement will be reported.

2.2 Stabilization of soil subjected to freeze-thaw cycles

Hazirbaba and Gullu investigated the effect of geofier and synthetic fluid as stabilizing materials on the California Bearing Ratio (CBR) of fine-grained soil [12]. The experimental work conducted tests for the specimens considering freeze-thaw action and compared the results with the samples tested without freeze-thaw action. Also, soaked and unsoaked conditions were applied. Significant effects in the CBR performance were observed for the samples tested under wet conditions, especially for the samples stabilized with both material, fiber, and synthetic fluid. In the case of the soaked condition, the samples treated with only geofiber showed better performance. The results of freeze-thaw testing also showed the same trend. Moreover, cyclic freezing and thawing negatively affected the CBR value.

Liu et al. examined the cement and lime treated soil behavior under freezing and thawing conditions since these materials are becoming widely used as a fill material for the construction of railways [13]. This experimental research work was conducted using triaxial tests on cement and lime-treated soil. Furthermore, the deviator stress and resilient modulus parameters of the soil samples were analyzed. The findings of experimental work illustrated that the modified soil samples show better performance in comparison with the soil samples without modification before repeated cyclic freezing and thawing. The cement-treated soil samples have shown better results than lime-treated soil.

The study conducted by Ghazavi and Roustaie investigated the impact of freezing and thawing cycles on the physical and mechanical characteristics of fiber-reinforced clay soil [14]. For experimental work, kaolin clay was reinforced by steel and polypropylene fibers. The unconfined compressive test was applied to test the modified and unmodified soil samples. The results of the experiment were that with the increase of freeze-thaw cycles the strength values decrease. Moreover, the reinforcement of soil samples improved the strength values and reduced the influence of cyclic freezing and thawing in comparison with not-modified soil samples.

The research of Aldood et al. examined the mechanical and mineralogical behavior of the gypsum-containing soil that was treated with lime considering the freeze-thaw action. The study focused on the stability and durability of subgrade design for the long-term period. The various gypsum contents and fixed lime content were tested using unconfined compression strength (UCS) and wave velocity (UPV) tests. Moreover, other parameters, such as pH, water content, and changes in volume were also considered. The analysis of obtained results from experimental work shows that repeated cyclic freezing and thawing negatively influence the strength of treated soil samples. Furthermore, the increase in water content is observed after cyclic freezing and thawing, which leads to the volume increase of soil specimens. For the explanation of such changes, the scanning electronic microscope (SEM) was applied for microstructural analysis. From the SEM analysis, the dissolution of gypsum, formation of ettringite, and formation of cracks were observed, due to the action of cyclic freezing and thawing. Consequently, changes in the mineralogy and structure of soil samples weakened the soil strength and lead to a reduction in durability.

The fat and lean clays treated with the Portland cement under freeze-thaw action were investigated by Escisar et al. [15]. Two different curing conditions were tested, which are 7 and 28 days. For the assessment of stabilizing method, UCS and UPV tests were applied. The increase of strength with the increase of cement content was observed from the analysis of the results of experimental work. Moreover, soil samples with the lowest water content showed the highest strength. From the obtained results the relationship curve between the UCS and UPV test results was plotted, and it showed a linear correlation. Overall, the paper's findings concluded that the cement treatment method could be suggested for the treatment of fat and lean clay to enhance the durability of soil under cyclic freezing-thawing conditions.

Expansive soil stabilization with lime exposed to cyclic freezing and thawing was examined by Hotineanu et al. [16]. The experimental work studied the mechanical characteristics of two types of soils, which are bentonite and kaolinite, treating them with lime. The durability, soil strength, changes in volume, and porosity were assessed after curing days and cyclic freezing and thawing. A significant increase in volume was observed immediately after the first cycle. Also, an improvement in strength values was determined with the increase in the curing period. The cyclic freezing and thawing had more effect on the bentonite clay than kaolinite. From the microstructural analysis, the impact of freeze-thaw action was observed with the increase in pore size of soil samples.

Zhang et al. explored the influence of cyclic freezing and thawing and the effectiveness of stabilization methods using various additives. Paper suggests the effective method of soil stabilization to avoid undesirable consequences of freeze-thaw action that may lead to the damage of pavement due to the changes in the foundation layers [17]. For the experimental work, soil was stabilized with three different additives, which are fly ash, cement, and polymer fibers. The addition of stabilizers improved the frost susceptibility property of soil.

The soft soil treated with the Bassanite was studied by Kamei et al. taking into account the influence of cyclic freezing and thawing on its durability and strength [18]. Bassanite is a product recycled from gypsum waste. The results of experimental testing show that exposure to freeze-thaw action reduced the UCS value and durability index of stabilized soil. Increasing Bassanite content reduced the content of water and increased the dry unit weight value of soil samples exposed to the freeze-thaw cycles. Considerable volume change was observed after the action of the first cyclic freezing and thawing. Overall, the application of Bassanite as a stabilizing binder for very soft clay helped to reach the acceptable strength value and durability of soil that was exposed to F-T cycles.

Overall, by the summary of recent previous studies, it can be concluded that the exposure of soil to freeze-thaw cycles negatively affects the soil performance, which shows the importance of considering of the frost susceptibility of soil during the design process. This is an important criterion, especially for regions with cold weather. Moreover, it should be noted that improvements in the soil properties could be achieved by applying various treatment methods.

2.3 Stabilization of soil with Calcium Sulfoaluminate cement

Calcium Sulfoaluminate cement (CSA) is widely used in the concrete industry, but it is a comparatively new material in the soil stabilization area. Some research works studied the application of CSA cement for soil stabilization purposes. This part will discuss papers written about the effectiveness of CSA cement usage as a stabilizing material.

The sand treated with cement by the addition of fine particles was investigated by Moon et al. [6]. OPC and CSA cement with three different contents (3%, 5%, and 7%) were tested. Kaolin powder is used as a fine material with 4 different content, which are 0%, 1%, 3%, and 5%. For the evaluation of soil performance treated with cement, the UPV, UCS, and shear wave velocity tests were conducted. The findings of experimental work show that the addition of fine material even at a negligent proportion affects soil performance. The increase in strength and stiffness were observed with the addition of fine material. Moreover, the CSA cement added soil samples showed better performance in comparison with the OPC cement treated soil, which shows the rapid strength gain property of CSA cement.

The paper written by Gin et al. investigated the performance of soil treated with two types of cement, CSA cement, and OPC, exposing them to different temperatures [19]. Different OPC and CSA ratios were used for experimental work. Thermogravimetric analysis (TG), X-ray powder diffraction, macroeconomic imbalance procedure (MIP), calorimetry, and Scanning electronic microscope (SEM) were used to define the heat flow, hydration products, and distribution of pore size, and changes in the microstructure of soil samples. A higher percentage of CSA cement resulted in a decrease in setting time. Also, the strength of soil increased with the increase of CSA cement content at early ages, which is favorable for the improvement of soil resistance at early age frost. However, at later ages, the decreasing trend of strength was observed at higher dosages of CSA cement, which was explained by lower curing temperature. Because of the fast hydration process of CSA cement, the pore structure of cement is impaired, when the CSA content was higher than 5%. However, after the exposure to the early age frost, the microstructure of soil was improved, due to the higher resistance of CSA treated soil at early ages. Overall, the paper concluded that the use of CSA cement at a suitable content helps enhance the resistance against frost damage in the early stage.

Subramanian et al. highlighted the importance of CSA cement usage for the replacement of OPC cement. CSA cement emits less carbon into the atmosphere and can replace OPC, whereas other additives used for soil stabilization purposes can partially replace the OPC. The paper determines the optimum content of gypsum for the CSA stabilized sand, to reach a higher early strength gain value. The results of experimental work show that the replacement of 30% of CSA cement will provide high initial strength. Soil samples treated with CSA cement have shown high strength gain in comparison with the OPC treated soil samples under dry and wet curing conditions.

The research work conducted by Pooni et al. examined the performance of CSA cement-stabilized expansive soil [20]. For the evaluation of the effectiveness of stabilizing material, the mechanical properties and microstructural characteristics were analyzed. From the analysis obtained data from UCS testing, the addition of CSA cement improved the strength of the soil. Also, the microstructural analysis of CSA treated problematic soil showed that with the hydration of cement spacing between clay sheets was reduced since hydration products were formed that enhanced the soil strength.

Moreover, for the evaluation of the effect of freeze-thaw cycles on CSA cement-treated soil, Jumassultan et al tested quartz sand [10] by evaluating the physical characteristics of the soil. Soil samples were prepared with 4 different cement content, 2%, 5%, 7%, and 10%. Prepared samples were cured for 7 and 14 days, taking into consideration the early strength gain properties of CSA-treated soil. The UCS and UPV test was carried out in the experimental part. The main findings of experimental work were that with increased freeze-thaw cycles, the strength, and pulse velocity values reduced, but an improvement in cement content increased the strength values. A decreasing trend was also observed for the durability index, but the reduction was insignificant, which shows the effectiveness of CSA cement as a stabilizing material. In addition, the SEM pictures obtained after cyclic freeze-thaw shows changes in the microstructure of soil samples, where the crack formation and pore size increase were observed.

Furthermore, a numerical study conducted by Bisserik et al. analyzed the CSA cement stabilized soil using the three-dimensional discrete element method (DEM) [9]. Fixed values for the cement and water contents were used, which are 7% and 10%, respectively. The results of the simulation show the improvement in strength, where after 1 curing day, the CSA treated soil samples gained 72% of the total strength that was obtained for 7 days of cured samples. From the analysis of results, the rapid strength gain property of CSA cement can also be observed from the simulation using DEM, and it coincides with the results of experimental works.

Table 2.1 presents the comparison of parameters and conditions used for freezing and thawing tests for experimental works in different research studies. By the review of previous research works and analysis of *Table 2.1*, curing conditions and other variables for the current experimental work were decided.

Table 2.1: Analysis of papers based on the selected parameters for testing

Author	**Curing time**	**Freezing time and temp.**	**Thawing time and temp.**	**Freeze-thaw cycles**
Gullu and Hazirbaba [21]	28 days	-20C, 24 hours	+20C, 24 h	0, 1
Liu et al. [13]	28 days	-15C, 12 hours	+5C, 12 h	0, 1, 3, 6, 8, and 10
Hazirbaba and Gullu [12]	28 days, soaking 96 h	−20 °C, 24 h	21 °C, 24 h	
Kamei et al. [18]	28 days	−10 °C, 24 h	24 h, room temperature	0,1, 2, and 5
Ghazavi and Roustaie [14]	No	−20 °C, 6 h	+25 °C, 6 h	0, 1, 3, 5, and 10
Gullu and Khudir [22]	No curing	−18 °C, 24 h	18 °C, 24 h	1, 2 and 3
Zhang et al. [17]	7 days	-10 °C, 24 h	at room temp., 24 h	5
Hotineanu et al. [16]	7 days	-10 °C, 18 h	23 ± 2 °C, 6 h	0, 1, 5, and 10
Kravchenco et al. [23]	7 days	-15 °C, 12 h	+20 °C, 12 h	0, 2, 5, 8, 10, 15
Lake et al. [24]	28 days	−10 °C (±1 °C), 24 h	22 °C (±1 °C), 24 h	3
Bozbey et al. [25]	7, 28, and 56 days	−23 °C, 24 h	23 °C, 23 h	12

Chapter 3 – Experimental Work

3.1 Introduction

In this chapter, the materials used for experimental work will be described, and the sample preparation process, testing procedure, and tests performed will be explained in detail.

3.2 Materials

The materials used for the experimental part of the research are natural soil, Calcium Sulfoaluminate (CSA) cement, gypsum, and water.

3.2.1 Natural Soil

Natural soil (*Figure 3.1*) utilized for the experimental work was obtained from a land excavation site in Nur-Sultan, Kazakhstan. The soil color is light brown and the size was not uniform, as shown in *Figure 3.1*. Before the start of the experimental work, soil samples were oven-dried. For the evaluation of grain sizes and to conduct other testing, soil grinders were used to get more uniform soil samples and to provide precision in the analysis of results.

Figure 3.1: Natural soil sourced from land excavation

Figure 3.2: Grinded natural soil

Since the size of the soil was not uniformly distributed, the soil was grinded before use, and the picture of grinded soil was provided in *Figure 3.2*.

The particle size distribution curve of the soil used in this study is shown in *Figure 3.3*.

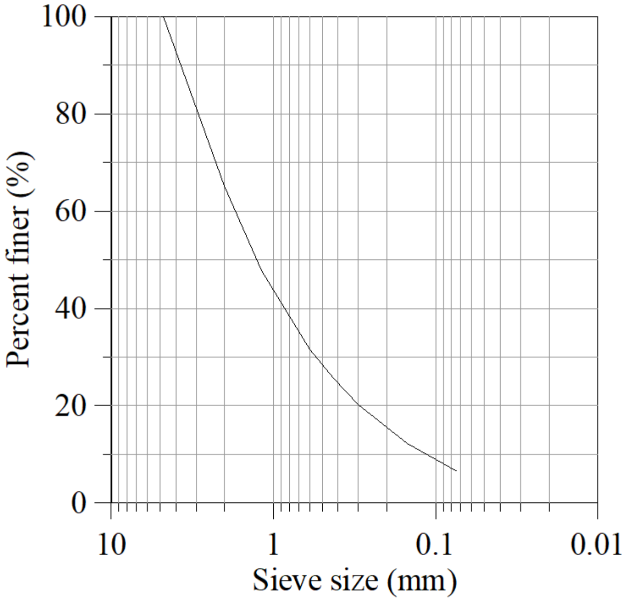


Figure 3.3. Particle size distribution curve for the sand used in this study

The geotechnical soil properties are demonstrated in *Table 3.1*. Based on the Unified Soil Classification System (USCS), the soil is defined as a well-graded sand with silt (SW-SM). For the evaluation of uniformity of soil samples, the main parameters were obtained from the soil particle size distribution curve, which is particle diameters at 10% (D10), 30% (D30), 60% (D60) in the cumulative distribution, coefficient of uniformity, and the coefficient of curvature (*Table 3.1*).

Table 3.1: Physical properties of the soil used in this research

Property	Value	Standard
D10 (mm)	0.11	ASTM D1921
D30 (mm)	0.55	ASTM D1921
D60 (mm)	1.8	ASTM D1921
Coefficient of curvature, C_U	1.53	ASTM D1921
Coefficient of uniformity, C_C	16.36	ASTM D1921
USCS classification	SW-SM	ASTM D1921
Optimum Moisture Content (%)	16.5	ASTM D698
Maximum dry density (kN/m^3)	1.75	ASTM D698
Plastic Limit (%)	40.35	ASTM D4318
Liquid Limit (%)	44.31	ASTM D4318
Plasticity Index (%)	3.96	ASTM D4318

3.2.2 Calcium Sulfoaluminate cement

CSA cement is used as a stabilizing material. It was made by mixing several materials such as limestone, bauxite, clay, and gypsum or anhydrate. It is widely used in China for construction purposes for about 30 years and now it is in demand all over the world [26, 27]. Several advantages of CSA cement are described below.

- The amount of consumed energy during the manufacturing process is much lower than OPC cement since the firing temperature of clinker is lower at 200C than for Portland cement [28].
- Based on research conducted by Sharp et al. [26] the manufacturing process of CSA cement emits about 40% less CO_2 gases than Ordinary Portland cement.
- Moreover, the addition of CSA cement can provide high early strength development in a short time [28].

3.2.3 Gypsum

Gypsum is used to partially replace the CSA cement, since the CSA cement used in this research consists of ye'elimite, belite, and gelignite, without gypsum, as it was obtained from X-ray diffraction (XRD) analysis, as shown in *Figure 3.4*. The XRD test results of previous research work were used since the CSA cement was provided by the same supplier [10]. According to Subramanian et al. [8] replacement of 30% of CSA cement content by gypsum provides high initial strength gain and better strength development. Therefore, 30% of the total mass of CSA cement was replaced by gypsum.

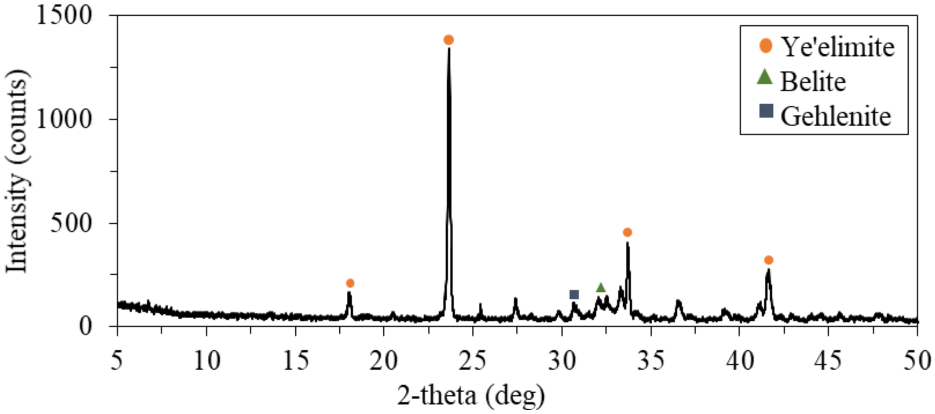


Figure 3.4: X-ray diffraction analyses of CSA cement **[10]**

3.3 Mix Design and Sample Preparation

The cement-soil mixture was prepared using the optimum moisture content (OMC) defined by Standard Proctor Test [29]. The results of OMC and maximum dry density (MDD) are shown in *Table 3.2* for soil samples with 3%, 5%, and 7% cement contents, which are 20.8 %, 21.0 %, and 22.0 % for OMC, 1.67, 1.62, and 1.56 kN/m^3 for MDD, respectively. From the results obtained, a certain trend can be traced, when with an increase in the cement content, the MDD decreases, and the OMC increases. Dry soil was used to prepare the soil-cement mixture since for the Standard Proctor test dry soil is applied.

Table 3.2: Standard Proctor Test results of CSA treated soil

Cement content	Optimum moisture content (%)	Maximum dry density (kN/m^3)
3%	20.8	1.67
5%	21.0	1.62
7%	22.0	1.56

In this experimental work, the cement content is determined as the ratio of cement mass to the total mass of dry soil, while the gypsum content is calculated as the ratio of gypsum mass to the total mass of binder, which consists of CSA cement and gypsum. The water content is calculated as the ratio of water mass to the total solid weight, which includes the mass of soil and binder. The mixture is prepared in two stages: first, dry materials are mixed, and then water is added. After completing the preparation of the mixture, cement-treated soil samples were made in a mold with a diameter of 50 mm and a height of 100 mm in three layers using undercompaction method [30]. Each layer was compacted 25 times using the hand rammer, and the top and middle layers were scarified to ensure contact between layers. Undercompaction technique is used to keep the uniform density in all layers of the soil sample. Three soil specimens were made for each combination in order to get more reliable results. After 1 day of curing, samples were extruded from molds and wrapped with plastic film, and left for curing. Three different curing periods were tested, which are 3, 7, and 14 days, applying the dry curing method at room temperature.

3.4 Experimental Methods

The experimental program consisted of four main tests, which are freeze-thaw (F-T), ultrasonic pulse velocity (UPV), unconfined compressive strength (UCS) tests, and scanning electronic microscope (SEM) tests.

3.4.1 Freeze-thaw test

At the end of curing periods, soil samples were put into the freeze-thaw chamber, where the humidity and temperature are controlled, as shown in *Figure 3.5*, and cement-soil specimens were tested under 0, 1, 3, 5, 7, 10, and 15 freeze-thaw (F-T) cycles. A closed system was used for the freeze-thaw tests, where there is no additional water supply, except the water in the voids of soil. In many cases, a closed system is mostly considered, since it has similar conditions to the field conditions [31]. The freezing temperature was –20 °C, because it is the minimum temperature of the soil in Nursultan, Kazakhstan [32], and the thawing temperature was taken as the room temperature (23 °C), based on the analysis of previous studies [33]. Freeze and thaw cycles lasted for 12 hours each. The duration of one cycle of freezing and thawing was 24 hours.

3.4.2 Ultrasonic Pulse Velocity test

As shown in *Figure 3.5*, after cyclic freezing and thawing ultrasonic pulse velocity (UPV) test was performed. The UPV test was applied for the evaluation of stiffness development of CSA cement-treated samples. Since the UPV test is considered non-destructive, the experiment can be carried out several times on the same sample and samples subsequently can be used for other testing purposes. For this experimental work, the ultrasonic pulse velocity test is conducted using the portable ultrasonic non-destructive digital indicator tester (PUNDIT) device. Before the start of testing, the device was calibrated using the cylindrical block. *Figure 3.5* shows the schematic of the experimental setup, where two transducers of PUNDIT apparatus with a diameter of 50 cm were attached to the surface of samples from both sides. The UPV value is determined using the travel time along the length of the soil sample. The pulse velocity values are determined from the pulse traveled along the length of the sample [34].

3.4.3 Unconfined Compressive Strength test

Following the UPV tests, the UCS test was performed to evaluate the cement-treated soil properties with and without exposure to the freezing and thawing cycles. The UCS test was conducted using the universal compression equipment at a standard strain rate of 1mm/min, according to ASTM/2166 Standard [35]. For the proper analysis of the UCS test findings, the average of the results of three samples was used to get more accurate and reliable results, while the analysis of obtained data.

3.4.4 Scanning Electronic Microscope test

A scanning electron microscope test (SEM) was conducted to observe the microstructure of cement-treated soil specimens before and after cyclic freezing and thawing. Zeiss Crossbeam 540 (*Figure 3.5*) high-resolution SEM equipment is used for the microscopic analysis of soil samples. Before taking SEM pictures, the soil specimens were gold-coated to obtain high-quality images.

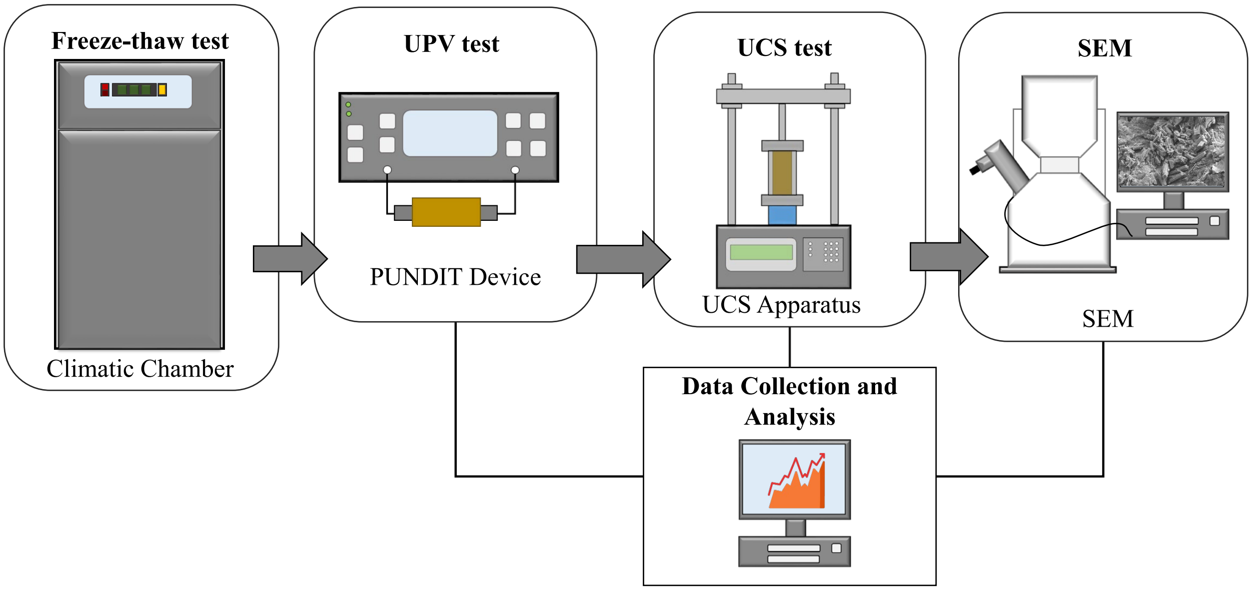


Figure 3.5: Schematic of the experimental setup

Chapter 4 – Results and Discussions

4.1 Ultrasonic Pulse Velocity

Figure 4.1 shows the results of UPV testing for 3, 7, and 14 cured samples under cyclic freezing and thawing. It can be seen that with the increase in freeze-thaw cycles, the pulse velocity values decrease. In addition, it can be highlighted that the UPV value of soil specimens with 7% cement content considerably decreased after the 7th freezing and thawing cycle by 80 % (*Figure 4.1* (a)). Such a reduction in the UPV values can be explained by the effect of cyclic freezing and thawing on soil samples. The impact of freeze-thaw cycles causes the formation of microcracks and an increase in the volume of voids, which causes a change in the structure of the samples. This will reduce the ability of the sample to transmit ultrasonic pulses.

Moreover, there is an increase in pulse velocity with the cement content growth from 3% to 7%. Furthermore, the increase in pulse velocity can be observed with the increase in curing days. For instance, the pulse velocity values increased from 1872 m/s to 2340 m/s, 2090 m/s to 2373 m/s, and 2340 m/s to 2453 m/s, with the increase of curing days from 3 to 14, at 3%, 5%, and 7% of cement content, respectively. Such an increase in UPV values can be explained by the effect of cementation. Thus, increasing the cement content increases strength.

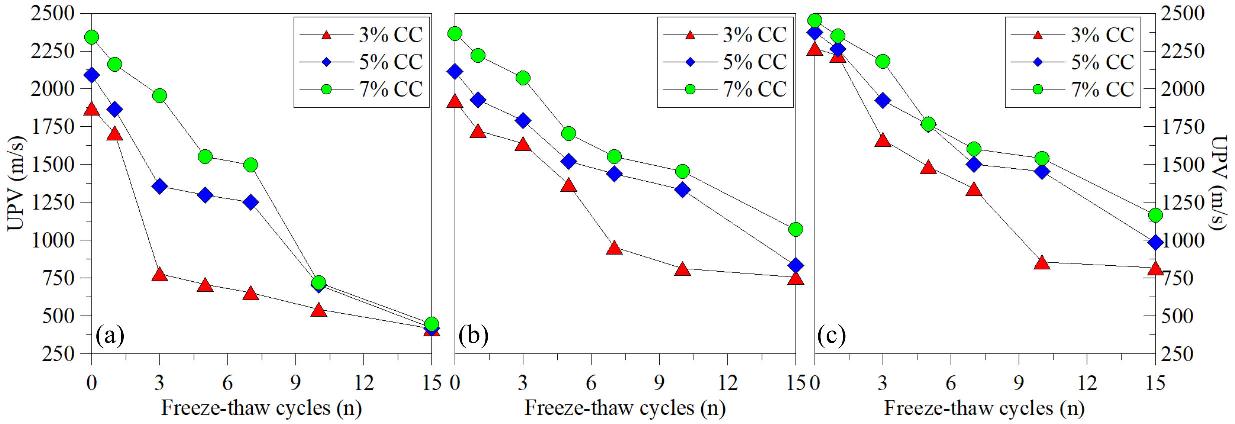


Figure 4.1: UPV test results (a) 3 day curing, (b) 7 day curing, and (c) 14 day curing (Note: CC – cement content)

4.2 Stress-strain behavior

Figure 4.2 shows the stress-strain response of 14 days of cured soil samples treated with CSA cement under cyclic freezing and thawing. It is clearly can be observed that in all three cement contents freeze-thaw cycles negatively affect the peak stress values of soil samples. The stress values decreased from 143 MPa to 49 MPa at 3% cement content, from 212 MPa to 67 MPa at 5% cement content, and from 296 MPa to 111 MPa at 7% cement content with the increase of cycles of freezing and thawing from 0 to 15. Moreover, it can be noticed that an increase in cement content from 3% to 7% increased the peak stress of soil specimens.

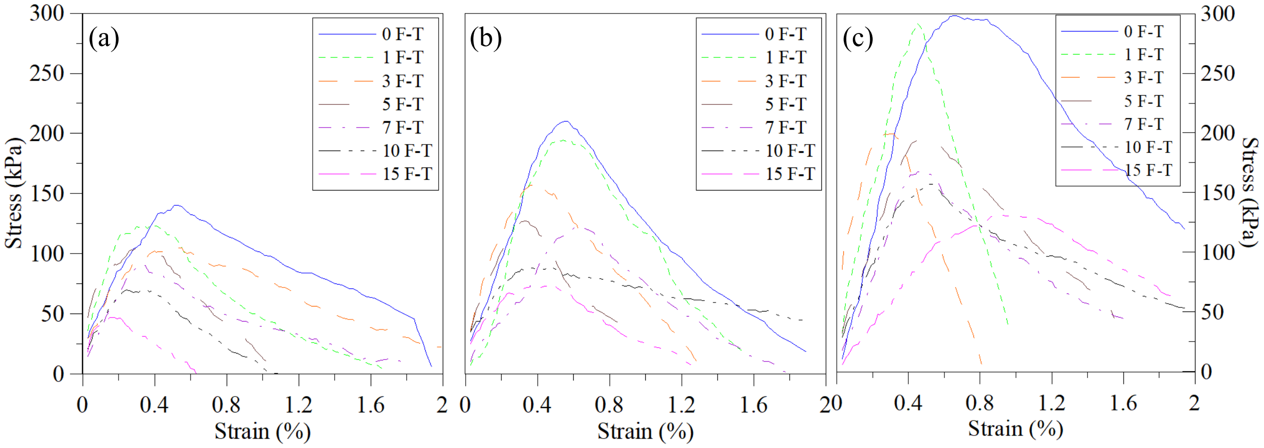


Figure 4.2: Stress-strain response of 14-day cured soil samples treated with CSA cement for all freeze-thaw cycles, (a) 3 % CC, (b) 5 % CC, and (c) 7 % CC

4.3 UCS performance

Figure 4.3 shows the results of the UCS test for 3, 7, and 14 days of cured cement-treated samples exposed under freeze-thaw cycles. The results of UCS tests show the same trend as the results of the UPV test. The loss of strength is observed at all curing periods, with increasing cycles of freezing and thawing. The UCS values of soil samples without exposure to cyclic freezing and thawing have shown an increase in strength with the increase of cement content and curing days. It can be observed that the strength values of soil specimens with 3% cement content at 0 freeze-thaw cycles increased by 35% when cured from 3 days to 14 days. Beyond 7% cement content, there is another considerable improvement in strength by 37% at 14 days cured samples in comparison with the 3 days cured samples. This can be explained by the cementation of soil, due to the hydration of gypsum in the CSA cement that produces ettringite and calcium silicate hydrate (CSH) [36].

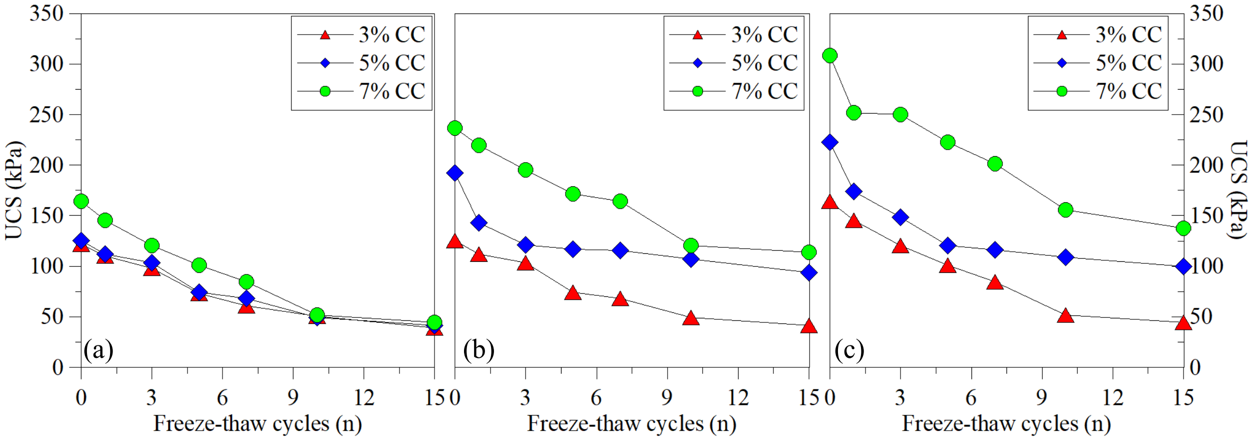


Figure 4.3: UCS test results (a) 3 day curing, (b) 7-day curing, and (c) 14 day curing

As an illustration of failure planes, the pictures of typical failure modes of cement-treated soil specimens were taken after UCS testing as illustrated in *Figure 4.4*. The failure plane of cement-treated specimens has been obtained along the shear plane, which shows brittle failure mode and it is comparable to the stress-strain curve in *Figure 4.2*.

Figure 4.4: Failure planes of soil samples treated with CSA cement

In addition, exposure to freezing and thawing negatively influences the strength of soil samples. Such changes could be explained using the microstructural analysis. The ice crystals formed during freezing process melt after the thawing, and that leads to the shrinkage of soil samples. The findings of experimental work are consistent with the results of previous studies that examined the effect of cyclic freezing and thawing of cement-treated soil samples. [10, 13, 17, 18].

Based on the obtained data from the UCS and UPV tests, the graph for the correlation between the results of these two tests was plotted, as shown in *Figure 4.5*, which demonstrates the increase of UCS with the increase of UPV values. As can be seen from *Figure 4.5* the exponential relationship was established, where the R square value is equal to 0.76. In this plot, the results of specimens subjected to cycles of freezing and thawing were also considered. Therefore, it may be a problem to achieve a better correlation between UCS and UPV tests, since the possible formation of internal cracks in the samples after the F-T cycles can cause fluctuations in the signals in the UPV test. To overcome such problems, the UPV test was repeated several times on the same sample.

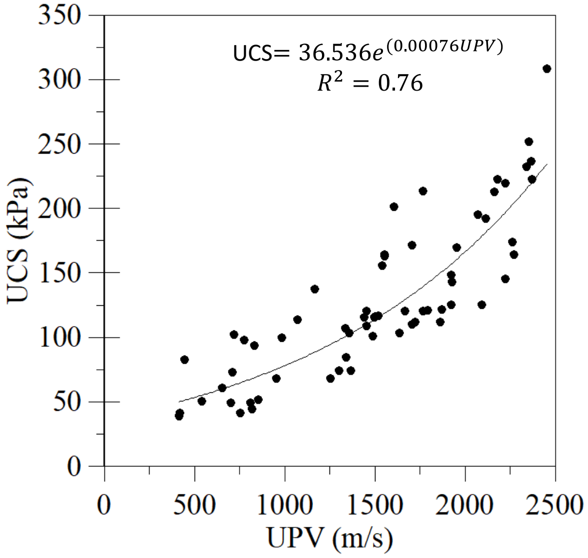


Figure 4.5: UCS and UPV relationship

4.4 Strength loss/gain

Applying the results of the UCS test the loss and gain of strength values of cement-treated soil from cyclic freezing and thawing were calculated using Eq. 1 below [3].

$$Strength\frac{loss}{gain}(\%) = \frac{UCS - UCS_{FT}}{UCS} \qquad (1)$$

where UCS is the compressive strength value before the freeze-thaw cycle and UCS_{FT} is the compressive strength value after cyclic freezing and thawing [3]. *Figure 4.6* illustrates the strength loss/gain graph for cement-treated soil after F-T cycles. The highest loss of strength can be observed for soil samples with 3% cement content, where strength values decreased by 67%, 68%, and 73% after 25 F-T cycles, for 3, 7, and 14 days cured samples. For the soil with 5% and 7% cement content, the strength loss values were almost the same after 15 F-T cycles. The reduction in strength loss can be proof of the formation of hydrated particles that improve the strength performance of cement-treated soil.

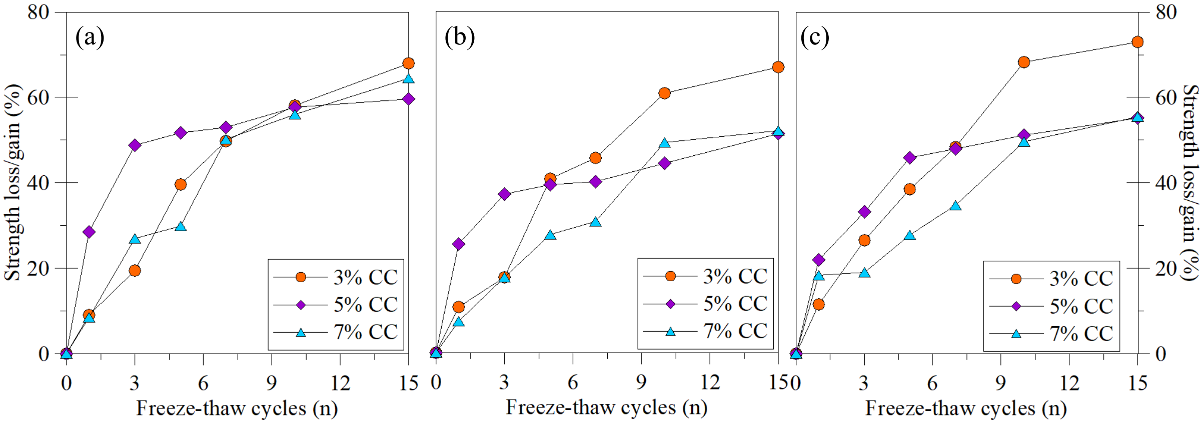


Figure 4.6: Strength loss/gain (a) 3-day curing, (b) 7-day curing, and (c) 14-day curing

Furthermore, it is important to note that for all cases, where different cement content and curing days were tested, after the 10th and 15th F-T cycle there was an inconsiderable loss in strength in comparison with the previous cycles, which means that after the 15th cycle changes in mechanical and physical properties of stabilized soil will be insignificant.

4.5 Estimation of resilient modulus

The resilient modulus is considered one of the significant parameters for the subgrade of soil, especially in the design of pavement. It is determined using the repeated load triaxial method and calculated from the ratio of applied deviator stress to the resilient strain [22]. However, this method is time-consuming and economically inefficient to conduct and, since it requires sophisticated laboratory test facilities and routine tests [3]. Therefore, the resilient modulus can be correlated with other strength parameters, such as unconfined compressive strength. Lee et al. proposed an indirect way to estimate resilient modulus using the UCS data [37]. Eq. 2 shows the regression-based model for the estimation of resilient modulus.

$$M_R = 606.6S_{U1\%} \tag{2}$$

where $S_{U1\%}$ is the stress value at 1% of axial strain in the UCS test, it is in psi (1psi = 6.8 kPa). This model is recommended for soils that have $S_{U1\%}$ values less than 241 kPa. Applying Eq. 2 the estimated values for resilient modulus were calculated and obtained data is shown in *Figure 4.7*. The results show that exposure to cyclic freezing and thawing negatively affects the resilient modulus, and with the increase of cement content, a considerable decrease in resilient modulus can be seen with the increase of F-T cycles. Moreover, it can be observed that as the cement content increase the resilient modulus values also increase, especially it can be observed for samples with 7% cement content. The same trend can be seen with the increase in curing days. However, the estimated values of resilient modulus for the soil samples with 3% and 5% cement content have shown nonlinear soil behavior with the growth of cyclic freezing and thawing, and there is a slight increase in resilient modulus when the curing days increase from 3 to 14 days. Therefore, to deeply explain this behavior of the soil, it is necessary to carry out separate research work.

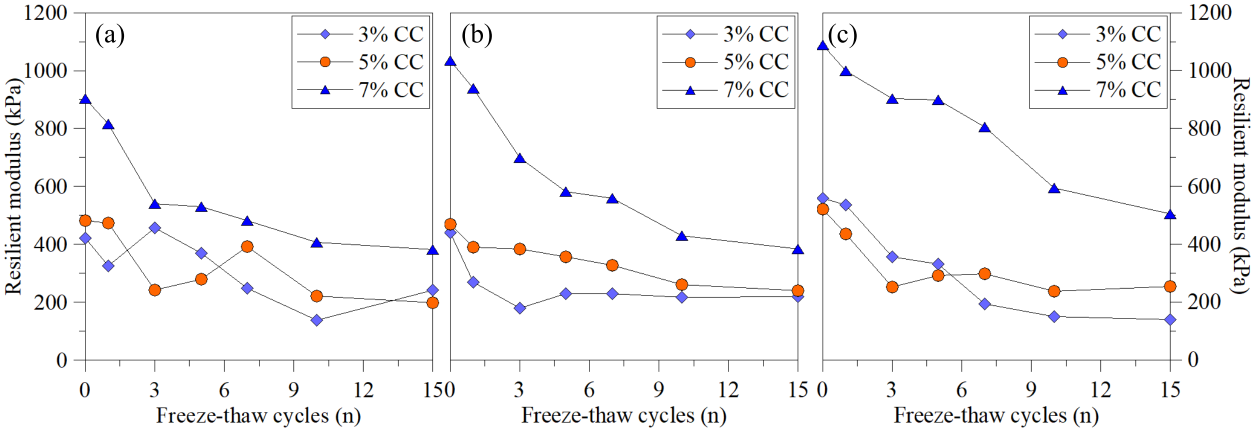


Figure 4.7: Resilient Modulus, (a) 3-day curing, (b) 7-day curing, and (c) 14-day curing.

4.6 SEM observation

Scanning Electronic Microscopic pictures were taken to analyze the microstructural characteristics of soil samples in order to explain changes in the mechanical behavior after F-T cycles. SEM pictures were taken for 14 days of cured samples with 7% cement content. *Figure 4.8* shows the SEM picture of soil samples that were not subjected to F-T cycles. From *Figure 4.8* the needle-shaped structure can be seen, which is ettringite that is produced from the hydration of ye’elimite. The ettringite formation results from the initial high strength gain of CSA cement-stabilized soil [36].

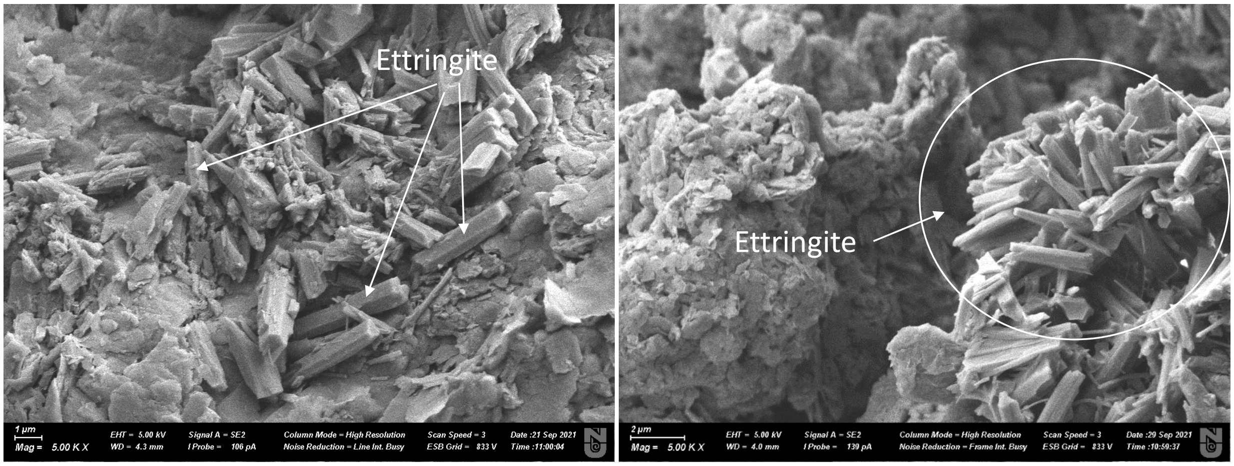
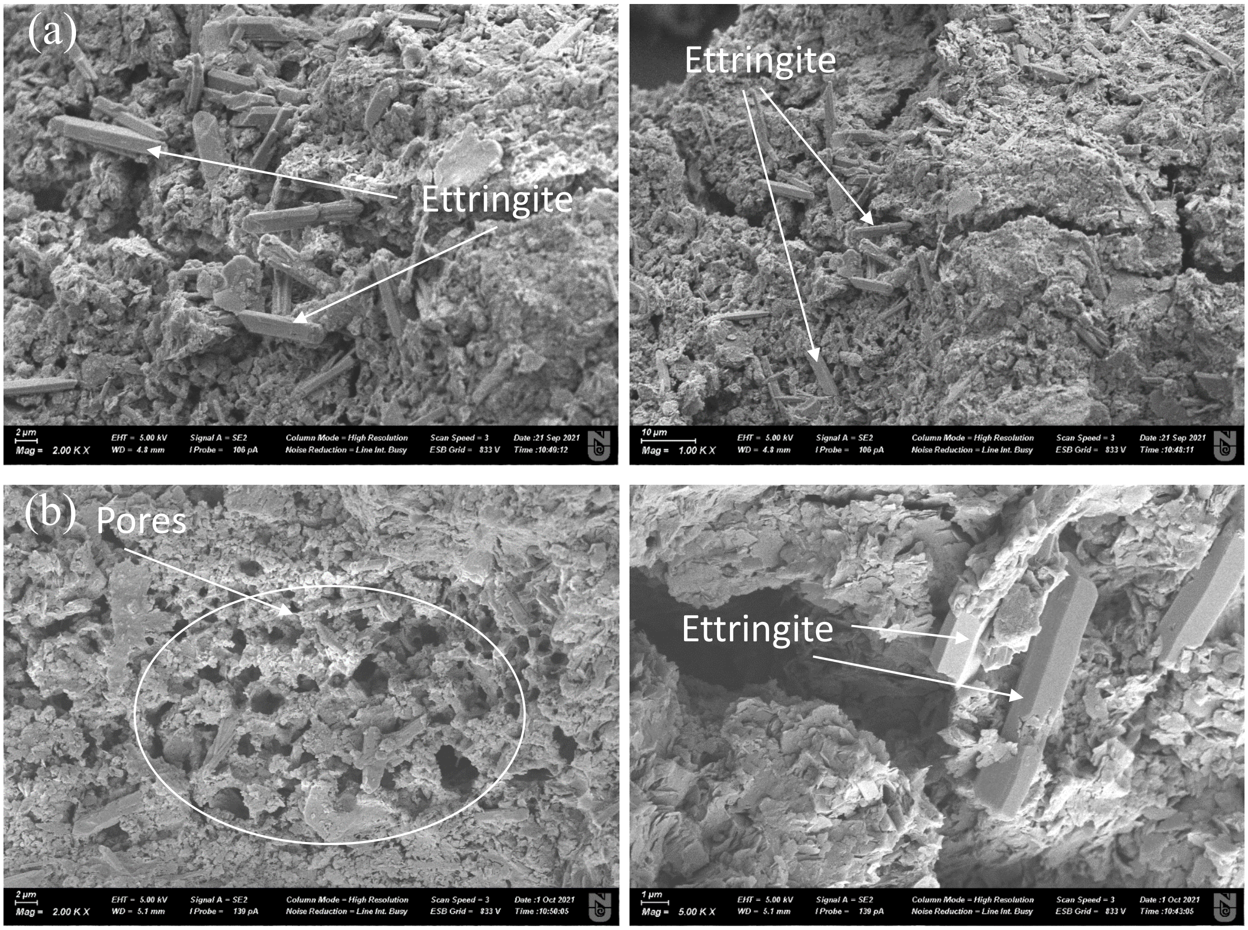
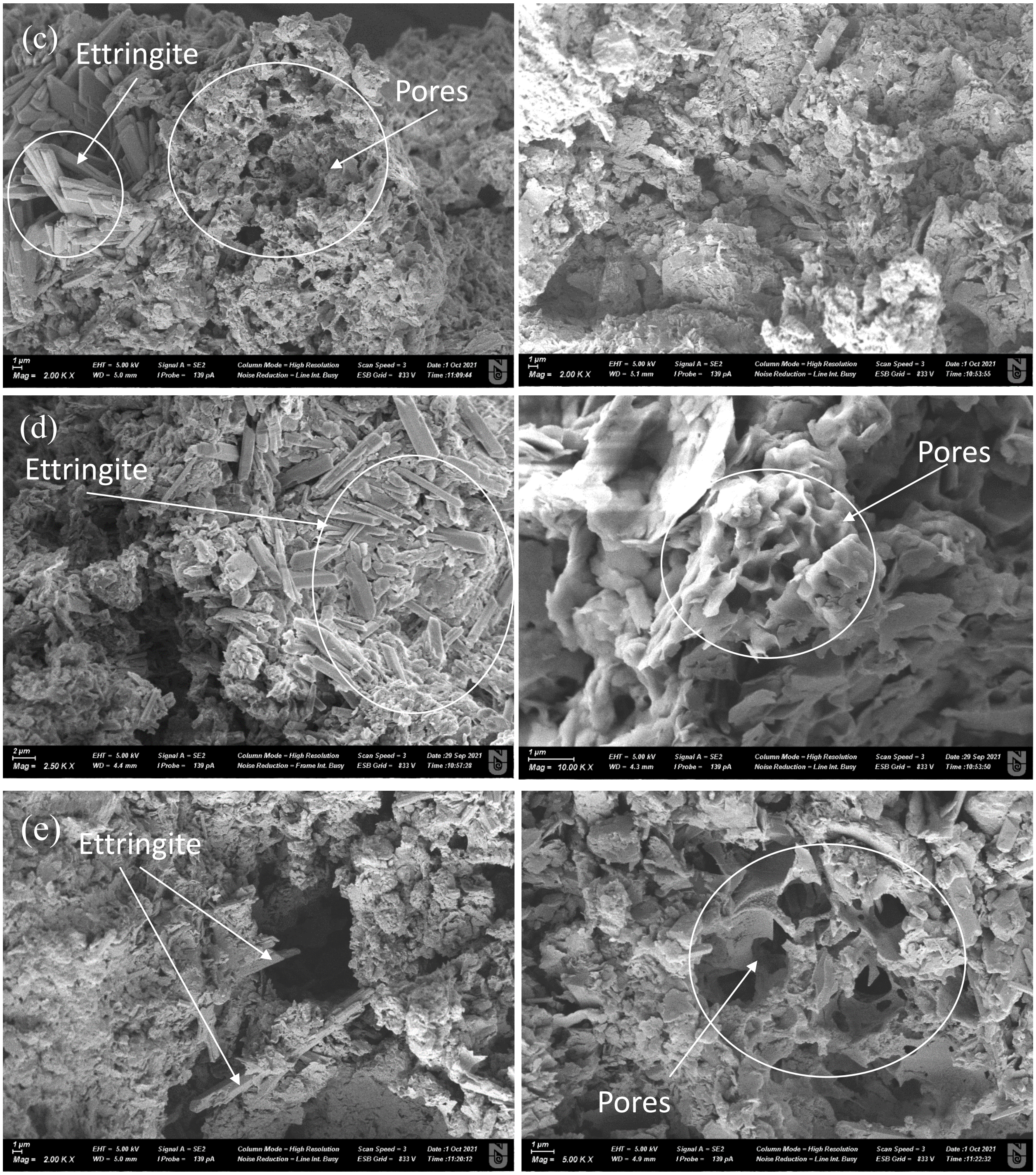


Figure 4.8: SEM images of soil samples treated with 7% CSA cement cured 14 days at 0 F-T.




(c)
Ettringite
Pores
(d)
Ettringite
Pores
(e)
Ettringite
Pores

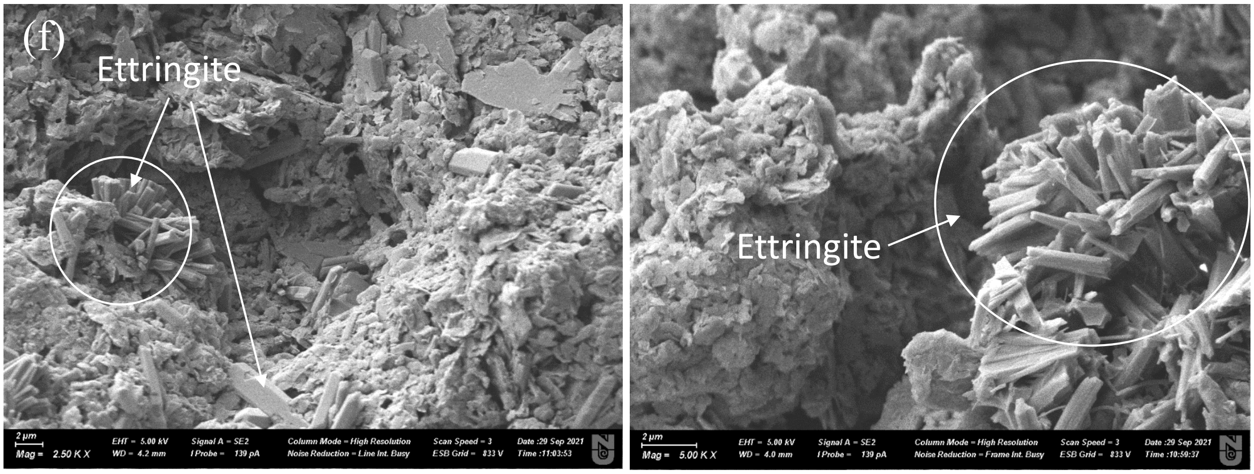


Figure 4.9: SEM images taken after F-T cycles, (a) 1 F-T, (b) 3 F-T, (c) 5 F-T, (d) 7 F-T, (e) 10 F-T, and (f) 15 F-T.

Figure 4.9 shows SEM images obtained after F-T cycles, which clearly show the presence of pores and the formation of cracks after F-T cycles. This can be explained by the formation of ice crystals during freezing and the total porosity increase of the soil, which subsequently leads to an increase in pore size.

Chapter 5 – Conclusions and Future Scope

The research work is conducted to assess the influence of cyclic freezing and thawing on the mechanical and physical properties of silty sand treated with CSA cement. For the evaluation freeze-thaw effect and stabilizing material, UPV and UCS tests were performed. The main findings of the research were:

- Treating the soil with CSA cement improved the characteristics of the soil, and with the increase of cement content, the strength of the soil increased even after 3 days of curing, indicating the early strength gain property of CSA cement.
- By increasing the number of freeze-thaw cycles, linear soil behavior was observed from the UPV test result. The UPV values of all samples reduced with the F-T cycle increase.
- The results of UCS testing showed the same trend as the UPV test.
- In addition, the calculated values for the strength loss/gain using the UCS test results shows the effect of cementation, where soil samples with higher cement content showed better performance than those with lower cement content.
- Moreover, the results of the estimated resilient modulus showed that the addition of cement for stabilization purposes improves the resilient modulus, which is important for subgrade design.
- Furthermore, after the 10th F-T cycle there was an insignificant reduction in strength in comparison with the results after 15th F-T, and it can be assumed that after 15th cycle changes there will not be considerable changes in mechanical and physical properties of stabilized soil.
- Microstructural analysis performed using SEM images, which were taken before and after the F-T cycle, explained the impact of F-T cycles on soil structure. The formation of pores due to the melting of ice crystals leads to the change in the soil characteristics that further causes the strength loss of soil.

Overall, from the summary of the findings of the conducted research work, the application of CSA cement is preferred for soil stabilization purposes, especially in regions with cold climate, due to environmental considerations and therefore the use of CSA cement clinker for earthworks in cold regions should be encouraged.

The long-term performance of CSA cement treatment under cyclic freeze-thaw action should be examined in the future in order to test the effectiveness of CSA cement over a long period.

References

1. Arasan, S. and O. Nasirpur, *The effects of polymers and fly ash on unconfined compressive strength and freeze-thaw behavior of loose saturated sand.* Geomechanics and Engineering, 2015. **8**((3)): p. 361-375.
2. Yilmaz, F. and D. Fidan, *Influence of freeze-thaw on strength of clayey soil stabilized with lime and perlite.* Geomechanics and Engineering, 2018. **14**(3): p. 301-306.
3. Güllü, H. and H.I. Fedakar, *Unconfined compressive strength and freeze-thaw resistance of sand modified with sludge ash and polypropylene fiber. Geomechanics and Engineering*, 2017. **13**((1)): p. 25-41.
4. Swain, K., *Stabilization of soil using geopolymer and biopolymer*. 2015.
5. Vinoth, G., et al., *Early strength development in cement-treated sand using low-carbon rapid-hardening cements.* Soils and Foundations, 2018. **58**((5)): p. 1200-1211.
6. Moon, S.-W., et al., *Effect of fine particles on strength and stiffness of cement treated sand.* Granular Matter, 2020. **22**((1)): p. 9.
7. Subramanian, S., et al., *CSA-treated sand for geotechnical application: microstructure analysis and rapid strength development.* Journal of Materials in Civil Engineering, 2018. **30**(12): p. 04018313.
8. Subramanian, S., Q. Khan, and T. Ku, *Strength development and prediction of calcium sulfoaluminate treated sand with optimized gypsum for replacing OPC in ground improvement.* Construction and building materials, 2019. **202**: p. 308-318.
9. Bisserik, A., et al. *Characterization of CSA cemented-treated sands via discrete element method*. in *AIP Conference Proceedings*. 2021: AIP Publishing LLC.
10. Jumassultan, A., et al., *Performance of cement-stabilized sand subjected to freeze-thaw cycles.* Geomechanics and Engineering, 2021. **25**(1): p. 41-48.
11. Petry, T.M. and D.N. Little, *Review of stabilization of clays and expansive soils in pavements and lightly loaded structures—history, practice, and future.* Journal of Materials in Civil Engineering, 2002. **14**(6): p. 447-460.
12. Hazirbaba, K. and H. Gullu, *California Bearing Ratio improvement and freeze–thaw performance of fine-grained soils treated with geofiber and synthetic fluid.* Cold Regions Science and Technology, 2010. **63**((1-2)): p. 50-60.
13. Liu, J., D. Chang, and Q. Yu, *Influence of freeze-thaw cycles on mechanical properties of a silty sand.* Engineering Geology, 2016. **210**: p. 23-32.
14. Ghazavi, M. and M. Roustaie, *The influence of freeze–thaw cycles on the unconfined compressive strength of fiber-reinforced clay.* Cold Regions Science and Technology, 2010. **61**(2-3): p. 125-131.
15. Eskişar, T., S. Altun, and İ. Kalıpcılar, *Assessment of strength development and freeze–thaw performance of cement treated clays at different water contents.* Cold Regions Science and Technology, 2015. **111**: p. 50-59.
16. Hotineanu, A., et al., *Effect of freeze–thaw cycling on the mechanical properties of lime-stabilized expansive clays.* Cold Regions Science and Technology, 2015. **119**: p. 151-157.
17. Zhang, Y., A.E. Johnson, and D.J. White, *Laboratory freeze–thaw assessment of cement, fly ash, and fiber stabilized pavement foundation materials.* Cold Regions Science and Technology, 2016. **122,**: p. 50-57.
18. Kamei, T., A. Ahmed, and T. Shibi, *Effect of freeze–thaw cycles on durability and strength of very soft clay soil stabilised with recycled Bassanite.* Cold Regions Science and

Technology, 2012. **82,**: p. 124-129.

19. Qin, L., X. Gao, and A. Zhang, *Potential application of Portland cement-calcium sulfoaluminate cement blends to avoid early age frost damage.* Construction and building materials, 2018. **190**: p. 363-372.
20. Pooni, J., et al., *Novel use of calcium sulfoaluminate (CSA) cement for treating problematic soils.* Construction and building materials, 2020. **260**: p. 120433.
21. Gullu, H. and K. Hazirbaba, *Unconfined compressive strength and post-freeze–thaw behavior of fine-grained soils treated with geofiber and synthetic fluid.* Cold Regions Science and Technology, 2010. **62**(2-3): p. 142-150.
22. Güllü, H. and A. Khudir, *Effect of freeze–thaw cycles on unconfined compressive strength of fine-grained soil treated with jute fiber, steel fiber and lime.* Cold Regions Science and Technology, 2014. **106**: p. 55-65.
23. Kravchenko, E., et al., *Performance of clay soil reinforced with fibers subjected to freeze-thaw cycles.* Cold Regions Science and Technology, 2018. **153**: p. 18-24.
24. Lake, C.B., M.A.-M. Yousif, and R.J. Jamshidi, *Examining freeze/thaw effects on performance and morphology of a lightly cemented soil.* Cold Regions Science and Technology, 2017. **134**: p. 33-44.
25. Bozbey, I., et al., *Effects of soil pulverization level on resilient modulus and freeze and thaw resistance of a lime stabilized clay.* Cold Regions Science and Technology, 2018. **151**: p. 323-334.
26. Sharp, J., et al. *Cementitious systems for encapsualation of intermediate level waste*. in *International Conference on Radioactive Waste Management and Environmental Remediation*. 2003.
27. Zhang, L., M. Su, and Y. Wang, *Development of the use of sulfo-and ferroaluminate cements in China.* Advances in cement research, 1999. **11**(1): p. 15-21.
28. Zhou, Q., N. Milestone, and M. Hayes, *An alternative to Portland cement for waste encapsulation—the calcium sulfoaluminate cement system.* Journal of hazardous materials, 2006. **136**(1): p. 120-129.
29. ASTM/D698, *Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort*, in *American Society for Testing and Materials*. 2007: Philadelphia, PA.
30. Ladd, R., *Preparing test specimens using undercompaction.* Geotechnical testing journal, 1978. **1**(1): p. 16-23.
31. Wong, L.C. and M.D. Haug, *Cyclical closed-system freeze–thaw permeability testing of soil liner and cover materials.* Canadian Geotechnical Journal, 1991. **28**((6)): p. 784-793.
32. Askar, Z. and S. Zhanbolat, *Experimental investigations of freezing soils at ground conditions of Astana, Kazakhstan.* Sciences in Cold and Arid Regions, 2015. **7**((4)): p. 399-406.
33. Ding, M., et al., *Effects of freeze-thaw cycles on mechanical properties of polypropylene fiber and cement stabilized clay.* Cold Regions Science and Technology, 2018. **154**: p. 155-165.
34. ASTM/597-09, *Standard Test Method for Pulse Velocity Through Concrete*, in *American Society for Testing and Materials*. 2009: International, USA.
35. ASTM/2166, *Standard test method for unconfined compressive strength of cohesive soil* in *American Society for Testing and Materials*. 2003: Philadelphia.
36. Glasser, F.P. and L. Zhang, *High-performance cement matrices based on calcium sulfoaluminate–belite compositions.* Cement and concrete research, 2001. **31**((12)): p.

1881-1886.

37. Lee, W., et al., *Resilient modulus of cohesive soils and the effect of freeze–thaw.* Canadian Geotechnical Journal, 1995. **32**(4): p. 559-568.